Microstructure-Dependent Dynamic Flow Stress in Metallic Alloys

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MICROSTRUCTURE-DEPENDENT DYNAMIC
FLOW STRESS IN METALIC ALLOYS

BY

JUSTIN SPIRDIONE

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE
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OF

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2015
ABSTRACT

A constitutive relationship for the dynamic flow stress in metallic alloys has been derived as a function of strain, strain rate, temperature and microstructure parameters. The microstructural features of interest in this study are the secondary phase and the grain size in dual and single phase materials, respectively. The role of the secondary phase has been investigated in Carbon steels by determining the relative contribution of pearlite colonies to the evolving flow stress. In this regard, several steels have been studied including as-received A572 steel, composed of α-ferrite matrix phase and pearlite colonies, and a heat treated A572 comprised of carbide particles dispersed within a ferrite matrix. In order to investigate the role of grain size being a strengthening mechanism on the flow stress, a fine and coarse grained Al6061 alloy have been investigated. An Equal Channel Angular Press was designed and built as a tool to refine the grain size of a coarse grained Al6061 material. Testing in the dynamic loading level was completed using a Split Hopkinson Pressure Bar at strain rates of $10^2$ to $10^4\text{s}^{-1}$ at temperatures of 20, 300, 500 and 650°C for the carbon steel and 20, 50, 100 and 200°C for the aluminum alloy. Result of the experimental testing in the form of true stress-true strain curves were used to model the stress-strain relationship as the sum of two independent components; athermal and thermal. The athermal stress component, which is due to the interaction of dislocations with stress fields generated by long range barriers has been described as a function of strain as well as relevant microstructural features. Sources of long range barriers include grain boundaries, large second phase particles, and dislocations on parallel slip planes. The thermal component of the flow stress as described as a function of strain rate and
temperature and is the result of dislocation interactions with thermally activated barriers or short range barriers; dominant sources include Peierls-Nabarro barriers in BCC metals and forest dislocations in FCC metals. The presence of the dislocation forest as a short range barrier requires the inclusion of strain in the derivation of the thermal component of stress. These two stress components, thermal and athermal, are derived as explicit functions of loading and microstructure parameters. For the carbon steel it is determined that the thermal stress is a function of strain rate and temperature while the athermal component is dependent on the pearlite phase measured in terms of its volume fraction. For the single phase aluminum alloys, both the athermal and thermal stresses are shown to be grain size dependent. Upon reaching a critical grain size, the thermal component becomes increasingly sensitive to grain size refinement thus indicating a change in the deformation mechanism. This increased influence of the grain size on the thermal stress is accounted for by considering the evolution of the short range barrier source. This treatment, furthermore, provides knowledge of the different grain size scales encompassing, coarse, fine and ultra-fine each of which is defined by a deformation mechanism that reflects the specifics of dislocation/barrier interactions. These mechanisms are identified and numerically simulated.

A validation procedure of the final form of the proposed dynamic flow stress model which was derived as explicit function of loading and microstructure has been carried out though a numerical simulation using loading and material conditions that were not involved in the generation of the model parameters. A comparison of the simulation results and those experimentally obtained are described and discussed.
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PREFACE

This thesis is prepared using manuscript format.

Chapter 1 is an introduction and provides an overview of the research work completed in this thesis. This chapter reviews constitutive dynamic flow stress models which are considered an important tool in many engineering efforts involved in material development, characterization, and design. The implementation of these models into predictive numerical simulations requires the ability to describe the material behavior under a wide range of loading conditions. A drawback in majority of these models is that they are only valid for the material for which model parameters are generated. In order to obtain a generalized form of a flow stress relationship, it should therefore be developed as an explicit function of microstructure; the goal of this thesis. Finally, the approach in which the goals of the thesis research are achieved are presented.

Chapter 2 is a manuscript presented following guidelines for the *Journal of Material Science and Engineering A*, for which the chapter has been submitted for publication. In this chapter a physics based dynamic flow stress model is presented. The model is derived based on the interaction of dislocations with various types of stress barriers. In this regard the model examines the second phase as a long range barrier in carbon steels with different pearlite volume fraction. Experimental stress-strain relationships for these materials have been generated using the Split Hopkinson Pressure Bar testing technique. Results of these tests are used to identify
microstructurally sensitive parameters to be incorporated into the governing model equations.

Chapter 3 is a manuscript presented following guidelines of the *Journal of Material Science and Engineering A*, for which it has been submitted for publication. In this paper, the role of grain size on the dynamic response of single phase materials is investigated. For this purpose, a coarse-grain Al6061 has been refined using a severe plastic deformation method known as Equal Channel Angular Pressing. The flow stress of both the coarse and fine grain materials is obtained for a range of strain rates and temperatures using the Split Hopkinson Pressure Bar. Results of these experimental tests are utilized to generate model parameters that are explicit functions of grain size, strain, strain rate and temperature.

Chapter 4 presents major conclusions of the thesis work as well as detailing future recommendations.

The thesis includes two appendices that describe the two experimental facilities designed and constructed at the Mechanics of Materials Research Laboratory for completion of the research goals of the thesis. Appendix A details the Split Hopkinson Pressure Bar apparatus with high temperature capabilities. Appendix B gives the details of the design and construction of an Equal Channel Angular Press utilized in the generation of fine grain material.
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CHAPTER 1

INTRODUCTION

1.1 Statement of Problem

The study of material performance for use in structural designs must consider loading conditions spanning a range of parameters including strain, strain rate and temperature. The case of high strain rate loading, above $10^2$, has been of great interest as the material response is known to change significantly in this dynamic loading regime as opposed to lower rate loading conditions. In order to investigate a materials behavior subjected to high strain rate loadings, several models have been developed for identifying the corresponding stress-strain relationship as a function of the loading parameters mentioned above. These models include the well-known Johnson-Cook (JC) and Zerilli-Armstrong (ZA) relationships [1,2] which, while shown to accurately predict the dynamic response, their parameters lack a direct correlation with microstructure characteristics. The absence of this correlation, limits the predictive ability of these models to materials and loading conditions for which the model parameters are determined experimentally. Dynamic deformation mechanisms have been shown to depend, in addition to loading parameters, on microstructure features such as grain size, secondary phase particles, dislocation density and homogeneity as well as the alloying elements of the solid solution. The success in formulating models that account for the loading and microstructure conditions would, in addition of establishing fundamental understanding of the dynamic deformation mechanism, provide the ability to tailor microstructures to obtain a desired dynamic response numerically, without the necessity to develop and test
materials through an expensive and lengthy experimental trial-and-error approach. To overcome this deficiency, the work in this thesis has focused on introducing microstructure parameters into a physically based constitutive dynamic flow stress model. This has been achieved by completing the following goals:

i) Incorporation of material dependent parameters that reflect the role of pearlite being a second phase strengthener in carbon steels, into a physically based constitutive relationship of the dynamic flow stress. The generalized model utilized in this thesis is developed on the basis of interactions between dislocations and barriers to their motion. These interactions have been shown to be the source of the plastic strength which is separated into two independent stress components; athermal and thermal. The athermal component is only a function of strain and microstructure while the thermal component is strain rate and temperature dependent and is sensitive to the crystalline structure of the material. Barrier sources are categorized into two separate stress field types depending on their range of influence. These are long range and short range. The former barriers are those with effective stress fields greater than 10 atomic distances [3], examples of which include grain boundaries and second phase particles which when interact with dislocations, generate an athermal flow stress. Short range barriers are those with generated stress fields being less than 10 atomic distances [4]. These are mainly Peierls-Nabarro barriers in BCC metals which when interact with dislocations result in a thermal flow stress. The role of these two barriers, long and short, have been investigated in relation to the influence of second phase particles on the dynamic flow stress of low carbon steel. This has been achieved by examining microstructures with and without the pearlite phase. The steels utilized in this thesis work are, A572, 1018
and 1060 carbon steels with varying volume fraction of pearlite. To determine the model parameters for both the thermal and athermal stresses as a function of the pearlite volume fraction, a series of high strain rate compression tests have been conducted using Split Hopkinson Pressure Bar in a range of strain rate and temperature.

ii) The second microstructure feature to be considered in relation to the dynamic flow response of materials is the grain size which is known to influence the athermal stress component via the Hall-Petch relationship, in which the flow stress is inversely proportional to the grain size [5]. The validity of this relationship breaks down as grain size is refined, particularly below 10μm [6], where dislocation/barrier interaction may no longer be the dominant source for deformation accommodation. Different grain size scales have therefore been identified in terms of the corresponding deformation mechanism [7]. These scales include coarse grain (>1μm), ultra-fine grained (20nm-1μm) and nano-grained (<20nm). The transition between each of these scales while is not well defined, is expected to be a material dependent. It is therefore, important that a mechanistic based flow stress model to not only take into account a direct grain size parameter but also have the capability of transitioning through the different grain size scales. This type of model could provide insight into deformation mechanisms associated with each of these scales as well as a procedure to determine the grain size at which transition between the different scales would occur. In order to achieve grain refinement of a coarse grained material, a severe plastic deformation method, Equal Channel Angular Pressing (ECAP), is applied on an aluminum alloy, Al6061. Similar to the work on LCS, the model parameters for both the thermal and athermal stresses as
a function of the grain size, are determined by conducting a series of high strain rate compression tests using SHPB in a range of strain rate and temperature.

The experimental testing and numerical modeling which is carried out to investigate the role of second phase particles and grain size in low carbon steel (BCC) and aluminum alloy (FCC) are detailed in chapters 2 and 3 of the thesis, respectfully. The significance of constitutive equations to model the stress-strain response of a material at high strain rates and a range of temperatures is reviewed in the following sections, followed by the justification of the research presented and carried out in this thesis.

1.2 Models of Dynamic Flow Response of Metals

Material models are utilized in order to predict a materials response under different loading conditions in order to simulate mechanical responses which generally include yield strength, strain hardening, and strain rate and temperature effects. Typically, yield strength defines the level of stress at which linear elastic properties no longer exist and a plastic behavior is defined. Strain hardening is the rate at which a material increases in flow stress with an increase in strain. Temperature effects, in a general sense, include thermal softening, where an increase in temperature results in a decrease in flow stress, until a saturation of this relationship occurs at a critical temperature and flow stress. Strain rate effects in most metals include an increase of flow stress with an increase in strain rate. There exist two separate and distinct linear relationships between flow stress and strain rate, usually a less sensitive region that exists from very low strain rates \(10^{-12}\text{ s}^{-1}\) to approximately \(10^{0}-10^{1}\text{ s}^{-1}\). The other distinct region includes a relatively high degree of sensitivity, usually above \(10^{2}\text{ s}^{-1}\), considered to
medium/high strain rate testing regime. The increase in sensitivity has been shown to be due to an enhanced rate of dislocation generation [8-10]. Due to this transition of rate sensitivity and its mechanistic cause, it is important to develop specific dynamic constitutive equations that are capable of accurately predicting this typical material response. The two most widely used material models are the Johnson-Cook (JC) flow stress equation and the Zerilli-Armstrong (ZA) material models. An explanation of these two models, their formulations and examples of their use will be presented in the following.

The JC material model for the flow stress as a function of strain, strain rate and temperature is expressed as in equation 1-1 [1],

$$\sigma = (A + B\varepsilon^n)(1 + C\ln\dot{\varepsilon}^*)(1 - T^m)$$  

(1-1)

The first set of terms represents the effect of strain hardening, the second set of terms represents the strain rate hardening while the third is representative of the thermal softening effects. The parameters of $A$, $B$, $C$, $n$ and $m$ must all be experimentally determined, where $\sigma$ is the dynamic flow stress, $\varepsilon$ is the plastic strain, $A$ is the yield stress at a reference temperature and strain rate, $B$ is the coefficient of strain hardening, $n$ is the strain hardening exponent and $C$ and $m$ are material constants that represent strain rate hardening and thermal softening respectfully. $\dot{\varepsilon}^*$ is the dimensionless strain rate given by $\dot{\varepsilon}^* = \dot{\varepsilon}/\dot{\varepsilon}_o$ in which $\dot{\varepsilon}$ is the strain rate of a given condition and $\dot{\varepsilon}_o$ is a reference strain rate at which parameters are determined. $T^*$ is the homologous temperature which is expressed as;
\[
T^* = \frac{T - T_{\text{ref}}}{T_m - T_{\text{ref}}}
\]  

(1-2)

where \( T \) is the testing temperature in absolute, \( T_m \) is the melting temperature and \( T_{\text{ref}} \) is the reference temperature at which parameters will be determined.

To determine the model parameters one must start with a test condition at a reference temperature and strain rate. This reduces equation 1-1 to the following;

\[
\sigma = A + B\varepsilon^n
\]  

(1-3)

In this reduced form, \( A \) is considered to be the yield stress at zero plastic strain. Using flow stress data at different intervals of strain, \( \ln(\sigma - A) \) versus \( \ln(\varepsilon) \) can then be plotted in a linear form for which the parameters \( B \) and \( n \) are determined as the coefficient and the exponent of the power law fit, respectively. The parameter \( C \) in equation 1-1, is determined by considering the form of this equation at the reference temperature which results in the elimination of the temperature sensitivity term, thus:

\[
\sigma = (A + Be^n)(1 + C\ln\dot{\varepsilon}^*)
\]  

(1-4)

By utilizing data of flow stress from varied levels of strain rate at one particular strain value, a plot of \( \ln(\sigma/(A + Be^n)) \) versus \( \ln\dot{\varepsilon}^* \) can be generated, in which the slope of this line represents the parameter \( C \).

To determine parameter \( m \), a consideration is made at the reference strain rate in which the equation 1-1 becomes equation 1-5 by removing the strain rate hardening effects.
\[
\sigma = (A + Be^\sigma)(1 - T^{-m})
\] (1-5)

A plot of \( \ln\left[1 - \left(\frac{\sigma}{(A + Be^\sigma)}\right)\right] \) VS \( \ln T^* \) is generated using flow stress data at a constant strain level at an elevated temperature. A power law fit is created in which the coefficient is representative of \( m \).

The ZA model as function of strain, strain rate and temperature is used to describe the flow stress behavior of materials has been derived to be equation 1-6 for FCC microstructures [2].

\[
\sigma = \sigma_0 + kd^{\gamma_2} + C_1 \exp(-C_3T + C_4T \ln \dot{\varepsilon}) + C_5\dot{\varepsilon}^n
\] (1-6)

The equation predicts the high temperature material characteristics considering isotropic hardening, temperature softening, strain rate hardening and coupled effects of temperature and strain and strain rate and temperature. \( \sigma \) is the dynamic flow stress, \( \varepsilon \) is the plastic strain, \( \dot{\varepsilon} \) is the strain rate of a given condition and similarly for \( T \), the temperature. The parameters of \( C_1, C_3, C_4, C_5 \) and \( n \) are material constants that must be determined using experimental data and a procedural analysis. By assuming that the portion of the equation of \( C_0 = \sigma_0 + kd^{\gamma_2} \), is independent of temperature and therefore an athermal component of stress, one can plot flow stress versus temperature for multiple strain levels and strain rates. It can be seen that at a critical temperature, the flow stress will saturate with temperature and strain. This value of stress is determined to be \( C_0 \).

With \( C_0 \) determined and assuming the yield stress at zero plastic strain and taking the natural logarithm, equation 1-6 is simplified to equation 1-7,
\[
\ln(\sigma - C_0) = \ln C_1 - C_3 T + C_4 T \ln \dot{\varepsilon}
\] (1-7)

By plotting \(\ln(\sigma - C_0)\) Vs \(\dot{\varepsilon}\) for a varied range of temperatures and applying a 2-parameter logarithm fit to each temperature curve, parameter \(C_4\) can be determined to be the coefficient that saturates with temperature. The second parameter of the logarithmic fit of the data, denoted \(S_f\), is concluded to be \(\ln C_1 - C_3 T\). By generating a plot of \(S_f\) Vs \(T\) and applying a linear fit, parameters \(C_1\) and \(C_3\) can be determined. To determine the final two parameters, \(C_5\) and \(n\), the critical temperature at which the flow stress saturates is considered once again at the reference strain rate. With this consideration, equation 1-6 simplifies to:

\[
\sigma = \sigma_y + C_5 \varepsilon^n
\] (1-8)

By plotting \(\sigma - \sigma_y\) versus \(\varepsilon\), and fitting a power law relationship to this data, \(C_5\) and \(n\) can be determined. The utilization of these models is prevalent throughout literature. This is most likely due to not only the proven accuracy of the models, but also the relative ease of model parameter generation, through the use of experimental stress-strain curves in a range of strain rates and temperatures and followed by the procedural analyses as presented.

Johnson and Cook [1] presented the constitutive model for use in computations in 1983 that includes the effects of strain and strain rate hardening and temperature softening. Model parameters were determined and confirmed for many materials that include copper, brass alloys, steel alloys, iron, tungsten alloys and aluminum alloys. Continued research of the models validity and use has been completed on other materials.
by many other researchers. Wang et al [11] studied the validity of the JC material model by predicting the response of Ti-6Al-4V under high speed ball impacts. Experimental impact craters were generated and results compared to finite element results in which the JC model was employed. Results showed good agreement between the experimental and computational results, and thus provides a validation of the JC model for this material to be utilized in other various simulations. DeMange et al [12] utilized the JC model to investigate blunt projectile penetration of a nickel-based super alloy. The investigation included the need of generating two separate sets of model parameters, first for an annealed case and secondly for a precipitate hardened microstructure of the same super alloy. Individually each set of model parameters resulted in good agreements when compared between FEA computational results with experimental high strain rate compression top hat shear tests. Khan and Liang [13] made comparisons of both the JC and ZA models to low strain rate experimental tests of tantalum. It was stated that each constitutive relationship was unable to accurately describe the decrease of work hardening with increasing strain rate. Therefore attempts were made to generate another model that has the ability to handle the coupled effects of strain and strain rate hardening for the tantalum studied. A similar result was seen by Khan et al [14] when comparing JC model predictions to experimental quasi-static and dynamic test results. Mishra et al [15] developed JC model parameters for three different material conditions of copper in which grain size was varied. Although results were seen to provide good agreement between computational and experimental results, three completely separate sets of model parameters had to be generated to describe each material condition individually.
Zerilli and Armstrong developed their constitutive relationships in 1986 in which two separate equations were derived for FCC and BCC microstructures [2]. Model parameters for these equations were determined for copper (FCC) and iron (BCC). Comparisons were made between experimental and simulated Taylor impact tests, and it was noted that the ZA model predicted the material response more accurately then the JC model, with the reasoning that the ZA model is based on dislocation mechanisms, rather than simple phenomenological methods as the JC model. Further investigations and validations of the ZA model have since been completed. Lee and Lin [10] investigated the impact response of a stainless steel with and without an applied pre-strain before impacting, thus modifying the dislocation network. The ZA model should good agreement with high strain rate experimental results for both material conditions that required the determination of independent parameters for each condition. Lee and Liu [16] generated ZA model parameters for steels with varying carbon contents (low, medium and high). Each set of parameters when utilized with the model should good agreement with experimental stress strain curves. He et al [17] developed model parameters of both the ZA and JC constitutive equations for a steel alloy. The results of this investigations showed that the ZA model was more accurate in the predictions made when comparing to experimental high strain rate tests, but it was noted that the computational time to develop the parameters of the ZA model was much more intensive. A comparative investigation of the JC model and ZA models ability to model behavior of a steel alloy was completed by Samantaray et al [18]. Within this investigation it was stated that the accuracy of a numerical model is highly dependent on the accuracy of the constitutive relationships that describes the mechanical behavior of the material being
represented. It was shown that the JC model as originally derived is unable to accurately predict the material behavior due to the lack of the coupled effect of strain and temperature and strain rate and temperature within the derivation. On the other hand the ZA model was acceptable in predicting experimental results, noting also that the ZA does have more parameters required to determine.

In summary, the work reviewed above shows that the physics-based ZA model provides a more accurate prediction of the dynamic flow response of many different materials with varying compositions and microstructures, as opposed to the phenomenological JC model. This improved accuracy is attributed to the fact that the model formulation accounts for the deformation mechanisms by considering the nature of dislocation interactions depending on the crystal structure. This has been accomplished by providing different model equations for the BCC and the FCC materials. It is important, however, to observe that from application point of view, while JC model parameters can be determined with relative ease, the ZA model is more complex and requires a larger set of material parameters.

More recently a model has been developed by Nemat-Nasser et al [19-21] in which the flow stress is expressed as the sum of two separate stress components each of which is dependent on the corresponding barrier sources to dislocation motion. The model has been applied to predict materials’ responses considering effects of strain, strain rate and temperature. In these works, the authors point out to the fact that their model formulations allow the incorporation of microstructure related parameters, such as grain size, secondary phase particles, alloying content, dislocation density as well as crystalline
structure. Results of the JC and ZA models, as discussed above, show that these microstructural characteristics have a direct effect on the model prediction response.

1.3 Thesis Justification

In the past several decades, a large amount of research efforts has been carried out to examine the dynamic response of materials with particular emphasis on the hardening characteristics and related flow stress patterns. These studies have shown that the evolution of the flow stress is affected significantly by strain, strain rate and temperature. Many attempts have been made to quantify these relationships through models that account for both loading and material conditions. These models are generally required as a design tool to predict materials performance under real-life scenarios including dynamic loadings with or without temperature. Examples of high strain rate loadings, among others, are explosive detonation, automobile crashes or ballistics. Additionally, these dynamic flow models are employed to investigate deformation mechanisms that occur during loading events considering effects of strain and strain rate hardening, and temperature sensitivity. Successful models in this regard would provide a tool to develop materials with tailored microstructures suitable for dynamic loading resistance. An important outcome of these studies is the observation that the strain rate sensitivity of the flow stress changes significantly at strain rates higher than $10^2\text{s}^{-1}$. This strain rate sensitivity transition is attributed to the distinct increase of the dislocation generation rate when compared with that at lower rate loadings ($<10^0-10^1\text{s}^{-1}$). The increase in dislocation generation rate has been shown to be due to the competition between dislocation generation and annihilation, in which the increase in strain rate decreases the time available for annihilation (recovery) and dislocation rearrangement processes, such as
cross-slip, to occur. This transition phenomenon is illustrated in Figure 1-1 for three different materials; aluminum [22], stainless steel [10] and copper [23].

Furthermore, it was shown that the flow stress is strongly dependent on microstructural characteristics such as grain size, secondary phases, alloying content, dislocation networks and crystalline structure [10,22,23]. At high strain rate levels, this dependency is explained in terms of the dislocation motion, being a source of deformation accommodation, as it is affected by interactions with microstructure related stress barriers. This correlation between flow stress and microstructure indicates that an accurate dynamic flow stress model must account for these dislocation interactions by considering the governing microstructure features. Development of such a model is the primary goal of this thesis with a focus on two strengthening microstructure characteristics; secondary phases and grain size. The two materials used to study the roles of these characteristics are carbon steel in relation to the second phase pearlite volume fraction and an aluminum alloy in relation to grain size refinement.
Figure 1-1: True Stress plotted versus log strain rate of data obtained from literature for a) an aluminum alloy [22] and stainless steel [10] and b) copper [23], noting the sharp increase in strain rate sensitivity at around $10^1$-$10^2$ s$^{-1}$.
Carbon steel is selected due to its dual phase microstructure in which the strength is governed by its carbon content. The proposed dynamic flow stress model presented in this thesis will be applied to A572 grade 50 low carbon steel which consists of alpha ferrite matrix with secondary phase pearlite colonies in the form of a lamella structure of alternating platelets of cementite and alpha-ferrite. The relative contribution of pearlite, being a strengthening phase, has been studied by D. Edmonds [24], where the quasi-static tensile strength of steels was measured as a function of the carbon weight percent which determines the pearlite volume fraction of the alloy. In addition, Campbell [25] has shown that the strength of low carbon steel is the sum of contributions due to grain size, pearlite and solid solution, see Figure 1-2. For the same material, while the role of the grain size is generally modeled by the Hall-Petch law [2,5], a constitutive relationship between the dynamic flow stress and pearlite content has yet to be established. This type of relationship, as discussed above, would need to be based on physical parameters that represent events taking place during the dynamic deformation process, specifically dislocation/barriers interactions.
Figure 1-2: Quasi-static flow stress versus carbon weight. This shows that pearlite, outlined in blue, is the most effective manner to increase the strength of the steel. Grain size, outlined in red, also being a source of strength, but relatively insensitive to carbon weight percent [24].

Models such as Johnson-Cook or Zerilli-Armstrong [26], are widely used in predictions of the dynamic flow stress. Parameters of these models, while include temperature and strain rate sensitivities, lack physical representations and are phenomenological in nature. To overcome this deficiency, other approaches, such as the thermal activation theory [4,8,20,21,27-29] has been used to derive constitutive models
capable of expressing the mechanical response of metals over a broad range of strain rates and temperatures on the basis of deformation mechanisms evolving due to dislocation motion. This theory is based on the thermally assisted movement of dislocations reaching, penetrating and passing through a barrier; thus leading to plastic deformation. Barriers to dislocation motion are described as two types; short range and long range. Short range barriers include phonons, forest dislocations, dislocation jogs and kinks and Peirs-Nabarro stress. Long range barriers include grain boundaries, large second phase particles and dislocations on parallel slip planes [3,8]. The flow stress due to high strain rate loadings can thus be considered as the summation of individual stress components, each of which represent a physical flow phenomenon. Short range barriers to dislocation motion and interactions of dislocations with these barriers result in a thermal stress component, generally dependent on the strain rate and temperature loading conditions. The long range barriers and their interactions with dislocations result in an athermal stress component which is dependent on strain as well as microstructural features such as grain size, microstructural phases and alloy elements [20]. The summation of these two stress components is shown graphically in Figure 1-3, in which the larger low frequency hills represent the long range stress fields, while the smaller more frequent hills represent the stress fields due to short range barriers. Here it can be seen that the maximum stress due to short range barriers, σ_o, will occur at 0K, known as the mechanical threshold stress at which no thermal fluctuations aid in the passing of the barrier. With the addition of thermal fluctuations caused by an increase of temperature, the mechanical stress, σ, that is needed to overcome the short range barrier and its stress field, σ_{th}, is lowered. With further increasing of the temperature, the totality of this hill
will diminish (shaded area in the Figure 1-3) and thus the mechanical stress becomes athermal in nature, $\sigma_{\text{ath}}$, at which any further increase of temperature will not lower the stress required to overcome the barrier.

![Figure 1-3: Internal stress fields encountered by a dislocation moving through the crystal lattice [4]](image)

A third component of stress that can be included into this summation is due to viscous drag of dislocations moving through the solid solution from barrier to barrier during plastic deformation [8,21]. This only occurs at very high strain rates ($>10^4 \text{s}^{-1}$) where the plastic deformation of the material is no longer dominated by stress-assisted thermally activated processes as previously expressed, but rather dominated by the
viscous drag of dislocations and the duration in which it takes for mobile dislocations to move from barrier to barrier [4,10]. Since strain rates above $10^4\text{s}^{-1}$ are not considered in this thesis, stresses due to viscous drag has not been taken into account. This restriction represents an upper limit of the applicability of the constitutive flow model presented in this thesis.

Ogawa et al [28] used quasi-static data to generate model parameters for a constitutive model based on thermally activated motion of dislocations that is capable of predicting high strain rate response that would be seen in SHPB testing. These results showed that using thermal activation theory, and the separation of flow stress into components to explain mechanical behavior, is an accurate method to generate a constitutive model with physically based parameters. Nemat-Nasser et al [21,27] has extensively studied the dynamic response of several materials including vanadium, tantalum and alloys, titanium, copper, molybdenum and niobium all utilizing the thermal activation theory in which the separation of flow stress into components was utilized to generate constitutive model parameters with the interest in addressing the plastic deformation as a function of material microstructure, strain rate and temperature based on the motion of dislocations. Results summarized include the initial microstructure affects the initial plastic flow of metals, that alloying of materials effects the flow response of materials, and that the procedure of analysis using separation of flow stress into components based on physical phenomenon of dislocation interactions with different barrier sources is a valid and accurate manner in generating a constitutive model.

Lee and Liu [16] studied the dynamic behavior of different steels with varying weight percent of carbon with respect to the effects of temperature and strain rate. Results
of their work showed, as in many BCC metals, their exists an inverse relationship of flow stress with increasing temperature and an increase in flow stress with increasing strain rate. Results also showed that there exists an increasing relationship with carbon content in which flow stress increases with increasing carbon content, and an increase in temperature sensitivity with increasing carbon content. Lee and Lin [10] studied the effect of pre-strain on 304 stainless steel, and results showed that there is a sensitivity to the dynamic response of the steel to adjustments in the microstructure of the steel via pre-straining, which adjusts the dislocation network and density inherent in the material. They too noted the relationship of increasing flow stress with strain rate and decreasing with increasing temperature, but also showed that there is a sensitivity with these properties with the microstructure of the material, this case dislocation network and density. Bardelcik et al [30] studied the effect of cooling rate on the high strain rate response of steel, showing that the changes in microstructure with phases including martensite, bainite, pearlite and ferrite, had a noticeable effect in the dynamic response of the steel, showing the microstructure sensitivity to dynamic response.

As discussed above, the second strengthening microstructure feature to be considered in the current analysis is the role of grain size. Grain refinement in materials has been of research interest for several decades as it is considered a means by which the strength of a material can be enhanced, which in combination with post-processing heat treatments, allows for the material to retain much of its original ductility [6,31-43]. The ability to accurately model the flow response of a material as a function of grain size is of great importance in order to further understand the corresponding dynamic deformation mechanisms. K. Muszka et al [44] has studied the effect of grain size on the dynamic
mechanical properties of micro-alloyed steels and showed that the mechanical response strongly depends on the grain size. The work of Q. Wei [6] has shown that the strain rate sensitivity of UFG BCC metals to be highly reduced compared to that of coarse grain microstructures. On the other hand, the strain rate sensitivity of FCC microstructures was shown to increase with decreasing the grain size.

The understanding of mechanisms that occur during the deformation of materials with refined granular structures has proved to be difficult, as this requires models capable of expressing the evolving deformation mechanisms as the grain size changes from coarse (<10μm) to ultra-fine (>10μm). H. Miyamoto et al [32], explained the observed constant flow stress in UFG material by the suggestion that dislocation emission and annihilation occurs at non-equilibrium grain boundaries of UFG, thus resulting in a constant dislocation density. H.S. Kim et al. [33] studied the plastic deformation of UFG materials and the deviation from the typical Hall-Petch relationship. Many explanations of this deviation have been proposed including grain-boundary sliding, contribution of triple junctions, grain boundary creep, phase transformation, grain boundaries as sources and sinks for dislocations. To accurately model this deviation, a phase mixture model, in which the material is considered a composite material, was shown to provide accurate results.

The general conclusion from the review of the current literature in relation to the dynamic flow stress models, as presented above, shows that their predictive abilities are limited to the material, temperature and loading conditions for which the model parameters are generated. The goal of this thesis, therefore, is to develop a microstructure-explicit flow stress constitutive model that accounts for the fundamental
deformation mechanisms occurring in materials subjected to dynamic loadings. The two important parameters that will be investigated in this thesis are the role of a second phase in dual phase materials such as carbon steels and the role of grain size in single phase materials such as aluminum alloys.

1.4 Thesis Approach

As discussed above the goal this thesis is to identify the dynamic flow stress as a function of governing microstructure features. To achieve this, a constitutive flow stress model based on the separation of thermal and athermal stress components has been adopted. The two microstructural features of interest are the second phase volume fraction in dual phase materials and grain size in single phase alloys. The materials investigated in this regard are carbon steel where the volume fraction of pearlite is considered to be the strengthening phase and aluminum alloys where hardening is proportional to the grain size. Model formulations for these two materials, carbon steel and aluminum, are developed where related parameters are determined by obtaining the corresponding dynamic stress-strain relationships as a function of pearlite volume fraction and grain size obtained in its fine form. Variations in the pearlite volume fraction were obtained by a series of heat treatments performed on steels with different carbon content. The control of grain size in aluminum alloys was carried out using a severe plastic deformation approach. In order to obtain the stress-strain curves as well as refine the grain size, two experimental tools are designed and built in the Mechanics of Materials Research Lab. These are the Split Hopkinson Pressure Bar (SHPB) and an
Equal Channel Angular Press (ECAP), detailed descriptions of which are given in Appendix A and B, respectively.

The SHPB is a widely used testing system for generating high strain rate stress-strain curves [45-49]. The SHPB designed and built in this thesis has several unique capabilities including the ability to test materials at temperatures ranging from the liquid nitrogen level, using a liquid bath method, to above 900°C using an induction heating system. Included in the extreme temperature testing setup is the use of a specially designed actuator for automatic positioning of loading bars. The system is precisely timed using an Eaton timing relay that also controls the firing of the gas gun projectile used as a means in causing the dynamic loading event. The timing relay is capable of timing the entire system to a millisecond resolution. By use of a data acquisition system in conjunction with strain gauge measuring techniques and Matlab coding, experimental stress-strain curves can be generated.

In order to investigate the role of pearlite colonies on the dynamic flow stress of low carbon steel through the derivation of a constitutive model and its parameters, presented in chapter 2, heat treatment procedures are developed to control the pearlite content. The desired result of the heat treatment of A572 grade 50 steel, is the removal of the secondary phase, pearlite colonies that account for a volume fraction of approximately 9% of the material. The heat treatment includes a dual stage procedure in which the result includes the primary alpha-ferrite matrix with spheroidized carbides residing along the grain boundaries of the primary phase.
In order to investigate the effect of grain size of the flow stress of dynamically loaded Al6061, an ECAP system was designed and built, in which a total of 400% strain has been applied to the Al6061 material in order to refine its coarse granular structure from 100μm, in the annealed condition, to a fine grain scale of 5μm. The ECAP system consists of a high strength tool steel die equipped with heating, displacement control and a data acquisition system and is mounted within a 100 ton hydraulic press. The Al6061 material generated by the severe plastic deformation processing as well as that of the annealed condition is further analyzed using SHPB testing techniques under different high strain rates and temperatures.

The model utilized to describe the dual phase carbon steel in chapter 2 and the single phase Al6061 in chapter 3, is based on the thermal activation theory which defines the thermal resistance of barriers to dislocation motion. Barrier sources and its resultant stress field that allow a thermally-assisted dislocation to pass through are short range in nature and have stress fields that are less than 10 atomic distances. The flow stress that results from the interaction of dislocations with these types of barriers is thus termed, the thermal component of stress and is generally a function of strain rate and temperature. Barriers in which thermally assistant has no affect in whether or not a dislocation can pass, have resultant stress fields greater than 10 atomic distances. The resultant flow stress generated by dislocations interacting with short range barrier types is termed the athermal component of stress, due to its inherent temperature insensitivity. The athermal component is defined as being strain dependent, while also being sensitive to microstructural characteristics such as grain size or volume fraction of large precipitates. The summing contributions of athermal, \( \sigma_a \), thermal, \( \sigma_{th} \), and also the inclusion of
dislocation drag, \( \sigma_d \), stress components, each arising from individual mechanisms involving dislocation interactions with different types of barriers, is given as in equation 1-9. The drag stress is not included further in this derivation due to the lack of flow stress dependency until above \( 104 \text{s}^{-1} \), not within the scope of this work.

\[
\sigma = \sigma_a + \sigma_{th} + \sigma_d
\]  

(1-9)

The athermal stress, \( \sigma_a \), as shown by Taylor et. al [5] is a function of strain and other microstructural parameters. The athermal stress could thus be written as a function of plastic strain \( \varepsilon \):

\[
\sigma_a = \sigma_a^* \varepsilon^n + \sigma_M
\]  

(1-10)

Where \( \sigma_a^* \) and \( n \) are material constants and \( \sigma_M \) represents the stress due to the microstructural characteristics. Furthermore, the athermal stress is indirectly affected by temperature through the temperature dependency of the elastic and shear moduli. In order to remove this dependence the athermal stress with the temperature dependent elastic modulus, the stress component is normalized by the elastic modulus function as follows:

\[
\bar{\sigma}_a = \sigma_a \frac{E_o}{E(T)}
\]  

(1-11)

\( E_o \) is the reference elastic modulus at room temperature and \( E(T) \) is the shear modulus at a given temperature.

The thermally active thermal stress component, \( \sigma_{th} \), as mentioned above, is due to interactions between dislocations and short range barriers. Due to the nature of
dislocation forest density increasing with increasing strain, the thermal component of stress in FCC metals includes the role of strain. To attain a general relation between temperature, strain rate and the thermal stress component, the energy required for a dislocation to overcome a barrier by thermal activation is written as:

$$\Delta G = G_o \left[ 1 - \left( \frac{\sigma_{th}}{\hat{\sigma}} \right)^p \right]^q$$  \hspace{1cm} (1-12)

p and q are constants that define the profile of the barrier to be crossed, where $0 < p \leq 1$ and $0 \leq q \leq 2$. $G_o$ is the energy required to overcome a barrier solely by thermal activation and is given by

$$G_o = \hat{\sigma} \lambda lb$$  \hspace{1cm} (1-13)

$\hat{\sigma}$ is the stress required for a dislocation to overcome a barrier without any thermal assistance, $\lambda$ and $l$ are the average effective barrier width and spacing respectively. It is assumed that strain rate is related to $\Delta G$ by the following,

$$\dot{\varepsilon} = \dot{\varepsilon}_r \exp \left( -\frac{\Delta G}{kT} \right) \text{ where } \dot{\varepsilon}_r = \rho_m b \omega_o l$$  \hspace{1cm} (1-14)

$k$ is the Boltzmann's constant, $\omega_o$ is the attempt frequency for a dislocation to overcome a barrier, $b$ is the burgers vector and $\rho_m$ is the mobile dislocation density. Combining equation 1-12 and 1-14 and solving for $\sigma_{th}$, one can obtain an expression for the thermal stress as:
\[
\sigma_{th} = \hat{\sigma}_{th} \left[ 1 - \left\{ \frac{-kT \ln \frac{\dot{\varepsilon}}{\dot{\varepsilon}_0}}{G_o} \right\}^{1/q} \right]^{1/p}
\]  

(1-15)

The thermal stress component, as can be seen from equations 1-13 and 1-14, depends on the dislocation spacing, \( l \). To include this into the derivation of the equation for a more general sense to include FCC metals where the dominant short-range barrier is dislocation forests, means that \( l \) must be an evolving function of the average dislocation spacing. This is expressed as [20]:

\[
l = \frac{l_o}{f(\varepsilon, T)}
\]  

(1-16)

where \( l_o \) is the initial average dislocation density. From this relation it can be expressed that:

\[
\hat{\sigma}_{th} = \sigma^o f(\varepsilon, T) \quad \text{with} \quad \sigma^o = \frac{G_o}{b\lambda l_o}
\]  

(1-17a,b)

\[
\dot{\varepsilon}_r = \frac{\dot{\varepsilon}_0}{f(\varepsilon, T)} \quad \text{with} \quad \dot{\varepsilon}_0 = \rho_m b \omega l_o
\]

It is reasonable to assume that the average dislocation spacing increases with further straining in a manner similar to work-hardening effects and decreases with increasing temperature as in annealing effects. These trends are written as

\[
f(\varepsilon, T) = \frac{l_o}{l} = 1 + a \left[ 1 - \left( \frac{T}{T_m} \right)^2 \right] \varepsilon^n
\]  

(1-18)

By substituting equations 1-17a,b and 1-18 into equation 1-15, the thermal stress can be expressed as:
\[ \sigma_{th} = \sigma^o \left[ 1 - \left( \frac{-kT}{G_o} \ln \frac{\dot{\varepsilon} f}{\dot{\varepsilon}_o} \right)^{1/q} \right]^{1/p} f \]  

(1-19)

From this the constitutive relationship for flow stress as a function of strain, strain rate, temperature and grain size can be assembled in the following form:

\[ \sigma = \sigma^* \dot{\varepsilon}^n + \sigma_M + \sigma^o \left[ 1 - \left( \frac{-kT}{G_o} \ln \frac{\dot{\varepsilon} f}{\dot{\varepsilon}_o} \right)^{1/q} \right]^{1/p} f \]  

(1-20)

It is noted that this relationship still remains true for BCC metals, where from equation 1-18, \( f(\varepsilon, T) = \frac{l_{\varepsilon}}{l} \), would simply be equivalent to 1. Here, for BCC metals, the initial dislocation barrier distance does not evolve with strain. To examine the validity of the flow stress equation as expressed above requires the identification of the material parameters, \( \sigma^*, n, \sigma_M, \sigma^o, p, q, a \) and \( m \), through the use of experimentally generated stress-strain curves. This model will be shown in chapter 2 and 3 to be extremely useful tool in completing the major goals of the thesis research.

1.5 Thesis Overview

The thesis is divided into four chapters and two appendices. Chapter 1 is an introduction which summarizes the goals of the thesis and presents a review of dynamic flow responses of metals which lead to thesis justification. This chapter also reviews the modeling and experimental approach utilized in the thesis to achieve its objectives. Chapter 2 describes the determination of parameters used within a constitutive relationship for carbon steel. Model parameters are generated as a direct function of the microstructural characteristics, specifically the volume fraction of pearlite being the
second phase strengthener, as well as strain, strain rate and temperature. Chapter 3 describes the development of a dynamic flow stress model and identifies the model parameters for the transition through multiple grain size scales each of which is characterized by a distinct deformation mechanism. The material of concern is the single phase aluminum Al6061 in both coarse and fine grain size. The model formulations include effects of grain size, strain, strain rate and temperature.

Chapter 4 presents the major conclusions obtained in the thesis work as well as gives recommendations for future studies in the dynamic flow stress of metals. Additionally the thesis includes two appendices describing in details the design and operation of the two experimental tools used in the thesis. Appendix A describes the Split Hopkinson Pressure Bar (SHPB) which is used for the generation of high strain rate stress-strain curves, with the ability to test within a large range of temperatures. Appendix B describes in details an Equal Channel Angular Press (ECAP) designed and built to generate materials with refined granular structure, through sever plastic deformation of coarse grained microstructure,

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CHAPTER 2

"Role of Pearlite Colonies on Dynamic Flow Stress of Low Carbon Steel"

by

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2.0 Abstract

The dynamic deformation of low carbon steel (LCS) is examined within a range of strain rates and temperature conditions. The materials studied are as-received coarse grained steel, composed of α-ferrite and pearlite phases, and a heat treated LCS comprised of carbide particles dispersed within a ferrite matrix. Testing in the dynamic regime is completed using a Split Hopkinson Pressure Bar at strain rates of $10^2$ to $10^4$ s$^{-1}$ with testing temperatures of room temperature, 300, 500 and 650°C. The correlation between strain rate and corresponding flow stress showed a distinct transition delineating the quasi-static regime as dominated by the thermally activated flow stress and that of the dynamic regime. The strain rate and basic characteristics of the thermal and athermal stresses, are studied to determine the mechanisms of deformation as a function of the microstructure, strain rate and temperature. The relative influence of short and long range barriers on the flow stress components is studied in a LCS microstructure in which the pearlite colonies, an effective long range flow stress barrier, are removed. Separation of flow stress components is utilized in a constitutive equation based on thermal activation theory to predict flow stress as a function of strain, strain rate, temperature and explicit microstructure variables including pearlite volume fraction (VFP) and grain size. Results of this model will be compared with those obtained experimentally on both the as-received and heat treated LCS.
2.1 Introduction

Low carbon steel (LCS), is the primary reinforcing phase in civil structures due to its diverse nature of mechanical properties relative to its economical feasibility. Consideration of this material in structural designs must account for its effective properties under conditions resulting in abnormally high loading rates and elevated temperatures. These properties are generally studied utilizing models which incorporate strain, strain rate and temperature dependent parameters. Several models exist for calculating the stress-strain response under these conditions. One of the common models is that of Johnson-Cook (JC) [1] in which the material constants are determined from empirical fitting of experimental dynamic flow stress obtained from at various strain rates and temperatures. These constants do not include microstructure related parameters such as the grain size, phases and dislocation homogeneity and density which are known to influence the material dynamic response [2-5]. Another model is the Zerilli-Armstrong (ZA) that includes the effect of grain size by utilizing the Hall-Petch relationship for both the BCC and FCC crystal structures [6]. A third widely used phenomenological model is the Mechanical threshold stress (MTS) [7,8] which is based on a physical mechanism of thermally activated dislocation motion and focuses on the determination of mechanical threshold stress. The flow stress is expressed in terms of an athermal component due to barriers of thermally activated dislocation motion, the strain hardening due to dislocation accumulation, and strain-rate and temperature dependent scaling factors based on normalized activation energies. The model parameters do not include explicit microstructure dependent terms [9]. DeMange et al, [10] utilized the JC model to simulate the dynamic response of Inconel-718 in the annealed and precipitate-hardened
states. The hardening parameter in the model is written as a strain rate independent which could explain the fact that the model outcomes over predict the flow stress response at high strain rates. The model also did not accurately predict the saturation of the flow stress with increasing strain, a feature that was observed in the experimental results. Daridon et al. [11], studied effects of adiabatic shear band spacing on the dynamic response of HY-100 steel and Ti-6Al-4V alloy and concluded that the physically based MTS model was in better agreement with experimental results than that of the JC model. The authors reasoning being that the adiabatic shear bands act as thermal activation barriers for dislocation motion, a physical phenomenon that the JC model does not account for.

Lee and Liu [2] studied the dynamic behavior of different steels with varying weight percent of carbon with respect to effects of temperature and strain rate. Results of their work showed that the flow stress and temperature sensitivity increase with increasing carbon content. Lee and Lin [3] studied the effect of pre-strain on the 304 stainless steel where results showed that the dynamic deformation response is sensitive to pre-straining. They interpreted this effect in terms of the role of pre-straining in modifying arrangements and density of the dislocation network within the microstructure. Bardeleijk et al. [12] studied the effect of cooling rate on the high strain rate response of steel, showing the changes in microstructural phases including martensite, bantite, pearlite and ferrite, had a noticeable effect on the dynamic response of the steel by adjusting the UTS and hardening rate. Odeshi et al. [13] examined effects of the high rate loading of low alloy steel and showed that the plastic deformation is governed by two occurring processes. In the early stage, strain hardening which is strain rate dependent, is
dominant. As deformation progresses, adiabatic heating occurs causing thermal softening to dominate. Zhang et al [14] and Lee and Chen [15] have recently investigated the role that Al$_3$Sc precipitates have on the dynamic response of the material. It was shown that the secondary particles had a significant effect on the microstructural evolution during dynamic loading conditions. Zhang studied the condition of high speed projectile impacts while Lee and Chen utilized SHPB techniques to characterize the dynamic response. They similarly concluded that the particles had two effects; the first is related to the stabilization of the matrix and the second is due to effect of the particles acting as a major source of dislocations, thus resulting in an increase in the strain-induced hardening.

DeMange et al [10] studied the dynamic deformation response due to blunt projectile penetration, dynamic compression and top-hat dynamic shear testing of annealed and precipitate-hardened Inconel-718. During blunt projectile testing, using plate impacts, the annealed material had a higher resistance to penetration than that of the precipitate-hardened state. During dynamic compression tests, the annealed state resulted in a much lower over-all flow stress than the latter material, but showed considerably superior work hardening than the precipitate-hardened Inconel. The reason for the lowered work hardening in altered state of the Inconel was validated by top-hat geometry dynamic shear tests. Results of these tests showed that the precipitated hardened material readily forms shear bands, leading to localization which lowers the load capacity as compared to the annealed state of the material.

Ogawa et al [16] studied the high strain rate of low carbon steel employing a model based on the concept of separating the flow stress into two physically-based components. The first is an athermal component which is only a function of strain and is
modeled as a simple power law relation. The second is a thermal component that is a function of temperature and strain rate and is modeled assuming that deformation obeys a single thermal activation process. The combined dependence of the stress on strain rate and temperature is based on the Larson-Millar parameter. In a similar approach, Nemat-Nasser et al [4,5,17] has studied the dynamic response of several metals and alloys including vanadium, tantalum alloys, titanium alloys, aluminum alloys, OFHC copper, molybdenum and niobium and further considered the drag stress as an additional component in the flow stress calculations. The attractive feature of these models is that the different components of the flow stress being derived on the basis of dislocation dynamics as a function of strain rate and temperature would allow the inclusion of microstructure sensitive parameters.

The current work examines the dynamic flow stress of the low carbon steel as a function of pearlite volume fraction. Formulations of this stress are achieved by considering its separate thermal, athermal and drag stress components. The as-received microstructure of this alloy consists of equaixed alpha-ferrite grains with evenly dispersed pearlite colonies. The strength of the steel is due to the contributions of grain size, the pearlite phase and solid solution [18,19]. The pearlite colonies are a harder secondary phase than that of its ferrite matrix and would thus act as an effective long range barrier to dislocation motion [20,21]. In order to examine this effect, the flow stress of the as-received microstructure (9% volume fraction of pearlite), is assessed along with three other steels having 0%, 20% and 72% of pearlite volume fraction. The first part of the paper describes the materials used in the study followed by details of a dynamic flow stress model. The third part is the experimental program for determining parameters
required for the identification of the stress components. The last part of the paper is a discussion of the role of the pearlite volume fraction on the basis of the model outcomes.

2.2 Material

The material of study is A572 Grade 50 LCS which consists in the as-received condition of equaixed alpha-ferrite grains with evenly dispersed pearlite colonies of a lamella structure of alternating alpha-ferrite and cementite platelets as shown in Figure 2-1a. The composition of the steel in weight percent is 0.23 max Carbon, 0.005-0.05 Columbium, 1.35 max Manganese, 0.04 max Phosphorus, 0.04 max Silicon, 0.05 max Sulfur, 0.01-0.15 Vanadium, 0.015 max Nitrogen, balanced Iron, with an average grain size of approximately 25μm [22,23]. As mentioned above, pearlite colonies being a hard phase are considered long range dislocation barriers.
Figure 2-1: Micrographs of the microstructures with different pearlite volume fraction being studied: (a) As-received A572 with a grain size of 25μm, consists of pearlite colonies (dark phase) having a volume fraction of 9% and alpha-ferrite (light phase) equiaxed grains. The insert is a magnification of a pearlite colony showing the cementite-ferrite lamella configuration. (b) Heat treated microstructure with pearlite colonies being dissolved and dispersed along the grain boundaries of the ferrite matrix. The grain size is 38μm. (c) 1018 steel with a 20% volume fraction of pearlite and grain size of 9μm, (d) 1060 steel with grain size of 7μm grain size, consists of pearlite colonies shown as light regions with 72% volume fraction and alpha-ferrite phase being the dark regions.
In order to study the effect of pearlite volume fraction in relation to the dynamic flow stress, an attempt is made to generate a microstructure with no pearlite colonies. This is achieved by a heat treatment carried out on the as-received condition in order to disperse the carbon in speriodized form evenly throughout the alpha-ferrite matrix. In the initial stage of this procedure, as-received specimen is heated to 750°C, above the eutectic point, for one hour followed by rapid quenching in an ice-brine solution. This stage of the heat treatment dissolves both the alpha-ferrite and pearlite colonies bringing the material into a fully austenitic microstructure. This stage of heat treatment results in a ferrite-pearlite microstructure with the pearlite being altered. The cementite platelets within the pearlite become refined into very fine lamellar structure as opposed to the coarser lamellar structure of the as-received condition. In the second stage of the heat treatment, the alpha-ferrite and fine pearlite structured LCS is tempered at 720°C, below the eutectic point, for 72 hours, then furnace cooled. This process diffuses the fine cementite structure into speriodized carbides which allow the carbides to become dispersed throughout the alpha-ferrite matrix, forming speriodized carbide globules around the grain boundaries of the ferrite grains. The resultant heat treated material consists of equaixed ferrite grains with an average grain size of 38μm, as shown in Figure 2-1(b). The hardness of the heat treated material is measured as 75HRB compared with 83 HRB in the as-received condition.

In addition to the as-received and heat treated A572 microstructures, dynamic deformation tests are carried out on the 1018 and 1060 steels' with two different pearlite volume fraction. The 1018 steel was received in the annealed condition and consisted of a dual phase ferrite-pearlite microstructure with a 20% volume fraction of pearlite and
average grain size of 9μm, see Figure 2-1(c), while the 1060 steel was annealed to achieve a dual phase ferrite-pearlite microstructure that consisted of a 72% volume fraction of pearlite with an average grain size of 7μm, see Figure 2-1(d).

2.3 Dynamic Flow Stress Model

Modeling the flow stress as the sum of separate stress components is based on the concept that stresses developed in the material as a result of dynamic loading are related to the different mechanisms associated with dislocation/barrier interactions. This sum is written as:

$$\sigma = \sigma_a + \sigma_{th} + \sigma_D$$

(2-1)

$\sigma_a$ is the athermal component of stress which is the result of dislocation interactions with long range barriers such as grain boundaries and second phase particles. This stress is shown to be strain dependent [2-5]. The thermal component, $\sigma_{th}$, arises due to stress generated by short range barriers mainly that due to Peirls-Nabarro stress but also includes phonons, forest dislocations, dislocation jogs and kinks [4,5,21,24]. This stress is a function of thermal dependent effects mainly the strain rate and temperature. The viscous drag stress component $\sigma_D$ is due to the resistance of dislocation motion through a lattice by the lattice potential as well as interactions between phonons, electrons, radiation and point defects. In BCC metals, viscous drag effects are significant only at strain rates above $10^4\text{s}^{-1}$, which is higher than those applied in this study [25,26]. As such the drag stress component will be neglected in the current analysis.
The athermal component is indirectly affected by temperature through the
temperature dependency of the elastic and shear moduli. To account for this, \( \sigma_u \) is
normalized by the elastic modulus function as expressed in equation 2-2. \( E_o \) is the
reference elastic modulus at room temperature and \( E(T) \) is the shear modulus at a given
temperature.

\[
\bar{\sigma}_u = \sigma_u \frac{E_o}{E(T)}
\]  

(2-2)

In the remaining of the paper, \( \sigma_u \) refers to the normalized athermal stress which,
following the work of [2,4,5] can be described by a simple power law equation as a
function of strain in addition to a term, \( \sigma_o \), representing the influence of the grain size as
well as strength of the individual phases. This is expressed as:

\[
\sigma_u = \sigma_u^* \varepsilon^n + \sigma_o
\]  

(2-3)

\( \sigma_u^* \) and \( n \) are material constants. The second term of the above equation can be
written in a Hall-Petch type form to account for the pearlite volume fraction by
considering a weighted average of the individual strengths as follows.

\[
\sigma_o = V_f \frac{K_f}{\sqrt{d_f}} + V_p \frac{K_p}{\sqrt{d_p}}
\]  

(2-4)

where \( V_f, K_f \) and \( d_f \) are the volume fraction, Hall-Petch constant and grain size of the
ferrite phase respectfully and \( V_p, K_p \) and \( d_p \) are those related to the pearlite phase. Taking
d to be the average of \( d_f \) and \( d_p \) and considering \( V_f + V_p = 1 \), the above equation can be
rewritten as

45
\[ \sigma_a = \frac{1}{\sqrt{d}} \left( K_f + V_p \left( K_p - K_f \right) \right) \]  

(2-5)

Substituting equation 2-5 into equation 2-3, the athermal component of stress can be expressed as:

\[ \sigma_a = \sigma_a^* \epsilon^n + \frac{1}{\sqrt{d}} \left( K_f + V_p \left( K_p + K_f \right) \right) \]  

(2-6)

Turning attention to the thermal stress component, it is, as mentioned above, a product of interactions between dislocations and short range barriers, mainly Peierls-Nabarro barriers. Here it is assumed that the activation energy, \( \Delta G \), required for a dislocation to surpass a barrier by a single thermally activated process is written as:

\[ \Delta G = G_o \left[ 1 - \left( \frac{\sigma_{th}}{\dot{\sigma}_{th}} \right)^p \right]^q \]  

(2-7)

where \( G_o \) is the activation energy of 1eV/atom, and \( p \) and \( q \) are parameters that represent the profile of the dislocation barrier [17]. Furthermore, the strain rate, \( \dot{\epsilon} \), is related to \( \Delta G \) by the following relationship:

\[ \dot{\epsilon} = \dot{\epsilon}_r \exp \left( \frac{-\Delta G}{kT} \right) \]  

(8)

where \( \dot{\epsilon}_r \) is the reference strain rate and \( k \) is Boltzmann's constant. Substituting equation 2-7 into equation 2-8, yields an expression for the thermal stress component written as:

\[ \sigma_{th} = \hat{\sigma}_{th} \left[ 1 - \left( \frac{-kT \ln \frac{\dot{\epsilon}}{\dot{\epsilon}_r}}{G_o} \right)^{1/q} \right]^{1/p} \]  

(2-9)
The flow stress equation can now be written in terms of $\sigma_a$ expressed by equation 6 and $\sigma_{th}$ expressed by equation 2-9 as:

$$\sigma = \sigma_a^* \varepsilon^n + \frac{1}{\sqrt{d}} \left( K_f + V_p \left( K_p - K_f \right) \right) \sigma_{th}$$

$$+ \frac{-kT}{G_0} \ln \left( \frac{\dot{\varepsilon}}{\dot{\varepsilon}_r} \right)^{1/p}$$

(2-10)

The model parameters of equation 2-10 are determined using experimental dynamic stress strain curves generated each of the materials with varying pearlite volume fraction using SHPB tests performed at a range of strain rates up to $5.4 \times 10^3$ s$^{-1}$ and temperatures up 650°C. The experimental procedure and related analysis are described in the following sections.

2.4 Experimental Dynamic Flow Stress and Model Parameters

Dynamic testing has been carried out using a SHPB system capable of strain rates from $10^2$ - $10^4$ s$^{-1}$. High temperature testing utilized Tungsten inserts with the impedance matched to that of the maranging steel split Hopkinson bars assuming that no effects are induced to the loading pulse. The specimens were heated using a 5kW induction system. Cylindrical SHPB specimens with an L/D ratio of 0.5 for all tests where used and loading surfaces are ground to ensure parallelism while also to achieving a smooth, defect free surface, and reducing frictional effects. High temperature graphite lubrication was used in between the loading bars and specimen to reduce friction and ensure little to no barreling of the specimen will occur, resulting in an undesirable unsteady loading. Pulse
shaping techniques were utilized to ensure steady state loading occurred before plastic deformation, where aluminum pulse shapers where utilized with a L/D ratio of 0.25.

Tests are carried out on the as-received and heat treated conditions of the A572 steel at strain rates ranging from 7.3E2 to 5.4E3\text{s}^{-1} and temperatures of 20, 300, 500 and 650°C. Due to the nature of the dynamic experiments, oscillations were present in the stress strain curves. These curves were smoothed using a curve fitting procedure in order to facilitate data analysis. Figs. 2-2 and 2-3 show experimental curves as well as their smoothed trends for the as-received and heat treated material respectfully. The flow stress versus strain rates for different strains are plotted in Figure 2-4 along with quasistatic data for the as-received material. It can be seen that there is a distinct transition in strain rate sensitivity, represented as the slope of lines connecting data points, at approximately strain rates of 10^2\text{s}^{-1}.
Figure 2-2: True stress versus true strain for as-received condition a) at 20°C for various strain rates b) at a strain rate of 2.4E3s⁻¹ for various temperatures
Figure 2-3: True stress versus true strain for heat treated material a) at 20°C and b) at a strain rate of 2.5E3s⁻¹
Figure 2-4: Flow stress extracted at values of strain 1%, 3% and 5% at temperatures of a) 20°C and b) 500°C. Note the rapid change in strain rate sensitivity, $\beta$, at approximately $10^2$ s$^{-1}$. 

\[ \beta = 6.7 \]

\[ \beta = 42.5 \]

\[ \beta = 11.7 \]

\[ \beta = 77.2 \]
This transition from a low sensitivity to a high sensitivity with strain rate has been attributed to the increased rate of dislocation generation [3,27], which would cause the barriers that impede the motion of the dislocations to become a significant factor in the deformation of the material. The increase in dislocation generation is the cause in the increase in flow stress, or increase in strain rate sensitivity. Figure 2-7 shows that that there is a approximately 81% change in strain rate sensitivity between the loading regions at 20°C, and similarly at 500°C.

2.4.1 Athermal Flow Stress Parameters

The flow stress is extracted at constant values of strain for the as-received and heat treated A572 conditions from the dynamic stress strain curves and is plotted versus temperature in Figure 2-5 for three different strain rates for the as-received and heat treated conditions discussed above. These results show that the flow stress decreases rapidly with the increase in temperature, until saturation at a critical temperature, T_{cr}. The trends in this figure indicate that the curves corresponding to the three different strain rates would converge to a single value of stress. This saturated stress represents the athermal stress component, see [2,4,5].
**Figure 2.5:** Flow stress at 2% of strain versus temperature at different strain rates for the a) as received and b) heat treated materials.
Figure 2-6: Experimental athermal stress versus strain (circles) fitted into a three parameter power law (solid line) for the a) as-received b) heat treated material
The athermal component of stress determined from Figure 2-5 are normalized as expressed in equation 2-2 and is plotted versus the corresponding strain for the as-received and heat treated materials as shown in Figure 2-6. The experimental stress-strain points follow a power law relationship, \( \sigma_a = \sigma_a^* \varepsilon^n + \sigma_o \), which is used to determine the parameters \( \sigma_a^* \), \( n \) and \( \sigma_o \). These are 231 MPa and 0.25 and 105MPa for the as received condition and 231 MPa, 0.25 and 105MPa for the heat treated material.

As mentioned above, two additional steels are considered in this study; 1018 and 1060 steels. They consist of dual phase microstructures with volume fractions of pearlite determined to be 20 and 72% respectfully. The athermal components of these materials are generated in a manner similar to that described above and are shown in Figure 2-7. The values of \( \sigma_a^* \) and \( n \) are found to be consistent with the as-received and heat treated conditions, while \( \sigma_o \) assume values of 120 for the material with 20% volume fraction of pearlite and 144 for that with 72% volume fraction.
**Figure 2-7:** Plots of the normalized athermal stress versus strain for a) 1018 steel, and b) 1060 steel. Circles are experimental points which are fitted into a three-parameter power law plotted as solid line.
From the above analysis which provide a full identification of the term $\sigma_o$ for the four materials, the remaining parameters of equation 2-6 are determined. The values of which are determined to be 310 MPa/μm$^{1/2}$ for $K_f$, and a value of 1078 MPa/μm$^{1/2}$ for $K_p$, considering values from literature as well [28,29].

2.4.2 Thermal Flow Stress Parameters

Since the flow stress is presented as the sum of two components, the thermal stress can be calculated as:

$$\sigma_{th} = \sigma - \sigma_o$$

(2-11)

Where $\sigma$ is the normalized flow stress, it is shown in equation 2-9 that $\sigma_{th}$ is a function of $T^{p/q}$. This relationship is plotted in Figure 2-8, by selecting p/q to be 3/2 to obtain a simplified linear correlation [2,4,5].
Figure 2-8: Normalized thermal stress calculated using equation 8 versus temperature for a range of strain values for the a) as-received material and b) heat treated material.
From this figure, the values of the y-intercept are $\hat{\sigma}_{th}$ and the slope is $\frac{-k\hat{\sigma}_{th}}{G_o}\ln\hat{\epsilon}$. These are used to calculate the reference strain rate, $\hat{\epsilon}_r$, with known values of $k = 8.63E-5$ eV/K and $G_o = 1eV$ [4,5]. The values of $\hat{\sigma}_{th}$ are calculated to be 922 and 964 MPa and $\hat{\epsilon}_r$ is determined as $2.0E8$ s$^{-1}$ and $1.73E8$s$^{-1}$, for as-received and the heat treated materials, respectfully. These values are shown to be similar for all the steels which indicate that the thermal flow stress is independent of microstructural variation. The above analysis for both the athermal and thermal flow stress components provide a full identification of the dynamic flow stress expressed by equation 2-10. The next section is a simulation of this equation to examine the stress sensitivity to pearlite volume fraction.

### 2.5 Model Simulations

Parameters generated from the analysis detailed in the last section were numerically optimized and are presented in table 2-1 and can be used in conjunction with equation 2-10 to simulate the flow stress-strain curve as a function of pearlite volume fraction for different strain, strain rate and temperature.
Table 2-1: Optimized model parameters for the four steels studied with vary VFP, see equation 2-10

| Constant | Value       | Unit          |
|----------|-------------|---------------|
| $\sigma_d^*$ | 346.5       | MPa           |
| n        | 0.2304      |               |
| $K_p$    | 718.67      | MPa/μm$^{1/2}$|
| $K_f$    | 465         | MPa/μm$^{1/2}$|
| $\hat{\sigma}_{th}$ | 950 | MPa          |
| p        | 1           |               |
| q        | 1.5         |               |
| k        | 8.63E-5     | eV/K          |
| $G_0$    | 0.92        | eV            |
| $\dot{\varepsilon}_y$ | 1.865E8 | s$^{-1}$     |
In these simulation efforts, it is important to recognize that due to the large amount of energy converted into heat during dynamic loading, adiabatic heating would develop during plastic deformation causing material softening during loading. This can be accounted for during the simulation work by altering the temperature range $\Delta T$ during the loading history following the expression [4,16]:

$$\Delta T \approx \frac{\eta}{\rho} \int_{\gamma_0}^{\gamma} \frac{\tau}{C_v} d\gamma$$  \hspace{1cm} (2-12)

where $\rho$ is mass density which is assumed to remain constant at 8065 kg/m$^3$, $\eta$ is a constant representative of the amount of energy converted into heat, and $C_v$ is the temperature dependent heat capacity of the material, initially 0.420 J/gK. The variation of $\Delta T$ is taken in consideration during simulation steps of the flow stress calculations carried out at different strain rates and temperature conditions. Results of these simulations for isothermal and adiabatic stress-strain curves compared with those experimentally obtained but were not utilized in parameter identification, are shown in Figure 2-9. This figure shows the good agreement between these simulated and experimental curves for the as-received (9% volume fraction of pearlite) and heat treated conditions (0% volume fraction of pearlite) at all loading conditions. The comparison between the experimental and modeled stress-strain curves shows that the parameters generated, Table 2-1, are valid in representing the experimental dynamic response of the material under the tested range of strain rates and temperatures.
Figure 2-9: Plotted are comparisons between the simulated stress strain curves using optimized model parameters and experimental curves, in both the isothermal and adiabatic conditions. For the as-received material a) $1.3 \times 10^3 \text{s}^{-1}$ at $300^\circ \text{C}$ and b) $5.0 \times 10^3 \text{s}^{-1}$ at $20^\circ \text{C}$ and for the heat treated material c) $5.0 \times 10^2 \text{s}^{-1}$ at $650^\circ \text{C}$ and d) $8.0 \times 10^2 \text{s}^{-1}$ at $650^\circ \text{C}$.
Figure 2-10 shows the models sensitivity to strain rate and temperature, indicating an increase in the flow stress with increasing strain rate at a constant temperature and a decrease in flow stress with increasing temperature at a constant strain rate. Additionally, Figure 2-10(c) represents the models sensitivity with changing volume fraction of pearlite, in which the heat treated material which has 0% volume fraction has a lower flow stress, than that of the 1060 which has the highest volume fraction of pearlite.
Figure 2-10: Plotted are simulated adiabatic stress-strain curves using parameters in table 1, a) as-received LCS at an initial temperature of 500°C at strain rates of 2E2, 1E3, 5E3 and 1E4s⁻¹, b) as-received LCS at a strain rate of 5E2s⁻¹ and initial temperatures of 20, 150, 300 and 500°C, c) at an initial temperature of 300°C and strain rate of 5E2s⁻¹ for varying volume fractions of pearlite (0%, 9%, 20% and 72%)
2.6 Significance of Pearlite Volume Fraction

By utilizing the constants gathered for equation 2-10, it is possible to investigate the role that pearlite colonies play in the deformation response during high strain rate loading. Figure 2-11 shows a plot of equation 5 for \( \sigma_o \), the stress component due to pearlite, versus volume fraction of pearlite for constant grain sizes values.

This figure shows that as the volume fraction of pearlite increases there is an increase in the \( \sigma_o \) value. An interesting result obtained from Figure 2-14 is that with a decrease in grain size there is an increase in the rate at which \( \sigma_o \) changes with volume fraction of pearlite, shown by the increase in slope from 40 at 40\( \mu \)m to 80 at 10\( \mu \)m. Meaning that as the grain size of the material is refined there is an increased effectiveness in the strengthening ability of the pearlite phase of the material. This is clearly illustrated in Figure 2-12 which plots \( \sigma_o \) versus grain size at two extremes, 100% volume fraction of pearlite and 0% volume fraction of pearlite.
Figure 2-11: Plotted is equation 5, for $\sigma_o$, the stress component due to pearlite, as function of volume fraction for four different values of grain size.

Figure 2-12: $\sigma_o$, the stress component due to pearlite versus grain sizes at two different extremes, fully ferritic state (0% VFP and fully pearlitic material (100% VFP).
The value of $\Delta \sigma_o$ at 45$\mu$m is 112 with the value of 187 at 15$\mu$m, leading to a 60% increase in contribution of pearlite to flow stress, in just over a 30% decrease in grain size. The pearlite volume fraction becomes an increasing contributor as a strengthening phase within the low carbon steel. These figures show that the pearlite colonies are more effective as long-range barriers to dislocation motion, in that they provide an increase in the $\sigma_o$-parameter with increasing volume fraction of pearlite than that of a greater increase due to a decrease in grain size would cause. The pearlite colonies are there for a stronger barrier to dislocation than that of grain boundaries themselves.

2.7 Conclusions

The role of pearlite colonies of the dynamic flow response has been studied for dual phase steels consisting of ferrite-pearlite microstructures by conducting a series of SHPB tests in a range of strain and temperatures. Major outcomes of the study are listed below.

- A constitutive model is developed based on thermally activated dislocation interaction with barriers. The model separates the flow stress into two stress components; thermal and athermal. The athermal component is related to the interaction of dislocations with long range barriers and is a function of strain and microstructure of the material. Long range barriers include grain boundaries, precipitates and second phase particles. The thermal component accounts for the interaction of dislocations with short range barriers that include phonons, forest
dislocations, dislocation jogs and kinks and Peirls-Nabarro stress. This stress component is shown to be a function of strain rate and temperature.

- A heat treatment procedure is carried out on the as-received A572 LCS to modify the ferrite-pearlite microstructure into a ferrite-sperlized LCS. This was carried out in order to compare the dynamic flow stress as a function of pearlite volume fraction.

- A series of SHPB dynamic loading tests were conducted on 1018 (20% VFP), 1060 (72% VFP), as-received A572 (9% VFP) and heat treated A572 (0% VFP) steels. The as-received and heat treated conditions were tested at temperatures: 20°C, 300°C, 500°C and 650°C for strain rates ranging from 7E2 to 5E3 s⁻¹. The 1018 and 1060 steels were tested at 500°C and 650°C at 1.5E3s⁻¹

- A procedural analysis is performed in order to generate model parameters for the athermal and thermal components of stress of the steels mentioned above. It is shown that only the athermal component of stress is affected by the volume fraction of pearlite.

- A parameter representing the stress contribution due to pearlite within the athermal component of stress is derived as a function of volume fraction of pearlite. In addition, a weighted average of the Hall-Petch type relationship with respect to volume fraction of pearlite is generated. Furthermore the Hall-Petch constants for pearlite and ferrite are generated.

- The constitutive model has been applied for different microstructure, strain, strain rate and temperature. Results compared well with those experimentally generated.
The effectiveness of pearlite as a strengthening phase in steel is evaluated as a function of volume fraction of pearlite. A parametric study is carried out to examine the role of pearlite as a strengthening phase in low carbon steel. It is shown that this role is more effective as grain size decreases.

2.8 References

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CHAPTER 3

"Modeling of Dynamic Flow Stress of Ultra-Fine Grain Material"

by

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3.0 Abstract

A fine grained Al6061 generated using equal channel pressing techniques is studied in terms of the dynamic deformation within a range of strain rates and temperatures. Testing within the dynamic regimes is completed using a split Hopkinson pressure bar at strain rates of $10^2$-$10^4$ s$^{-1}$ with testing temperatures of 20, 50, 100 and 200°C. A constitutive relationship and its model parameters for the dynamic flow response are generated as an explicit function of grain size while also including strain, strain rate and temperature effects. The constitutive relationship is derived on the basis of the separation of flow stress into two physically based components, athermal and thermal, each depending on the differing barrier types to dislocation motion. The predictive ability of the model is used to predict dynamic test results not utilized in the parameter generation, showing good agreement with these results. The athermal component of stress is shown to be related to grain size through the typical Hall-Petch relationship while also relating the hardening parameter to grain size. This is shown to decrease in a Morrison type relationship. The thermal component of flow stress is shown to be increasingly dependent on grain size due to the transition in deformation mechanisms at a critical grain size, from coarse-grain regime into the ultra-fine grain. The material studied is shown to exists in a fine grain regime, in which competing dominant deformation mechanisms exists from both the coarse and ultra-fine grain regimes.
3.1 Introduction

Grain refinement of a material, its generation, performance and microstructural characteristics has been of great interest of researchers for the past few decades. This is due the enhancement in mechanical properties with refinement in grain size. Severe plastic deformation methods are used for the manufacturing of fine (FG), ultra-fine (UFG) and nano-crystalline (NC) materials; these include equal channel angular pressing (ECAP) [1], high pressure torsion (HPT) [2,3], accumulative roll bonding (ARB) [4], tubular channel pressing (TCP) [5] and dissimilar channel angular pressing (DCAP) [6]. These methods lead to effective manner in which the process can be scaled from the laboratory level into full-scale production modes, with the ECAP method receiving the highest level of interest. With the increased production and future use of such a material, it is important to have the ability to derive constitutive relationships that can predict the corresponding mechanical stress-strain response in a wide range of strain, strain rate and temperature loading conditions. In addition, microstructure parameters, particularly the grain size, phases and dislocation configuration including density and homogeneity, are shown to have a significant influence on the refined material response [7-10]. A widely used model is the Johnson-Cook relationship in which material constants are derived from empirical relations of experimental dynamic flow stress carried out under varying strain-rates and temperatures [11]. These constants are general in nature and microstructure independent. Zerilli-Armstrong (ZA) model includes the effect of grain size by utilizing the Hall-Petch relationship. The model is applied with different formulations tailored to BCC and FCC microstructures [12]. A third model is the mechanical threshold stress, which is a phenomenological model based on the
consideration of thermally activated flow stress. This model does not include explicitly dependent microstructure terms [13,14]. A recent study by Spirdione et al. [15] has developed microstructure-sensitive flow stress formulations that have shown promising ability to predict the stress-strain response in a wide range of strain rates and temperatures for both FCC and BCC crystalline structures. This model, similar to the work of Nemat-Nasser et al [9], is based on the separation of the total flow stress into separate components, athermal and thermal, that are resultants of different physical barriers to dislocation motion. The athermal component is due to long range barrier sources such as grain boundaries and second phase particles, that generate stress fields that are 10 atomic diameters or larger. The thermal component on the other hand is attributed to the stress that arises as dislocations interact with short range barriers, stress fields generated less than 10 atomic diameters, mainly Peierls-Nabarro stress in BCC materials and dislocation-forests in FCC structures [16,17].

May et al [18] investigated UFG aluminum and aluminum alloys in regards to its mechanical properties, dislocation density and grain structure. This work showed that the precipitate and alloy content in the course grain (CG) state of the material contributes to the ability of the material to be refined into UFG; with an increase in alloying elements, the grain size decreased after ECAP processing, along with an increase in dislocation density within the grain interior. This effect is due to the inclusion of impurities that hinders dynamic recovery and re-crystallization during processing. Similarly, Chowdury et al, [19] showed that Al2024 and Al5056 had an increase in strength with an increase in the number of passes through an ECAP system. While with an increase in alloy content in Al7034, there was an initial decrease in strength with the number of passes, then
subsequent increase in strength with further processing. This is attributed to the effect that precipitates have on the texturing of the grain structure during UFG processing. This effect is due to the rate at which low angle boundaries, which are at a weaker non-equilibrium states, convert into the more equilibrated high-angle grain boundaries that do not allow dislocation transport as readily. Kahn et al [20] studied the thermo-mechanical response of Al6061 with and without ECAP processing. It was found that in the quasi-static loading regime, the strain rate sensitivity (SRS) increases with the increase in the number of ECAP passes. The increase in the flow stress while is directly dependent on the number of passes as well as the strain rate, reaches a saturation at 125°C. For the same number of passes, SRS was observed to increase with an increase in temperature. Kunimine et al [4] have investigated the temperature and strain rate sensitivities in UFG copper. The work shows that there is an increase in SRS in a FCC material with a decrease in grain size but also shows that there is a SRS increase with an increasing temperature testing condition. Lee et al [21] showed that in the CG state of Al606-T6 the relationship with SRS and temperature is similar to the of Kunimine et al and Wei et al. Also that temperature sensitivity increases with both an increasing strain rate and temperature in CG Al6061-T6.

Wei et al [22] investigated effects of the crystalline structure at the NC and UFG scales would have on the mechanical response, including the strain rate sensitivity. It is observed that SRS increases with the decrease in the grain size of the FCC microstructure while the opposite trend occurs in the BCC structure. This response is attributed to the fact that in the FCC microstructure, the thermally activated barrier that interacts with mobile dislocations is mainly in the form of dislocation forests. As a decrease in the grain
size occurs, reaching a critical transitional grain size, defined as when the grain size is equivalent to the inverse root of dislocation density, the grain interior becomes nearly dislocation free, while the grain boundaries exhibit an increase in the dislocation density. As such, dislocation forest cutting is no longer the rate controlling deformation mechanism. Consequently grain boundaries become the dominant thermally activated barrier which could then be the source of the increase in the SRS. The change in the controlling deformation mechanism is evident by the steep decrease in the deformation related activation volume with the decrease in the grain size. The transition in deformation mechanisms as a function of grain size is shown in the work of Kim et al [23] where the plastic behavior of UFG materials is described utilizing a phase mixture model. The material is treated as dual-phase material; a grain interior surrounded by grain boundary. The grain interior consists of three deformation mechanisms dislocation glide, lattice diffusion and boundary diffusion. The grain boundary deformation is treated as being in an amorphous state with a diffusional mass transport mechanism. This work shows that at a grain size larger than that of the critical size, deformation will include dislocation and diffusional mechanisms, while below this size, only diffusional mechanisms are considered. Hiyamoto et al [24] studied the viscous nature of deformation in UFG Al in which results suggest that the major deformation mechanism is dislocation emission and annihilation occurring at grain boundaries. The equivalent rate of annihilated and emitted dislocations at grain boundaries results in a constant dislocation density which would in turn explain the observed nearly non-existent hardening. Estrin et al [25] performed quasistatic and stress-jump tensile tests on UFG Al at 0.52K. Results showed the typical increase in the flow stress and decrease in the strain
hardening rate generally observed in UFG materials as compared to their CG counterparts. The change in the hardening rate of the stress-strain curve from parabolic in the CG to a linear in UFG is due to the fact that at this low temperature, deformation depends only on initial dislocation density and distribution within the crystal.

The studies discussed above illustrate the significance of the grain size on the dynamic flow stress response of materials. This significance is due to the change in dominant deformation mechanisms that act as thermal barriers to dislocation motion. In the CG microstructure of FCC crystalline materials, the dominant rate controlling mechanism is dislocation forest cutting. This differs in the FCC UFG materials in which the dominant thermally activated barrier becomes grain boundaries at a critical grain size. This change in mechanistic behavior of the material, makes it therefore imperative that constitutive relationships that are used in a predictive sense are a direct function of grain size and must be capable of evolving in their formulation. The current work aims at providing a constitutive relationship that takes into account the effect of grain size on the dynamic flow stress response of a grain size refined material.

The first part of the paper describes the materials of study, an annealed Al6061 and an FG Al6061. The FG Al6061 is generated using an ECAP system which is discussed in detail. Secondly the flow stress is formulated in a constitutive equation which considers the separate stress components of athermal and thermal stress, where each stress component is dependent on separate barriers to dislocation motion. The mechanisms of dislocation interactions with barriers to their motion are discussed relative to a variance in grain size. The constitutive equation is derived as general function of strain, strain rate and temperature, as well as with a capability to evolve with differing
crystalline structures. Next, in order to determine model parameters, experimental tests using SHPB methods are used to generate the true stress-strain response of both the CG and FG microstructures. Experimental data is analyzed in a procedural manner to generate constitutive model parameters. Several parameters from both the athermal and thermal components within the constitutive equation are shown and defined to be a function of grain size. Equations are derived to describe parameters that are shown to change with grain refinement. Finally, model comparisons with experimental test results are discussed in terms of validity of the model derived. Also discussed is a physical understanding of the effect that grain size plays on the dynamic response of FG aluminum. A model is now defined to be a function of strain, strain rate, temperature, grain size to predict the dynamic flow stress.

### 3.2 UFG Material and Processing

Aluminum 6061 was received in the T6 temper with a composition of (wt%) Al-1.01, Mg-0.49, Si-0.31, Cu-0.24, Fe-0.06, 0.06Cr [5]. To improve formability and ease of pressing, ECAP specimens were annealed from the T6-condition to the O-condition. This was done by heating specimen to 412°C for 2 hours and subsequently furnace cooled. The annealed condition consists of Fe$_3$SiAl$_{12}$ matrix with evenly dispersed Mg$_2$Si particulates as shown in Figure 3-1.
Figure 3-1: Micrographs of the O-condition Al6061 showing an average grain size of 100μm. The light regions consist of Fe$_3$SiAl$_{12}$ with evenly dispersed Mg$_2$Si particulates appearing as dark regions. In order to refine the grains of the aluminum, an ECAP system was built. The system was designed to generate approximately 1.0 strain per pass. This is achieved by utilizing an interior angle, $\phi$, of 95° and an exterior angle, $\psi$, of 85° which are correlated as given by equation 3-1 below: [26]

$$\varepsilon_n = \frac{2N}{\sqrt{3}} \left[ \cot \left( \frac{\phi + \psi}{2} \right) + \psi \csc \left( \frac{\phi + \psi}{2} \right) \right]$$

(3-1)

where $\varepsilon_n$ is the total strain applied during ECAP pressing and N is the number of passes. The die accepts a 4.5 inch long specimen having a half inch square cross-section. The exit channel of the die is tapered from the interior outward to allow for the relaxation of elastic stresses within the specimen on exit. A schematic of the die detailing the dimensions is shown in Figure 3-2.
Figure 3-2: Schematic representing the ECAP system utilized to generate UFG Al6061

The die consists of 5 separate plates that are pinned and bolted together, in which three half-inch thick interior plates form the die channel. These plates are assembled using M42 tool steel pins and A2 tool steel sleeves, to hold the channel in its proper configuration during pressing. This stack of the three channel plates are then placed between two four inch thick A2 tool steel blocks. The five plates are bolted together using six grade-8 steel bolts, each of which are torqued to 600ft-lbs, ensuring that the plates do not separate during pressing. MoS$_2$ high temperature/pressure lubricant is painted onto the channel and specimen surfaces prior to each pass. The entire die is heated to an interior temperature of 150°C by use of four 1000W cartridge heaters.
pressed into two heater plates that are attached to the exterior of the die. The die is then surrounded by one inch ceramic insulation to improve heating time and thermal uniformity. The increase in temperature for pressing, improves formability by lowering the shear stress required to deform the material. The work of Khan et al and Gazder et al [20,27] has shown that route B_c, in which the specimen is rotated 90° about the Z-axis after every pass, is the most effective in generating equaixed low-angle fine grained structures. This route was chosen in the processing of Al specimens for this study. The FG material obtained after four passes in the ECAP system described, is shown in Figure 3-3.

![Micrographs](image)

**Figure 3-3:** Micrographs of the FG Al6061 showing an average grain size of 5μm. The light regions consist of Fe_3SiAl_{12} with evenly dispersed Mg_2Si particulates appearing as dark regions.
From the micrograph in Figure 3-3, it can be seen that the initial average grain size before ECAP processing of the annealed 6061 in Figure 3-1 is approximately 100μm, in an equiaxed form. Similarly, the average grain size of the FG material is shown to be approximately 5μm.

3.3 Microstructure Dependent Dynamic Flow Stress Model

The flow stress as a result of dynamic loadings has been shown to be the summing contributions of athermal, \( \sigma_a \), thermal, \( \sigma_{th} \), and drag, \( \sigma_D \), stress components each arising due to individual mechanisms involving dislocation interactions with different types of barriers. The drag stress is not included in this derivation due to the lack of its flow stress dependence until above \( 10^4 \) s\(^{-1} \), not within the scope of this work. The summing of these contributions can be written as:

\[
\sigma = \sigma_a + \sigma_{th} + \sigma_D
\]  \hspace{1cm} (3-2)

\( \sigma_a \) is the stress resultant from interactions between dislocations and long range barriers which could include dislocations, grain boundaries, second phase particles and defects. The athermal stress as shown by Taylor et. al [28] is a function of strain and other microstructural parameters, mainly being the grain size. The athermal stress could thus be written as a function of plastic strain \( \varepsilon \) :

\[
\sigma_a = \sigma_a^* \varepsilon^n + \sigma_G
\]  \hspace{1cm} (3-3)

Where \( \sigma_a^* \) and \( n \) are material constants and \( \sigma_G \) represents the stress due to the grain size. Furthermore, the athermal stress is indirectly affected by temperature through
the temperature dependency of the elastic and shear moduli. In order to remove this
dependence the athermal stress with the temperature dependent elastic modulus, the stress
component is normalized by the elastic modulus function as follows:

$$\bar{\sigma}_a = \sigma_a \frac{E_o}{E(T)}$$  \hspace{1cm} (3-4)

$E_o$ is the reference elastic modulus at room temperature and $E(T)$ is the shear modulus at
a given temperature The thermal component,

The thermally active thermal stress component, $\sigma_{th}$, as mentioned above, is due to
interactions between dislocations and short range barriers. Major sources of these barriers
are Peirls-Nabarro stress in BCC metals and dislocation forests in FCC metals. The
thermal component is dependent on strain rate and temperature. In addition, this
dependency in FCC metals includes the role of strain. To attain a relation between
temperature, strain rate and the thermal stress component, the energy required for a
dislocation to overcome a barrier by thermal activation is written as:

$$\Delta G = G_o \left[ 1 - \left( \frac{\sigma_{th}}{\bar{\sigma}} \right)^p \right]^q$$  \hspace{1cm} (3-5)

$p$ and $q$ are constants that define the profile of the barrier to be crossed, where $0 < p \leq 1$
and $0 \leq q \leq 2$. $G_o$ is the energy required to overcome a barrier solely by thermal
activation and is given by

$$G_o = \dot{\varphi} \lambda lb$$  \hspace{1cm} (3-6)
\( \dot{\sigma} \) is the stress required for a dislocation to overcome a barrier without any thermal assistance, \( \lambda \) and \( l \) are the average effective barrier width and spacing respectively. It is assumed that strain rate is related to \( \Delta G \) by the following,

\[
\dot{\varepsilon} = \dot{\varepsilon}_r \exp \left( -\frac{\Delta G}{kT} \right) \text{ where } \dot{\varepsilon}_r = \rho_m b \omega \nu \lambda \quad (3-7)
\]

\( k \) is the Boltzmann's constant, \( \omega_0 \) is the attempt frequency for a dislocation to overcome a barrier, \( b \) is the burgers vector and \( \rho_m \) is the mobile dislocation density. Combining equation 3-5 and 3-7 and solving for \( \sigma_m \), one can obtain an expression for the thermal stress as:

\[
\sigma_{th} = \dot{\sigma}_{th} \left[ 1 - \frac{kT}{G \omega} \ln \frac{\dot{\varepsilon}_r}{\dot{\varepsilon}_r} \right]^{1/p}
\]

The thermal stress component, as can be seen from equations 3-6 and 3-7, depends on the dislocation spacing, \( l \). In FCC metals where the dominant short-range barrier is dislocation forests, means that \( l \) must be an evolving function of the average dislocation spacing. This is expressed as in [9].

\[
l = \frac{l_o}{f(\varepsilon, T)} \quad (3-9)
\]

where \( l_o \) is the initial average dislocation density. From this relation it can be expressed that:
\[ \hat{\sigma}_{th} = \sigma^o f(\varepsilon, T) \text{ with } \sigma^o = \frac{G_o}{b\lambda l_o} \]

3-10(a,b)

\[ \dot{\varepsilon}_r = \frac{\dot{\varepsilon}_0}{f(\varepsilon, T)} \text{ with } \dot{\varepsilon}_0 = \rho_o b\omega_o l_o \]

It is reasonable to assume that the average dislocation spacing increases with further straining in a manner similar to work-hardening effects and decreases with increasing temperature as in annealing effects. These trends are written as

\[ f(\varepsilon, T) = \frac{l_o}{l} = 1 + a \left[ 1 - \left( \frac{T}{T_m} \right)^2 \right] \varepsilon^n \]

(3-11)

By substituting equations 3-10 and 3-11 into equation 3-8, the thermal stress can be expressed as:

\[ \sigma_{th} = \sigma^o \left[ 1 - \left( \frac{-kT}{G_o} \frac{\dot{\varepsilon} f}{\dot{\varepsilon}_o} \right)^{1/q} \right]^{1/p} \]

(3-12)

From this the constitutive relationship for flow stress as a function of strain, strain rate, temperature and grain size can be assembled in the following form:

\[ \sigma = \sigma^o \varepsilon^n + \sigma_D + \sigma^o \left[ 1 - \left( \frac{-kT}{G_o} \frac{\dot{\varepsilon} f}{\dot{\varepsilon}_o} \right)^{1/q} \right]^{1/p} \]

(3-13)

Examination of the validity of the flow stress as expressed above requires the identification of the material parameters, \( \sigma^o_a, n, \sigma_o, \sigma^o, p, q, a \) and \( m \). These will be determined using experimental flow stress curves generated for each of the materials described above; FG and CG materials having grain sizes of 5 and 100\( \mu \)m, respectfully.
These curves will be obtained using SHPB tests performed at strain rates up to 5.0E3 s\(^{-1}\) and temperatures up to 200°C. The analytical procedure and model parameters determined are described in the following section.

### 3.4 Model Parameters Determination

Testing the CG and FG conditions of the Al6061 are carried out at strain rates ranging from 1.0E3 to 5.0E3 s\(^{-1}\), and temperatures ranging from 20°C to 200°C. Experimental flow stress curves were generated and smoothed using a curve fitting procedure in order facilitate data analysis. Figure 3-4 a,b show examples of the fitted curves for the CG and FG Al6061, respectfully, imposed on the original experimental data. Figure 3-4a,b show flow stress curves for both the CG and UFG materials which have a trend typical of that of metals, in which there is an inverse relationship between stress and temperature at a given strain rate. Figure 3-5 illustrates the strain rate sensitivity where the CG material exhibits a positive strain rate sensitivity (SRS) at a given temperature with the FG material following a similar trend. Figure 3-6 shows the flow stress versus strain state at two different strains; 10% and 20% at 20°C and 100°C for both CG and FG materials.

The SRS of a material is expressed as the change in flow stress with respect to the change in the corresponding strain rate. This ratio as obtained from Figure 3-7 is plotted versus grain size corresponding to the two test materials. The SRS versus grain size relationship indicates that for the same temperature, SRS increases as the grain size decreases. These observations which are supported by the previous work [22], will be discussed later in this paper.
Figure 3-4: True stress versus true strain for a) CG Al6061 - strain rate of 2.8E3s\(^{-1}\) b) FG Al6061 - strain rate of 1E3s\(^{-1}\)
Figure 3-5: True stress versus true strain for CG Al6061 at 100°C.
Figure 3-6: Flow Stress versus Strain Rate at strains of 10% and 20% and at temperatures of 20°C and 100°C for the a) CG Al6061 and b) FG Al6061. Solid lines are for the black data points and dashed lines are for white data points.
Figure 3-7: Strain Rate Sensitivity, $\beta$, versus Grain Sizes of the CG and FG Al6061, at temperatures of 20°C and 100°C
3.4.1 Athermal Flow Stress Parameters

Figure 3-8 a,b show the flow stress for the CG and FG obtained at strain rates 1.5E3 and 1.0E3s\(^{-1}\) respectfully for five strain levels of 5, 10, 15, 20 and 25\%. For each of these strain rates, the flow stress decreases as the temperature increases, reaching a plateau at a critical temperature of 473°C. The athermal stress is the quantitative level of the saturated flow stress which shows independence of temperature. The normalized values of the athermal stress are plotted as a function of strain in Figure 3-9a,b for the two material conditions. From this figure, the athermal stress versus strain is fitted into a two parameter power law to determine parameters \(\sigma_a, n\) and \(\sigma_G\).

As can be seen the \(\sigma_G\) parameter changes between the two grain sizes. Using the Hall-Petch relationship, the \(\sigma_G\) parameter takes the form of \(K/d^{1/2}\). Using the known grain size of the annealed CG Al6061 to be 100\(\mu\)m, the K parameter can be determined. Also noted is the variance in the hardening parameter, \(n\), with grain size. This parameter is will be discussed further at a later time.
Figure 3-8: Flow stress versus Temperature for a) CG Al6061 - at strain rate of 1.5E3s$^{-1}$ and b) FG Al6061 - at a strain rate of 1E3s$^{-1}$
Figure 3.9: Normalized Athermal Stress versus True Strain for a) Annealed Al6061 and b) FG Al6061
3.4.2 Thermal Flow Stress Parameters

The identification of the thermal component of stress, equation 3-12, requires, in addition to the constants $k$ and $G_0$, the knowledge of $\dot{\varepsilon}_o$, $\sigma^o$ and the $a$-parameter in equation 3-11. Using equation 3-10b and setting the burgers vector, $b=2.86E-5$cm, the initial dislocation density for commercially pure aluminum, $\rho_m = 9.1E9/cm^2$ [29], $l_o = 500$ lattice spacing and $\omega_o = 10^{11}s^{-1}$ [9] the reference strain rate, $\dot{\varepsilon}_o$, is determined to be $5.28E-8s^{-1}$. This parameter, as shown in Table 3-1, is invariant of microstructure. From literature it is expected that $\sigma^o$ and the $a$-parameter will both be functions of the grain size [9]. In order to determine the $a$-parameter from equation 3-11, the relationship between $l$ and $\rho_c$, is assumed to be [9,22].

$$l \approx \rho_c^{-\frac{1}{2}}$$

(3-14)

Therefore, the ratio between initial $l_o$ and $\rho_o$ and the current conditions, $l_c$ and $\rho_c$, is written as:

$$\left(\frac{\rho_o}{\rho_c}\right)^{-\frac{1}{2}} = \frac{l_o}{l_c} = f(\varepsilon, T) = 1 + a \left[1 - \left(\frac{T}{T_m}\right)^2\right] \varepsilon^m$$

(3-5)

The work of Mujica et al [29] on commercially pure aluminum provided the dislocation densities at three different strain levels at room temperature (circles in Figure 3-10a). These densities were utilized, in addition to the assumption that $m=0.5$, to generate a three parameter power law (solid curve in Figure 3-10a) by which the constant $a$ is determined to be $2.963$ for the CG condition. To carry out similar calculations for the FG material, the dislocation density, as will be discussed later in this paper, is
expected not to vary drastically with strain. An initial dislocation density for FG Al6061, determined as 2E11/mm$^2$ [29], is used to normalize the solid data points in Figure 3-10a. These normalized dislocation densities are then fitted into a power law relationship (solid curve in Figure 3-10b) and utilized to determine the parameter $a$ for FG material, which is calculated as 0.0266.

To determine the parameter $\sigma^\circ$, the relationship given in equation 3-2 is rearranged as:

$$\sigma_{th} = \sigma - \sigma_u$$

(3-16)

By subtracting the normalized athermal stress values from the total normalized flow stress, the normalized thermal stress component, $\sigma_{th}$, is determined and then used in equation 3-12, to determine the average $\sigma^\circ$ using three different strain rates for each material condition. This calculation is carried out at 20°C, being the lowest temperature testing condition, to satisfy the physical definition of, $\sigma^\circ$, being the stress above which a barrier is crossed without any thermal assistance. Values of $\sigma^\circ$ for the CG and FG conditions were calculated as 73 and 303MPa respectfully.
Figure 3-10: Ratio of initial and current dislocation densities Vs Applied Strain, with a three parameter power law fit to acquire model parameters for a) pure Aluminum and b) FG Al6061
3.5 Grain Size Dependent Flow Stress Parameters

In order to accurately model the flow stress as expressed in equation 3-13, it is important to investigate each of its parameters in relation to grain size. To start with, the hardening parameter, $n$, of the athermal stress component is shown to be inversely related with the grain size following a Morrison type law in UFG steels [30].

$$n = \frac{m_n}{b_n + d^{\frac{1}{2}}}$$  \hspace{1cm} (3-17)

The $n$ versus grain sizes as obtained in section 4.1 (solid circles in Figure 3-11) is fitted into equation 17 (solid line in Figure 3-11) and the parameters $m_n$ and $b_n$ can then be determined. The parameters $\sigma_d$, $\sigma_o$ and $a$ are examined with respect to the grain size, $d$, by assuming a Hall-Petch type relationship:

$$P = \frac{m_x}{d^{\frac{1}{2}}} + b_x$$  \hspace{1cm} (3-18)

where $P$ is the parameter of interest, and $m_x$ and $b_x$ are material constants. Figure 3-12a-c shows the plots of $\sigma_d$, $\sigma_o$ and $a$ versus $d$ respectfully (solid circles in Figure 3-12a-c). Equation 18 is fitted to the data in each of these plots (solid line in Figure 3-12a-c) and values of $m_x$ and $b_x$ for the corresponding $\sigma_d$, $\sigma_o$ and $a$ are determined; these are shown in each of the figures. The $\sigma_d$ is seen as the direct Hall-Petch relationship in which $m_{C}$ is equivalent to $K$, normally used in this type of form. Parameters $\sigma_o$ and $a$ dependency with grain size is due to the distance between barriers for a dislocation, $l$, which as expressed
in an FCC material, is expected to vary with the grain size. These determined relationships are then implemented into the constitutive flow stress equation 3-13.

\[ n = \frac{0.25}{0.23 + d^{-1/2}} \]

**Figure 3-11**: Hardening parameter \( n \) for the FG and CG conditions of Al6061 versus \( d^{-1/2} \), with a fitted curve in the Morrison Type form to generate model parameters \( m_n \) and \( b_n \).
Grain Size Parameter - $\sigma_D$ (MPa)

Exp. Determined Parameter
Linear Fit

\[ \sigma_D = \frac{137}{d^{1/2}} \]

Thermal Parameter - $\sigma_o$ (MPa)

Exp. Determined Parameter
Linear Fit

\[ \sigma_o = \frac{664}{d^{1/2}} + 6.24 \]
Figure 3-12: Plot of determined parameters (solid circles) from section 4.1 versus grain size, in which a general form of the Hall-Petch type relationship seen in equation 18 is fitted to this data to determine linear constants for a) $\sigma_c$, b) $\sigma^o$ and c) $a$.

Equation: 

$$a = \frac{-8.7}{d^{1/2}} + 3.92$$
3.6 Flow Stress Simulation

Throughout the previous section, the flow stress with its two components; thermal and athermal, is described in terms of material parameters that are determined through series of high strain rate tests. These parameters were numerically optimized using the Matlab function *fmincon*, which attempts to find a constrained minimum of a scalar function of several variables starting at an initial estimate. Results are presented in Table 3-1.

These parameters are used to simulate flow stress curves for loading conditions that are not used in the calculation of the parameters. A comparison between these simulated curves and those experimentally obtained, determines the validity of the developed flow stress model represented by equation 3-13 and associated parameters. These validated parameters, can be used to simulate the true-stress strain curve for FG aluminum alloy 6061 as a function of grain size for different strain, strain rate and temperatures.
Table 3-1: Optimized model parameters to be used to simulate material response as a function of strain, strain rate, Temperature and grain size of Al6061

| $\sigma^*$ | $m_n$ | $b_n$ | $K$ | $m_o$ | $b_o$ | $k$   |
|---------|-------|-------|-----|-------|-------|------|
| (MPa)   | (MPa/μm$^{1/2}$) | (eV/K) |
| 96.25   | 0.227 | 0.25  | 138 | 1560  | -10.6 | -8.63E-5 |

| $G_o$ | $\dot{\varepsilon}_o$ | $T_m$ | $m_a$ | $b_a$ | $m$ | $p$ | $q$ |
|-------|----------------------|------|-------|------|-----|-----|-----|
| eV    | l/s                  | K    |
| 1.38  | 5.28E8               | 861  | -11.3 | 5.05 | 0.2875 | 0.15 | 1 |
During simulations it is important to include adiabatic heating which is expected to develop during high rate plastic deformation and would cause thermal softening during loading. This can be accounted for by considering a temperature term which is expressed as a function of plastic strain in the manner shown below [31]:

\[
\Delta T \approx \frac{\eta}{\rho_0 C_v} \int \sigma d\varepsilon
\]

(3-19)

\( C_v \) is the temperature dependent heat capacity of the material, given by the function \( \log[0.8816J/gK + 0.0063T] \). \( \rho \) is the mass density which remains constant at 2700 kg/m\(^3\), \( \eta \) is a constant representative of the amount of energy converted into heat. The variation of \( \Delta T \) is taken in consideration during the simulation of the flow stress carried out at different strain rates and temperature conditions. Results of these simulations are presented in Figure 3-13 which also include the corresponding experimentally obtained curves. The two sets of curves show good agreement for both material conditions, annealed Al6061 (CG) and the ECAP Al6061 (FG) for a few temperatures and strain rates. With the model parameters validated by the good agreement made between simulation and experimental curves, it is now possible to investigate the relationship that testing conditions play in the predicted stress-strain response of both material conditions. Figure 3-14 a-e shows the model results as a function of grain size, strain rate and temperature.

Results indicate an increase in flow stress with a decrease in grain size, a decrease in flow stress with increasing temperature and an increase in flow stress with an increase in strain rate.
Plastic Strain (mm/mm)
0.0 0.1 0.2 0.3 0.4 0.5 0.6
True Stress (MPa)
0 50 100 150 200

Experimental Model

\[ \dot{\varepsilon} = 4.0 \times 10^3 s^{-1} \]
\[ T = 50^\circ C \]

CG

Experimental
Model

Plastic Strain (mm/mm)
Figure 3-13: Plotted are comparisons between the simulated stress strain curves using optimized model parameters and experimental curves. For Al6061-O CG condition a) 4.0E3 s⁻¹ at 50°C and b) 4.0E3 s⁻¹ at 150°C and the Al6061-ECAP FG condition c) 3.0E3 s⁻¹ at 50°C.
Figure 3-14: Plot showing model simulations of True Stress Vs True Strain a) 100°C and 1.0E3s⁻¹, in which grain size if varied between the annealed(100μm) and FG (5μm) conditions, b) for the CG (100μm) microstructure at 200°C for varied strain rates, c) for the FG (5μm) at 100°C at varied strain rates, d) for the CG (100μm) microstructure at 2.0E3s⁻¹ at varied temperatures, e) for the FG (5μm) at 4.0E3s⁻¹ for varied with temperature
3.7 Discussion

The work presented in this paper aimed at obtaining an explicit form of the flow stress components, thermal and athermal, at high strain rate as a function of loading parameters; strain, temperature and strain rate, as well as microstructural variables, particularly the grain size. Formulations of the flow stress model have been carried out by considering the interactions between dislocations and various barrier types and their associated stress fields which evolve with grain size. The basic model parameters have been determined utilizing results of SHPB tests performed on two Al-6061 microstructures differing in their grain size, a coarse (100μm) and fine (5μm), at temperatures ranging from 20°C to 200°C and strain rates between 1.0E3 to 5.0E3s⁻¹. From this work the sensitivity of the flow stress parameters in relation to grain size have been determined. An important outcome in these results is the observation that, while the athermal stress, as expected, has a constant grain size dependence, the thermal stress component which generally does not depend on long range barrier sources such as grain boundaries will, at approximately 10μm, increasingly become grain size dependent, as shown in Figure 3-15. The results of this study may represent the approach of a transitional regime between the mechanisms that control the deformation in the coarse grain material to that of the ultra-fine grain.

Cheng et al [32], presented a deformation mechanism map for FCC metals, in which the differing deformation mechanisms with grain size are separated into four regimes. The smallest grain size regime occurs roughly at 10nm and denoted Nano-1 in which deformation occurs solely by grain boundary processes such as grain boundary sliding or Coble creep. The lack of dislocation based activities is assumed to be due to the
absence of dislocations within the grain interior. The inclusion of grain boundary processes as the dominant deformation mechanism explains the inverse Hall-Petch, or grain size refinement softening, seen at this grain size scale [23,33-38]. The second scale, Nano-2, exists with grain sizes larger than 100nm. At this range, grains are sheared by twining or by Shockley partial dislocations which are absorbed in the opposite grain boundaries, leaving intrinsic stacking faults behind. The third regime, Ultra-fine, consists of grains larger then 20-35nm up to 200-1000nm, depending on the material. Here grains are sheared by lattice dislocations which are nucleated within grain boundaries. A trailing partial can be nucleated before the entire grain is sheared by a leading partial. Therefore UFG deformation occurs by the motion of perfect dislocations that are emitted from grain boundary sources. The difference between the UFG and the final regime, coarse scale, in which classical mechanical response is expected, is that, in the UFG regime, the dislocation source can only be the grain boundaries. In the coarse grain scale, dislocation sources include grain boundaries as well as intra-granular sources, other dislocations, precipitates etc. Cheng concluded that little research has been conducted to identify the transition between grain boundary sources and mixed sources, so the exact UFG/coarse transition is not well defined.

This type of classification of materials based on grain size scale and associated source of dislocations, indicates that in the UFG regime, even with the removal of intra-granular dislocation sources, the deformation remains thermally activated due to presence of grain boundary emitted dislocations. Dao et al [39] proposed several deformation mechanisms that can occur in UFG, provided that the mechanism must have a small activation volume, a condition seen in NC and UFG metals [22,32]. These mechanisms
include the punching of a mobile dislocation through a dense bundle of excess grain boundary dislocations, defect assisted dislocation nucleation, and the de-pinning of dislocations that are pinned at boundary obstacles. Results presented in the current study assume that the transition from the traditional regime to the UFG must involve a transitional regime characterized by a fine grain scale. In this fine grain regime, deformation accommodation, which encounters a decrease of dislocation sources within the grain, would require an increase in dislocations originating from grain boundary sources. This could explain the observation, Figure 3-15a, that the thermal stress component in the aluminum material studied is inversely proportional to the grain size and the sensitivity of which increases with further refinement. This effect is possibly due to the increase of thermally activated barriers from grain boundaries sources with a decrease in grain size. The residual athermal stress with further refinement into the nanoscale, may be the reason in which dislocation activities cannot be utilized during the deformation and only grain boundary processes, as this stress would be too high to allow a dislocation to be emitted from the grain boundary. The athermal component of stress still depends on the grain size, Figure 3-15b, in both the fine and the UFG regimes due to the observed fact that Hall-Petch relationship maintains its validity in these two regimes.
Figure 3-15: Plot of the a) Thermal Component c) Athermal Component of stress versus grain size at strain of 5, 10 and 15% and a strain rate of 2.3E3s⁻¹
To further understand this transitional region between grain sizes, one can investigate the parameters within the model that evolve within grain size. The proportional nature of the hardening parameter, \( n \), with grain size, see Figure 3-11, may lead \( n \) to approach zero with further grain refinement, thus removing the strain dependence of the athermal component. This can be understood by the fact that a decrease of mobile dislocations within the grain with further grain refinement leads to a saturation of the athermal stress, which is dependent on long range barrier sources, resulting in a constant residual long range stress field. In the development of the flow stress model presented above, the thermal component has been viewed to be the outcome of dislocation/dislocation-forest interactions which is the active hardening mechanism in FCC materials. This is accounted for by the use of equation 3-11, for a CG microstructure, in which the function \( f \) is equivalent to the ratio of the initial to current dislocation spacing, \( l_0 / l_c \), where an increase in dislocation density during plastic straining would cause a decrease in barrier spacing. This ratio is set to be an function of strain and of temperature. Whereas in FG microstructure the barrier spacing is not altered by the increase in the strain and the function \( f \) stays nearly constant, approximately equal to 1 which leads to setting \( a \) in equation 11 to be nearly zero. On the other hand in the CG microstructure, where \( f \) is an evolving function, the parameter \( a \) reaches approximately 3.

The decreasing nature of the \( a \)-parameter as the grain size decreases is of great interest as it identifies the transition in the rate controlling mechanism of the thermal stress component with respect to the grain size. This transition is due to the change in the source of thermally activated dislocations. This can be explained by recognizing that the activation volume of thermally activated dislocations is given as:
\[ v^* = b \times \lambda \times l \]  

(3-20)

where \( b \) is the Burgers vector, \( \lambda \) and \( l \) are the average effective barrier width and spacing respectively. As has been presented in the model derivation, the average forest length spacing, \( l \), is a function of the dislocation density which in turn is a function of the evolving strain hardening. The length spacing, as shown by Wei et al [22] , can have two possible limits related to each of the acting deformation mechanisms: \( l_1 = \alpha / \sqrt{\rho} \) and \( l_2 = \chi d \). The first limit is associated with large grain size and high dislocation density, while the second corresponds to a very small grain size and small dislocation densities. The transition in deformation mechanisms would occur at a critical grain size, \( d_{\text{critical}} \), that can be calculated by setting \( l_1 = l_2 \). Assuming that \( \alpha \) and \( \chi \) are of the same order, then, \( d_{\text{critical}} \approx 1 / \sqrt{\rho} \). This indicates that once the grain size drops below a critical transition size, the rate controlling mechanism is no longer forest cutting, but rather grain boundaries or sub grain boundaries acting as the dominant source of dislocations.

This is explained by the fact that as a grain size is decreased the dislocation density within the grain is expected to become very low, thus removing sources of forest dislocations, whereas the obstacle density associated with grain boundaries becomes very high. It is thus possible that the controlling intersection obstacles are the grain or sub-grain boundaries. It was shown experimentally in heavily deformed copper [22], that as grain size was decreased a sharp decrease was measured in the activation volume indicating a change in the rate controlling mechanism in the thermally activated process. This is supported by results of the current work, see Figure 3-6, which shows that as the grain size is decreased, there is an increase in strain rate sensitivity, \( m \), which is inversely
proportional to activation volume $v^*$, by the relation, $m = k_b T / \tau v^*$. This relationship is further expressed in terms of $l$, yielding strain sensitivity expressions for both the coarse grains, $m_{co}$, and fine grains, $m_{fg}$, written as:

$$
m_{CG} = \frac{k_b T}{\tau \lambda b \sqrt{\rho} \chi} \quad , \quad m_{UFG} = \frac{k_b T}{\tau \lambda b \chi d}
$$

The parameters determined provide a manner in which for the model used in this work for the flow stress as function of strain, temperature, strain rate and grain size to be sensitive and capable of evolving with the transition in deformation mechanisms that occur with grain refinement. These transitions are shown by the resultant change in strain rate sensitivity due to the changing mechanisms of deformation, related to the activation volume.

The parameters determined provide a means by which the flow stress can be accurately predicted through transitional grain sizes, where rate controlling mechanisms evolve with differing grain size regimes. These transitional regimes are experimentally observed by the increase in SRS with grain refinement, and explained by the sharp change in activation volume, an indication of a change deformation mechanisms. An attempt has been made to add a defined transitional regime, fine grain, between the CG and UFG materials. In the CG, the traditional mechanical response is expected, and in the UFG the deformation mechanisms differ in which intra-granular dislocation sources are no longer present. This fine grain regime of deformation, shares mechanisms that occur in both the CG and UFG materials, but exists at a point in grain refinement at which the dominant rate controlling mechanism is evolving from intra-granular sources of
dislocations to mainly grain boundary sources. The change in dislocation sources results in the thermal stress component having an increased dependency with grain size as refinement moves from the CG into the UFG.

3.8 Conclusion

The effect of grain size on the dynamic flow stress of an Aluminum alloy has been investigated by conducting a series of split Hopkinson pressure bar (SHPB) tests in a range of strain rates and temperatures

- A constitutive relationship is derived based on dislocation interactions with barriers to their motion. The model is based on the separation of flow stress into individual components that are dependent on the type of barrier that a dislocation will encounter. The first component, athermal stress, develops from dislocation interactions with long range barriers, and is derived as a function of strain and microstructure of the material. The second component, thermal stress, arises from the interaction of dislocations with short range barriers. The sources of barriers changes depending on the crystalline structure of the material, in this case an FCC material, which has been shown to be mainly due to dislocation forests in the course grain microstructure.

- Fine Grained (FG) Al6061, 5μm, was generated using an Equal Channel Angular Press (ECAP) system, in which the grain size was refined from an annealed Al6061 course grained (CG) condition, 100μm. Using these two
different material conditions the effect of grain size on model parameters are determined and defined.

- The CG and FG Aluminum alloys were tested using SHPB at temperatures ranging from 20°C to 200°C and strain rates from $10^2$ to $10^4$ s$^{-1}$. Outcomes of these tests in the form of true stress-true strain curves are used to identify the role of different variables on the dynamic response of the test material.

- Using a procedural analysis, constitutive model parameters for the athermal and thermal stress components are generated in terms of the grain size. These relationships are used to investigate the evolution of dislocation barrier types that exists as grain size is refined.

- The FG material produced in this work is shown to exist as an intermediate scale between the CG and UFG scales. These three scales are identified by a deformation mechanism in which the thermal component of stress, increasingly becomes grain size dependent. This is explained by the fact that as the grain size is refined, a higher portion of dislocation forests barriers are removed from the grain interior, causing the grain boundary to become increasing a dominant thermally activated dislocation source.

- The constitutive model has been applied for strain, strain rates, temperatures and grain size conditions which were not utilized in the model parameter generation. Results of these applications in terms of true
stress-strain curves are compared well with those experimentally generated curves.

3.9 References

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CHAPTER 4
MAJOR CONCLUSIONS AND FUTURE RECOMMENDATIONS

4.1 Major Conclusions

The objective of this research project was to develop a physics-based constitutive relationship for the dynamic flow stress in metals with the model parameters being derived as explicit functions of microstructural features of the material. This model is intended as a numerical tool that can be used to tailor the material’s microstructural characteristics in relation to a given set of dynamic loading conditions. To achieve this, combined experimental, analytical and numerical studies were performed on two different materials, BCC pearlite-ferrite carbon steels with different pearlite volume fractions and an FCC aluminum alloy in both coarse and fine grained microstructure. A constitutive model is developed based on thermally activated dislocation interaction with barriers. The model separates the flow stress into two stress components; thermal and athermal. The athermal component is related to the interaction of dislocations with long range barriers and is a function of strain and microstructure of the material. Long range barriers include grain boundaries, precipitates and second phase particles. The thermal component accounts for the interaction of dislocations with short range barriers, that include phonons, forest dislocations, dislocation jogs and kinks and Peirils-Nabarro stress. This stress component is shown to be a function of strain rate and temperature. Experimental work included high strain rate \(10^2\text{-}10^4\text{s}^{-1}\) compression tests within at temperatures ranging from 20°C-650°C for carbon steels, and 20°C-200°C for the
aluminum alloy. Results of these experiments were presented in the form of true stress-true strain curves.

Utilizing the experimental data in conjunction with the models analytical formulations, parameters were generated as functions of materials and loading variables. For carbon steels, these parameters were defined in terms of pearlite volume fraction while for the aluminum alloy they were generated as functions of the grain size through Hall-Petch type laws. This law has been shown to breakdown during the transition from the coarse grain scale (>10microns) to the ultra-fine grain scale (1micron-10microns). This delineation from the original Hall-Petch effect has been shown to be due to the change in the deformation mechanism associated with each of these scales. The two-component flow stress model presented in this thesis is developed to have the ability to bridge this transition by considering each grain scale and associated deformation mechanism.

A summary of conclusions made from the experimental, analytical and numerical work are listed.

**Influence of pearlite volume fraction in the dual phase carbon steel**

- A dual stage heat treatment of A572 was developed to obtain a microstructure free of pearlite colonies. This treatment consists of heating to 750°C which is well above the eutectic point of the steel at which a phase transformation would occur. The alpha-ferrite and pearlite grains transform into a fully austenitic phase. The material was held at this temperature for one hour, followed by a rapid quenching in an ice-brine solution. This rapid cooling allows the material to revert back into
the dual phase structure consisting of alpha-ferrite and pearlite, though the rapid quenching does not allow the pearlite to form into as well organized colonies as in the as-received condition. This material is then tempered below the eutectic, at 720°C for 72 hours. This process diffuses the fine cementite structure into speriodized carbides, mostly residing at alpha-ferrite grain boundaries.

Optical and scanning electron microscopy was utilized to characterize the volume fraction of pearlite, as well as grain size. The as-received A572 steel was shown to have a volume fraction of 9% and a grain size of 25µm, with the heat-treated condition resulting in a 0% volume fraction and grain size of 38µm. The other two steels 1018 and 1060 showed volume fractions of 20% and 72% and grain sizes of 9µm and 7µm respectfully.

A Split Hopkins Pressure Bar (SHPB) apparatus was designed and constructed in order to experimentally test metals at high strain rates. The system includes the ability to test in a wide range of temperatures ranging from the liquid nitrogen temperature to temperatures up to 900°C. The SHPB components include a loading gas gun which fires a projectile to create a dynamic event, the loading bars along which strain pulses are measured, a thermal heating system as well as a data acquisitions system which allows for data to be collected from strain gauges bonded to the loading bars.

A series of SHPB dynamic loading tests were conducted on as-received A572 (9% VFP), heat treated A572 (0% VFP), 1018 (20% VFP) and 1060 (72% VFP) steels. The as-received and heat treated conditions were tested at temperatures:
20°C, 300°C, 500°C and 650°C for strain rates ranging from $7 \times 10^2$ to $5 \times 10^3$ s$^{-1}$. The 1018 and 1060 steels' were tested at 500°C and 650°C at $1.5 \times 10^3$ s$^{-1}$.

- Experimental test results for each material were investigated as a function of strain rate and temperature. The as-received A572 steel showed an increase in strain rate sensitivity as compared with the quasi-static response. The dynamic strain rate sensitivity was determined to be 42.5 MPa/s at room temperature and 77.2 MPa/s at 500°C. The other carbon steels examined in the thesis showed a similar strain rate effect. These steels have also shown that their flow stress decreases with temperature until a critical temperature is reached, at which the decrease in flow stress saturates.

- An analytical procedure is performed in order to generate model parameters for the athermal- and thermal-stress components of the steels mentioned above. The athermal component at varied levels of strain is determined to be the corresponding saturated stress when plotted versus temperature. The thermal component of stress which are calculated as the difference between the athermal stress and the total flow stress, are then investigated in relation to the strain rate and temperature.

- A rule of mixture type weighted Hall-Petch relationship was utilized to define the flow stress evolution with volume fraction of pearlite. Furthermore the Hall-Petch constants for pearlite and ferrite phases are determined.

- The constitutive model has been applied for different microstructure, strain, strain rate and temperature. Results of these comparisons showed a good agreement
with experimental curves that have not been used in the generation of the model parameters.

- A parametric study is carried out to examine the relative contribution of pearlite as a strengthening phase and showed that this contribution increases with the decrease in the grain size.

Influence of grain size in the single phase aluminum alloy

- An equal channel angular press (ECAP) was designed and constructed in order to refine the grain size of an aluminum alloy (Al6061) through severe plastic deformation. The ECAP system includes a high strength tool steel die with a channel angle of 95° to apply approximately 1.0 strain per pass, a 100 ton hydraulic press as a loading system, a die heating system to allow for the ability of high temperature pressing and a data acquisition system for measurements of pressing force, displacement and rate.

- A fine grained (FG) Al6061 (5μm) was generated using the ECAP system using processing route Bc, at a temperature of 150°C and at a rate of approximately 1inch/minute. The material in its original state from which it was refined was an annealed Al6061 coarse grain (CG) condition (100μm).

- The CG and FG Aluminum alloys are tested using SHPB techniques at temperatures of 20, 50, 150 and 200°C and strain rates 1.5E3, 2.8E3, 4.7E3s⁻¹. Dynamic true stress-true strain curves were generated during these tests.

- Using a the same analytical procedure applied to carbon steel, constitutive model parameters for the athermal and thermal stress components are generated for both the FG and CG Al6061 material conditions. By comparing parameters obtained
for each of the Al microstructures, it is concluded that both the athermal and thermal stress components are grain size sensitive.

- It is shown that the FG material produced in this work exists as a distinctive scale that lies between the coarse and ultra-fine grain size levels. As such the deformation mechanisms of the fine grain scale have features that are associated with those of the coarse and the ultra-fine grain materials. In this type of transition, as the grain refinement reaches the FG scale, the thermal component of stress, becomes increasingly grain size dependent. This is explained by the fact that a high degree of dislocation forests barriers are removed from the grain interior, causing the grain boundary to become increasing dominant as the thermally activated dislocation source.

- The constitutive model has been applied for different strain, strain rates and temperatures and for both coarse and fine grain size conditions and results are compared with true stress-strain curves that had not been utilized in the model parameter generation and optimization. Results of this comparison showed good agreement with those experimentally generated.

4.2 Future Recommendations

The work completed in this thesis provided a constitutive relationship that describes the dynamic stress-strain response of materials as a function of strain, strain rate and temperature as well as microstructural features, in particular, second phase particles and grain size. The model materials utilized in this work are low carbon steel (BCC) and an aluminum alloy (FCC). A continuation of this work would be to apply the
developed model and the manner by which corresponding parameter are generated to other materials. Several additional concepts should be examined in relation to future research efforts, these are:

- The impact loading utilized in both the LCS and Al work was designed to provide a maximum strain rate lower than $10^4 \text{s}^{-1}$ which would exclude the role of dislocation drag as a flow stress contributor. At rates higher than this limit, the drag stress becomes an efficient contributor and possibly the dominant stress component. In order to examine this role, modification to the existing SHPB must be made to increase its capabilities to provide higher strain rates.

- A possibility of extending the current model is to investigate its parameters in relation to Hexagonal Closed Pack (HCP) crystalline structures which are shown to follow either the BCC or FCC dynamic response. This is explained by the fact that the most probable rate controlling obstacles in the HCP materials vary between forest dislocations or Peierls-Nabarro.

- The model should also be examined in relation to other microstructural features such as other secondary phases (carbides or gamma prime) or dislocation density. These types of secondary phases could affect the nature of dislocation/barrier interactions and bring about further modifications to the athermal stress component of the dynamic flow stress. While on the other hand the modification of the athermal stress can also be studied through dislocation density, which can
be modified by investigation an as-received, annealed and previously strained material conditions. All of which should have a varying density of dislocations.

- This thesis has examined the role of the grain size by exploring the range from the coarse grained level to the fine grain level. Results of this work was extrapolated theoretically to the ultra-fine size. It is then important to validate this prediction by carrying out an experimental program involving grain sizes of this scale well as nano-crystalline materials. At these grain levels it is known that the deformation mechanisms differ from that of the coarse grain, in which sources of dislocations become only the grain boundaries.

- The effect of grain size specifically in BCC metals would be of great interest the behavior of this crystalline structure has been shown to differ from that of the FCC material studied in this work. This change is flow stress behavior is the decrease in strain rate sensitivity, as opposed to in FCC metals, grain size reduction results in an increase in strain rate sensitivity. The application of the model produced could therefore provide insight as to why this response occurs.
APPENDIX A

SPLIT HOPKINSON PRESSURE BAR

A.1 Split Hopkinson Pressure Bar Apparatus

This appendix section will present an overview and background of the history of dynamic testing as it pertains to the use of the split Hopkinson pressure bar (SHPB) in the dynamic loading field. Also a general description of the conventional SHPB will be given to aid in the understanding of following material.

A.1.1 Brief History

The split Hopkinson pressure bar has become the most widely used tool in high strain rate mechanical testing ($10^2$-$10^4$ s$^{-1}$). This is true because of the accuracy and repeatability that the tests can achieve. Data obtained from these tests can be utilized in many different fashions, such as determining conventional material properties like yield strength, strain hardening and ultimate strength while also being used in more advanced manners to study temperature and strain rate effects or to generate material models, as it has become common knowledge that these mechanical properties will change at higher strain rates. Most material properties found within handbooks and design manuals are determined at the quasistatic testing regime, it is important in some cases, in order to ensure proper performance, that designs be based on the material response in the high strain rate regime, examples include vehicle collisions, explosive detonation or the inevitable dropping of an electronic device [1].
The development of the SHPB system was due to the collective work of several researchers over several decades. The first was Bertram Hopkinson [2] (1913) who developed a manner in which to determine pressure-time relationships due to an impact produced by an explosive. Components of the experiments included an explosive impactor, a long steel rod, a short steel billet, and a ballistic pendulum. Impact occurs at the rod generating a compressive wave which travels to the far end where the short billet is in contact, and reflected at the free-end of this short billet as tensile wave. When this pulse returns to the long rod interface, the pulse wave is ended. The wave that was now fully-generated within the long rod impacts a ballistic pendulum, located at the other end of the long rod. The motion of the ballistic pendulum would lead to the determination of the maximum pressure and total duration as well as an ill-defined pressure-time curve.

Davies [3] (1948) greatly improve the accuracy of this system by the addition of condensers to measure the strain generated within the pressure bar, the long steel rod in Hopkinson apparatus. The output of the condensers is relatable to the pressure-time relation within the bars, as long as the elastic limit is not reached within the bars. Kolsky [4] (1949) the first person to utilize this system in order to determine the stress-strain response of a material under high strain testing conditions. The manner in which Kolsky measured the pressure within the bars was similar to Davies. The major addition to the setup was the adding a second bar, which sandwiched a specimen between the first loading bar. Kolsky noted the importance of specimen dimensions, lubrication of the specimen as well as a detailed analysis in determining the stress-strain response from data collected using the condenser microphones. The system has been mostly unchanged in its most basic compressive form with the addition of the use of strain gauges by Krafft [5]
(1954) to measure the signals generated within the pressure bars, as well as the addition of a gas gun to propel a striker into the pressure bars rather than the explosive detonation.

A.1.2 General Compressive SHPB

The SHPB has become the tool of choice to characterize materials at strain rates typically between $10^2$-$10^4 \text{s}^{-1}$. The SHPB as explained has evolved over the past century from a simple explosively controlled apparatus to a complex, accurate and repeatable system that can precisely define a materials properties. There are several variants that have been developed which include a tensile form of the system, systems used for high and low temperature conditions as well as automation of the system, please see the following references for a comprehensive SHPB literature review, [1,6-14]. To begin it is necessary to investigate the general compressive SHPB system.

The typical SHPB system consists of three main components which include the loading bars, a gas gun utilized as a loading device and a data acquisition system. During a SHPB test a short cylindrical specimen is placed between two long loading bars, typically made from high strength maranging tool steel. Dimensions of the loading bars are typically 0.75" in diameter and around 5 feet long. This ration of length to diameter is important for the wave interaction during a loading event. The loading bars are denoted as the incident and transmitted bars, in which a striker that is propelled by the gas gun, towards the incident bar. A gas gun used in a SHPB typically consists of a gas source, a gas chamber to hold gas before firing, a manner in which to fire the gas gun, usually a high rate solenoid valve, and a barrel to track the striker towards the incident bar. This impact between the incident bar and the striker generates a compressive pulse within the
two bars respectively. The pulse reaches the free end of the striker and reflects fully thus reversing the sign of the pulse to a tensile wave. When this tensile wave reaches the incident/striker interface, it effectively ends the pulse, therefore the length of the striker determines the duration of the pulse generated within the incident bar. The fully developed incident pulse now travels down the length of the incident bar until it reaches the sandwiched specimen. Due to the miss-match of impedance between the specimen and incident bar, a defined portion of the pulse is transferred into the specimen and the remaining is reflected back into the incident bar. The transmitted portion of the pulse continues through the specimen and into the transmitted bar. Some reflections do occur at the transmitted/specimen interface due to the miss-matched impedance. These reflections are what causes the initial unsteady state stress state of the specimen, thus making early elastic data of the specimen unreliable, and are also the source of oscillations within the pulses collected using the data acquisition system. These oscillation and the initial unsteady stress state can be mitigated using pulse shaping techniques, but will be ignored at this point. The pulse that is transferred into the transmitted bar, travels along its length where it meets a momentum stopper to capture the remaining energy of the pulse. The strain histories of the two loading bars, which include the incident, reflected and transmitted pulses, can be recorded using two strain gauges. One mounting on the incident bar, which collects the incident and reflected pulses. The other is placed on the transmitted bar for the transmitted pulse. Typical data acquisition systems include the before mentioned strain gauges, a manner in which to excite, filter and amplify the gauges and a recording system, usually a high-speed oscilloscope or computer. These three pulses as, defined by Kolsky, can be used to generate the stress-strain response of
the specimen that was sandwiched between the loading bars. A derivation and explanation of these equations is expressed in the following section.

A.2 Design of SHPB at Mechanics of Materials Research Laboratory

A.2.1 SHPB Governing Equations

The compression form of the split Hopkinson bar has been well established in the dynamic testing field, where most compression machines are similar in nature with a few iterations being applied by different researchers depending on the application [7]. The theory of the wave propagation with detailed analysis is largely found in literature and with the level of common knowledge of the subject [15]. A short derivation of the major equations used in the SHPB analysis will be presented. The solution to the one dimensional wave equation, A-1, for the incident and transmitted bars, denoted by subscripts 1 and 2 respectfully is shown to be in equations A-2 and A-3

\[
\frac{\partial^2 u}{\partial x^2} = \frac{1}{c_b^2} \frac{\partial^2 u}{\partial t^2} 
\]

\[
u_1 = f(x - c_b t) + g(x + c_b t) = u_i + u_r \quad (A-2)
\]

\[
u_2 = h(x - c_b t) = u_i \quad (A-3)
\]

where \(c_b\) is the longitudinal wave speed of loading bar material given by \(\sqrt{\frac{E}{\rho}}\), where E is the elastic modulus and \(\rho\) is the density. \(u_1\) and \(u_2\) is displacement for the incident and transmitted bars respectfully. f, g and h are resultant differentiation functions and \(x\) and \(t\) are differentiating terms of distance and time. \(u_i\), \(u_r\), and \(u_i\) are the displacements of the
incident, reflected and transmitted pulses. Differentiating equations A-2 and A-3 with respect to x is shown to be,

\[
\frac{\partial u_i}{\partial x} = \varepsilon_1 = \varepsilon_i + \varepsilon_r \tag{A-4}
\]

\[
\frac{\partial u_t}{\partial x} = \varepsilon_2 = \varepsilon_i \tag{A-5}
\]

where, \( \varepsilon_i \) is the strain induced with the corresponding subscripts for the incident and transmitted bars, 1 and 2 respectfully and the incident, reflected and transmitted pulses, i, r and t respectfully. Referring to Figure A-1, equation A-4 can be seen graphically, in which the X-t diagram shows the interaction of the pulses generated from the striker impact with the specimen and incident and transmitted bars.

\[\text{Figure A-1: X-t diagram representing the wave interaction within the SHPB testing apparatus. A wave is generated by the striker during initial impact, which travels through the incident, into the specimen where a reflected and transmitted pulse is generated}\]

By differentiating equations A-2 and A-3 with respect to time the following is shown to be
\[
\frac{\partial u_1}{\partial t} = \dot{u}_1 = c_b(-f' + g') = c_b(-\varepsilon_i + \varepsilon_r) \quad (A-6)
\]

\[
\frac{\partial u_2}{\partial t} = \dot{u}_2 = -c_b\varepsilon_i \quad (A-7)
\]

Furthermore, assuming that the interface between the two loading bars is in a state of equilibrium during a given experiment, the forces \( F_1 \) and \( F_2 \) are therefore seen to be equal in which,

\[
F_1 = A\sigma_1 = AE\varepsilon_1 = AE(\varepsilon_i + \varepsilon_r) \quad (A-8)
\]

\[
F_2 = A\sigma_2 = AE\varepsilon_2 = AE\varepsilon_i \quad (A-9)
\]

where \( A \) and \( E \) are the cross sectional area and elastic modulus of the loading bars respectfully and therefore be equating these two equilibrium forces it can be shown that

\[
\varepsilon_i = \varepsilon_i + \varepsilon_r \quad (A-10)
\]

The strain rate, \( \dot{\varepsilon}_s \), of the specimen being loaded can be described as

\[
\dot{\varepsilon}_s = \frac{\Delta \varepsilon}{\Delta t} = \frac{\dot{u}_1 - \dot{u}_2}{l_s} \quad (A-11)
\]

where \( l_s \) is the length of the specimen. The strain rate, using equations A-7, A-8, equations A-11, A-12, can be written as

\[
\dot{\varepsilon}_s = \frac{2c_b\varepsilon_i}{l_s} \quad (A-12)
\]

The engineering strain of the specimen can be found by integrating equation A-12 with respect to time as shown by
Continuing with the assumption that the specimen is in a state of equilibrium, \( F_1 = F_2 \), then therefore \( F_2 \) is the force that is applied upon the specimen and the engineering stress, \( \sigma_s \), can be written as

\[
\sigma_s = \frac{AE \varepsilon_s}{A_s}
\]

where \( A_s \) is the initial cross sectional area of the specimen. To calculate the true stress and strain, \( \sigma_T \) and \( \varepsilon_T \) respectfully from the engineering stress and strain the following equations are used.

\[
\sigma_T = \sigma_s (1 - \varepsilon_s)
\]

\[
\varepsilon_T = -\ln(1 - \varepsilon_s)
\]

From equations A-32 - A-36 it is therefore shown that during a given experiment, the magnitudes of the three loading pulses, incident, reflected and transmitted can be used to characterize the material during high strain loading by the generation of the true stress-strain curve. The method and apparatus used for the work presented within this thesis is presented in the following sections.

A.2.2 Design of SHPB Components

The components of the SHPB system and it's subsystems each consist of several different design considerations when the system is first begin conceptualized. For the loading bar system, incident and transmitted bars, a few of these considerations are the
length and diameter and the ratio between them, the material to be tested, a mounting system, the available laboratory space, testing temperature and material of the bars themselves. Each of these parameters are interrelated making the design of the SHPB a complex, multi-variable problem, the most important of which is the material to be tested. A system that is to be used mainly for testing plastics, composite or concrete would be very different then that meant for testing metals as in this case, in which the bar material, diameter and lengths would all be adapted for the material to be tested, and in turn the gas gun and data acquisition system would also have to reflect these differences. The gas gun has several design considerations as stated, that are based upon the material to be tested, maximum pressure and velocity, the bar material and dimensions, projectile design and barrel length. The data acquisition system is less affected by the other variable of the system, but still has a few design considerations that must be taken into account, mainly being the strain gauges used to measure the loading pulses and their positioning along the length of the bars based on the length of projectiles being used, the amplifier-conditioning system and data capturing system also need to be capable of handling the high rate of data with loading pulses being in the hundreds of nanosecond time range. The details in the design of the SHPB used in this research is expressed in this chapter.

A.2.2.1 Overview of SHPB at MMRL

For the purposes of referencing, an summary of the design completed and built at the MMRL is given. The final design of the SHPB consists of 0.75” diameter Maranging 350 hardened steel bars for the incident, transmitted and strikers. The bars are center-less ground to high tolerance and the bars ensured in straightness to within +/-0.001/3ft along their length. A gas gun was designed and built as a loading apparatus capable of 500psi
and projectile velocities exceeding 90m/s, with care taken not to exceed the yield strength of the striker and loading bars. Strikers of varying lengths were machined, as means to vary the strain rate and strains induced upon the specimen coupled with varying pulse lengths, giving an achievable range of 20 to 100 microseconds. Coupled with a variable gas gun pressure, and in turn striker velocity, a wide, repeatable and precise strain rate testing range is possible, from $10^2$ to $10^4\text{s}^{-1}$. A schematic representing the SHPB built is shown in Figure A-2.
Figure A-2: SHPB schematic showing major components of loading system: gas gun, incident and transmitted bars
A high rate data acquisition system is assembled using strain gauges, a Vishay signal-conditioning amplifier with a built-in Wheatstone bridge and a Tektronix digital phosphorus oscilloscope for data recording. Mechanical drawings of each part generated for the construction and manufacture of the SHPB is shown in a following subsection of this appendix.

A.2.2.2 Gas Gun

The gas gun component of the SHPB system is used to propel a striker bar at the incident bar, creating the loading pulse that is used to load the specimen at a certain desired strain rate. A typical gas gun consists of a holding chamber, a firing mechanism, a manner in which to fill the chamber and barrel to direct the projectile or striker into the loading bars. In use, the gas gun chamber is filled to a desired pressure, held in a state of pressure by utilizing a valve, when the valve is released, this pressure is applied to the striker, thus propelling the projectile into the incident bar.

The chamber must be designed to be able to withstand the high pressures utilized to propel the striker. In order to safely fill and release the pressure within this chamber, a conservative safety factor is applied, making the chamber much stronger and resilient than would be necessarily needed. The chamber is composed of a cylinder capped with two end pieces and bolted together using 4 1"-20 grade 8 bolts. The cylinder that creates the main body of the chamber is machined from 316 stainless steel with an ID of 4.5" and OD of 5.5", resulting in a thickness of 0.5". Using classical pressure vessel analysis it was determined that this thickness would result in a safety factor of approximately 13 at 500Psi. The end caps are machined from stainless 440C, a high strength hardened steel
alloy. Again this piece is designed to ensure safety during use. A series of o-ring groves are machined to seal in the pressurized gas, preventing any leaking to occur. A set of brackets were created to fix the gas gun to the support structure, not allowing any movement during firing. Additionally a funnel was created that is set into the main body at the front of the chamber. This allows for the pressurized gas to have a smooth transition from the chamber and into the gas gun barrel. This is a 3" tall, solid funnel that tapers from the 4.5"ID of the chamber down to the 0.75" barrel diameter.

The gas gun barrel is 38" long with an OD of 1.5" with the ID of 0.75” was precisely bored and honed to a R16 smoothness. This allows for a smooth fit of the striker, not allowing for any gas to escape around the projectile during firing. Beginning 20" down the length of the barrel, a set of 8 pairs of 1/4-20NPT taped holes, 2 inches apart are machined into the barrel. This allows for pressure behind the striker to be release when contact is made with the loading bars as well as giving the ability to effectively change the length of the barrel if needed. The barrel is attached to the gas gun chamber using the pressure release valve to fire the gun and mounted to the support structure using two mounting braces. The type of valve utilized is a quick release 12V-DC solenoid valve. Solenoid valves are electrically controlled valves that use the collapse of a magnetic field to move a plunger that was previously blocking the path of travel of the gas. A solenoid valve was utilized because of its quick, repeatable and reliable release of pressure. Care was taken to ensure full sealing of this valve, while also allowing for the fastest possible opening. This was done by customizing the valve with specific teflon seal and spring strength for the gas used and pressure range with help from the manufacturer. A simple switch can be used to apply the proper voltage to the valve to
cause the opening of the valve, or a more complex sequencing can be used as in this case, presented when extreme temperature considerations are discussed. A series of pressure lines, an emergency release valve, a pressure regulator, digital pressure gauge and a large pressurized nitrogen tank are used to fill the gas chamber when testing is to begin. A figure of the actual gas gun used in the SHPB is shown in Figure A-3.
Figure A-3: Gas Gun apparatus used to propel projectile striker at loading bars, generating a loading pulse: Components included gas chamber, filling and emergency release, solenoid valve, firing switch and barrel.

Figure A-4: Plot showing the 8" striker velocity versus the gas gun pressure experimentally determined (points) and the theoretical curve generated (line).
A series of test firings were completed on several of the strikers to establish and validate theoretical curves for gas gun pressure versus striker velocity curves. These curves are then used to decided upon a pressure to achieve a desired strain rate testing conditions. The velocity was measured by using the measured stress in the incident bar during impact, measured by the data acquisition system. The theoretical model for the incremental velocity down the length of the barrel is described as

\[ V_n = \sqrt{V_{n-1}^2 + 2a_n d_n} \]  \hspace{1cm} (A-17)

where \( V_n \) is the incremental velocity at a given point, \( V_{n-1} \) is the velocity at the previous increment, \( d_n \) is the incremental distance and \( a_n \) is the acceleration given by equation A-38 and is founded on the classical \( F = ma \) equation where the force applied by the pressurized gas is subtracted by the force due to drag.

\[ a_n = \frac{P_n A_{cr} - 1/2 \rho_{nit} C_d V_{n-1}^2}{m_{st}} \]  \hspace{1cm} (A-18)

where \( P_n \) is the incremental pressure based on the changing volume as the distance down the length of the barrel increases, \( A_{cr} \) is the cross-sectional area of the barrel, \( \rho_{nit} \) is the density of the nitrogen gas used as a firing propellant, \( m_{st} \) is the mass of the striker used and \( C_d \) is a drag coefficient that is adjusted to match the theoretical curve to the experimental data points determined by testing. The plot of theoretical striker velocity versus initial gas gun chamber pressure with experimentally determined data points for the 8" striker is shown in Figure A-4.
A.2.2.3 Loading Bars: Incident and Transmitted Bars

The loading bar system typically consists of an incident bar, transmitted bar, minimal friction mounting apparatus and momentum trap at the free end of the transmitted bar. The bars are typically the same diameters, lengths and material. They must be ideally straight and free to move in any supports they may have while being accurately aligned, sharing a common longitudinal loading axis to ensure one dimensional wave propagation. Based on length of pulses typical upwards of 20 inches, and ratios of length to diameter of the bars which are 80 or greater, in the 5 foot range in length. This is to ensure one dimensional wave propagation and no over-lapping of loading pulses during data acquisition.

The SHPB being designed is to be used to dynamically test metal materials, meaning that they are generally higher in strength as opposed to a system meant for testing plastics. Equations derived for SHPB analysis are based on the assumption that a generated elastic wave is being applied onto smaller, softer specimen to plastically deform the material. To ensure that the loading bars remains elastic, a high yield, linear elastic material must be used. This sets an upper limit at which the level of stress from the striker can be applied on the incident bar, with a safety factor to ensure that a plastic wave is not generated within the loading bars, causing damage to the system.

The incident, transmitted and striker bars are made from maranging 350 grade steel that has been hardened to have a high yield stress of 2GPa and linear elastic behavior with an elastic modulus of 207.7GPa. This high yield stress ensures that the bars can be loaded to the stress necessary reach strain rates up to $10^4\text{s}^{-1}$ and has become a
popular material choice for the loading bars of SHPB. The diameter of 0.75" was chosen using the ratio of 80 of length to diameter ratio ensure one dimensional wave propagation, using a length of 5ft, a common length used in SHPB design, but also based on laboratory space constraints. The small diameter also reduces the force necessary applied by the gas gun to achieve the strain needed to deform the specimen, but still allowing for the specimen to be substantial enough for deformation analysis post testing, such a microscopic and hardness analysis. The bars where center-less ground to a tolerance of +/- 0.001" and ensured to be straight within 0.0005" per foot of length, this is to ensure one dimensional wave propagation. The bars a supported by three mounting blocks each, which included pressed fit slider bearings of 863 bronze, to ensure a frictionless support allowing for restrictive free axial bar motion. These bearings are also greased with a light grease to further improve the reduction of friction. The mounting blocks a machined from stock aluminum 6061, and was chosen due to its ease of machining as well an acceptable strength. Each mounting block is bolted to the main support structure and are fully adjustable in all directions to allow for accurate alignment of the two bars as well as with the gas gun, this as well ensures one dimensional wave propagation. A picture of the actual loading bar component can be seen in Figure A-5.
Figure A-5: Loading bar components used in SHPB, showing major parts which include incident and transmitted bars, a momentum trap, frictionless support system for bars and the specimen loading area

Figure A-6: Representation provided by Vishay micro-measurement group portraying the averaging effect of the strain gauges dependent on the length of the gauge, a major design consideration
A momentum trap is utilized at the free end of the transmitted bar to catch the residual momentum from the test. The entire system is supported by a rigid structure that consists of a structural steel W-beam with welded steel leg supports to raise the system to a comfortable user height with adjustable feet for fine adjustment of the height. The structural integrity of this component is important to ensure that all energy within the system is utilized in the testing, and not lost to any undesirable motion of the system. A separate W-beam section of the same type is used to mount the gas gun, and it too is structurally adequate to handle the force caused from the gas gun firing. The separation of the gas gun from the loading bars, ensures that any vibrations caused from the release of pressure during firing is isolated from the loading bars and the sensitive strain gauges used to measure axial displacement of the bars, as well as allowing for accurate alignment with the rest of the system.

A.2.2.4 Data Acquisition System

The data acquisition system utilized in the SHPB has several important considerations. The typical components used to acquire data are a set of strain gauges, Wheatstone bridge, signal amplifier and a manner to save and capture data typical a high rate oscilloscope or A/D computer interface. Strain gauges are used to capture the displacements of the loading bars caused by the passing of the applied and resultant stress pulses during testing, with a gauge being bonded on either. The gauges must be capable of withstanding the high amount of strain induced, properly bonded for accurate measurement, and accurately placed an adequate distance from either ends of the bars, to ensure an overlapping of pulses is not measured. They must also be compatible with the Wheatstone bridge and amplifier system utilized. The amplifier and oscilloscope must
have high frequency response in order to record the appropriate data, usually shorter than a millisecond in duration. To ensure all components used are capable of doing so, a minimum of 100kHz frequency response should be established.

The strain gauges used to measure axial displacement of the loading bars are 0.125" dynamic strain gauges. These gauges a designed for dynamic use and are fully encapsulated iso-elastic with high endurance lead wires. The iso-elastic constantan alloy, designated alloy D by the supplier has superior fatigue life desirable for dynamic use as opposed to other alloys used for static cases. The gauges also have a high gauge factor, 3.3, which improves signal to noise ratio, in which high noise levels are typically seen in dynamic use. The size of the gauge selection is of importance. A gauge of smaller size is desirable so that the strain across the gauge is less affected by averaging over the length and thus able to handle measuring of high frequency load, but to small of a size and the gauge becomes difficult to bond to the bars and handle in general. This averaging phenomenon is represented in Figure A-6 from Vishay micro-measurement group.

They strain gauge utilized in the apparatus is a half-bridge configuration and has resistance of 350Ω, a higher resistance than the other common resistance of 120 Ω. This was chosen due to the advantages that include decrease of lead wire effects, reduction of heat generation by a factor of 3 and an improvement of the signal to noise ratio. Generally strain gauges work by having a constant supplied voltage, in which when a resistance change occurs, as occurs when a strain is applied, the voltage measured across the gauge can be related to the amount of strain applied as given,
\[ \varepsilon = \frac{4V}{V_s G_C(GF)} \]  

where \( V \) is the measured voltage, \( G_C \) is the gain applied by the signal-amplifier, \( V_S \) is the supplied voltage and GF is the gauge factor, 3.3 for these gauges. The resultant measured voltage is in the millivolt range, so a signal-conditioner system with a built in Wheatstone bridge is utilized, where a specified gain, \( G_C \), can be applied to amplifier the signal into the Volt range of measurement.

The placement of the strain gauges is a critical step in the experimental setup of the SHPB. Placement of the gauges should be placed as close as possible to the specimen loading bar interfaces. This is to reduce wave dispersion effects that are caused by the physics of wave motion in an elastic media. From the nature of SHPB testing, a short rise time and step nature of the impact loading pulse, the elastic wave produced is a superposition of high and low frequencies. The higher frequencies travel at slower velocities than that of the lower frequency that maintain a velocity approaching one dimensional elastic wave velocity. Due to this difference in velocities, the wave over a long length will begin to spread and disperse. There exists another condition though that makes the ideal case of placing the gauge close to the specimen-loading bar interfaces, and this is that the loading pulse has a finite length. If the gauge is placed too close to this interface, the reflected and incident pulse would overlap when measurement is to be taken. To ensure this does not occur, the gauges should be placed at minimum, a distance equal to the length of the loading pulse from specimen-loading bar interface, or twice the length of the striker used. Furthermore, another concern in placing the gauges is to ensure that they are only measuring in the axial direction, meaning they are accurately placed to
be parallel with the longitudinal axis of the bars. This is carefully done during mounting of the gauges, using tick markings supplied on the gauge body and use of paper wrapped around the loading bars to mark a line that is perpendicular to the longitudinal axis to which the tick markings are aligned. To bond the gauges to the loading bars, a M-200 bonding kit from Vishay is used. This is a quick curing epoxy, which strongly adheres the gauges to the bars, leading to accurate measurement of the displacement caused by the loading pulses; also a protective coating is utilized to ensure no damage occurs to the gauges during use. It is important to use the proper epoxy for gauge bonding, this epoxy transfers the displacement from the bars into the gauge for measurement, if this epoxy is too stiff, the epoxy will crack causing failure during loading and if too flexible the epoxy will absorb a portion of the displacement leading to incorrect strain measurement.

The system used to control the excitation voltage and amplify the signal generated from the strain gauges during testing is the Vishay 2310B signal conditioning amplifier which is specifically designed for dynamic use, and has the capability to accept quarter or half bridge strain gauges and has maximum frequency response of 300kHz, well above the accepted minimum of 100kHz. The voltage data is sent via BNC outputs from the signal conditioning amplifier to the data recording system, a Tektronix DPO 3034 digital phosphorus oscilloscope. The oscilloscope has a maximum recordable frequency of 300MHz and a sampling rate of 2.5 giga-samples per second. A trigger function is utilized once a pulse is sensed, in which when there is a slope change above a certain voltage level, the system begins recording for a set period of time, this allows for a repeatable and reliable manner to record the data. A picture of the data acquisition system used is shown in Figure A-7. The data can then be saved onto a flash drive, or also sent
through a closed network, and moved to a computer where data can be analyzed and processed to generate the stress-strain curve. A series of Matlab programs written by the author are utilized to complete this process.
Figure A-7: Showing data acquisitions system designed for SHPB experimental apparatus. System includes high rate Tektronix digital phosphorus oscilloscope, Vishay dynamic signal conditioning amplifier with built in Wheatstone bridge and firing system power supply.
A.2.2.5 High and Low Temperature Testing

The deformation of metal is known to be dependent upon temperature, in which the flow stress decreases with an increasing temperature. In generating model parameters that take this temperature sensitivity into account and in order to qualitatively define this sensitive it is therefore important to have the ability to test the material of investigation within a wide range of temperatures. There arise some difficulties that come with testing at extreme temperature conditions, which include achieving a constant uniform temperature of the specimen, the mechanical properties of the loading bars with changing temperatures mainly the elastic modulus' sensitivity with temperature, ensuring that heat damage does not occur to the loading bars and the temperature sensitivity of strain gauges used to measure the strain pulses. There are many different manners in which researchers have handled this issue these include automatic specimen positioning systems which load the heated specimen just before testing, the use of replaceable impedance matched inserts on the loading bar-specimen interfaces that protect the bars from any damage, and localized heating systems that reduce the heat transfer into the rest of the system. Considering all of these factors a sub-system was devised that would take these concerns into consideration.

As mentioned having the specimen at a precise constant temperature is of importance to ensure that the data collected is accurate and not changing during testing. The difficulty in keeping the specimen at a constant temperature is heat loss, sources of which include the outside environment, causing heat convection loss to the surrounding air and also through heat conduction in which heat is transferred from the specimen into the loading bars. The heat loss through convective processes can be easily avoided by
constantly applying a source of heat to the specimen, that is equal to this loss. The source of heating in the SHPB system used is 5kW induction system, that includes a 240V power supply, a feed-back temperature control system, a copper induction coil to generate a magnetic field that excites electrons within the material, causing heating and a cooling system that cycles water through the copper coil ensuring melting of this coil does not occur. This system provides ample heating that can easily overcome any convective heat loss. The other major source of heat loss, conduction through contact with the loading bars can be alleviated through another manner.

To reduce the amount of heat loss through conduction, which in turn would cause heating of the loading bars, a pneumatic actuator bar positioning system was devised. This system provides precisely timed motion to the loading bars, within a millisecond before striker impacts the loading bars and positioning them in contact with either side of the specimen. Figure A-8 shows a schematic of the piston/cylinder system which is attached to each loading bar.
**Figure A-8:** A schematic representing the pneumatic actuator system used to apply motion to the

**Figure A-9:** The programmable timing relay used to begin the firing sequence, in which the pneumatic actuators and gas gun solenoids are precisely timed. The sequence begins using the ignition switch shown on the right of the image.
Using a high rate solenoid valve, a burst of approximately 40 to 60Psi of Nitrogen is applied to the back of actuator pistons, on the transmitted and incident loading bars. This moves each bar approximately 1 inch into contact with the specimen after specimen has reached the testing temperature.

In order to control the timing of the actuator system with the firing of the gas gun to apply the loading pulse, a programmable Eaton timing relay was used as shown in Figure A-9. This system allows for the excitation voltage to the solenoid valve that controls the actuator system to be timed within a millisecond to the solenoid valve that controls the gas gun. The sequence is started with a toggle switch that applies an input voltage into the timing relay. When the relay recognizes this input voltage from the switch it begins the timing sequence. The systems sends the excitations voltage to the actuator solenoid valve 5 seconds after the input switch voltage, with the timing relay then sending the excitation voltage to the gas gun solenoid valve 3ms after the firing of the actuator solenoid. This allows for time for the loadings bars to move into position, just before the loading pulse is generated by the firing of the gas gun.

In order to protect the bars from the possibility of being damaged, tungsten carbide inserts are utilized. Theses inserts are placed on either side of the specimen and in-between each loading bar. The inserts are mounted using a support system as shown in Figure A-10. The support mount allows for accurate alignment with the loading bars and through the use of bearings, allows the tungsten carbide inserts to move freely. Tungsten carbide was chosen as the material for the inserts due to the mechanical properties remaining relatively constant with temperature, mainly the wave speed, which is directly dependent on the elastic modulus. Extreme care was taken to match the
impedance with the impedance of the loading bars. If there existed a impedance mismatch proper transmission of the loading pulse from the incident bar into the incident tungsten rod would not occur, as a reflection at this interface would result and similar as the loading pulse moved into the transmitted bar. This is done geometrically by decreasing the diameter of the tungsten inserts smaller then the loading bar diameter, as the tungsten has a higher density as shown in equation A-20 and include the inserts to be centerless ground for high precision of the dimension.

\[
\left( \rho c \pi r^2 \right)_{\text{bar}} = \left( \rho c \pi r^2 \right)_{\text{insert}} \quad (A-20)
\]

where \( \rho \) is the density of the material, \( c \) is the wave speed and \( r \) is the radius of the bar.

Figure A-10: Photograph of the high temperature tungsten carbide insert setup, which includes an incident and transmitted insert and support system including bearings. This system is utilized in extreme temperature testing to protect loading bars.
A.2.3 Mechanical Drawing and Specifications

This appendix section contains all drawings generated for the design and manufacture of the SHPB at the MMRL facilities.
Figure A-11: A schematic representing the final assembly of the SHPB system. From right to left this includes the gas gun assembly, the loading bar systems and below is the I-beam used to support the entire system.
Figure A-12: Bar mount used to mount and support the two loading bars to the SHPB frame. This included pressed brass slider bearings to allow for horizontal free motion of the loading bars during testing.
Figure A-13: Actuator mount, similar to the bar mount, in which is serves as a support for the loading bars, but is specifically placed as the center mount of the loading bars and has an additional machining procedure to allow for the application of the pneumatic actuator piston.
Figure A-14: Dash pot mount is used as a support for the momentum stopper placed at the end of the transmitted bar.
**Figure A-15:** A schematic representing the gas gun assembly of the SHPB system, in which major parts include the barrel, barrel mounts, solenoid valve, chamber, chamber insert, chamber end caps and filling attachments
Figure A-16: Barrel mount is used for supporting the gas gun barrel to the SHPB frame, allowing for accurate alignment and aiming of the gas gun to the loading bars.
Figure A-17: Gas gun barrel with precisely honed interior diameter for repeatable firing of the striker
Figure A-18: Gas Gun Chamber which is designed to be capable of being filled to 500Psi safely
Figure A-19: Gas Chamber insert is attached to the from end cap of the gas gun. This funnel allows for the rapid transition of gases from the chamber necked down the barrel diameter.
Figure A-20: Front End cap of the gas gun chamber, this includes several o-ring grooves for proper sealing of the chamber as well as exit hole for air passage during firing. Also 4 - 1" grade 8 bolts are used in conjunction with the front end cap and chamber to complete the gas gun assembly
Figure A-21: Back end cap of the gas chamber, this includes an o-ring groove to allow for proper sealing of the gas chamber. Also 4 - 1" grade 8 bolts are used in conjunction with the front end cap and chamber to complete the gas gun assembly.
Figure A-22: Schematic representing the high temperature specimen holder. This system is used to isolate the high temperature of the specimen from the loading bars. This is done by using two tungsten rods mounted in this apparatus, with the specimen being placed in between the two tungsten rods.
Figure A-23: Tungsten High Temperature inserts used to hold the specimen and create a temperature barrier between the specimen and loading bars.
Figure A-24: Base of the high temperature inset holder, used to precisely align the tungsten rods with the loading bars
**Figure A-25:** High Temperature Insert Base Feet which are attached to the base to allow for a sturdy, movement free system when clamped to the I-beam of the SHPB system
Figure A-26: High temperature insert bearing clamp used to fix the bearings in which the tungsten rods move horizontally within
Figure A-27: High temperature insert bearing clamp used to fix the bearings in which the tungsten rods move horizontally within
Figure A-28: A schematic representing the electrical wiring and Nitrogen gas lines of the firing system utilized in the SHPB system
A.2.4 Testing, Data Processing and Software

This appendix section contains the description of the testing procedure for dynamic testing using the split-Hopkinson pressure bar system. This include specimen preparation, testing procedure and data processing. Data processing is completed using a series of Matlab codes, taking the raw data collected from the high speed oscilloscope and ending with the final true stress-strain curve for the given test.

A.2.4.1 Experimental Procedure

The procedure utilized during a experimental SHPB setup and testing is presented in the following section. The importance of following this procedure is to ensure repeatability and accuracy of each test. The specimen preparation, experimental and equipment setup, and data capture will be discussed in detail.

Proper specimen preparation will ensure that each test will perform in a repeatable manner, that can be successfully used for material characterization. With-in this study two classes of alloys were used, low carbon steel and aluminum alloy, both of which a prepared in the same manner. The major concerns in generation specimens for testing in a SHPB compression system are the accurate dimensioning of specimens for proper wave interactions (length to diameter ratio) as well as surface finish on loading surface of the specimen, to reduce any frictional effects, to ensure 1D loading condition. Specimens should be machined in batches to ensure the same procedure and conditions were the same to generate them. Specimens in this case where created using raw stock, where was turned cylindrically using a machining lathe, to a diameter of 0.475" ±0.001". It is important to use lubrication not only to keep the specimen cool during machining but also
to achieve a defect free surface. Once the diameter is within the given tolerance, the specimens were rough parted using the lathe and then faced within the lathe, with great attention placed on the final surface finish, ensuring the highest reduction in frictional effects as possible. The length of the specimen was 0.3875" ±0.001". The tolerances are held at a high level to ensure repeatability between tests. Finally a high-grit (1200) sanding was completed to further smooth the specimen loading surface. Pulse shapers used during testing where generated the same manner.

The alignment of the entire system is of great importance for the accuracy of testing with the alignment being re-checked on a regular basis. To align the system all the mounting blocks of the loading bars should have bolts slightly loose to allow for precise-small increment movements. Starting at the far end of the system, end of transmitted bar, the mound blocks should be incrementally tightened, repeatedly ensuring proper motion of the transmitted bars through the bearings. Once this position has been determined, a final tightening of the bolts should be done. Now moving to the incident bar, the loading surfaces, where the specimen is placed during testing, should be ensured to match up diametrically. This can be done by using a calipers, ensuring that the transition from the transmitted to incident bar on the outer surface is continuous, this ensures that the bars share the same axis. A similar procedure should be completed to the incident bar's mounting blocks as the transmitted bar, always checking proper motion of the bar and that the loading surfaces are aligned properly. Finally the gas gun needs to be aligned properly with the loading bar axis. This is done in a similar manner as the specimen loading surface alignment, in which a striker is used to match the continuity of the axis. All mounting blocks of the gas gun should be tightened fully. This procedure is complete
for room temperature testing where the inclusion of high temperature tungsten rods are not included. To include these for elevated temperature testing, a similar procedure should be included for this additional apparatus, but the alignment of the transmitted and incident bars should not be adjusted during.

To run a test power must be supplied to the oscilloscope, strain gauge conditioning amplifier and programmable timing relay. It is important to allow the strain gauge conditioning amplifier ample time to come to a temperature equilibrium for accurate strain gauge amplification. Once temperature equilibrium is reached, approximately 20 minutes, the gauges should be balanced and settings of this system checked. The important settings to check before testing include the filter and gain settings, as these directly affect the data collected. All other settings should be referred to system manual. The oscilloscope should now be set for data collection. Use of the oscilloscope should be referred to the system manual, but major considerations include channel selections, trigger setting, voltage scale, time scale and collection offset. The trigger should be set to a 1.0V level, with downward sloped pulse with a duration longer then 50μs. The voltage scales for each channel should be set to 1.0V/division scale, with the time scale being 200μs/division. The system should have two channels activated, the first being the incident/reflect strain gauge and the second being the transmitted strain gauge. A collection offset of 10% is utilized, in which when triggered, 10% of the total data will include data before the event. This data is used later in data processing sequence. The data capturing system should be complete and armed by setting "single" on the oscilloscope.
The specimen should be lubricated using graphite, high pressure type lubrication. This aids in the reduction of frictional effects. The lubricated specimen should be centered between the two loading bars or in the high temperature condition, in between the two tungsten rods. This can be done by using the specimen centering tool presented in previous section. This precisely places the specimen directly in the middle of the loading bars, important for uniaxial loading. The lubrication should aid in holding the specimen in place when lightly squeezed between the loading bars. For high temperature testing, a thermocouple needs to welded to the end of the incident tungsten high temperature rod, closet to the specimen loading surface. This thermocouple is used to control the induction heating system.

For the remainder of the procedure for purpose of brevity, the test will be assumed to be at an elevated temperature, as this is the most complex set of testing and therefore includes all necessary steps for a room temperature test as well. Room temperature testing does not include the induction system and the high temperature tungsten rod setup. A pulse shaper should now be placed at the incident bar striker interface, using the same lubricant that is used for the specimen. The striker should be pushed down the length of the barrel into its initial firing position. The incident and transmitted bars should be placed into their initial positions before they are to be mobilized using the pneumatic bar positioning system. Their position should be based on a 0.5" gap between each bar and their adjacent tungsten rods. The regulator for the pneumatic bar positioning system should be set to 40-60Psi. The gas gun chamber can now be filled to the desired pressure determined to provide a desired striker velocity and therefore desired strain rate. Once the chamber has been filled, the heating of the
specimen can begin. This can be done using the induction system which includes the power supply, heating coil apparatus, coil cooling system and the temperature controller. The temperature controller is set to the desired temperature. Once the specimen has reached its desired temperature, the testing can begin. To reduce noise in the data collection system, it is important to turn off the induction power supply just before firing of the gas gun. Once the induction system is shut off, the user should quickly begin the firing sequence by using the timing relay switch. The system should first move the bars into position just before the gas gun is fired. Data will be automatically collected using the triggering system of the oscilloscope. Data should be saved via a flash drive where it can then be transferred to the computer for data analysis.

A.2.4.2 Data Analysis

By utilizing the fundamental equations of the SHPB testing equipment, a series of Matlab codes were developed to convert the raw experimental collected during each test, and generate a stress-strain curve from the experiment. The raw data is saved via a flash-drive from the digital phosphorus oscilloscope used to capture the data during the experiment. This raw data, saved in a .csv text file is run through the first Matlab program, Read_Data_SHPB.m. The programs written consist of three separate programs, the first program, Read_Data_SHPB.m, reads the raw data, a .csv file, line by line and converts the voltage signal to strain data using equation A-19 where input variables include the gain, gauge factor and excitation voltages. The raw data includes two separate .csv files, one for each strain gauge, in which one includes the incident and reflected pulse and the second contains the transmitted voltage data, and both contain a time string with its respected voltage value. The program then outputs three separate text files, one
for each pulse, incident, transmitted and reflected. Each outputted text file is labeled accordingly and contains three columns that include time, voltage and strain.

\textit{Read\_Data\_SHPB.m}

% Program Read\_SHPB\_Data.m
% By Justin Spirdione
% Description: This program is to read a .csv file line by line and
% convert the voltage signal to strain. It will output 3 separate text
% files one for the incident pulse, reflected pulse and transmitted
% pulse. Each text file contains 3 columns, time, voltage and strain.
% 
% Clear Command Window
% to give you a clean screen
cle;
% Clear Workspace
% to clear any previously stored variables
clear;

Start = datestr(now) % print date and time
LOG = fopen('log.txt','w+'); % name of log file, w+ opens or creates file
% for reading and writing and discard existing contents, if any
fprintf (LOG, \n\n','********** Log for Read\_Data\_SHPB.m **********

fprintf (LOG, \n\n','Start Time is: 

fclose(LOG);

FigureCount = 0; % Initialize Figure Count
Font\_Size = 20; % Set Font Size

Filename1 = 'tek0002CH1'; % name of .csv file for channel 1
Filename2 = 'tek0003CH2'; % name of .csv file for channel 2
% Note: Oscilloscope is set up to record at a rate of 50M points/sec (this is equal to 50 points/microsec), a buffer size of 6500 points will store 130 microseconds of data. The buffer size needs to be sufficiently long enough to capture the length of the pulse.

\[
\text{buffer} = 40000; \quad \text{number of points in buffer}
\]

\[
\text{points} = 1000; \quad \text{number of points to store before trigger}
\]

\[
\text{trigger\_inc} = -250\times10^{-6}; \quad \text{(sec) time trigger to store data}
\]

\[
\text{trigger\_ref} = 200\times10^{-6}; \quad \text{(sec) time trigger to store data}
\]

\[
\text{trigger\_trans} = 200\times10^{-6}; \quad \text{(sec) time trigger to store data}
\]

\[
\text{Nheader} = 18; \quad \text{number of header lines}
\]

% Input Channel Gains, Gage Factor and Excitation Voltage

\[
\text{Gain\_Ch1} = 450; \quad \text{(unitless) gain for channel 1}
\]

\[
\text{Gain\_Ch2} = 500; \quad \text{(unitless) gain for channel 2}
\]

\[
\text{Vex\_1} = 9.94; \quad \text{(V) excitation voltage ch 1}
\]

\[
\text{Vex\_2} = 9.95; \quad \text{(V) excitation voltage ch 2}
\]

\[
\text{GF} = 3.3; \quad \text{(unitless) gage factor}
\]

% Read initial data prior to pulse to calculate the shift

\[
\text{for Channel} = 1:2
\]

\[
\text{clear temp tline k cc time V flag bcount Gain strain}
\]

\[
\text{clear Name OutputFile Filename}
\]

\[
\text{if Channel == 1} \quad \text{Incident and Reflected Pulse}
\]

\[
\text{Filename = Filename1; \ name of .csv file for channel 1}
\]

\[
\text{Name = 'Channel 1'; \ Channel Name}
\]

\[
\text{FigName = 'Ch1'; \ Channel Name}
\]

\[
\text{Gain = Gain\_Ch1; \ (unitless) gain for channel 1}
\]

\[
\text{Vex}=\text{Vex\_1}; \quad \text{(V) excitation voltage ch 1}
\]

\[
\text{else} \quad \text{Transmitted Pulse}
\]

\[
\text{Filename = Filename2; \ name of .csv file for channel 2}
\]

\[
\text{Name = 'Channel 2'; \ Channel Name}
\]

\[
\text{FigName = 'Ch2'; \ Channel Name}
\]

\[
\text{Gain = Gain\_Ch2; \ (unitless) gain for channel 2}
\]

\[
\text{Vex}=\text{Vex\_2}; \quad \text{(V) excitation voltage ch 2}
\]

\[
\text{end}
\]

\[
\text{temp = fopen(strcat(Filename, '.csv')); \ open file}
\]
for q = 1:Nheader % in header line
tline = fgets(temp); % read lines 1 to Nheader
end
tline = fgets(temp); % read the next line (first line of data)
k = 1; % start count
cc = str2num(tline); % convert to number
time(k) = cc(1); % (sec) time
V(k) = cc(2); % (V) voltage
flag = 1;
while ischar(tline) % returns true, 1, if the line is a character
    % returns false, 0, if the line is NOT a character, i.e. the end of
    % the file
    tline = fgets(temp); % read the next line
    if tline ~= 0 % if it is not the end of the file
        k = k + 1; % update count
        cc = str2num(tline); % convert to number
        if k <= buffer % fill buffer with initial size
            time(k) = cc(1); % (sec) time
            V(k) = cc(2); % (V) voltage
        else % k > buffer
            flag = 3;
        end
    else
        end
    end
if flag == 3
    break % exit loop (you don't need to read the rest of the file)
end
fclose(temp); % close file

% Convert the voltage signal to strain using equation for 1/4 bridge
for g = 1:buffer % 1 to total number of data points (1 to buffer size)
    strain(g) = (4*V(g))/(Gain*GF*Vex); % (unitless) strain
end
% Calculate average voltage and strain signal
sum_V = 0; % (V) voltage
sum_strain = 0; % (unitless) strain
for g = 1:buffer
    sum_V = sum_V+V(g); % (V) voltage
    sum_strain = sum_strain+strain(g); % (unitless) strain
end
avg_V = sum_V/buffer; % (V) average voltage
avg_strain = sum_strain/buffer; % (unitless) average strain

FigureCount = FigureCount + 1;
figure(FigureCount)
subplot(2,2,1)
plot(time*10^6,V,'-k',...)
    [time(1)*10^6;time(end)*10^6],[avg_V;avg_V],'-ro',...  
    'LineWidth',2)
xlabel('time (\musec)','FontSize',Font_Size)
ylabel('Voltage (V)','FontSize',Font_Size)
title(strcat(Name,' Signal'),'FontSize',Font_Size)
set(gca,'FontSize',Font_Size-4)
gtext on
subplot(2,2,2)
plot(time*10^6,strain*100,'-k',...)
    [time(1)*10^6;time(end)*10^6],...
    [avg_strain*100;avg_strain*100],'-ro',...
    'LineWidth',2)
xlabel('time (\musec)','FontSize',Font_Size)
ylabel('Strain (%)','FontSize',Font_Size)
title(strcat(Name,' Signal'),'FontSize',Font_Size)
set(gca,'FontSize',Font_Size-4)
gtext on
% % SHIFT CHANNEL'S SO VOLTAGE AND STRAIN ARE ZERO INITIALLY
V       = V - avg_V;    % (V) shifted voltage
strain  = strain - avg_strain;  % (unitless) shifted strain
%
% Still Same Figure
subplot(2,2,3)
plot(time*10^6,V,'-k','LineWidth',2)
xlabel('time (\musec)','FontSize',Font_Size)
ylabel('Voltage (V)','FontSize',Font_Size)
title({strcat(Name,' shift is:'),...
    strcat(num2str(avg_V),' volts')},...  
    'FontSize',Font_Size)
set(gca,'FontSize',Font_Size-4)
gtext on
subplot(2,2,4)
plot(time*10^6,strain*100,'-k','LineWidth',2)
xlabel('time (\musec)','FontSize',Font_Size)
ylabel('Strain (%)','FontSize',Font_Size)
title({strcat(Name,' shift is:'),...
    strcat(num2str(avg_strain*100),'% strain')},...  
    'FontSize',Font_Size)
set(gca,'FontSize',Font_Size-4)
gtext on
%
saveas(gcf,strcat(FigName,'_Shift.fig')) % Save Figure
%
189
% Save shift values and write to screen
if Channel == 1
    disp('The shift values for Channel 1 (volts and strain) are :')
    Shift_Ch1_V = avg_V % (V) average voltage shift
    Shift_Ch1_strain = avg_strain % (unitless) average strain shift
else % Channel == 2
    disp('The shift values for Channel 2 (volts and strain) are :')
    Shift_Ch2_V = avg_V % (V) average voltage shift
    Shift_Ch2_strain = avg_strain % (unitless) average strain shift
end
LOG = fopen('log.txt','a+'); % name of log file, a+ opens or creates file
% for reading and writing and appends data to the end of the file
fprintf (LOG, '%s
',strcat(Name,' shift is:',num2str(avg_V),' volts'));
fprintf (LOG, '%s
',strcat(Name,' shift is:',num2str(avg_strain*100),'% strain'));
fclose(LOG);
end
%
% Read Data and Write to Separate .txt Files
for Signal = 1:3
    clear temp tline k cc time V flag bcount Gain strain
    clear Name OutputFile Filename
    if Signal == 1 % Incident Pulse, Channel 1
        trigger = trigger_inc; % time trigger to store data
        Filename = Filename1; % name of .csv file for channel 1
        Name = 'Incident'; % Pulse Name
        Gain = Gain_Ch1; % (unitless) gain for channel 1
        Shift_V = Shift_Ch1_V; % (V) average voltage shift in channel 1
        Shift_strain = Shift_Ch1_strain; % (unitless) average strain shift in channel 1
        Vex = Vex_1; % (V) excitation voltage ch 1
    elseif Signal == 2 % Reflected Pulse, Channel 1
        trigger = trigger_ref; % time trigger to store data
        Filename = Filename1; % name of .csv file for channel 1
        Name = 'Reflected'; % Pulse Name
        Gain = Gain_Ch1; % (unitless) gain for channel 1
        Shift_V = Shift_Ch1_V; % (V) average voltage shift in channel 1
        Shift_strain = Shift_Ch1_strain; % (unitless) average strain shift in channel 1
        Vex = Vex_1; % (V) excitation voltage ch 1
    else % Signal == 3 % Transmitted Pulse, Channel 2
        trigger = trigger_trans; % time trigger to store data
        Filename = Filename2; % name of .csv file for channel 2
        Name = 'Transmitted'; % Pulse Name
        Gain = Gain_Ch2; % (unitless) gain for channel 2
        Shift_V = Shift_Ch2_V; % (V) average voltage shift in channel 2
        Shift_strain = Shift_Ch2_strain; % (unitless) average strain shift in channel 2
        Vex = Vex_2; % (V) excitation voltage ch 2
temp = fopen(strcat(Filename, '.csv')); % open file
for q = 1:Nheader % in header line
tline = fgets(temp); % read lines 1 to Nheader
end
tline = fgets(temp); % read the next line (first line of data)
k = 1; % start count
cc = str2num(tline); % convert to number
time(k) = cc(1); % (sec) time
V(k) = cc(2); % (V) voltage
flag = 1;
while ischar(tline) % returns true, 1, if the line is a character
    % returns false, 0, if the line is NOT a character, i.e. the end of % the file
tline = fgets(temp); % read the next line
if tline ~= 0 % if it is not the end of the file
    k = k + 1; % update count
    cc = str2num(tline); % convert to number
    if k <= buffer % fill buffer with initial size
        time(k) = cc(1); % (sec) time
        V(k) = cc(2); % (V) voltage
    else % k > buffer
        cc = str2num(tline); % convert to number
        % update buffer
        for i = 1:(buffer-1)
            time(i) = time(i+1); % (sec) time
            V(i) = V(i+1); % (V) voltage
        end
        time(buffer) = cc(1); % (sec) time
        V(buffer) = cc(2); % (V) voltage
    end
if cc(1) > trigger && flag == 1;
    Triggered = datestr(now) % print date and time
    %
    LOG = fopen('log.txt','a+'); % name of log file, a+ opens or creates file
    % for reading and writing and appends data to the end of % the file
    fprintf (LOG, '%s

',strcat('Triggered ',Name,' Pulse at: ', Start));
    fclose(LOG);
elseif flag == 2;
    bcount = bcount + 1;
    if bcount == (buffer-points) % store variables
flag = 3;
else
if flag == 3
  break % exit loop (you don't need to read the rest of the file)
end
end
fclose(temp); % close file

% Convert the voltage signal to strain using equation for 1/4 bridge
for g = 1:buffer % 1 to total number of data points (1 to buffer size)
  strain(g) = (4*V(g))/(Gain*GF*Vex); % (unitless) strain
end

% SHIFT CHANNEL'S SO VOLTAGE AND STRAIN ARE ZERO INITIALLY
V = V - Shift_V; % (V) shifted voltage
strain = strain - Shift_strain; % (unitless) shifted strain

OutputFile = strcat(Name, '_t_V_strain.txt'); % name of .csv file
temp = fopen(OutputFile, 'wt'); %write text
for g = 1:buffer % Number of data points
  fprintf (temp, '%-20.10f \t %-20.10f \t %-20.10f \n',
    time(g), V(g), strain(g)); % column 1 is time (sec)
end
fclose(temp);

FigureCount = FigureCount + 1;
figure(FigureCount)
subplot(1,2,1)
plot(time*10^6,V,'-k','LineWidth',2)
xlabel('time (\musec)','FontSize',Font_Size)
ylabel('Voltage (V)','FontSize',Font_Size)
title(strcat(Name, ' Pulse'),'FontSize',Font_Size)
set(gca,'FontSize',Font_Size-4)
grid on
subplot(1,2,2)
plot(time*10^6,strain*100,'-k','LineWidth',2)
xlabel('time (\musec)','FontSize',Font_Size)
ylabel('Strain (%)','FontSize',Font_Size)
title(strcat(Name, ' Pulse'),'FontSize',Font_Size)
set(gca,'FontSize',Font_Size-4)
grid on
```matlab
saveas(gcf, strcat('Original_', Name, '_Pulse.fig')) % Save Figure
End = datestr(now) % print date and time
LOG = fopen('log.txt', 'a+'); % name of log file, a+ opens or creates file
% for reading and writing and appends data to the end of the file
fprintf(LOG, '%s

', strcat('End Time is: ', End));
fprintf(LOG, '%s

', '********** End of Read_Data_SHPB.m **********');
fclose(LOG);
% End of Read_Data_SHPB.m
%
```

The second program, `Clean_strain_pulses.m`, is adapted from another program previously developed at the laboratory facilities, in which the program reads, line by line, each of the three text files outputted by the previous program. This program takes the strain data from those three text files and smoothes and blocks the data to remove the noise inherent within the system. The smoothing process takes place first and is essentially a running average procedure, which takes the running average of 7 points and replaces the points with the average of the 7 points. The block process takes the now smoothed data and reduces the number of data points to 1 point generated from the average of a block of data points. The program then outputs three text files labeled accordingly to be used by the third program, one for each pulse.

`Clean_strain_pulses.m`

% This Program smooths, blocks and smooths again the strain pulses from the
% SHPB
% By: Justin Spirdione
% Created on July 29, 2007
% This Program is based on file SMOOTH.BAS by Dian Zheng that smooths
% the data points based on a seven point moving average method
% %
% Created on August 7, 2007
% This Program is based on file block2.m by Dian Zheng that takes smoothed
% data and reduces the number of data points
1. Clear Command Window
to give you a clean screen

2. Clear Workspace
to clear any previously stored variables

3. Start = datestr(now) % print date and time

4. LOG = fopen('log.txt','a+'); % name of log file, w+ opens or creates file

5. fprintf (LOG, '\n%s
\n', strcat('********** Log for Clean_strain_pulses.m **********'));

6. fprintf (LOG, '%s

', strcat('Start Time is: ', Start));

7. fclose(LOG);

8. FigureCount = 0; % Initialize Figure Count

9. Font_Size = 20; % Set Font Size

10. for Signal = 1:3

11. \indent clear Atemp a N Y temp Name

12. if Signal == 1 % Incident Pulse

13. \indent Name = 'Incident'; % Pulse Name

14. elseif Signal == 2 % Reflected Pulse

15. \indent Name = 'Reflected'; % Pulse Name

16. else % Signal == 3 % Transmitted Pulse

17. \indent Name = 'Transmitted'; % Pulse Name

18. end

19. % Call N and a from experimental data file

20. Atemp = load(strcat(Name, '_t_V_strain.txt'));

21. % create column vector N and column vector a

22. N = Atemp(:,1); % (sec) time

23. a = Atemp(:,3); % (unitless) strain

24. P = size(N); % Total Number of Data points

25. M = P(1,1) - 3;

26. % Calculate Y a column vector of a, a 7 point average

27. J = 4; % Initialize J
while J <= M
    Y(J) = (a(J-3) + a(J-2) + a(J-1) + a(J) + a(J+1) + a(J+2) + a(J+3))/7;
    J = J + 1;
end

% Calculate Y 1, 2, 3, P, P-1, P-2 (the numbers that we had to skip before)
Y(1) = (13*a(1) + 10*a(2) + 7*a(3) + 4*a(4) + a(5) - 2*a(6) - 5*a(7))/28;
Y(2) = (5*a(1) + 4*a(2) + 3*a(3) + 2*a(4) + a(5) - a(7))/14;
Y(3) = (7*a(1) + 6*a(2) + 5*a(3) + 4*a(4) + 3*a(5) + 2*a(6) + a(7))/28;
B_1 = (a(P-6) + 2*a(P-5) + 3*a(P-4) + 4*a(P-3) + 5*a(P-2));
Y(P-2) = (B_1 + 6*a(P-1) + 7*a(P))/28;
B_2 = (-a(P-6) - 2*a(P-5) + a(P-4) + 4*a(P-3) + 5*a(P-2));
Y(P-1) = (B_2 + 5*a(P))/14;
B_3 = (-5*a(P-6) - 2*a(P-5) + a(P-4) + 4*a(P-3) + 7*a(P-2));
Y(P) = (B_3 + 10*a(P-1) + 13*a(P))/28;

figure(FigureCount) plot(N*10^6,a*100,'-r',N*10^6,Y*100,'-ok','LineWidth',2)
xlabel('time (musec)','FontSize',Font_Size)
ylabel('Strain (%)','FontSize',Font_Size)
legend('original','smoothed')
title(strcat(Name,'Pulse'),'FontSize',Font_Size)
set(gca,'FontSize',Font_Size-4)
grid on
saveas(gcf,strcat(Name,'_Pulse_s.fig')) % Save Figure

% print file with N vs. Y(a, average of 7 points)
temp = fopen(strcat(Name,'_t_strain_smooth.txt'),'wt'); %write text
for i = 1:P %Number of data points
    fprintf(temp,'%-20.10f %-20.10f
', N(i), Y(i));
end
fclose(temp);

XX = datestr(now); % print date and time
LOG = fopen('log.txt','a+'); % name of log file, w+ opens or creates file
% for reading and writing and appends data to the end of the file
fprintf(LOG,'%s
',strcat('Completed Smoothing Original Data at: ', XX));
fclose(LOG);

% Block the Smoothed-Original-Data

195
% for Signal = 1:3
    clear Atemp A N temp Name
    clear b2 d1 s1 s2 s3 p1 i3 i v1 bb jj N1 A1 NR AR
    if Signal == 1 % Incident Pulse
        Name = 'Incident'; % Pulse Name
    elseif Signal == 2 % Reflected Pulse
        Name = 'Reflected'; % Pulse Name
    else % Signal == 3 % Transmitted Pulse
        Name = 'Transmitted'; % Pulse Name
    end
    % Call N and a from smoothed data file
    Atemp = load(strcat(Name, '_t_strain_smooth.txt'));
    % create column vector N and column vector a
    N = Atemp(:,1); % (sec) time
    A = Atemp(:,2); % (unitless) strain
    %
    P = size(N); % Total Number of Data points
    P = P(1,1);
    %
    % Define Block
    % Example: 1 2 3 4 5 6 7 8 9
    % #1 Block size 4 will take points 1 through 4
    % #2 Increment 2 will advance to 3 and take points 3 through 6
    % #3 will be points 5 through 8, etc
    b2 = 20; % 6The smallest block size (default=4)
    d1 = 0.005; % 4The increment of each block (default=2)
    %
    s1 = (1-2.*b2/d1);
    s2 = s1^2.;
    s3 = 8.*P/d1;
    p1 = (s1+sqrt(s2+s3))/2.;
    i3 = fix(p1); % fix Rounds the number to the integer part, 1.3 to 1, 1.9 to 1
    % Initialize Numbers for Counting
    i = 0; % initialize i
    v1 = 0; % initialize v1
    bb = 0; % initialize bb
    jj = 1; % initialize jj (count for number of reduced data points)
    %
    % Calculate Reduced N and a
    while bb <= P
        if i3 >= 0
            b1 = fix(b2+(i3-1)*d1);
            for k = 1:b1
                v1 = v1 + A(bb+k);
if k == b1
    v1 = v1/b1;
end
if (bb+k) > P
    v1 = v1/k;
    exit;
end
N1(jj) = ((N(bb+1)+N(bb+b1))/2); % Reduced Cycle Number N
A1(jj) = v1; % Reduced Crack Length a
jj = jj+1; % Step jj (count for number of reduced data points)
else
    break
end
v1 = 0;
i3 = i3 - 1;
bb = bb + b1;
end
P_reduced = jj - 1; % Number of points (without initial and final)
% Add in N=0 and a0, and N=final and afinal
NR(1) = N(1); % Initial Cycle
NR(P_reduced + 2) = N(P); % Final Cycle
AR(1) = A(1); % Initial Crack Length a0
AR(P_reduced + 2) = A(P); % Final Crack Length af
for k = 1:(P_reduced)
    NR(k+1) = N1(k);
    AR(k+1) = A1(k);
    k = k + 1; % Step k
end

FigureCount = FigureCount + 1;
figure(FigureCount)
plot(N*10^6,A*100,'-xr',NR*10^6,AR*100,'-ok','LineWidth',2)
xlabel('time (musec)','FontSize',Font_Size)
ylabel('Strain (%)','FontSize',Font_Size)
legend('original','blocked')
title(strcat(Name,' Pulse'),'FontSize',Font_Size)
set(gca,'FontSize',Font_Size-4)
grid on

saveas(gcf,strcat(Name,'_Pulse_sb.fig')) % Save Figure
%
% print file with N vs. a (reduced number of points)
temp = fopen(strcat(Name, '_t_strain_smooth_block.txt'), 'wt'); %write text
for i = 1:(P_reduced+2) %Number of data points
    fprintf (temp, '%-20.10f \t %-20.10f \n', NR(i), AR(i));
end
% time tab strain new line
end
fclose(temp);
end
%
XX = datestr(now); % print date and time
LOG = fopen('log.txt','a+'); % name of log file, w+ opens or creates file
% for reading and writing and appends data to the end of the file
fprintf (LOG, '%s

',strcat('Completed Blocking the Smoothed Data at: ', XX));
fclose(LOG);
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
End = datestr(now) % print date and time
LOG = fopen('log.txt','a+'); % name of log file, a+ opens or creates file
% for reading and writing and appends data to the end of the file
fprintf (LOG, '%s

',strcat('End Time is: ', End));
fprintf (LOG, '%s

',strcat('********** End of Clean_strain_pulses.m **********'));
close(LOG);
%
% End of Clean_strain_pulses.m

The final program, Calculate_Specimen_Stress_Strain_SHPB.m, aligns the data of the incident, reflected and transmitted strain pulses by using a procedure that notes when the pulse begins by noting when the strain level deviates from zero and then aligns these points of three pulses. Then the program calculates the stress, strain and strain rate for the given test using equations A-32-A-36. This program also outputs a series of figures to ensure alignment of the pulses, important for receiving the proper stress-strain curve, the stress-strain curve itself as well as strain versus time, representing the strain history of the specimen during loading. The program has a series of user inputs for the loading bars and specimen needed to complete the calculations. These include the length and diameters of both and the elastic modulus, density and wave speed of the loading bars. Furthermore, the program compares the data using the smooth-blocked data created in the previous program, with the results of the original non-smooth and blocked data,
this is to ensure that none of the major characteristics of the stress-strain curve are lost during the smooth-blocking procedure.

*Calculate_Specimen_Stress_Strain_SHPB.m*

% By: Justin Spirdione
% Description: This program is to align the incident, reflected and transmitted strain pulses and calculate the stress, strain and strain rate in the specimen. It will output the aligned pulses as a function of time and the specimen stress-strain data.
% 
% NOTE: This code is written assuming your incident pulse is negative, your reflected pulse is positive and your transmitted pulse is negative.
% 
% Clear Command Window
% to give you a clean screen
cle;
% Clear Workspace
% to clear any previously stored variables
clear;
%
Start = datestr(now) % print date and time
LOG = fopen('log.txt','a+'); % name of log file, w+ opens or creates file
% for reading and writing and appends data to the end of the file
fprintf(LOG, '

********** Log for Calculate_Specimen_Stress_Strain_SHPB.m **********

Start Time is: ', Start));
fclose(LOG);

FigureCount = 0; % Initialize Figure Count
Font_Size = 20; % Set Font Size
%
% Input Name of Files to Analyze
Filename = '_t_strain_smooth_block_2.txt';
cc = 2; % column with strain
%
% Input Constants -----------------------------------------------
%
% Incident and Transmitted Bar Dimensions and Material Properties
d_bar = 1.89992E-2; % (m) diameter of the bars
L_bar = 1.524; % (m) length of the bars
rho_bar = 8065;  
E_bar = 1.94409E11;  
C_bar = 5767;  
L_st = 8*0.0254;  

d_sp = 0.3875*0.0254;  
L_sp = 0.170*0.0254;  
%

r_bar = d_bar/2;  
A_bar = pi*r_bar^2;  
r_sp = d_sp/2;  
A_sp = pi*r_sp^2;  
%

% Load text files

% Call time and strain from incident pulse

time_inc = temp(:,1);  
strain_inc = temp(:,cc);  
clear temp

% Call time and strain from reflected pulse

time_ref = temp(:,1);  
strain_ref = temp(:,cc);  
clear temp

% Call time and strain from transmitted pulse

time_trans = temp(:,1);  
strain_trans = temp(:,cc);  
clear temp

P = size(time_inc);  
P = P(1,1);

orig_shift_inc = time_inc(1);  
orig_shift_ref = time_ref(1);  
orig_shift_trans = time_trans(1);

time_inc = time_inc - orig_shift_inc;  
time_ref = time_ref - orig_shift_ref;  
time_trans = time_trans - orig_shift_trans;
% Determination of Alignment criteria-using the Standard deviation of the % smoothed data before the pulse begins as criteria for which the pulse % begins

% time_buffer = 5/10^6; % (sec) time buffer that the strain is allowed to deviate from the STDEV
dt = time_inc(end)/P; % (sec/point) time between data points
points_buffer = round(time_buffer/dt); % Number of points in buffer
era_points = round((1/10^6)/dt); % Number of points at start of pulse (for 1 microsecond)
Nendpnt = 10; % extra_points*Nendpnt Number of points at end of pulse
for Signal=1:3
    if Signal == 1 % Incident Pulse
        Name = 'Incident'; % Pulse Name
    elseif Signal == 2 % Reflected Pulse
        Name = 'Reflected'; % Pulse Name
    else % Signal == 3 % Transmitted Pulse
        Name = 'Transmitted'; % Pulse Name
    end

    % Call N and a from smoothed data file
    Atemp = load(strcat(Name, '_t_strain_smooth_2.txt')); % create column vector N and column vector a
    N = Atemp(:,1); % (sec) time
    A = Atemp(:,2); % (unitless) strain
    B = size(N); % Total Number of Data points
    B = B(1,1);
dt_B=N(end)/B; % (sec/point) time between data points
    points_buffer_B = round(time_buffer/dt_B); % Number of points in buffer
    for i=1:points_buffer_B
        A1(i)=A(i);
    end
    if Signal == 1 % Incident Pulse
        STDEV_inc=max(abs(A1))*1.5; %standard deviation of the incident pulse
    elseif Signal == 2 % Reflected Pulse
        STDEV_ref=max(abs(A1))*1.6; %standard deviation of the reflected pulse
    else % Signal == 3 % Transmitted Pulse
        STDEV_trans=max(abs(A1))*1.5; %standard deviation of the transmitted pulse
    end
end

% Align Incident pulse ----------------------------------------
% Note: this is written for a negative incident pulse

% k = 0; % initialize buffer
flag = 1; % at start
for i = 1:P
    if strain_inc(i) < -STDEV_inc && flag == 1  % when pulse becomes lower than STDEV
        k = k + 1;  % count number of points in buffer
    else  % -abs(strain_inc(i)) > 0
        k = 0;  % reset counter
    end
    if k == points_buffer && flag == 1
        inc_start = i - points_buffer;  % increment number at start of pulse
        flag = 2;  % inside pulse
    else
    end
    if strain_inc(i) >= 0 && flag == 2
        inc_end = i + extra_points*Nendpnt;  % increment number at end of pulse
        flag = 3;  % end of pulse
    else
    end
    if flag == 3
        break  % exit loop (you don't need to read the rest of the file)
    end
end
% Calculate slope of line in beginning of incident pulse
linefit = polyfit(time_inc(inc_start:inc_start+points_buffer),...
    strain_inc(inc_start:inc_start+points_buffer),1);
m = linefit(1,1);  % slope of curve (strain rate)
b = linefit(1,2);  % intercept
time_inc_start=-b/m-time_buffer*2;  % instance in which fit line to pulse intercepts x-axis (y=0)
% TimeStart_Inc = time_inc_start*10^6 + orig_shift_inc*10^6;  % (microsec) Time that Incident Pulse Starts
% flag = 1;  % at start
count = 1;  % initialize count
for i = 1:P
    if time_inc(i)>= time_inc_start && flag == 1  % start storing data
        shift_time_inc(count) = time_inc(i);  % (sec) time
        shift_strain_inc(count) = strain_inc(i);  % (unitless) strain
        flag = 2;  % inside pulse
        count = count + 1;  % update count
        inc_start=i;
    elseif i == inc_end && flag == 2  % store last point and break
        shift_time_inc(count) = time_inc(i);  % (sec) time
        shift_strain_inc(count) = strain_inc(i);  % (unitless) strain
        flag = 3;  % end of pulse
    elseif i < inc_end &&& time_inc(i)> time_inc_start
shift_time_inc(count) = time_inc(i);  % (sec) time
shift_strain_inc(count) = strain_inc(i);  % (unitless) strain
count = count + 1;  % update count
end
if flag == 3
    break  % exit loop (you don't need to read the rest of the file)
end
end
N_pulse=size(shift_time_inc);
N_pulse=N_pulse(1,2);

shift_time_inc  = shift_time_inc - shift_time_inc(1);  % (unitless) shifted time (to start at zero)
%
FigureCount = FigureCount + 1;
figure(FigureCount)
subplot(2,2,1)
plot(time_inc*10^6,strain_inc*100,'-k',...  
    time_inc(inc_start:inc_end)*10^6,...  
    strain_inc(inc_start:inc_end)*100,'-r',...  
    'LineWidth',2)
xlabel('time (\musec)','FontSize',Font_Size)
ylabel('Strain (%)','FontSize',Font_Size)
title(strcat('Incident Strain Pulse Started at:',...  
    num2str(round(TimeStart_Inc)),' \musec'),...
    'FontSize',Font_Size)
set(gca,'FontSize',Font_Size-4)
grid on
%
% Align transmitted pulse  -----------------------------------------------%
% Note: this is written for a negative transmitted pulse
%
k = 0;  % initialize buffer
flag = 1;  % at start
for i = 1:P
    if strain_trans(i) < -STDEV_trans && flag == 1  %when pulse becomes lower than STDEV
        k = k + 1;  % count number of points in buffer
    else  % -abs(strain_inc(i)) > 0
        k = 0;  % reset counter
    end
    if k == points_buffer && flag == 1
        inc_start = i - points_buffer;  % increment number at start of pulse
        flag = 2;  % inside pulse
    else
        end
if flag == 2
    break % exit loop (you don't need to read the rest of the file)
end
end

% Calculate slope of line in beginning of incident pulse
linefit = polyfit(time_trans(inc_start:inc_start+points_buffer),... 
    strain_trans(inc_start:inc_start+points_buffer),1);

m = linefit(1,1); % slope of curve (strain rate)
b = linefit(1,2); % intercept

 earns

%instance in which fit line to pulse intercepts x-axis (y=0)

TimeStart_Trans = time_trans_start*10^6 + orig_shift_trans*10^6; % (microsec) Time
that Inciden

t

flag = 1; % at start
count = 1; % initialize count

for i = 1:P
    if time_trans(i)>= time_trans_start && flag == 1 % start storing data
        shift_time_trans(count) = time_trans(i); % (sec) time
        shift_strain_trans(count) = strain_trans(i); % (unitless) strain
        flag = 2; % inside pulse
        count = count + 1; % update count
        inc_start=i;

    elseif count == N_pulse && & flag == 2 % store last point and break
        shift_time_trans(count) = time_trans(i); % (sec) time
        shift_strain_trans(count) = strain_trans(i); % (unitless) strain
        flag = 3; % end of pulse
        inc_end = i;

    elseif count < N_pulse && & time_trans(i)>= time_trans_start
        shift_time_trans(count) = time_trans(i); % (sec) time
        shift_strain_trans(count) = strain_trans(i); % (unitless) strain
        count = count + 1; % update count

    end

if flag == 3
    break % exit loop (you don't need to read the rest of the file)
end
end

% shift_time_trans  = shift_time_trans - shift_time_trans(1); % (unitless) shifted time (to
start at zero)

shift_strain_trans=shift_strain_trans+0.5E-5;
strain_trans=strain_trans+0.5E-5;

% same figure
subplot(2,2,2)
plot(time_trans*10^6,strain_trans*100,'-k',...
   time_trans(inc_start:inc_end)*10^6,...
   strain_trans(inc_start:inc_end)*100,'-r',...
   'LineWidth',2)
xlabel('time (\text{musec})','FontSize',Font_Size)
ylabel('Strain (%)','FontSize',Font_Size)
title(strcat('Transmitted Strain Pulse Started at:',...
   num2str(round(TimeStart_Trans)), '\text{musec}'),...
   'FontSize',Font_Size)
set(gca,'FontSize',Font_Size-4)
grid on

% Align reflected pulse --------------------------------------------------------
% Note: this is written for a positive reflected pulse
%
k = 0; % initialize buffer
flag = 1; % at start
for i = 1:P
   if strain_ref(i) > STDEV_ref && flag == 1
      k = k + 1; % count number of points in buffer
   else
      k = 0; % reset counter
   end
   if k == points_buffer && flag == 1
      inc_start = i - points_buffer; % increment number at start of pulse
   else
   end
   if flag == 2
      break % exit loop (you don't need to read the rest of the file)
   end
end
% Calculate slope of line in beginning of incident pulse
linefit = polyfit(time_ref(inc_start:inc_start+points_buffer),...
   strain_ref(inc_start:inc_start+points_buffer),1);
   m = linefit(1,1); % slope of curve (strain rate)
b = linefit(1,2); % intercept
time_ref_start=-b/m-time_buffer*2;
%time_ref_start=time_trans_start; %instance in which fit line to pulse intercepts x-axis (y=0)
% TimeStart_Ref = time_ref_start*10^6 + orig_shift_ref*10^6; % (microsec) Time that Incident Pulse Starts
% flag = 1; % at start
count = 1; % initialize count
for i = 1:P
    if time_ref(i) >= time_ref_start && flag == 1 % start storing data
        shift_time_ref(count) = time_ref(i); % (sec) time
        shift_strain_ref(count) = strain_ref(i); % (unitless) strain
        flag = 2; % inside pulse
        count = count + 1; % update count
        inc_start = i;
    elseif count == N_pulse && flag == 2 % store last point and break
        shift_time_ref(count) = time_ref(i); % (sec) time
        shift_strain_ref(count) = strain_ref(i); % (unitless) strain
        flag = 3; % end of pulse
        inc_end = i;
    elseif count < N_pulse && time_ref(i) >= time_ref_start
        shift_time_ref(count) = time_ref(i); % (sec) time
        shift_strain_ref(count) = strain_ref(i); % (unitless) strain
        count = count + 1; % update count
    end
    if flag == 3
        break % exit loop (you don't need to read the rest of the file)
    end
end
shift_time_ref = shift_time_ref - shift_time_ref(1); % (unitless) shifted time (to start at zero)
strain_ref = strain_ref;
shift_strain_ref = shift_strain_ref;
% same figure
subplot(2,2,3)
plot(time_ref*10^6,strain_ref*100,'-k',...
    time_ref(inc_start:inc_end)*10^6,....
    strain_ref(inc_start:inc_end)*100,'-r',...
    'LineWidth',2)
xlabel('time (\musec)','FontSize',Font_Size)
ylabel('Strain (%)','FontSize',Font_Size)
title(strcat('Reflected Strain Pulse Started at: ',...
    num2str(round(TimeStart_Ref)), '\musec'),...
    'FontSize',Font_Size)
set(gca,'FontSize',Font_Size-4)
grid on
% LOG = fopen('log.txt','a+'); % name of log file, w+ opens or creates file % for reading and writing and appends data to the end of the file fprintf(LOG, '%s\n',strcat('Time that Incident Pulse Starts is: ', num2str(TimeStart_Inc), '\musec'));
fprintf (LOG, 'Time that Reflected Pulse Starts is: ', num2str(TimeStart_Ref, 'microsec'));
fprintf (LOG, 'Time that Transmitted Pulse Starts is: ', num2str(TimeStart_Trans, 'microsec'));
fclose(LOG);

% Compare the three shifted pulses --------------------------------------
% same figure
subplot(2,2,4)
plot(shift_time_inc*10^6,shift_strain_inc*100,'-k',
     shift_time_ref*10^6,shift_strain_ref*100,'-r',
     shift_time_trans*10^6,shift_strain_trans*100,'-b',
     'LineWidth',2)
xlabel('time (musec)', 'FontSize', Font_Size)
ylabel('Strain (%)', 'FontSize', Font_Size)
legend('
epsilon_I','epsilon_R','epsilon_T','Location','SouthEast')
title('Strain Pulses After Aligning', 'FontSize', Font_Size)
set(gca,'FontSize', Font_Size-4)
grid on
saveas(gcf, strcat('All_Shifted_Strain_Pulses.fig')) % Save Figure

XX = datestr(now); % print date and time
LOG = fopen('log.txt', 'a+'); % name of log file, w+ opens or creates file
% for reading and writing and appends data to the end of the file
fprintf (LOG, 'Completed Aligning Pulses at: ', XX);
fclose(LOG);

% Calculate the stress and strain in specimen --------------------------
for i = 1:(N_pulse)
    % Calculate stress in specimen
    sig_sp(i) = -(E_bar*A_bar/A_sp)*
        shift_strain_trans(i); % (Pa) stress in specimen
    
    % Calculate strain rate in specimen
    % using reflected pulse only
    edot_sp_r(i) = (2*C_bar/L_sp)*(shift_strain_ref(i)); % (1/sec) strain rate in specimen
    % using incident and transmitted pulse
    % Note: strain_trans = (strain_inc + strain_ref), therefore
    % strain_ref = (strain_trans - strain_inc)
    edot_sp_ti(i) = (2*C_bar/L_sp)*(shift_strain_trans(i)-shift_strain_inc(i)); % (1/sec) strain rate in specimen
    
    strain_rate_avg(i) = (edot_sp_r(i) + edot_sp_ti(i))/2; % (1/sec) strain rate in specimen
end
% Calculate strain in specimen
if i == 1
    e_sp(i) = 0; % (unitless) strain in specimen
else
    e_sp(i) = e_sp(i-1) + edot_sp_avg(i)*dt; % (unitless) strain in specimen
end
end
% Calculate Strain Rate for test
% take the slope of the strain versus time in the middle quarter of the pulse
k = 0; % initialize buffer
flag = 1; % at start
for i = round(N_pulse/2):N_pulse
    if strain_ref(i) < STD_EV_ref && flag == 1
        k = k + 1; % count number of points in buffer
    else % -abs(strain_inc(i)) > 0
        k = 0; % reset counter
    end
    if k == points_buffer && flag == 1
        ie = i - points_buffer; % increment number at end of pulse
        flag = 2; % inside pulse
    else
        break % exit loop (you don't need to read the rest of the file)
    end
end
% is=round(2*time_buffer*dt); % time increment start
% mid = (ie-is)/2;
% %start of middle 1/4
% i1=round(mid-mid/8);
% %end of middle 1/4
% i2=round(mid+mid/8);
% i1=30;
% i2=75;
% linefit = polyfit(shift_time_inc(i1:i2),e_sp(i1:i2),1);
% m = linefit(1,1); % slope of curve (strain rate)
% b = linefit(1,2); % intercept
linefit = polyfit(shift_time_inc(round(N_pulse/3):round(2*N_pulse/3)),...
    e_sp(round(N_pulse/3):round(2*N_pulse/3)),1);
    m = linefit(1,1); % slope of curve (strain rate)
    b = linefit(1,2); % intercept

% Rename Data
time_blocked = shift_time_inc; % (sec) time blocked

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sig_blocked = sig_sp; % (Pa) stress blocked
edot_sp_r_blocked = edot_sp_r; % (sec^-1) strain rate blocked
edot_sp_ti_blocked = edot_sp_ti; % (sec^-1) strain rate blocked
edot_sp_avg_blocked = edot_sp_avg; % (sec^-1) strain rate blocked
avg_rate = m; % (sec^-1) avg strain rate blocked
strain_blocked = e_sp; % (unitless) strain blocked

XX = datestr(now); % print date and time
LOG = fopen('log.txt','a+'); % name of log file, w+ opens or creates file
% for reading and writing and appends data to the end of the file
fprintf (LOG, '%s 


',strcat('Completed Calculating Stress and Strain in Specimen at: ', XX));
fclose(LOG);

%---------------------------------------------------------------------------------------------------
% Smooth Stress Data ------------------
%---------------------------------------------------------------------------------------------------

N = time_blocked*10^6; % (microsec) time
a = sig_blocked/10^6; % (MPa) stress

P = size(N); % Total Number of Data points
P = P(1,2);
M = P(1,1) - 3;

% Calculate Y a column vector of a, a 7 point average
% Skips the first 3 and last 3
J = 4; % Initialize J
while J <= M
    Y(J) = (a(J-3) + a(J-2) + a(J-1) + a(J) + a(J+1) + a(J+2) + a(J+3))/7;
    J = J + 1;
end

% Calculate Y 1, 2, 3, P, P-1, P-2 (the numbers that we had to skip before)
Y(1) = (13*a(1) + 10*a(2) + 7*a(3) + 4*a(4) + a(5) - 2*a(6) - 5*a(7))/28;
Y(2) = (5*a(1) + 4*a(2) + 3*a(3) + 2*a(4) + a(5) - a(7))/14;
Y(3) = (7*a(1) + 6*a(2) + 5*a(3) + 4*a(4) + 3*a(5) + 2*a(6) + a(7))/28;
B_1 = (a(P-6) + 2*a(P-5) +3*a(P-4) + 4*a(P-3) + 5*a(P-2));
Y(P-2) = (B_1 + 6*a(P-1) + 7*a(P))/28;
B_2 = (-a(P-6) - 2*a(P-5) + a(P-4) + 4*a(P-3) + 7*a(P-2));
Y(P-1) = (B_2 + 5*a(P))/14;
B_3 = (-5*a(P-6) - 2*a(P-5) + a(P-4) + 4*a(P-3) + 7*a(P-2));
Y(P) = (B_3 + 10*a(P-1) + 13*a(P))/28;

XX = datestr(now); % print date and time
LOG = fopen('log.txt','a+'); % name of log file, w+ opens or creates file
fprintf (LOG, '%s
', strcat('Completed Smoothing Data at: ', XX));
fclose(LOG);
%
% Save Results to Text File -----------------------------------------------
%
temp = fopen('Specimen_stress_strain_Cleaned.txt', 'wt'); %write text
fprintf(temp, '%s
', 'Time','Strain','Stress','Smoothed Stress',... 
        'Incident Strain','Reflected Strain','Transmitted Strain');
fprintf(temp, '%s
',... 
        'microsec','unitless','MPa','MPa','unitless','unitless','unitless');
for g = 1:(N_pulse) % Number of data points
    fprintf (temp, '%-20.10f %-20.10f %-20.10f %-20.10f %-20.10f %-20.10f ...
        %-20.10f
', ... 
        shift_time_inc(g)*10^6,... % column 1 is time (microsec)
        e_sp(g),... % column 2 is specimen strain (unitless)
        sig_sp(g)/10^6,... % column 3 is specimen stress (MPa)
        Y(g),... % column 4 is smoothed specimen stress (MPa)
        shift_strain_inc(g),... % column 5 is aligned incident strain (unitless)
        shift_strain_ref(g),... % column 6 is aligned reflected strain (unitless)
        shift_strain_trans(g)); % column 7 is aligned transmitted strain (unitless)
end
fclose(temp);
%
% Plot Results -----------------------------------------------------------------
%
FigureCount = FigureCount + 1;
figure(FigureCount)
subplot(2,2,1)
plot(shift_time_inc*10^6,sig_sp/10^6,'-k',... 
    shift_time_inc*10^6,Y,'-r',... 
    'LineWidth',2)
xlabel('time (musec)','FontSize',Font_Size)
ylabel('Stress (MPa)','FontSize',Font_Size)
legend('Cleaned Pulses','Smoothed Stress','Location','South')
title('Cleaned Data','FontSize',Font_Size)
set(gca, 'FontSize',Font_Size-4)
grid on
subplot(2,2,2)
plot(shift_time_inc*10^6,edot_sp_r,'-k',... 
    shift_time_inc*10^6,edot_sp_ti,-'r',... 
    shift_time_inc*10^6,edot_sp_avg,-'b',... 
    [shift_time_inc(1)*10^6,shift_time_inc(end)*10^6],... 
    [m,m],'-xg','LineWidth',2)
xlabel('time (musec)', 'FontSize', Font_Size)
ylabel('Strain Rate (sec^-1)', 'FontSize', Font_Size)
legend('w/\epsilon_R', 'w/\epsilon_T-\epsilon_I', 'w/Avg', 'Avg \epsilon Rate', 'Location', 'South')
title('Cleaned Data', 'FontSize', Font_Size)
set(gca, 'FontSize', Font_Size - 4)
grid on
subplot(2,2,3)
plot(shift_time_inc*10^6, e_sp*100, '-k', ...
     shift_time_inc*10^6, (m*shift_time_inc+b)*100, '--r', ...
     'LineWidth', 2)
xlabel('time (musec)', 'FontSize', Font_Size)
ylabel('Strain (%)', 'FontSize', Font_Size)
legend('Incremental', 'fit to middle 1/4', 'Location', 'SouthEast')
title(strcat('Average Strain Rate, ', num2str(round(m)), 'sec^-1'))
set(gca, 'FontSize', Font_Size-4)
grid on
subplot(2,2,4)
plot(e_sp*100, sig_sp/10^6, '-k', ...
     e_sp*100, Y, '-r', 'LineWidth', 2)
xlabel('Strain (%)', 'FontSize', Font_Size)
ylabel('Stress (MPa)', 'FontSize', Font_Size)
legend('Cleaned Pulses', 'Smoothed Stress', 'Location', 'South')
title('Cleaned Data', 'FontSize', Font_Size)
set(gca, 'FontSize', Font_Size-4)
grid on
saveas(gcf, strcat('Stress_Strain_Data.fig')) % Save Figure

-------------------------------------------------------------------------
% Original Data (NOT smoothed or blocked) ---------------------------------%-------------------------------------------------------------------------
XX = datestr(now); % print date and time
LOG = fopen('log.txt', 'a+'); % name of log file, w+ opens or creates file % for reading and writing and appends data to the end of the file
fprintf (LOG, '%s\n', strcat('Started Examining Original Data (NOT smoothed or blocked) at: ', XX));
fclose(LOG);

% Input Name of Files to Analyze
% Comment out the ones you are not using
Filename = '_t_V_strain.txt'; % Use this if you are using the raw data
cc = 3; % column with strain

%
% Load text files
---------------------------------------------------------
% Call time and strain from incident pulse
temp = load(strcat('Incident',Filename));
time_inc = temp(:,1); % (sec) time (column vector)
strain_inc = temp(:,cc); % (unitless) strain (column vector)
clear temp
% Call time and strain from reflected pulse
temp = load(strcat('Reflected',Filename));
time_ref = temp(:,1); % (sec) time (column vector)
strain_ref = temp(:,cc); % (unitless) strain (column vector)
clear temp
% Call time and strain from transmitted pulse
temp = load(strcat('Transmitted',Filename));
time_trans = temp(:,1); % (sec) time (column vector)
strain_trans = temp(:,cc); % (unitless) strain (column vector)
clear temp
% Calculate the total number of points (this is the same for all three pulses)
P = size(time_inc); % Total Number of Data points
P = P(1,1); % Total Number of Data points
%
trigger_inc = TimeStart_Inc/10^6; % (sec) time trigger to store data
trigger_ref = TimeStart_Ref/10^6; % (sec) time trigger to store data
trigger_trans = TimeStart_Trans/10^6; % (sec) time trigger to store data
N_pulse_old = N_pulse; % number of points in buffer
dt_old = dt; % (sec/point) old time between data points
dt = (time_inc(end)-time_inc(1))/P; % (sec/point) time between data points
%
% Align incident pulse and determine pulse length
% Note: this is written for a negative incident pulse
%
flag = 1; % at start
for i = 1:P
    if time_inc(i) > trigger_inc && flag == 1
        inc_start = i; % increment number at start of pulse
        flag = 2; % inside pulse
    elseif time_inc(i) > (trigger_inc+N_pulse_old*dt_old) && flag == 2
        inc_end = i; % increment number at end of pulse
        flag = 3; % end pulse
    end
    if flag == 3
        break % exit loop (you don't need to read the rest of the file)
    end
end
%
N_pulse = inc_end - inc_start;
% flag = 1; % at start
count = 1; % initialize count
for i = 1:P
    if i == inc_start && flag == 1 % start storing data
        shift_time_inc(count) = time_inc(i); % (sec) time
        shift_strain_inc(count) = strain_inc(i); % (unitless) strain
        flag = 2; % inside pulse
        count = count + 1; % update count
    elseif i == inc_end && flag == 2 % store last point and break
        shift_time_inc(count) = time_inc(i); % (sec) time
        shift_strain_inc(count) = strain_inc(i); % (unitless) strain
        flag = 3; % end of pulse
    elseif i > inc_start
        shift_time_inc(count) = time_inc(i); % (sec) time
        shift_strain_inc(count) = strain_inc(i); % (unitless) strain
        count = count + 1; % update count
    end
    if flag == 3
        break % exit loop (you don't need to read the rest of the file)
    end
end
% shift_time_inc  = shift_time_inc - shift_time_inc(1); % (unitless) shifted time (to start at zero)
% FigureCount = FigureCount + 1;
figure(FigureCount)
subplot(2,2,1)
plot(time_inc*10^6,strain_inc*100,'-k',...
     time_inc(inc_start:inc_end)*10^6,....
     strain_inc(inc_start:inc_end)*100,'-r',...
     'LineWidth',2)
xlabel('time (\mu s)','FontSize',Font_Size)
ylabel('Strain (%)','FontSize',Font_Size)
title('Incident Strain Pulse (NOT smoothed or blocked)','FontSize',Font_Size)
set(gca,'FontSize',Font_Size-4)
grid on
% % Align transmitted pulse ---------------------------------------------------
% Note: this is written for a negative transmitted pulse
%
flag = 1; % at start
for i = 1:P
    if time_trans(i) > trigger_trans && flag == 1
inc_start = i; % increment number at start of pulse
flag = 2; % inside pulse
else
end
if flag == 2
    break % exit loop (you don't need to read the rest of the file)
end
end
inc_end = inc_start + N_pulse; % increment at end of pulse

flag = 1; % at start
count = 1; % initialize count
for i = 1:P
    if i == inc_start && flag == 1 % start storing data
        shift_time_trans(count) = time_trans(i); % (sec) time
        shift_strain_trans(count) = strain_trans(i); % (unitless) strain
        flag = 2; % inside pulse
        count = count + 1; % update count
    elseif i == inc_end && flag == 2 % store last point and break
        shift_time_trans(count) = time_trans(i); % (sec) time
        shift_strain_trans(count) = strain_trans(i); % (unitless) strain
        flag = 3; % end of pulse
    elseif i > inc_start
        shift_time_trans(count) = time_trans(i); % (sec) time
        shift_strain_trans(count) = strain_trans(i); % (unitless) strain
        count = count + 1; % update count
    end
if flag == 3
    break % exit loop (you don't need to read the rest of the file)
end
end

shift_time_trans  = shift_time_trans - shift_time_trans(1); % (unitless) shifted time (to start at zero)

shift_time_trans  = shift_time_trans - shift_time_trans(1); % (unitless) shifted time (to start at zero)

shift_time_trans  = shift_time_trans - shift_time_trans(1); % (unitless) shifted time (to start at zero)

plot(time_trans*10^6,strain_trans*100,'-k',...
     time_trans(inc_start:inc_end)*10^6,...
     strain_trans(inc_start:inc_end)*100,'-r',...
     'LineWidth',2)
xlabel('time (musec)','FontSize',Font_Size)
ylabel('Strain (%)','FontSize',Font_Size)
title('Transmitted Strain Pulse (NOT smoothed or blocked)','FontSize',Font_Size)
set(gca,'FontSize',Font_Size-4)
grid on
% Align reflected pulse ---------------------------------------------
% Note: this is written for a positive reflected pulse
%
flag = 1; % at start
for i = 1:P
    if time_ref(i) > trigger_ref && flag == 1
        inc_start = i; % increment number at start of pulse
        flag = 2; % inside pulse
    else
        end
    if flag == 2
        break % exit loop (you don't need to read the rest of the file)
    end
end
inc_end = inc_start + N_pulse; % increment at end of pulse
%
flag = 1; % at start
count = 1; % initialize count
for i = 1:P
    if i == inc_start && flag == 1 % start storing data
        shift_time_ref(count) = time_ref(i); % (sec) time
        shift_strain_ref(count) = strain_ref(i); % (unitless) strain
        flag = 2; % inside pulse
        count = count + 1; % update count
    elseif i == inc_end && flag == 2 % store last point and break
        shift_time_ref(count) = time_ref(i); % (sec) time
        shift_strain_ref(count) = strain_ref(i); % (unitless) strain
        flag = 3; % end of pulse
    elseif i > inc_start
        shift_time_ref(count) = time_ref(i); % (sec) time
        shift_strain_ref(count) = strain_ref(i); % (unitless) strain
        count = count + 1; % update count
    end
    if flag == 3
        break % exit loop (you don't need to read the rest of the file)
    end
end
%
shift_time_ref = shift_time_ref - shift_time_ref(1); % (unitless) shifted time (to start at zero)
%
% same figure
subplot(2,2,3)
plot(time_ref*10^6,strain_ref*100,'-k',...}
time_ref(inc_start:inc_end)*10^6,...
strain_ref(inc_start:inc_end)*100,'-r',... 
'LineWidth',2)
xlabel('time (musec)','FontSize',Font_Size)
ylabel('Strain (%)','FontSize',Font_Size)
title('Reflected Strain Pulse (NOT smoothed or blocked)','FontSize',Font_Size)
set(gca,'FontSize',Font_Size-4)
grid on
%
% Compare the three shifted pulses -----------------------------------
%
% same figure
subplot(2,2,4)
plot(shift_time_inc*10^6,shift_strain_inc*100,'-k',... 
    shift_time_ref*10^6,shift_strain_ref*100,'-r',... 
    shift_time_trans*10^6,shift_strain_trans*100,'-b',... 
    'LineWidth',2)
xlabel('time (musec)','FontSize',Font_Size)
ylabel('Strain (%)','FontSize',Font_Size)
legend('
epsilon_I','epsilon_R','epsilon_T','Location','SouthEast')
title('Strain Pulses After Aligning (NOT smoothed or blocked)','FontSize',Font_Size)
set(gca,'FontSize',Font_Size-4)
grid on
%
saveas(gcf,strcat('All_Shifted_Strain_Pulses_NOTsb.fig')) % Save Figure
%
XX = datestr(now); % print date and time
LOG = fopen('log.txt','a+'); % name of log file, w+ opens or creates file
% for reading and writing and appends data to the end of the file
fprintf(LOG, '%s

',strcat('Completed Aligning Pulses at: ', XX));
close(LOG);
%
% Calculate the stress and strain in specimen ------------------------
for i = 1:(N_pulse+1)
    % Calculate stress in specimen
    sig_sp(i) = -(E_bar*A_bar/A_sp)*...
        shift_strain_trans(i); % (Pa) stress in specimen
%
    % Calculate strain rate in specimen
    % using reflected pulse only
    edot_sp_r(i) = (2*C_bar/L_sp)*...
        shift_strain_ref(i); % (1/sec) strain rate in specimen
    % using incident and transmitted pulse
    % Note: strain_trans = (strain_inc + strain_ref), therefore
    % strain_ref = (strain_trans - strain_inc)
    edot_sp_ti(i) = (2*C_bar/L_sp)*(shift_strain_trans(i) -...
        shift_strain_inc(i)); % (1/sec) strain rate in specimen

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% taking average
edot_sp_avg(i) = (edot_sp_r(i) + edot_sp_ti(i))/2; % (1/sec) strain rate in specimen
%
% Calculate strain in specimen
if i == 1
    e_sp(i) = 0; % (unitless) strain in specimen
else
    e_sp(i) = e_sp(i-1) + edot_sp_avg(i)*dt; % (unitless) strain in specimen
end
avg_e_r(i) = (shift_strain_ref(i) +
... (shift_strain_trans(i) - shift_strain_inc(i)))/2; % (unitless) average reflected strain
end
% Calculate strain in specimen using trapezoidal method
e_sp_trapz = (2*C_bar/L_sp)*
... cumtrapz(shift_time_inc,avg_e_r); % (unitless) strain in specimen
%
% Calculate Strain Rate for test
% take the slope of the strain versus time in the middle quarter of the pulse
k = 0; % initialize buffer
flag = 1; % at start
for i = round(N_pulse/2):N_pulse
    if strain_ref(i) < STDEV_ref && flag == 1
        k = k + 1; % count number of points in buffer
    else % -abs(strain_inc(i)) > 0
        k = 0; % reset counter
    end
    if k == points_buffer && flag == 1
        ie = i - points_buffer; % increment number at end of pulse
        flag = 2; % inside pulse
    else
        end
    if flag == 2
        break % exit loop (you don't need to read the rest of the file)
    end
    end
% is=round(2*time_buffer*dt); % time increment start
% mid = (ie-is)/2;
% % start of middle 1/4
% i1=round(mid-mid/8);
% % end of middle 1/4
% i2=round(mid+mid/8);
% linefit = polyfit(shift_time_inc(i1:i2),e_sp(i1:i2),1);
% m = linefit(1,1); % slope of curve (strain rate)
% b = linefit(1,2); % intercept
%
linefit = polyfit(shift_time_inc(round(N_pulse/3):round(2*N_pulse/3)),...
e_sp(round(N_pulse/3):round(2*N_pulse/3)),1);
m = linefit(1,1); % slope of curve (strain rate)
b = linefit(1,2); % interce
XX = datestr(now); % print date and time
LOG = fopen('log.txt','a+'); % name of log file, w+ opens or creates file
fprintf (LOG,'%s

% for reading and writing and appends data to the end of the file
fprint' (LOG, 'Completed Calculating Uncleaned Stress and Strain in Specimen at:', XX));
fclose(LOG);

% Save Results to Text File ---------------------------------------------------------
% temp = fopen('Specimen_stress_strain_Original.txt','wt'); %write text
fprintf(temp,'%s

% The strain rate is',round(m),'
% sec^-1',...
'Time','Strain','Stress',...
'microsec','unitless','MPa','unitless','unitless','unitless');
for g = 1:(N_pulse+1) % Number of data points
  fprintf (temp, '%-20.10f %-20.10f %-20.10f %-20.10f %-20.10f

% column 1 is time (microsec)
% column 2 is specimen strain (unitless)
% column 3 is specimen stress (MPa)
% column 4 is aligned incident strain (unitless)
% column 5 is aligned reflected strain (unitless)
% column 6 is aligned transmitted strain (unitless)
end
close(temp);

% Plot Results -----------------------------------------------------------------------
% FigureCount = FigureCount + 1;
figure(FigureCount)
subplot(2,2,1)
plot(shift_time_inc*10^6,sig_sp/10^6,'-k',...
'LineWidth',2)
xlabel('time (musec)','FontSize',Font_Size)
ylabel('Stress (MPa)','FontSize',Font_Size)
title('Original Data (NOT smoothed or blocked)','FontSize',Font_Size)
set(gca,'FontSize',Font_Size-4)
grid on
subplot(2,2,2)
plot(shift_time_inc*10^6,edot_sp_r,'-k',...
shift_time_inc*10^6,edot_sp_ti,'-r',...
shift_time_inc*10^6,edot_sp_avg,'-b',... 
[shift_time_inc(1)*10^6,shift_time_inc(end)*10^6],...
[m,m],'-xg','LineWidth',2)
xlabel('time (musec)','FontSize',Font_Size)
ylabel('Strain Rate (sec^-1)','FontSize',Font_Size)
title('Original Data (NOT smoothed or blocked)','FontSize',Font_Size)
legend('w/epsilon_R','w/epsilon_T-epsilon_I','w/Avg','Avg epsilon Rate','Location','South')
set(gca,'FontSize',Font_Size-4)
grid on
subplot(2,2,3)
plot(shift_time_inc*10^6,e_sp*100,'-k',...
shift_time_inc*10^6,e_sp_trapz*100,'--b',...
shift_time_inc*10^6,(m*shift_time_inc+b)*100,'-r',...
'LineWidth',2)
xlabel('time (musec)','FontSize',Font_Size)
ylabel('Strain (%)','FontSize',Font_Size)
title('Average Strain Rate',num2str(round(m)),'sec^-1','FontSize',Font_Size)
set(gca,'FontSize',Font_Size-4)
grid on
subplot(2,2,4)
plot(e_sp*100,sig_sp/10^6,'-k','LineWidth',2)
xlabel('Strain (%)','FontSize',Font_Size)
ylabel('Stress (MPa)','FontSize',Font_Size)
title('Original Data (NOT smoothed or blocked)','FontSize',Font_Size)
set(gca,'FontSize',Font_Size-4)
grid on

saveas(gcf,strcat('Stress_Strain_Data_NOTsb.fig')) % Save Figure

% Compare blocked data to original data -------------------------------------

FigureCount = FigureCount + 1;
figure(FigureCount)
plot(e_sp*100,sig_sp/10^6,'-k',... 
strain_blocked*100,sig_blocked/10^6,'-r',...
strain_blocked*100,Y,'-b',...
'LineWidth',2)
xlabel('Strain (%)','FontSize',Font_Size)
ylabel('Stress (MPa)','FontSize',Font_Size)
title('Original','Cleaned','Stress Smoothed','Location','South')
legend('Original','Cleaned','Stress Smoothed','Location','South')
set(gca,'FontSize',Font_Size-4)
grid on

%
saveas(gcf, strcat('Stress_Strain_compare.fig')) % Save Figure

End = datestr(now) % print date and time
LOG = fopen('log.txt', 'a+'); % name of log file, a+ opens or creates file
% for reading and writing and appends data to the end of the file
fprintf(LOG, '%s
', strcat('End Time is: ', End));
fprintf(LOG, '%s
', '********** End of Calculate_Specimen_Stress_Strain_SHPB.m
**********
');
fclose(LOG);

% End of Calculate_Specimen_Stress_Strain_SHPB.m
A.2.4.3 Typical Experimental Results

An example of the typical experimental results generated during a split Hopkinson pressure bar compression test is presented. The data collected will be presented in terms of the oscilloscope output, the typical SHPB data analysis codes production and the generated SHPB stress-strain curve. Figure A-29 shows a screen shot of the oscilloscope data captured from a SHPB test of Al6061-O at 100°C and a strain rate of 5E3s⁻¹. From Figure A-30 the three pulses utilized to generate the experimental stress strain curve can be seen, incident, reflected and transmitted.

The data collected from the oscilloscope is transferred via flash drive and run through a series of Matlab programs that converts the data into the experimental stress-strain curve as previously explained in this appendix. After the raw data is converted from voltage to strain with extraneous data removed and then smoothed within the first two programs the third program aligns the three pulses and outputs the Figure A-30 as shown from Matlab.

The alignment of these three pulses are extremely important as this alignment adjust the alignment of the stress and strain time domains, and thus will have an extreme effect of the resultant stress-strain curve. The curve that was generated from this test is shown in Figure A-31 in true stress-true strain.
Figure A-29: Screenshot taken from the high rate oscilloscope utilized to capture SHPB strain gauge voltages, that represents the strain histories of the incident and transmitted loading bars of an experimental test of Al6061-O at 100°C and 5E3s⁻¹. The three pulses of incident, reflected and transmitted are then used to generated the strain-strain curves.
Figure A-30: Plots outputted as from the third Matlab program, Calculate_SHPB_strain_strain_curve.m, after captured data is converted from voltage to strain and smoothed. The three pulses are then aligned for strain-strain curve generation.
Figure A-31: Resultant True stress-True strain curve generated from the data as shown in Figure A-30, for a test of Al6061-O at 100°C and 5.0E3s⁻¹
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APPENDIX B

EQUAL CHANNEL ANGULAR PRESS SYSTEM

B.1 Introduction of Equal Channel Angular Press

The first equal channel angular press (ECAP) was developed by Segal et al [1] in the Soviet Union, in which work provided a manner to apply simply shear as the most optimum manner to achieve transformation of microstructures, in terms of refining the grain size. Sever plastic deformation (SPD) has long been known to be cause grain refinement in materials, defined as deformation that occurs in a material beyond a strain of 4.00. SPD systems include wire drawing, rolling, constrained-pressing and ECAP. The benefit of grain refinement in which abnormally high strength and ductility can be achieved within a material is characterized by the Hall-Petch relationship [2,3], in which with a decrease in grain size, a large increase in yield stress occurs. Therefore grain refinement processes have been of great interest of research for the past 50 plus years.

Wire drawing is a process in which a cylinder is pulled through a die with a reduced cross section. The material is pulled through the die, it is forced to deform at a high level of strain, in which when the process is completed several times the material reaches the level of severe plastic deformation as previously defined, and a thin- wire of material with a refined grain structure [4]. The process is use-full in some applications but a negative is that a large reduction in cross sectional is resulted, not allowing for a bulk material to be generated. This reduction in cross section is also the draw-back in rolling as a process of SPD. Rolling is a process in which a material is repeatedly compressed through rollers, reducing the thickness after each processing step, leaving a
nearly 90% reduction in cross section [5]. Constrained pressing does result in a bulk material, in which a sample is repeatedly forced in to a designed cavity and rotated and pressed again into the same cavity [6]. The difficulty is that the material is allows to bulge and therefore deform inhomogeneously, leading to the repeatability of the process to be difficult. The use of an ECAP system to cause SPD upon a material has shown to be the most viable, repeatable manner in which to cause grain size refinement within metals [7-13]. This can be expressed due to its use of the main deformation mechanism of shear to deform the material, allowing for the most optimum effect of plastic deformation upon a material. Segal showed that there are several requirements a process of SPD must apply to deform a material most efficiently. These include the uniformity of the stress and strain states, accurate control of the intensity of deformation and stress state, spatial development and texture formation control of the deformation and finally the capability to apply a ultra-high amount of deformation without resultant fracture.

The fundamentals of an ECAP is to pass a billet through two intersecting channels of equal cross section. These channels intersect at an angle anywhere from 90 to 120°, dependent of the desired amount of strain to be applied per pass, as schematically shown in Figure B-1 [14] . A well lubricated billet of the same cross-section is placed into the entrance of the top channel and a plunger or punch presses this billet into the second channel at constant velocity. The billet moves through the die as a rigid body, with deformation occurring ideally by simple shear at intersecting planes of the two channels, AO of Error! Reference source not found., showing the slip line field and velocity vectors.
From this figure, in which $P$ is the load applied by the plunger and $k$ is the material shear strength, and assuming no other forces acting upon the push rod, including friction, equations B-1 and B-2 can be determined.

$$\sigma = -k \cot \theta \quad \text{(B-1)}$$

$$P = 2k \cot \theta \quad \text{(B-2)}$$

where $\sigma$ is the average stress along line $AO$ and $\theta$ is the semi-angle between the channels. If friction is included into the system, then the equations become more complex, in which Figure B-2 [14] represents a diagram of the slip plane field and velocity vectors. The single plane slip line field in the frictionless assumption becomes a fanned circular section $AOB$ centered at point $O$ at within which predominantly all the shearing of the material occurs. With the frictional forces, $\tau$ included the total fanned region consists of four separate parts, $AOB$ is a rigid metal zone of the die, $AOB$ the region at which shearing occurs as expressed previously and $OBC$ and $OAD$: regions of uniform stress distribution.
Figure B-1: Schematic of the a) slip line diagram and b) velocity vector for ECAP process without friction [14]

Figure B-2: Schematic of the a) slip line diagram and b) velocity of ECAP process with friction forces [14]
The angle $\Psi$ is the angle of the outer curvature of the die and can be calculated using equation B-4, with $n$ representing the angle between slip line AO and BO, and $L/H$ representing the length to thickness ratio and $P$, the pressure applied by the plunger being calculated using equation B-3. These formulations are valid only when $\tau = k \cos 2\theta$.

$$P = 2k(\cot n + 2(n - \theta)) + \tau(\sin n(\sin n + \cos n))^{-1} + \frac{2\tau L}{H}$$ (B-3)

$$\psi = 2(n - \theta)$$ (B-4)

$$n = \frac{\pi}{2} - \frac{1}{2} \arccos \left( \frac{\tau}{k} \right)$$ (B-5)

In order to estimate the total accumulated shear strain developed during processing the die geometry is defined by the cross section and the angle, $\phi$, between the intersection of the two channels and the arc of curvature at the outer point or intersection, $\Psi$, in which frictional effects can be neglected. Shear is defined as a shape change of a cubic element being displaced along a single set of parallel planes.

Seen in Figure B-3 [14], a cubic element $abcd$ moves through the die defined by angles $\phi$ and $\Psi$ at a constant velocity, following slip lines e and f. The element after moving through the region of simple shear the element will transform into orthogonal element $a'b'c'd'$. Using the notion within Figure B-3, the shear strain can be shown to be

$$\gamma = \frac{a'u}{d'u} = r_c + as = \frac{\psi \cdot ad \cdot \cos ec \left( \frac{\phi}{2} + \frac{\psi}{2} \right) + ad \cdot \cot \left( \frac{\phi}{2} + \frac{\psi}{2} \right)}{ad}$$ (B-6)

and reduces to,
Figure B-3: Schematic of the shear deformation of element $abcd$ to $a'b'c'd'$ during ECAP processing [14]

Figure B-4: Rotation schemes A, $B_a$, $B_c$ and C, used in ECAP processing
\[ \gamma = \psi \cos \varepsilon \left( \frac{\phi}{2} + \frac{\psi}{2} \right) + 2 \cot \left( \frac{\phi}{2} + \frac{\psi}{2} \right) \]  

(B-7)

The Von misses shear strain, or equivalent strain can be calculated by dividing the shear strain by the square root of three, and considering the case of multiple passes, the strain of one pass can be multiplied by \( N \), the number of total passes. These inclusions lead to equation B-8 being shown to be.

\[ \varepsilon_{eff} = \frac{N}{\sqrt{3}} \left( \psi \cos \varepsilon \left( \frac{\phi}{2} + \frac{\psi}{2} \right) + 2 \cot \left( \frac{\phi}{2} + \frac{\psi}{2} \right) \right) \]  

(B-8)

Using equation 8 represents an estimated amount of strain applied during processing excluding strain hardening, frictional and strain rate effects. It has been shown by several authors [9,15-21] that the application of different routes of ECAP processing has a direct effect on the final generated material microstructure and characteristics. Routes are defined as the pattern in which each subsequent pass after the initial pass is performed in terms of the initial orientation of the billet. The four principle routes of processing in an ECAP are defined at A, C, Bc and Ba. Figure B-4 [22] gives a simple representation of these four routes.

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The initial coordinate system of the billet is defined as the transverse, extrusion and normal directions are equivalent to the X, Y, and Z axis respectively. Route A is defined as having no rotations along the extruding direction axis between each pass. Route C is characterized by being rotated 180° along the extrusion direction between each pass. This applies shear strain along the same shear plane for each pass, in which the direction is reversed with each rotation. The reversal of direction results in a reversal of the shear strain applied in the previous pass, which occurs every even pass. The Ba route
includes a 90° rotation in the clockwise direction with relation to the extrusion direction on even passes numbers and a 90° rotation in the counter clockwise direction with odd numbered passes. This processing route therefore applies a superposition of the shear strain onto X and Y planes. Route Bc includes a 90 clockwise rotation between each pass, applying shear to each spatial plane equally. Route Bc in several cases has been shown to be the most effective in generating ultra-fine grained materials. This is true because it is the only route that does not apply the strain repeatedly on the same shear plane(s). This causes dislocations generated on more slip planes each pass, rather than just moving the dislocations previously generated during the opposite direction shearing on the same plane. Figure B-5 [23] represents the routes discussed, showing the effect of the cubic element of each plan up to 8 passes. It can be seen in the case of Route A and C, that the Z plane does not have strain applied, which cause it to be less efficient in grain refinement. Routes Ba and Bc have strain applied to each plane after a number of passes. During processing using route Ba causes subsequent passes in the same direction on each of the planes, which in turn does not generate equaixed grain structures. This will cause the material to by anisotropic in nature, have grain elongated in a preferential direction. Route Bc not only applies strain on each plane equally, but after 4 passes, the direction at which the initial strain on a plane was applied, is also reversed. This is beneficial in generating an equaixed grain structure and has been shown to be the most efficient in the refinement process.
| Route | Plane | Number of pressings |
|-------|-------|---------------------|
|       |       | 0 1 2 3 4 5 6 7 8  |
| A     | X     | □ □ □ □ □ □ □ □ □ |
|       | Y     | □ □ □ □ □ □ □ □ □ |
|       | Z     | □ □ □ □ □ □ □ □ □ |
| B_A   | X     | □ □ □ □ □ □ □ □ □ |
|       | Y     | □ □ □ □ □ □ □ □ □ |
|       | Z     | □ □ □ □ □ □ □ □ □ |
| B_C   | X     | □ □ □ □ □ □ □ □ □ |
|       | Y     | □ □ □ □ □ □ □ □ □ |
|       | Z     | □ □ □ □ □ □ □ □ □ |
| C     | X     | □ □ □ □ □ □ □ □ □ |
|       | Y     | □ □ □ □ □ □ □ □ □ |
|       | Z     | □ □ □ □ □ □ □ □ □ |

**Figure B-5**: Schematics of the deformation of a unit element after n passes through a ECAP die
**B.2 Design of ECAP at Mechanics of Materials Research Laboratory**

The following appendix sections will present an overview of the Equal Channel Angular Press (ECAP) system designed and built at the MMRL. This system is utilized to generate fine-grain materials for the research within this thesis. This sections will include Abaqus FEM modeling for the design of the die and heating components, as well as explanation of the final system completed including mechanical drawings as well as an experimental manual for utilizing the system as described.

**B.2.1 Die Design: Abaqus Numerical Material Process Modeling**

The previous section of this appendix described the multiple variables of the ECAP processing technique. These include the channel angles of the inner, $\phi$, and outer, $\Psi$, frictional effects and the total amount of desired strain applied based on the characteristic angles. The analysis utilized to predict the strain applied omits known variables like surface effects due to friction, and material strain rate and strain hardening characteristics. Also not discussed previously is the effect of the bottom configuration of the billet, in which the geometry of the leading edge of the billet can affect the deformation characteristics in terms strain uniformity [24]. The different geometry types include tapered in either direction, squared, or rounded. For all of these factors it is valuable to model the ECAP process using FEA simulations where insight is given into the effect of these variables without the high cost of multiple design iterations and lessening of experimental testing. Once the overall processing is determined, in terms of design parameters, then simulations can be completed on the structural components of the die can be completed to insure that the die will be able to withstand the high loads seen
during ECAP processing. To do so, the commercially available Abaqus, a FEA simulation program, is utilized to aid in the design of the ECAP system at the MMRL. First a generalized model will be discussed, in which idealizations are made, and other complexities such effect of the channel angles, channel dimensions, addition of frictional effects, specimen dimensioning tolerances and specimen configurations can be investigated.

To develop the FEA model both the bottom (L-shaped region) and top (corner) die sections that make up the channel are modeled as 2D analytically rigid shells using a channel geometry of 90° for the inner and outer angles, and radius of 0.25" and 0.75" respectfully. These variables will be varied at a later point. The die sections are then assembled in their proper positions with reference to one another. A specimen is modeled as 2D planar deformable shell with exact dimensions of 4" long and 0.5" thick, with the assumption that it is perfectly fit into the die channel, and assembled in its initial position. A figure of this assembly can be seen in Figure B-6. A dynamic-explicit step is generated, at which the material loading will take place. An interaction is created where the contact between the billet and die will be seen to be frictionless for a simplified case. The analytical die will be fixed in all directions for the boundary condition of the die surfaces. This is a valid assumption as the die surface is expected to not deform during the pressing of the billet. The top surface of the billet will have a constant velocity boundary condition, to ensure that the specimen will move with a constant rate through the die, allowing for the resultant force that it would take to do so to vary freely. These boundary conditions can be seen in Figure B-6.
Figure B-6: FEA Abaqus assembly in which the 2D die is simulated as analytically rigid and the 2D specimen as a planar deformable part. Also shown are the boundary conditions where the 2D die has boundary conditions that restrict any motion, and the billet has a constant velocity boundary condition applied upon the top surface.
Figure B-7: FEA Abaqus simulation results showing plastic equivalent (PEEQ) strain contours, in which the maximum amount of strain is seen on the interior surface with respect to the die at a value of 78.13%
In order to accurately model the deformation of the material as it moves through the die, a plastic constitutive model is used. This allows for strain and strain rate hardening characteristics to be considered, these will affect the overall strain uniformity of the deformation, more accurately predicting the response in the real-world scenario. The built-in Johnson-Cook plasticity feature of Abaqus was used to do so, along with data obtained in literature [25]. A relatively coarse mesh was used on the specimen to improve computer simulation time, this mesh was held to be constant as to allow for proper comparison between each variable change. This mesh can be seen in Figure B-7 in its deformed state after the simulation where the resultant plastic equivalent strain contour plot is shown, in which each the distance between each node is equivalent to 0.125” in the initial un-deformed state.

With the model showing the ability to complete the simulation, it is now possible to systematically adjust variables and compare the result in terms of strain uniformity and the force required to deform the billet. First the most critical of the design criteria is the inner and outer angles, several angles will be simulated, the before mentioned 90° as well as 95° and 100°. With these simulation, all other variables will remain constant, including the radii and interactions. The equivalent plastic strain is measured along the length of the center axis beginning with the leading edge of the billet. The center axis is chosen as to avoid any surface effects. A plot of the collected data is shown in Figure B-8.
Figure B-8: Plot of the PEEQ versus position (in) along the length of the specimen beginning at the leading edge of the billet for varying channel angles of 90°, 95° and 100° obtain from FEA Abaqus simulations.

Figure B-9: Plot of the Force (Tons) required to move the billet through the die versus time obtained from FEA Abaqus results for varying channel angles of 90°, 95° and 100°.
It can be seen in Figure B-8, that the theoretically determined applied strains of 1.57, 1.49 and 1.42 for the 90°, 95° and 100° degree channels from equation (B-8) differs in the simulation, in which average values of strain 0.5, 0.48 and 0.46 are shown for the center region of the specimen, from 1-3 inches. This can be explained by the lack of strain and strain rate hardening effects in the predicted strain values, that were implemented by using the Johnson-Cook constitutive equation. Therefore the same die geometry would not necessarily be valid for varying materials. Also from the simulation it is possible to investigate the resultant force that occurred at the top surface of the billet, which represents the force that would needed to move the specimen through the die.

In Figure B-9, the average force for the steady state regions for the 90°, 95° and 100° degree angles are shown to be 2, 1.87 and 1.74 Tons. The force required, as simulated, is considerably lower then what would be expected in the actually pressing. This can be attributed to lack of friction involved. From this analysis it can be shown that with a 5° change in the channel angles, the reduction of force can be very beneficial with the effect of this reduction being amplified when frictional effects would be added. The change in the strain by only a difference of 0.23% from the 90° case to the 95° attains a nearly 1% decrease in the force necessary to press the billet between the two angles. With this reasoning it was determined that the 95° inner and outer channel angles would be utilized in the remainder of the simulation for that fact that it gives the best relationship of a high degree of strain applied but the benefit of a decrease in load applied, as well as will be utilized in the actual ECAP system designed.

With the channel angles determined it is now possible to investigate a few of the other variables involved. First the effect of friction is investigate, in which the importance
of proper lubrication of the die will be investigated by systematically adjusting the kinetic friction coefficient, $\mu_k$, within the interaction properties. This effect will investigated using the shear strain distribution along the length of the specimen, on node in from the right surface. This is done because friction is known to cause surface effects that extend into the interior of the specimen [24]. All other variables remain the same from the previous analysis.

It can be seen from Figure B-10a that although the increase in friction does cause an increase in the shear strain applied which is desirable, the distribution of the shear strain becomes less continuous with an increase in strain. This discontinuity is undesirable as the material will therefore not see a constant deformation along its length. The force necessary to move the billet is also affected by the increase in the friction coefficient, as shown in Figure B-10b.
Figure B-10: a) Plot of the shear strain (in/in) resultant a single pass through the simulated ECAP die obtained from FEA Abaqus results versus the position (in) along the length of the billet beginning at the leading edge of the billet. b) Plot of the Force (Tons) required to move the billet through the die versus time obtained from FEA Abaqus results for varying coefficient of kinetic friction, $\mu_k$, of 0, 0.1, 0.2 and 0.3
From Figure B-10b it can be seen that the increase in the frictional coefficient, as would be expected increases the load necessary to move the billet through the die at a constant rate. As was shown in the previous Figure B-10a, friction may actually apply some benefits in the terms that it increases the amount of strain within the specimen, the increase in the load from the friction is highly undesirable, as it may lead to failure of the other systems involved in terms of the actual structural components of the die. This leads to the knowledge that lubrication during the ECAP pressing is extremely important in the survivability of the die, and also attaining a uniformly deformed specimen.

It has been shown from other researchers [24] that changing the leading end of the billets' geometry has an effect on the overall strain uniformity, not just at the end at which the geometry is adjusted, but the distribution throughout the length. Therefore three separate cases will be investigated. These being the square end billet that has been used so far in the analysis and is typical in ECAP pressing, a geometry that matches the radius of the outer curvature of the die, and a tapered geometry where the billet is tapered from the inside edge to the outside, as shown in Figure B-12.
Figure B-11: Schematics of the varying billet end geometries studied where from right to left include, square, radiused and tapered. All were meshed using seed sizes of 0.125
From these simulations it is possible to again investigate the strain uniformity throughout, as shown in Figure B-13. It can be seen that the deformation is most uniform in the tapered specimen, followed by the square and then radiused geometry. This is determined by the uniformity of the PE12 contours, where majority of the bulk material of the tapered geometry specimen remains in one contour regime. This is not the only determinate facet though when investigating which geometry is best suited for the ECAP processing. From the taper geometry specimen, it can be noted that a large amount of deformation occurred at the leading edge. This material would have to be discarded and the taper re-machined for every pass. This large amount of material loss is not acceptable, and therefore it is determined that the square geometry specimen will be utilized as it gives the most uniformly deformed while also providing the largest amount of material to be utilized in following pressings.
Figure B-12: Shear strain contour plots in the 1-2 directions for the three differing billet end geometries
The final geometry of the die channel and summary of the above results is now given. It was determined by varying the inner and outer channel angles, that the 95° angle set would result in an acceptably uniformly distributed deformation while also lowering the force needed to extrude the billet compared to the more extreme channel angles. Therefore the channel dimensions will be 95 degrees, with radii of 0.25 on the interior and 0.75 on the exterior angle, this is done to retain a uniform cross-section throughout the channel, in which the channel thickness will be 0.5". The specimen must be heavily lubricated as well as the die walls, to ensure that friction is reduced. Friction can be advantageous in the sense that it result in a larger amount of stain applied caused by surface effects that transfer through the specimen but, these effect also cause a discontinuous deformation while also increasing the load to extrude the specimen. Therefore to ensure a repeatable experiment, where each billet produced has undergone the same deformation, and therefore grain refinement, it is important to remove the frictional effects as much as possible. A high temperature/pressure graphite based lubricant will be used. Finally it was determined that the square geometry of the billet results in an acceptable balance between even deformation of the material and retainable quantity of material between each pass. This is important as the bulk generation of a fine grain material will be later utilized for mechanical testing to determine material characteristics.
B.2.2 System Design

B.2.2.1 Overview of ECAP at MMRL

To facilitate an understating of the ECAP system designed and built at the MMRL, a brief summary of the system is given. The ECAP consists of five subsystems which include the tool steel die used to apply a desired amount of geometric strain per pass, the plunger and plunger elevator used to stabilize the application of the load to the billet, the hydraulic press used to apply and control the rate of loading, a heating system used to heat the entire die to a desired temperature, aiding in the controlled deformation of the material and a data acquisition system which gives the ability to collect experimental data of pressing force and rate over the duration of the experiment. A photograph of the entire system can be seen in Figure B-13.
**Figure B-13**: The ECAP system built at the MMRL in which major sub-systems are noted including the tool steel die, shown with heater plates attached, 100Ton hydraulic press, plunger and plunger elevator, heating system including control unit and heater plates and the LVDT apparatus used for data acquisition.
The ECAP die is easily the most important component utilized in the SPD system. The die needs to be accurately designed to provide the desired result in the generated refined granular structure of the material. The die must be very resilient to extreme levels of applied stress during pressing, while also capable of performing at elevated temperatures. The die must be easy disassembled and reassembled in a repeatable manner in order to remove the pressed billet after every pass. Also it is desirable that the die be designed in a modular fashion that will ensure, if failure occurs, the part can be easily replaced, while also able to fit within the constraints of the hydraulic press used during apply the load to the billet.

The major design consideration for the die as expressed previously in this appendix, is the geometry of the channel. Mainly the inner and outer channel angles and radii. Given by equation B-8, the inner and outer channel angles determine the amount of geometric strain that is applied per pass. The effect theses variables was investigated in B.2.2.1 of this appendix, and resulted in the determination that an inner and outer angle of 95°, with radii of 0.25" and 0.75" respectfully, resulted in the most uniform deformation of the billet material. The thickness of the specimen to be pushed was determined to be 0.5" thick, as this size is adequate for the future testing of the generated material, therefore that determining the channel width and thickness. It is possible to scale the entire system to a larger billet size, though this would cause an increase in load required to press the billet, and in turn an increase in load applied on the die.
It was determined that tapering the exit channel from the inner to the outer of the channel would be very advantageous as this would allow for the elastic stresses generated within the material of the billet during shearing and push outward onto the channel walls. The removal of these stresses allow for the ease of disassembly of the channel plates, as well as decrease the amount of friction. If the stress is decrease, due to their removal, then the force in turn against the walls of the die will increase, lowering the friction. The gradual widening of the channel was completed by flaring the upper and lower surfaces of the die a total of five thousands from beginning to end of the exit channel. This magnitude of flaring was determined to allow the full elastic strain to be released halfway through the exit channel.

The entire die, in general is 9"x9"9.5" tool steel die, as these dimensions fit into the criteria needed to use the hydraulic press, which will be discussed at a later point. The die is designed in a modular fashion in which the channel is created by the use of three separate plates, the central plate contains the major die geometry characteristics, the inner and outer angles and radii. This includes a bottom section of this plate in an "L-shape" and a corner section, this can be seen in Figure B-24 of the appendix. The center plate sections are capped on either side with two plates that create the side walls of the channel. To ensure that the two separate sections that create the center plate remain in the proper relation between one another, a set of pins and sleeves are used. The pins and sleeves are pressed into the stack of three plates, the holes of which are machined with high degree of precision using EDM machining techniques, in which 9 strategically placed pins are used and along with four sleeves. The sleeves are placed in 4 strategic holes, that are also allow for bolts to pass through them. In this configuration, the pin and sleeves are really
the source of strength of the die during pressing. As the billet is shearing and moving through the die, through Poisson's ratio, exerts a force against the die walls. This force therefore is transferred on to the pins and sleeves that connect the three inner plates. It is therefore important that these pins and sleeves be made out of high strength material. The pins are 3/8" center-less ground to within half a thousand and made from M42 tool steel, with a hardness of approximately 66HRC. The benefit of this material is that it retains this hardness at high temperatures. The sleeves where machined from annealed A2 tool steel, heat treated to a hardness of 62-63HRC and then ground to their final dimensions. The precision machining of the pins and sleeves are of great importance as any shifting of the center plates during pressing, would therefore change the geometry of the channel, and the resultant material would not be consistent from billet to billet. Also any shifting of these plates can cause a failure during pressing. Outside of the three plates that create the die channel, two 4-inch thick A2 tool steel blocks, of the same 9"x9" width and height of the three center plates, are used as bulk strength in the die assembly. These blocks allow for the stress that is generated during pressing within the channel and on the two capping channel plates, to be transferred out of those plates and into the large bulk material. This causes the two side plates of the channel, to remaining rigid and not bulge outward from the processing loads. Each plate of the die, was received in the annealed state, machined either using standard CNC or EDM methods depending on the tolerance and critical nature of the machining needed. All the plates' where then heat treated to an HRC of approximately 62-63, and subsequently surface ground on the mating surfaces. The surface grinding was essential to ensure that all plates where in perfect contact and square, so all stresses could be efficiently transferred.
The material that the die is to consist of is a critical design criteria in the survivability of the die. The material must be resilient to wear, able to withstand high loads and elevated temperatures, as well as easily machine-able. Through a literature search of other ECAP systems [26-29], it could be seen that tool steel has proven to be a suitable material for this application. Utilizing a 2D Abaqus simulation and material properties obtained for the D2 tool steel [30,31], simulations were completed to investigate whether the material would withstand the high loads applied to the die during an ECAP pressing. A schematic of this simulation can be seen below, in which all pins, sleeves and bolt holes were included, as these would create stress concentrations, a source of cracks during loading. Within the simulation a static distributed load is applied on the channel surfaces facing outward, with a magnitude equivalent to the relationship of the plastic Poisson's ratio of the billet material and the load applied by the plunger during an ECAP pressing. This loading results in a distributed load of 80ksi on each channel wall, or equivalent to the case in which 20Ton of force is applied to the specimen. This is worst case scenario, as can be seen from in the previous section of this appendix, the highest load expected with a high coefficient of friction would be approximately 10Tons. Also the entire surfaces of the die would not be loaded as in this case, the load would only be a pressure exerted on the channel walls from the length of the specimen, approximately 4-5 inches. A Von-Mises stress-contour plot of the result of this loading can be seen in Figure B-14, in which it can be seen that the resultant stresses do not exceed the yield stress of the die material (300ksi), thus tool steel is a valid material to be used. There results a safety factor of 1.3 even in this worst case scenario. Also from this analysis it can be seen that the placement of the pins and sleeves will not
affect the survivability of the die material, all though as expected, the pins and sleeves do act as sources of stress concentrations.

Another design consideration is the torque to be applied of the 6 grade 8 steel bolts that hold the die together, which includes the three inner plates that create the channel and the two four inch bulk material blocks. The purpose of these bolts is not to only hold these plates in place during a pressing, but also to place the die into a state of compression. The added state of compression, effectively adds an increase of load that the die plates can withstand, as the outward force generated during pressing must overcome the applied compressive load of the bolts before the load would be applied onto the plates of the die. Any bulging outward of these side plates would result in die separation, thus the billet material would extrude through these gaps. It was determined that a torque of 600ft-lb would be sufficient not to allow any plate separation, while also not exciding the bolt strength [28]. The use of high pressure/temperature anti-seize was used on each bolt and nut to ensure ease of disassembly as well as limiting the friction during the torque being applied.
Figure B-14: Von-Mises stress contour plot of a 2D simulated ECAP die under pressure loads on both channel walls of 80ksi, in which die is modeled using D2 tool steel material properties, pin are modeled using M42 tool steel material properties and sleeves are modeled using A2 tool steel material properties
B.2.2.3 Plunger Elevator System

A plunger elevator system is used to precisely align the plunger with the die channel. This is important as any miss-alignment of the tight fitting plunger with respect to the die dimensions, will result in failure of the plunger. The systems consists of several different components which include a base plate that bolts to the top of the die, a center plate in which the plunger is bolted and a top plate that allows for the passing of the ram through this system and also a serves as a fixing point for the four rods that connect these three plates together. These rods serve as a track that the center plate can move up and down freely upon as the press ram applies a load during pressing. The elevator system can be seen in Figure B-15. The height of this system is also of importance as, it needs to have the ability to have the plunger moved out of position while the specimen is inserted into the die and then move back into pressing position once this done. Also the plunger must be long enough to move the biller entirely through the die per pass. From this is was determined that the plunger would be approximately 5 inches long, and the elevator would be approximately 12 inches high to allow for ample room for the 4-5inch specimen to be inserted.
Figure B-15: Plunger and Plunger elevator used in the ECAP system. Parts include the base, center and top plates, alignment rods and tool steel plunger. This system allows for proper vertical motion of the plunger as it transfers load from the ram head into the plunger then onto the specimen.
The plunger directly applies all the load on top of the billet. Therefore the plunger must be extremely strong and precisely machined. The plunger is made from the same material as the die, therefore it will not have the ability to gouge or damage the die surface, as it would be able to if it was a harder material. It is therefore machined from annealed D2 tool steel to rough dimensions, heat treated to 62-63HRC and then surface ground to its final dimensions. The final dimensions are 0.495" +/- 0.0005" square. This allows for a minimal gap between the plunger and the die channel, in which to large of gap would allow material to extrude between the die and plunger. If this occurs it can cause failure of the system, as it would generate an extreme increase in load to move the billet, essentially binding the system. The plunger is bolted to the center plate of this elevator system. This center plate has four brass bearing pressing into each corner hole, that are lubricated and allows for smooth movement along the 1" ground rods. These bearings are initial pressed into the holes and undersized. They are then reamed to the precise dimension, therefore not allowing any wobble of this plate on the support/tracking rods, retaining its proper alignment with the die. The plates are machined from high strength 4041 chrome-moly steel. A high degree of care was taken to have these machined precisely using CNC methods so that the entire system would move as designed with the movement of the center plate being smooth and straight, with no binding of the center plate.

B.2.2.4 Hydraulic Press

The choice of a loading applying system is critical in the design of an ECAP system. Several design criteria include the maximum applicable load, the size of the workable area, the ram extension and the rate at which the load can be applied. It was
decided the most effect method would be to purchase a commercially available press. Through the design of the die and the size of the press is somewhat an interrelated problem, as the die cannot be to wide or tall to fit on the load applying apparatus, but yet the die has to be large enough to withstand the high loads experienced during pressing. Also to be able to have the die in place, insert a specimen into the die from the top and then have the plunger move into position to begin the pressing process, the press would need a relatively long stroke. From all these criteria it was determined that the Dake 100 ton hydraulic press would fit all the criteria needed, and can be seen in Figure B-16.

The hydraulic press used in the ECAP system is capable of applying 100 Tons (5074Psi) of pressure. This press has an adjustable table with a wide open base, suitable to be worked around. The press has a maximum ram travel of 19" which was very desirable as it gives the ability to assemble the die without the ram being in the way, but also is capable of extending enough to extrude the billet. It has a rapid advance speed of 52 inches per minutes and maximum pressing rate of 3 inches per minute. The rate of pressing can be varied by the used by use of a lever that when compressed further will move the ram with increasing rate, and when direction of this lever is reversed, the press will move in the opposite direction. The system comes with an analog pressure gauge, which is upgraded to an electronic pressure transducer to be used in the data acquisition system, which will be discussed later. All operating considerations of this system should refer to the operating manual, which should be reviewed before any new user of the system.
Figure B-16: 100 Ton hydraulic press utilized in the ECAP system in which major components include the pressing table which serves a mounting and support of the ECAP die, the rigid frame, the 19” stroke ram, and the control box and ram control lever.
Many researchers have shown that it is very beneficial that during the pressing process to have the die and billet at an elevated temperature, the exact temperature being material dependent [27]. The benefits include that having the die and billet at elevated temperatures makes the pressing “easier” with the fact that the shear yield strength will decrease with an increase in temperature. This is not the only result of heating the system; the final microstructure of the material will be affected by pressing at elevated temperatures. The temperature has been shown to, at above a certain experimentally determined material dependent level, will result in recovery of the grain size, thus not as efficiently refining the material. If the temperature of the system is to far below a certain processing temperature, the resulting microstructure will be in a non-equilibrated state, thus not allowing the material to be at its highest possible strength as well as having the possibility of cracking during processing. This important factor of the ECAP processing system, heating of the die and billet.

Design considerations such as the amount of heaters, wattage and the length of time at which it will take for the die to arrive at its desired, element size and placement are to be considered in the usage of the cartridge heaters. In order to solve the problem presented, a manufacturing specification and design handbook is utilized. To validate the manufacturing suggestions, a FEA model is established and the heating of the die is modeled.

Several different techniques can be found within literature on how to accurately heat the ECAP system. A. Yamashita et al and G. Colombo [27,32] utilized furnace
techniques in which the entire die was placed in a furnace heated by heating coils embedded within ceramic, as shown in Figure B-17a. Such methods, although valid and functional, can be seen as inefficient, with increased sources of heat loss by indirectly heating, heating the environment in which the die is placed. More efficient methods include the use of heating bands and cartridge heaters, such as in the work of J. Werenskiold [12], seen in Figure B-17b, in which the cartridge heaters are directly inserted into the ECAP die.

From the work of J. Werenskoid, it was concluded that the use of the cartridge heaters was a very accurate method of heating and controlling the temperature of the ECAP system. The benefit of utilizing cartridge heaters can be explained by the ability to have the source of heat be directly in contact with the material to be heated, where no loss of energy would occur, say from convection between the air and the heat source and then the air and the die. Another great benefit of cartridge heaters is the high level of wattage available commercially, this is desirable to increase initial heating time and ability to remain at a constant temperature. The manner in which the die is to be heated in this case is through the use of two heater plates which include the inserted cartridge heaters. These heater plates are fixed to two outer surfaces of the die to apply even heating to both sides.
Figure B-17: Examples of ECAP Die heating Techniques a) ECAP die is inserted into furnace using heating coils imbedded in ceramic b) ECAP die heated using cartridge heaters inserted directly into die
In any system that is to be heated or is within the process of heat transfer, a major area of interest is the temperature distribution through the system whether in the steady state or transient condition. Due to the complexity of the system it would be difficult to determine an exact closed form solution of the temperature distribution through the material. Therefore the manufacture has provided a set of equations to determine the amount of wattage necessary to heat the given material to a desired temperature within a certain amount of time. The predicted results based on these equations are presented below, these results will be compared to an FEA model generated using the Abaqus platform.

The manufacture of the cartridge heaters have provided a selection guide to aid in design and specifications of cartridge heater utilization, the Omegalux Cartridge Heat Selection Guide [33]. Within this guide an equation for determining the amount of wattage necessary is given in equation B-9,

\[ P = \frac{W_T C_p \Delta T}{(3412)H} \]  

(B-9)

where \( W_T \) is the weight of the material to be heated, \( C_p \) is the specific heat of the material, \( \Delta T \) is the change in temperature from initial to desired final temperature, in this case it is desired that the die would heat from room temperature to 350C, 3412 is a conversion factor from US units of BTU-lb-F to SI units of kW, and \( H \) being the amount of time to reach final desired temperature. From refs [30,31] the material properties used in the analysis are given in Table B-1. Using these material properties and the equation B-9, the amount of wattage necessary to heat the die within 2 hours can be determined to
be approximately 4KW with a safety factor of 2. This safety factor is used to include any heat lose effects.

To achieve the 4kW of power necessary to heat the die, four 1kW cartridge heaters will be used, based on Omegas available heaters, it was determined that the cartridge heaters would be 3/8” in diameter and 5” long. With predicted values of temperature and time based on the input wattage, an FEA model can be generated and compared to ensure the proper selection of cartridge heaters is made.

**Table B-1:** Material Properties of A2 and D2 tool steel used in Abaqus heat transfer FEA model

|                          | A2 Tool Steel | D2 Tool Steel |
|--------------------------|---------------|---------------|
| Thermal Conductivity (W/m-K) | 26            | 20            |
| Specific Heat J/Kg-K     | 460           | 460           |
| Density (kg/m³)          | 7750          | 7700          |

The Abaqus FEA platform is utilized to model the heating of a simplified ECAP die. As in any heat transfer problem a set of boundary conditions must be defined which also includes the loading conditions, in this case the cartridge heater output or heat flux. For simplification all exterior boundaries are seen to be insulated, the default setting for Abaqus heat transfer models, unless overridden by another boundary condition.

In order to simulate the transient process of heating the die, an initial step and loading steps are created. The loading step designated Step-1, this a heat transfer transient step that will simulate constant heating from the cartridge heaters for a time period of 7200s or 2 hrs. The loading step will include increments that automatically change during the duration of the simulation, but have the initial conditions of 0.1secs and the minimum
and maximum set to 1E-5 and 100 seconds respectfully. In order to model the interaction between the surfaces of the plates as they are assembled in real world application, contact properties are set between each matting surface, plate to plate, with some assumptions made. Each contact pair has the properties of the tangential behavior being frictionless, normal behavior being as hard contact and for thermal conductance between the surfaces, the contact is represented by having perfect conductance between surfaces. This last contact property, perfect thermal conductance, is an assumption that can be seen as valid for several reasons, being that in real world application, the mating surfaces are surface ground to ensure that each surface will be completely parallel with in a thousandth of an inch and have a smooth finish. Also the die itself is to be bolted together with each bolt being under a prescribed torque to ensure that no separation between the plates will occur.

Within the initial step mentioned previously, a predefined field must be generated. This pre-defined field is set as a temperature, simulating that the die begins its heating process in room temperature condition. The heat transfer transient step, Step-1, is where the loading condition is applied within four cylindrical holes representing the cartridge heaters. The chosen cartridge heaters to fit the application has a total wattage of 1000W/cartridge heater or a watt density of 170W/in^2. This loading has been applied to the parametrical surfaces of the cylindrical hole, not including the bottom surface as the cartridge does not supply heating power to this surface. This loading condition makes the assumption that all power supplied from the cartridge heater is perfectly transferred into the heater plates, this is valid as the cartridge heaters have a tight fit within these plates. The transient step is also were any other boundary conditions would be set, in this case
since any other forms of heat transfer are not considered, the outer surfaces of the die will be seen as insulated. This is a valid assumption because the actual setup will include an insulation system built around the entire die that should limit these losses.

The meshing used is a tetrahedral free type of mesh, with the element type being standard, linear heat transfer element, DC3D4, meaning it is a four node linear heat transfer tetrahedron. The Abaqus simulation was run to model and simulate the heating of the tool steel ECAP die, heated by 4-1000W cartridge heaters from the initial state of 20°C or room temperature for duration of 2 hours. The results in the form of a temperature contour plot can be seen in Figure B-19, in which the maximum temperature after 2 hours of this constant heating was found to be 835°C located at the source of the heat as would be expected.

The temperature contour plot results are as expected, noting the symmetry between either half of the die, which represents even heating and proper transfer of heat flux from the heating plates, where heating occurs moving into the center of the die. As expected the source of the heating, the cartridge heaters, are the positions that have the highest temperature. The following plot, Figure B-19, is a temperature versus time plot of two different positions of the die. The first being located on the exterior of the die, close to the cartridge heater, and the other being located in the interior of the die as shown in figure 8, where the channel in the ECAP die would fall.
**Figure B-18:** Nodal temperature contour plot resultant from Abaqus simulation in which the use of 4-1000W cartridge heaters are simulated in the positions as shown, left showing the entire die assembly and on the right showing the interior heating of the die. Temperature is given in Kelvin.

**Figure B-19:** Plot the temperature (°C) versus time obtained from FEA Abaqus heating simulation using 4-1000W cartridge heater sources for an interior and exterior positions. These positions are chosen due to the positions of the thermocouples that will be placed in these same positions.
These two positions will be monitored during actually pressing using thermocouple probes inserted into the die, therefore it is desirable to know the simulation results at this point. The experimental temperature for a pressing is considered the temperature at the interior point, as this will be the temperature in which the billet will be experiencing.

B.2.2.6 Data Acquisition

The acquisition of data during an ECAP test is important to be able to refer back to, and establish a consistent production of refined granular material, it was noted that there was seemingly lack of this type of data in literature. There are several experimental variables of interest to collect and analyze, which include the pressing force, the rate of pressing and the temperature at which the billet was produced at, previously discussed. In order to facilitate the data acquisition several apparatuses where added to the ECAP system. First a pressure transducer was implemented directly into the hydraulic lines of the press, used in conjunction with the analog gauge that was already present. The pressure transducer gives the ability to measure the pressure within the hydraulic lines and to be recorded digitally. From the hydraulic pressure it possible to make a relation to the amount of tonnage being applied on the billet top surface, and therefore the ability to generate this over the duration of the entire pressing. To measure the rate of the pressing, a linear variable differential transformer (LVDT) was used. This gave the ability to measure the displacement of the ram head, and utilizing the duration that the displacement occurs within, one can measure the rate simultaneously. The data was acquired using a Matlab data acquisition system, denoted Softscope. This built-in coding essentially acts as an oscilloscope. The benefit of this system is that multiple plots or
channels can be generated for each data set measured versus the same time domain, as can be seen in Figure B-21. Not only does it have this feature, but also it has the ability to take the data measured in one channel and convert it using a user defined equation, and thus generate a new channel or plot. This was utilized to convert the pressure transducer and LVDT raw data, both of which are initially in the form of a voltage. Using the provided conversions for the pressure transducer and LVDT from the manufactures, the data was converted into pressure measured in Psi and displacement in inches respectfully. The data of the pressure transducer was further converted into tonnage by utilizing the surface area at which the hydraulic pressure is applied, or simply the circular surface area of the ram head. The data of the displacement from the LVDT can also be further converted into rate. This is done by simultaneously and continuously calculating the difference between a block of displacement data, in this case 25pts, and dividing this by the total duration the block of data is collected at, based on the sampling rate of 5Hz. This gives the ability to receive the real-time pressing rate.

An apparatus was designed to adapt the LVDT for the ECAP used. This was done by adding a plate onto the end of the press ram that would receive an arm that would stick out perpendicular to the motion of the ram, at the end of this arm or rod, the LDVT was securely mounted. Also a table with another arm was fixed to the press frame. The arm attached to this table gave a position for which the LVDT could press against and compress as the ram was moved down during pressing. A figure of this can be seen in Figure B-22.
Figure B-20: Oscilloscope function utilized in Matlab, Softscope, for data acquisition, in which real-time data is collected during ECAP pressings. Data includes raw data of the pressure transducer and LVDT, determined displacement from LVDT data, determined hydraulic press from pressure transducer data and determined resultant tonnage from the hydraulic pressure. Also outputted measurements of pressing rate can be made, as shown in the measurements module of the figure.
Figure B-21: LVDT apparatus used to collect real-time displacement and rate data, in which major components include the LVDT, LVDT mound, LVDT mound arm, LVDT table and LVDT table arm.
B.2.3 Mechanical Drawings

This appendix section contains all drawings generated for the design and manufacture of the SHPB at the MMRL facilities.
Figure B-22: Schematic of the ECAP system utilized in this work. The schematic shows the three inner plates that create the channel, the outer tool steel plates that add strength to the die, the 7 bolts, that are torqued to keep the die plates from separating during pressing and the elevator apparatus used to align the plunger and die.
Figure B-23: 95° channel used in the ECAP system, this generates the channel used to deform the material for each pass. Plates of D2 tool steel were received oversized and in annealed state, at which the holes were drilled and honed. Then the plate was heat treated to a HRC 62-63, surfaced ground to the final thickness, and finally the channel geometry was machined using EDM processes.
Figure B-24: Side plates used to cap the outer sides to generate the full 3D channel of the ECAP system. Plates of D2 tool steel were received oversized and in annealed state, at which the holes were drilled and honed. Then the plate was heat treated to a HRC 62-63 and then surfaced ground to the final thickness.
Figure B-25: Bulk material used on either side of the ECAP inner plates that form the channel. These two plates add strength to the ECAP system, in order to be able to withstand the extreme loads. Plates of A2 tool steel were received oversized and in annealed state, at which the holes were drilled and honed. Then the plate was heat treated to a HRC 62-63 and then surfaced ground to the final thickness.
Figure B-26: Mechanical drawing of sleeves used to connect the three inner plates of the ECAP die as well as serving a structural and alignment member of the inner plates generating the die channel
Figure B-27: Schematic of the plunger elevator system, utilized to move the plunger into the die during pressing, ensuring that the alignment between the die and plunger remains constant for every pressing.
Figure B-28: Base plate of the elevator plunger system. This plate allows for the attachment of the elevator to the die itself, while also serving as support for the rods align the plunger system.
Figure B-29: Plunger plate used in the elevator system. The plunger is bolted to this plate, fixing its position. This plate has four brass slider bearings pressed into the plate allowing for the plate to ride on four rods during pressing.
Figure B-30: Top plate of the elevator system, which supports the rods that allow the motion of the plunger plate, as well as allow the press head to move into the elevator system.
Figure B-31: Alignment bars used in the elevator system. These rods are used to maintain the alignment of the plunger throughout the pressing, while also acting as a structural system of the elevator apparatus.
**Figure B-32:** The plunger shown is used to apply force to the specimen at a constant rate during ECAP pressing. The material of the plunger is annealed D2 tool steel, that was received over sized. The material was then machined oversized in the thickness and length, heat treated to a hardness of HRC 62-63, then surface ground to the final dimensions. The tolerances of the plunger is critical, not allowing any "blow-by" of the specimen material during pressing.
Figure B-33: The LVDT table shown, is used in the data acquisition system as a manner to mount a stable platform to measure displacement of the plunger using a LVDT.
**Figure B-34:** LVDT table arm which is attached to the LVDT table to create solid structure for which the LVDT to be pressed against during a ECAP pressing
**Figure B-35:** The LVDT mount is attached to the ram of the ECAP press, and serves as a manner in which to attach the LVDT to this ram
Figure B-36: This LVDT mount connects to the LVDT mount rod and holds the LVDT in place during an ECAP pressing.
Figure B-37: The heater plate shown in this figure is used on the bold head side of the die. This plate includes two 1000W cartridge heaters that are pressed into the plate. When the heaters are turned on, heat is transferred from the cartridge heater into the aluminum heater plate and then the heat is evenly distributed into the full face of the die.
Figure B-38: The heater plate shown in this figure is used on the bold head side of the die. This plate includes two 1000W cartridge heaters that are pressed into the plate. When the heaters are turned on, heat is transferred from the cartridge heater into the aluminum heater plate and then the heat is evenly distributed into the full face of the die.
B.2.4 Testing, Data Processing and Software

This appendix section contains the description of the experimental procedure for utilizing the equal channel angular press for generation of fine and ultra-fine grain material. This include specimen preparation, testing procedure and data processing. Data acquisition using the Matlab softscope oscilloscope code, with data collected from the pressure transducer and LVDT. An example of the typical experimental results is given.

B.2.4.1 Experimental Procedure

The procedure utilized during a experimental ECAP setup and testing is presented in the following section. The importance of following this procedure is to ensure repeatability and accuracy of each test. The specimen preparation, experimental and equipment setup, and data capture will be discussed in detail.

The assembly of the ECAP system begins with the preparation of the channel plates. This includes the painting of high pressure/temperature lubricant on each of the die channel surfaces as well on the specimen surfaces, the specifics of the specimen preparation before lubrication will be discussed shortly. Ample time for this lubricate to dry should be given, from 1-6hrs depending on environmental conditions (temperature and humidity). The lubrication of these surfaces is of great importance as the resultant material produced and as well as the survivability of the system during the pressing, as shown in this appendix, is a function of the resultant friction during pressing. The frictional effects have a great deal of effect on the deformation uniformity and amount of tonnage necessary to press the billet. Next the 9-M42 tool steel pins and the 4-A2 tool steel sleeves should be pressed into their appropriate positions of the three plates that
make the die channel. It is important to use an arbor press, as opposed to other methods such as hammering. The hammering can shock the brittle pins causing micro-cracks, that will lead to premature failure of the pins or sleeves during pressing. These three plates should not placed between the 2-4" tool steel blocks, noting that the edges on each face as aligned. All the plates as well as the mounting plates, that mount and fix the die to the press table should be lightly pressed together to prepare for the placing of the 6-1"diameter-12" long bolts. When bolts are placed into their positions, anti-seize should be heavily placed on the threaded end as well as the contact surface between the head of the bolt and die. The application of the anti-seize is importance in the ease of disassembly after each pass of the billet, as well as lowering the friction between the matting surface so the proper torque can be applied. Bolts and nuts should first be hand tightened, with the tightening of the mounting bolts to the die table. Now each bolt can be torqued in a star pattern, first to 300ft-lbs, then the final torque of 600ft-lbs. The star pattern and stepped increase of the torque aids the even application of the torque and proper alignment of the plates. The die assembly is now complete and the assembly of the other systems upon the die can begin.

Next in the assembly of the ECAP system is the plunger elevator. Before this subsystem can be attached and aligned to the die, proper function of the subsystem should be checked. First the plungers attachment to the center plate should be checked, for any movement within this plate, if this exists, the bolt used to attach the plunger should be tightened and then rechecked. The center plate should freely slide along the 4-1"diametre rods, and lubrication can be applied to aid in this motion. The subsystem can then be placed upon the die and the bolts used to attach the elevator to the die lightly
hand tightened. The alignment of the plunger to the channel is of great importance, as any miss alignment can cause catastrophic failure of the plunger or die channels. The alignment can be completed by slowly sliding the plunger in and out the die, once a position that feel smooth, the attaching bolts should be slightly further tightened in a X-pattern, the alignment checked again and this process continued until the bolts can no longer be tightened and the plunger has free and smooth movement in and out of the die.

The next step is to attach the two heater plates, noting that each is machined specifically depending on the nut or head side of the bolts used to torque the die. The mounting bolts used to mount the die to the press table must be removed to place the heater plates onto the die. Proper contact with the die surface should be checked. Three straps are used to pull the heater plates tightly onto the die faces. The mounting bolts can now be placed back into their proper position, but not tightened as the alignment of the die and elevator should now be made with the press ram. This can be done by carefully moving the ram head close to the elevator, a feel gauge should be used to assure that the ram head is aligned in the hold atop of the plunger elevator, with the entire die and plunger elevator being shifted to make adjustments, once the alignment as be corrected, the mounting bolts can be tightened and alignment check a final time. The two thermocouple wires can be threaded into place and checked to be functioning correctly, by powering the heating system, which should at this point be reading room temperature. The insulation system can then be placed around the die.

The ram head can be moved down beyond the top plate of the plunger elevator system so that the LVDT mounting arm can be threaded into the ram head. Once threaded into place the LVDT can be mounting into this apparatus, and the LVDT table arm placed
into position to make contact with the LVDT. The power supply that powers both the LVDT and pressure transducer must be powered using 120V AC - 12V DC adapter located behind the ECAP system. The data acquisition system can be opened by powering the supplied computer, and accessing Matlab and using the `softscope2('mcc.si')` function, which has been configured for the ECAP tests. Just before testing begins, the trigger button can begin and data acquisition will begin. The data acquisition and analysis will be discussed in the following section.

The specimen preparation is of great importance as it will affect the resultant material generated. Important factors include tolerance of fit into the die and the surface finish and proper lubrication, both of which will affect the friction between the specimen and die during pressing. The specimen must precisely be machined to the square dimensions of 0.500-0.495". This allows for a tight fit within the die, if specimen is at a smaller dimension then 0.495", the adverse effects such as cracking will occur. The surface of the specimen should be sanded to a high grit to remove any machining defects, and reduce friction. The specimen as mentioned must be fully coated with high pressure/temperature lubricant which is allowed to dry adequately. Each of these steps for the specimen preparation must be completed for each pressing through the die. After a pressing the ends must be machined to remove any deformation from the previous pressing, and return to a square configuration, as which was shown to be the most beneficial geometry for the ECAP pressing. The specimen can then be placed into the die, and the plunger slide into position to move the billet into its initial position, just before the radius of the channel.
With all components in proper alignment and specimen within the die, the heating process can begin. This is completed by powering the heating control unit, and utilizing the thermocouple closest to the heat plates as the feed-back controlling measurement, the desired temperature can be set and heating will begin. Depending on the temperature desired, this can take from 1.5-4-hours. Once the desired temperature has been reach the ECAP system can be utilized to press the billet and extrude it through the die. A secondary specimen, placed behind the first may be utilized to attain full extrusion of the primary billet, in order to attain the most extruded material as possible. With the test complete, heating system should be turned off and the entire system allowed to cool before disassembly.

**B.2.4.2 Data Analysis**

Utilizing the Matlab program and the built in softscope oscilloscope code, data is collected during the ECAP pressing. The program has been explained previously in this appendix, so this section will deal with a secondary program that converts the raw data collected and generates an excel and text file to be saved and analyzed at a later point. Once the press has be completed, using the file drop down menu, the captured data can be saved to the command window of Matlab. With the data now sent to the command window, a second program, ECAP_data_processing.m, can be utilized. This program creates a text file of the collected data, and also generates an excel file, both of which have the appropriate column labeling. The data can then be analyzed to determine the rate of pressing, total displacement and a force-time plot, examples of which will be shown in the following section. The data processing Matlab code is given as follows.
% ECAP Data Processing
% Justin Spirdione
% Nov. 2013

% This program acquired using softscope2('mcc.si') during ECAP testing
% Run this program after ECAP test, or using saved MAT-file from softscope

% Variable designation
% c0 - channel 0 - LVDT Voltage Data
% c1 - channel 1 - Pressure Transducer (PT) Voltage Data
% c3 - Math function - converts channel 0 data from LVDT into
% Displacement(in) - using calibration curve
% c2 - Math function - converts channel 1 data from PT into Pressure (PSI) -
% using calibration curve given from omega engineering
% c4 - Math Function - - converts c2(pressure in psi) into tonage, given
% the diameter of the ram

c0=c0';
c1=c1';
c2=c2';
c3=c3';
c4=c4';
displacement=c2;
displacement=c2-c2(1);
pressure=c3;
ton=c4;

% Calculation of Time using sampling rate
samplerate=5;  % Input sample rate used during test
sample_time=1/samplerate;
duration=(length(c0)-1)/samplerate;
time=[0:sample_time:duration];
time=time';

datae = [time, c0, c1, displacement, pressure, ton];
filename1='ECAP_test_1';
% Adds filetype .txt to filename so will save as a text file
filename1=strcat(filename1,'.txt');
% Formatting of Text File Header (title and column headers)
fid1=fopen(filename1,'w');
fprintf(fid1,'ECAP Test
');
fprintf(fid1,'Time(s)	LVDT(Volts)	PT(Volts)	Displacement(in)	Pressure(PSI)	Tons(Ton)
');
fprintf(fid1,'
');
fprintf(fid1,'%-4.4f\t%-4.4f\t%-4.4f\t%-4.4f\t%-4.4f\n',datae);
fclose(fid1);
header = { 'Time(s)', 'LVDT(Volts)', 'PT(Volts)', 'Displacement(in)', 'Pressure(PSI)', 'Ton(Tons)' };
name = { 'ECAP TEST' };
datew=datestr(now, 1);
sample_rate={ 'Sample Rate (Hz)' };
xlswrite('ECAP_Test.xls', name, 'TEST 1','A1');
xlswrite('ECAP_Test.xls', header,'TEST 1','A3');
xlswrite('ECAP_Test.xls', datae,'TEST 1','A4');
xlswrite('ECAP_Test.xls', sample_rate, 'TEST 1','A2');
xlswrite('ECAP_Test.xls', samplerate, 'TEST 1','B2');
xlswrite('ECAP_Test.xls', datew, 'TEST 1','B1');

B.2.4.3 Typical Experimental Results

An example of typical experimental results generated during the processing of an annealed Al6061 ECAP specimen for a single pass is presented. The data collected using the data acquisition system, as well as photographs of the resultant billets after pressing will be shown. Figure B-40 shows the force over the time during the pressing, collected from the pressure transducer, noting the rise to a maximum force of approximately 5 Tons, and then which this load continues through the duration of the pressing.

The displacement as a function of time during the pressing is shown in Figure B-41. Of importance to note from this figure is the linearity of the data, in which this shows that the specimen was pressed at a constant rate, determined to be approximately 1in/min. Typical results show similar force and displacement attributes. It is important to record and collect this data so it is assured that each pass is similar in nature in terms of rate of pressing, that has shown to have an effect of the material generated. Also this
establishes an load to press that should be noted, so if there is a sudden increase in load, it can be assumed that something may be occurring that is undesirable and should be checked, such as plate separation where the specimen extrudes between the die plates or specimen blow by where the specimen extrudes around the plunger. Figure B-42 shows the specimen the resultant specimen after subsequent pressing through the ECAP die for a total of 4 passes using Route Bc.

After the first pass it can be seen that deformation at the leading surface (right) and the end surface (left) is not uniform in nature, and will have to be machined to return to the square geometry. The center bulk of the specimen shows uniformity throughout. This shows that the specimen was experiencing uniform deformation during this period, and this is therefore the useable material for the next pass. The effect of the leading and tail ends can be seen for each subsequent pass, thus the useable material becomes shorter with each pass, until the result of the last pass, Figure B-42d, there remains approximately 1.25" of material. Also noted from each of these figures is the lack of any cracking or undesirable surface effects. Cracking upon the surface can lead to failure with further pressing.
Figure B-39: Plot of experimentally acquired force versus time for the first pass of an annealed Al6061 material through the ECAP, in which data of the force is captured using a pressure transducer.
Figure B-40: Plot of the experimental acquired data of the displacement versus time for the first pass of an annealed Al6061 material through the ECAP, in which the displacement is captured using an LVDT
Figure B-41: Resultant photos of an Al6061 specimen as it is processed through the ECAP system using processing Route Bc. a) first pass, b) second pass, c) third pass and d) final pass
B.3 References

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