Factor screening experiments using fractional factorial split plot designs and regression analysis in developing a top-down nanomanufacturing system for recycling of welding rod residuals

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ABSTRACT
A promising technology for integration of top-down nanomanufacturing is equal channel angular pressing, a process transforming metallic materials into nanostructured or ultra-fine grained materials with significantly enhanced performance characteristics. To bridge the gap between process potential and actual manufacturing output a novel prototype system identified as indexing equal channel angular pressing (IX-ECAP) was developed to capitalize on sustainable engineering opportunities of transforming spent or scrap engineering elements into key engineering commodities by recycling 4043 aluminium alloy welding rod residuals. A resolution III fractional factorial split-plot experiment assessed significance of predictors on the response, microhardness, with multiple linear regression used for model development. Five process parameters involving pressing temperature, number of passes, pressing speed, back pressure, and vibration were studied. Microhardness conversions allowed theoretical determination of grain sizes employing Hall-Petch relationships. IX-ECAP offered a viable solution whereby processing of discrete variable length work pieces proved very successful.

1. Introduction
Materials development, especially metallic systems, has been directed at attaining the highest performance characteristics per unit weight of material. This scenario is especially critical to industries such as aerospace, automotive, rail, and maritime. Each of these respective applications requires high performance materials of lightweight construction (Azushima et al., 2008). A key objective is the attainment of even higher material strength while at the same time retaining excellent ductility. Typically, these two performance characteristics are mutually exclusive whereby increased strength is achieved at the expense of ductility and vice versa. However, with a focus on performance gains from diverse applications of nanotechnology these mutually exclusive restrictions are being challenged and overcome (Valiev, Alexandrov, Zhu, & Lowe, 2002).
One particular method successful in overcoming traditional materials performance barriers is equal channel angular pressing (ECAP). Originally developed by Segal in Russia during the 1970s this process has proven capable of achieving significant improvements in performance characteristics in numerous metallic systems (Segal, 1999). Without altering dimensions of the submitted work piece, for example, metal components have experienced 323% increases in yield strength while retaining elongations of 34% after only one pass through ECAP (Qu et al., 2008). Other novel materials systems such as SiC/Al composites, NiTi shape memory alloys, and titanium dental implants have likewise been investigated for property enhancements employing ECAP (Figueiredo et al., 2014; Qian, Li, & Xue, 2015; Shahmir et al., 2015).

ECAP uses the principle of severe plastic deformation (SPD) to significantly alter the performance of materials, primarily metals. Typical metal working processes such as extrusion apply a force to a billet of larger form of solid metal to push or extrude the work piece through a die to form a smaller, similar, or alternate shaped product form. The traditional ECAP process, however, applies a force to a form of solid metal such as a rectangular or circular cross section to press it through entrance and exit channels of a die with equal cross sections. The metal sample or work piece is inserted into the entrance channel and simply pushed or pressed through the die using a plunger. The intersection angle of the entrance and exit channels in typical applications is 90°. An actual ECAP die is shown in Figure 1.

The ECAP manufacturing process induces SPD in the work piece resulting in grain refinement caused by imposition of simple shear and the response of resultant metallurgical microstructural reactions (Segal, 2004). Instead of flowing through the die the metal is sheared under intense strain as it traverses the turn at the bend of intersecting entrance and exit channels. These intense strains impose SPD in the intersection region subsequently reducing the metal's internal microstructural grain size into the ultra-fine (100–1000 nanometers) or nanoscale (≤100 nanometers) regimes (Zhu, Valiev, Langdon, Tsuji, & Lu, 2010).

ECAP efforts are motivated by a primary objective of reducing the grain size of the internal microstructure. Attainment of this goal affords significant potential material property enhancements prescribed by the Hall-Petch relationship which defines an inverse dependence between a material's grain size and the resulting yield strength at room temperature. As grain size is reduced yield strength of the material increases (Valiev & Langdon, 2006) as follows

\[
\sigma_y = \sigma_o + k_y d^{-1/2}
\]

where \( \sigma_y \) is the material’s yield strength; \( \sigma_o \) is the friction stress; \( k_y \) is a material constant for yielding; \( d \) is the material’s internal grain size.

Throughout years of development several modifications have been introduced to traditional ECAP to overcome inherent limitations of the process. Such prominent design alternatives include rotary die ECAP, con-shearing ECAP, equal channel angular rolling, conform process, and incremental equal channel angular pressing (I-ECAP) (Azushima et al., 2008; Valiev & Langdon, 2006). Even though a primary advantage of each of the processes is simplicity in material flow through the system methods oftentimes require extensive ancillary supporting facilities and equipment.

A process employing both simplicity in functionality and engineering design of supporting facilities and equipment is needed. To warrant further scientific and economic justification an application of the technology towards resolution of a sustainable engineering
problem was investigated. For example, one of the most common methods for joining structural components involves welding. The consequent nature of the welding process itself produces spent electrodes or scrap metal lengths of unused rods. These remaining residuals are typically discarded or recycled as metal scrap. However, implementation of the newly developed indexing equal channel angular pressing (IX-ECAP) process provides a critical alternative in transforming welding scrap into vital engineering materials with mechanical properties of vast potential importance. The feasibility of inducing SPD and subsequent strain hardening within these re-cycled product forms employing IX-ECAP manufacturing methodologies is presented from an initial factor screening perspective.

2. Materials and methods

2.1. Materials – alloy selection and work piece form

Work piece material and form consisted of fully annealed 4043 aluminium 3.175-mm diameter welding rod residuals. Proper sample selection based on metallurgical considerations

Figure 1. (a) Indexing equal channel angular pressing die disassembled after pressing. (b) Die assembled with pressed rod visible at the top. Entrance channel seen as the singular hole on the right die face.
plays a vital role in viability of the proposed manufacturing system as well as contributing towards commercial acceptance of nanostructured materials.

Several factors contributed to the selection of 4043 aluminium for this research. The first consideration is the extension of knowledge to existing research regarding SPD processes producing nanocrystalline materials. SPD using ECAP involves introduction of significant strains within a solid component through mechanical means without subsequent changes in cross-sectional dimensions. Research involving ECAP and cryomilling have provided detailed analyses involving non-ferrous metals such as aluminium and copper (Brochu, Zimmerly, Ajdelsztajn, Lavernia, & Kim, 2007; Haouaoui, 2005; Zhu, Lowe, & Langdon, 2004). Advances in aluminium processing using ECAP methods continue to offer significant opportunities in varied applications such as bolt manufacturing, consolidated nanocomposites, and joining of dissimilar metals (Casati, Vdani, Dellasega, Bassani, & Tuissi, 2015; Gudimetla, Kumar, Ravisandar, & Kumaran, 2015; Jafarlou, Zalnezhad, Ezazi, Mardi, & Hassan, 2015; Jin, Baek, Hwang, Im, & Jeon, 2012; Qian et al., 2015). A continuation of research promoting the transfer of such processes and materials from laboratory to commercialization is needed.

Of foremost importance in selecting alloy 4043 are the inherent properties of the material itself to nanocrystalline propensity and response to plastic deformation. Specifically, aluminium 4043 is a relatively soft metal possessing good ductility making these materials ideal for consideration in mechanical forming operations such as ECAP. The material also possesses inherent properties whereby microstructures remain stable as single-phase alloys from room temperature to elevated temperatures thereby providing continuity within processing operations. From a commercial familiarity perspective, aluminium represents the most available metallic element found on earth (Smith, 1993). Therefore, a commonality with these respective materials within the industrial community further encourages commercial acceptance of IX-ECAP products.

2.2. Facilities and equipment

IX-ECAP represents an alternative approach in the application of SPD processing. Design and development of the IX-ECAP process is an integration of traditional constructs of ECAP, modified ECAP processes, and alternative considerations aimed at overcoming commercialization barriers.

Several key design elements were required to build the prototype unit which included various die components, pressing and clamping force hydraulic systems, thermal system, back pressure components, and vibration elements. Design and development of the manufacturing system involved a focus from two primary perspectives. The first perspective considered the detailed elements comprising the die components through which the metal rods would pass; whereas, the second key emphasis involved the supporting or ancillary equipment providing the drivers and support structures for die components (Hester, 2015). Processing of metal work pieces involved four basic steps:

1. A horizontal stationary plunger rod was secured in a clamping die using a vertically mounted hydraulic cylinder.
2. A welding rod residual was placed into the entrance channel of a die insert secured inside a holding block attached to a pressing die.
(3) The pressing die containing the die insert and die insert holding block was indexed forward against a plunger rod using a horizontally mounted hydraulic cylinder to press the welding rod residual work piece through the system.

(4) The work piece was then removed from the exit channel of the die insert. The process was repeated with application of back pressure, heating, and vibration as required.

The completed manufacturing prototype unit is shown in Figure 2.

Figure 2. Indexing equal channel angular pressing (I₂-ECAP) system.
2.3. Materials characterization

2.3.1. Mechanical properties
An important determinant delineating traditional and nanoscale materials involves an assessment of performance characteristics inherently descriptive of metal components. Within this study standard mechanical properties were measured using Vickers micro-hardness. Test coupons for hardness testing were obtained along the pressed transverse work piece cross sections. Testing was performed in accordance with ASTM E384 Standard Test Method for Microhardness of Materials and E92 Standard Test Method for Vickers Hardness of Metallic Materials (ASTM, 1997a, 1997b).

2.3.2. Grain size
Initial baseline grain sizes were determined for unprocessed samples using optical light microscopy. This technique was used for grain size determinations and general morphology existing within optical resolution capabilities. Cross sections of the unpressed rods were cut for analysis. During sectioning of samples coolant such as water or cutting fluid was employed to ensure samples did not overheat. Metallographic samples were prepared and microstructural assessments were conducted for baseline grain size determination. Length and width grain size measurements were obtained on cross diagonals of microstructures as well as counting grain intercepts to obtain a mean lineal intercept distance to determine an average grain dimension. Final grain sizes for pressed samples were determined theoretically based on application of the Hall-Petch relationship employing known material constants and yield strength conversions from hardness values.

3. Experimental design

3.1. Fractional factorial split-plot
One-quarter fractional factorial split-plot (FFSP) design of experiments (DOEs) using five factors at two levels were conducted to assess statistical significance of processing parameters on the output response, micro hardness. Five predictor variables included:

1. pressing temperature
2. number of passes through the die
3. back pressure
4. pressing speed
5. externally applied vibrational energy

The required number of runs for a full factorial experiment with five factors at two levels (25) is 32 runs. However, based on the constraints associated with randomizing factor level combinations (FLCs) in the \( I_x \)-ECAP process a ¼ FFSP experimental design was selected requiring eight runs. For example, four of the factors were quite easily changed to provide randomization. These included pressing speed, number of passes through the die, back pressure applied to the pressed rod, and vibrational energy. However, pressing temperature was more difficult to change and required stabilization time for thermal equilibrium in the die and work pieces. Therefore, pressing temperature at two treatment levels was randomized within whole plots with the remaining four factors randomized at two levels within sub-plots. Based on available resources in actually running the process, as well as
subsequent product analysis, the split plot ¼ fraction factorial was the final design selection. Three replicates were run in blocks at the whole plot high and low temperatures in order to account for experimental noise resulting in a total of 24 observations.

With IX-ECAP being a new process, a commercial high purity 1100 aluminum alloy in a fully annealed softened condition was run through the complete ¼ FFSP design with three replicates at the whole plot level. The 1100 material served only to substantiate the feasibility of actually running the manufacturing unit safely and successfully producing output rods of acceptable surface integrity. As a result, no statistical analysis was conducted on the 1100 alloy. Assessing process performance involving analysis of variance and regression model development was conducted only for the 4043 material.

For the analysis of variance trials the dependent variable or output response was micro-hardness on the transverse cross section of the 4043 work pieces. Selected microstructures (average grain size) were also analysed. Based on results of experimentation Minitab was used to identify statistical significance at $\alpha = 0.05$. Factor levels studied as well as the experimental design, defining relation, and alias structure are presented in Tables 1 and 2. As noted from the alias structure resulting from a resolution III design main effects are confounded with two-way interactions as well as higher-order interactions. Likewise, two-way interactions are confounded with higher-order interactions.

### 3.2. Multiple linear regression

Regression analysis was performed for model generation. The multiple linear regression model representing the relationship between regressor and response variables is as follows (Montgomery & Runger, 2007):

$$Y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \cdots + \beta_k x_k + \epsilon$$

(2)

where $Y =$ response variable; $\beta =$ regressor coefficients; $x =$ independent regressor variables; $\epsilon =$ random error term.

Based on a consideration of experimental design constraints used in this research the ANOVA regression model was selected due to the complexities of the ¼ FFSP design.

| Table 1. DOE experimental factors and levels for 4043 aluminium alloy. |
|---------------------------------------------------------------|
| **Factor ID** | **Factor description** | **Low level (−1)** | **High level (+1)** |
|-------|------------------|------------------|------------------|
| A     | Pressing temperature (HTC) | 25 °C | 79.4 °C |
| B     | No. of passes | 1 | 3 |
| C     | Back pressure | 0 kg (0 MPa) | 4.536 kg (5.619 MPa) |
| D     | Speed | 25.4 mm/min | 762 mm/min |
| E     | Vibration | 0 cycles/min | 9490 cycles/min |

| Table 2. Fractional factorial split-plot design. |
|-------------------------------------------------|
| **Fraction** | 1/4 | **Defining relation:** | $I = ABCDE = ABE = CDE$ |
| **Resolution** | III | **Alias structure:** | $A + BE + ACDE + BCD$ |
| **No. of factors** | 5 | | $B + AE + BCDE + ACD$ |
| **Hard to change factor** | 1 | | $C + ABCE + DE + ABD$ |
| **Runs** | 24 | | $D + ABDE + CE + ABC$ |
| **Blocks** | 3 | | $E + AB + CD + ABCDE$ |
| **Design generator** | $D = ABCV, E = AB$ | | $AC + BCE + ADE + BD$ |
| **Whole plot generator** | $A$ | | $AD + BDE + ACE + BC$ |
Employing stepwise, forward, or backward variable selection methods would generate efficient regression equations. However, the algorithms would analyse the design as a factorial without delineating the impacts of blocking and randomizations at the whole plot and subplot factor levels. Regression coefficients would be generated; however, $p$-values for main effects would be incorrectly reported. The ANOVA regression equation provided in Minitab® correctly asserts the predictor coefficients and associated $p$-values for the FFSP design.

4. Results and discussion

The development of a top-down nanomanufacturing method transforming traditional materials such as spent or scrap welding rod residuals into nanocrystalline or ultra-fine grain materials for industrial use in specialty markets was investigated. Statistical experimentation (FFSP and multiple linear regression) provided initial factor screening and model development for further process development work.

4.1. FFSP analysis and regression model

A critical prerequisite in running split plot experiments involved planning of time constraints and a determination of available resources. Based on the original plan three replicates were to be conducted for the $1/4$ FFSP design. However, based on time constraints required to complete the eight runs required at the subplot level replicates had to be run on separate days of the week. Therefore, statistical analysis required the use of blocks in terms of days in order to complete the experiment.

To provide options for selection of the final experimental design and run order randomly generated design matrices were generated by Minitab®. The first randomizations occurred for the hard-to-change factor, temperature (A), at the whole plot level with subsequent randomizations generated for subplots within each whole plot for the remaining four easy-to-change factors. Upon review of the possible options the design matrix in Table 3 was selected based on randomized ordering of the temperature factor levels within each block which allowed four runs to be completed at the low temperature setting followed by four runs at the high setting within each block. This design facilitated physical constraints of experimentation for a process such as $I_x$-ECAP which involved thermal heating and cooling of system components while maintaining statistical validation through randomly generated design matrices. Run order, blocks, whole plots, and subplot treatments along with experimental values of the output response variable are provided in the design matrix in Table 3.

With the construction of a newly designed manufacturing prototype a resolution III design provided efficient and economical statistical validation as an initial screening experiment in studying the effects of main process factors. As seen from the alias structure main effects are free from aliasing with other main effects thereby allowing a statistical assessment of the primary factors controlling process performance. However, main effects are confounded with two, three, and higher-order factor interactions some of which could be statistically significant. Additionally, some of the two-factor interactions are confounded with other higher-order interactions. Therefore, subsequent experimentation is an area of future research in validating higher order factor interactions regarding $I_x$-ECAP processing.

Runs were completed based on run order from the design matrix. Each replicate consisting of eight runs, four at the low-temperature level and four at the high-temperature
level with randomizations within sub plots for the remaining four factors, were completed in blocks during different days. Runs were completed in the following sequence:

1. Pressing temperatures were set as defined by the design matrix.
2. Die insert and rod work piece were coated with molybdenum disulfide graphite based high pressure lubricant and dried with forced heated air.
3. Die components were assembled and placed in the pressing unit.
4. Work piece was inserted into the die insert.
5. Vibration level was set as per design matrix.
6. Back pressure level was set as per design matrix.
7. Hydraulic system was activated.
8. Clamping systems were activated.
9. Pressing of the work piece was performed.

For pressings requiring three passes through the system rod work pieces were polished prior to subsequent passes by external surface fine grinding to 600 grit. The procedure was then repeated for each of the respective runs within each block. Representative samples from runs requiring three passes through the system using the 25.4 × 25.4 × 25.4 mm die insert are shown in Figures 3–5.

Upon completion of all experimental runs the rods were sectioned normal to the pressing direction at mid-length and mounted in epoxy sample molds in the transverse and longitudinal directions. Vickers microhardness readings were obtained on all transverse sections of un-pressed and pressed samples; whereas, microstructural analysis was performed on baseline transverse sections. Table 4 provides the Minitab® analysis of variance (ANOVA) table. Statistical significance was determined at the 95% confidence level (α = 0.05).

Table 3. Design matrix and data display for split-plot ¼ fraction factorial.

| Run order | Block | Whole plot | A | B | C | D | E | Response (micro hardness) |
|-----------|-------|------------|---|---|---|---|---|--------------------------|
| 1         | 1     | 1          | − | + | + | − | − | 66                       |
| 2         | 1     | 1          | − | − | − | + | + | 63                       |
| 3         | 1     | 1          | − | + | − | + | − | 65                       |
| 4         | 1     | 1          | − | − | + | + | + | 60                       |
| 5         | 1     | 2          | + | + | − | − | − | 64                       |
| 6         | 1     | 2          | + | + | + | + | + | 60                       |
| 7         | 1     | 2          | + | − | − | + | − | 59                       |
| 8         | 1     | 2          | + | − | + | − | − | 60                       |
| 9         | 2     | 3          | − | + | − | − | + | 67                       |
| 10        | 2     | 3          | − | − | + | + | + | 64                       |
| 11        | 2     | 3          | − | − | − | − | + | 60                       |
| 12        | 2     | 3          | − | + | + | − | − | 66                       |
| 13        | 2     | 4          | + | − | − | + | + | 61                       |
| 14        | 2     | 4          | + | − | + | − | − | 57                       |
| 15        | 2     | 4          | + | + | − | − | + | 59                       |
| 16        | 2     | 4          | + | + | + | + | + | 63                       |
| 17        | 3     | 5          | − | + | + | − | − | 67                       |
| 18        | 3     | 5          | − | − | + | + | + | 61                       |
| 19        | 3     | 5          | − | − | − | − | + | 61                       |
| 20        | 3     | 5          | − | + | − | + | − | 68                       |
| 21        | 3     | 6          | + | + | − | − | + | 61                       |
| 22        | 3     | 6          | + | − | + | − | − | 62                       |
| 23        | 3     | 6          | + | + | + | + | + | 68                       |
| 24        | 3     | 6          | + | − | − | + | + | 61                       |

Notes: A = temperature, B = no. of passes, C = back pressure, D = pressing speed, E = vibration.
Based on a review of the $p$-values one main factor was statistically significant regarding the effect on microhardness. With a $p$-value of $0.001 < 0.05$, main factor B (number of passes through the system) was statistically significant. One very important consideration at the outset of experimental runs involved the possible impact of blocking as the three replicates were conducted over a three-day period. Replicates were performed on sequential days, and based on the $p$-value of $0.429 > 0.05$ the variance contribution from runs on different days as noted by blocks was statistically insignificant. Even though not statistically significant with a $p$-value of $0.086$, the impact of the hard-to-change factor, temperature, is noteworthy. Considering a significance level at $\alpha = 0.10$ temperature would be statistically significant. Plots for predictor variables versus the mean output response, microhardness, illustrate the effect of each of the respective factors as shown in Figure 6.

Regression analysis provided the estimated effects and coefficients for each of the regressor variables in developing a model for prediction of the response variable at factor levels
Figure 5. 4043 aluminium rod after 3 passes.
Note: Design matrix run order #1.

Table 4. ANOVA table for micro hardness (coded units) 4043 alloy.

| Source              | DF | Seq SS  | Adj SS  | Adj MS | F      | p-value |
|---------------------|----|---------|---------|--------|--------|---------|
| Block               | 2  | 12.000  | 12.000  | 6.000  | 1.33   | 0.429   |
| A [HTC] – Temperature| 1  | 45.375  | 45.375  | 45.375 | 10.08  | 0.086   |
| WP error            | 2  | 9.000   | 9.000   | 4.500  | 1.03   | 0.384   |
| B – No. passes      | 1  | 84.375  | 84.375  | 84.375 | 19.23  | 0.001   |
| C – Back pressure   | 1  | 1.042   | 1.042   | 1.042  | 0.24   | 0.634   |
| D – Speed           | 1  | 5.042   | 5.042   | 5.042  | 1.15   | 0.302   |
| E – Vibration       | 1  | 9.375   | 9.375   | 9.375  | 2.14   | 0.166   |
| SP Error            | 14 | 61.417  | 61.417  | 4.387  |        |         |
| Total               | 23 | 227.625 |         |        |        |         |

Figure 6. Main effects plot for response variable, microhardness, for 4043 aluminium.
established in the experimental design. Results of the ANOVA regression are provided in Table 5.

The regression equation provided by Minitab® employing all five factors is as follows:

$$Y = 62.625 + 1.875[B] - 1.375[A] - 0.625[E] + 0.458[D] + 0.208[C]$$  \(3\)

A consideration of the coefficients of partial determination for each factor provides additional insight to the development of the multivariate regression equation as each predictor variable is added to the model. For example, the five factors in order of \(p\)-values noted in Table 5 are as follows: \(B\) – number of passes \((p\)-value \(= 0.001\)), \(A\) – temperature \((p\)-value \(= 0.086\)), \(E\) – vibration \((p\)-value \(= 0.166\)), \(D\) – pressing speed \((p\)-value \(= 0.302\)), and \(C\) – back pressure \((p\)-value \(= 0.634\)). With additions of each of the factors the impacts on the error sum of squares (SSE) as well as the coefficients of partial determination for each predictor variable were assessed (Clark & Schkade, 1979). The SSE and coefficients of partial determination were calculated based on progressively adding the five factors as summarized in Tables 6 and 7.

As noted in Table 6, the SSE is reduced from 143.250 to 88.500 when the factors \(B\), \(A\), and \(E\) are progressively added to the model. This represents a 38.22% reduction in the SSE. Inclusion of the last two factors \(D\) and \(C\), however, offers minimal improved performance of the regression equation since the error is only reduced to 83.458 and 82.417, respectively.

The coefficients of partial determination also provide insight to the contributions from each of the respective factors. For example, 37.1% of the remaining variation is explained by factor \(B\) (number of presses) when no other factors are present in the model. With factor \(B\) contained in the regression equation the addition of factor \(A\) (temperature) is able to account for 30.9% of the remaining variation. Likewise, when factor \(E\) (vibration) is added to the model containing both factors \(B\) and \(A\), 10.6% of the remaining variation is explained by factor \(E\). Additional experimentation is warranted since factor \(E\) (vibration) with a \(p\)-value of 0.166 was not statistically significant. However, factor \(E\) was aliased with the \(AB\) two-factor interaction. Either factor \(E\) (vibration) may ultimately impact the process.

### Table 5. ANOVA regression – estimated effects and coefficients for 4043 alloy.

| Term          | Effect | Coef  | SE Coef | \(T\)  | \(p\)-value |
|---------------|--------|-------|---------|--------|-------------|
| Constant      | 62.625 | 0.4330| 144.63  | 0.000  |
| Block 1       | -0.500 | 0.6124| -0.82   | 0.500  |
| Block 2       | -0.500 | 0.6124| -0.82   | 0.500  |
| \(a\) – temp  | 3.750  | 1.875 | 0.4275  | 4.39   | 0.086       |
| \(B\) – no. passes | -1.375 | 0.4330| -3.18   | 0.086  |
| \(C\) – pressure | 0.417  | 0.208 | 0.4275  | 0.49   | 0.634       |
| \(D\) – speed | 0.917  | 0.458 | 0.4275  | 1.07   | 0.302       |
| \(E\) – vibration | -1.250 | 0.625 | 14.6   | 0.166  |

S = 2.09449 \(R\)-S\(\text{Sq}(\text{SP}) = 61.91\%\)
S (WP) = 0.168148 \(R\)-S\(\text{Sq}(\text{WP}) = 86.44\%\)

### Table 6. Error sum of squares for multivariate regression adding predictor variables.

| Error sum of squares for model containing X factors | Model factors | Error sum of squares, SSE |
|--------------------------------------------------|---------------|--------------------------|
| SSE\((X_o)\)                                     | No factors   | 227.625                  |
| SSE\((X_B)\)                                     | B            | 143.250                  |
| SSE\((X_B,X_A)\)                                 | B, A         | 99.000                   |
| SSE\((X_B,X_A,X_E)\)                             | B, A, E      | 88.500                   |
| SSE\((X_B,X_A,X_E,D)\)                           | B, A, E, D   | 83.458                   |
| SSE\((X_B,X_A,X_E,D,C)\)                         | B, A, E, D, C| 82.417                  |
at other factor levels or the AB interaction may also become statistically significant at other factor level settings of factors A and B.

At the factor levels selected for this research pressing speed, back pressure, and vibration were identified as statistically insignificant. However, each of these additional factors are reported to provide significant enhancements to processing efficiencies for ECAP processes (Ahmadi, Farzin, Meratian, Loeian, & Forouzan, 2015; Djavanroodi, Ahmadian, Naseri, Koohkan, & Ebrahimi, 2015; Langdon, 2007; Lapovok, 2005). For example, within this research the application of vibration was statistically insignificant and indicated a questionable inverse relationship between vibration and hardness. However, recent investigations have substantiated the benefits of vibration by realizing 9% reductions in punch loads, 51% reductions in grain size, 26.7% improvements in microstructure homogeneity, 16% increases in yield strength, and 12% increases in processed material hardness (Ahmadi et al., 2015; Djavanroodi et al., 2015). Therefore, further experimentation with IX-ECAP utilizing revised factor settings in conjunction with alternate or modified processing methods is warranted.

A run order plot of full model fitted values vs. the actual hardness values from Table 3 is provided in Figure 7.

To verify model adequacy residuals were examined to check underlying assumptions regarding normality, independence with mean equal to zero, and constant variance.

### 4.1.1. Normality
A normal probability plot (Figure 8) showed the residuals positioned along a straight line reflecting a closely approximated normal distribution. The normality assumption, therefore, appeared to be valid.

### 4.1.2. Independence
To verify the independence assumption a plot of residuals versus observation order was performed (Figure 9). The independence assumption also appeared to hold since a random distribution of points above and below the central line of zero was evident.

### 4.1.3. Constant variance
The constant variance assumption was assessed based on plots of residuals versus output response variable (microhardness), fitted values, and main factors. Plots of residuals versus measured hardness and fitted values (Figures 10 and 11) indicated structureless patterns of points above and below the zero line regardless of the low to high values of hardness and fitted values. Therefore, the constant variance assumption was supported.

To further check model adequacy regarding the constant variance assumption, residuals versus the regressors of temperature, number of passes, back pressure, pressing speed, and vibration were plotted as shown in Figures 12–16. With the exception of the plot for residuals
versus pressing speed and back pressure each of the plots indicated a possible presence of non-constant variance as the spread of the residual values at the low and high factor settings for temperature, number of passes, and vibration appeared to differ.

With possible invalidations regarding the constant variance assumption Levene’s test for equal variances was performed on each of the regressor variables at the low and high factor levels using a 5% significance level ($a = 0.05$) (Montgomery, 2001). Results are shown in Table 8.

Levene’s tests indicated that differences in variances for each of the regressors were statistically insignificant (Levene $p$ values > 0.05). However, main factor B (number of passes)
with a Levene’s $p$-value of 0.082 and main factor E (vibration) with a value of 0.054 were marginally statistically insignificant and could warrant further investigation. For example, this lack of potential equal variances for factor B (number of passes) makes intuitive sense due to the fact that I$_\chi$-ECAP is a strain hardening process resulting from simple shear imposed on the work pieces which alters the microstructures to develop finer grain sizes. As the number of passes through the system increases the microstructures continue towards increased levels of homogeneity resulting in less variability in hardness and decreased residual variance. However, true homogeneity may not be reached at three passes through the

Figure 9. Plot of residuals versus run order for 4043 aluminium.

Figure 10. Plot of residuals versus measured microhardness for 4043 aluminium.
system. Redistribution of the internal structures may be just beginning to become active at three passes. Additional passes through the system may be required to complete saturation of homogeneity in the microstructure and resulting hardness.

With $R^2$ values of 0.6191 and 0.8644 at the sub-plot and whole-plot levels, respectively, the model provided adequate insight to a first initial screening of the I_X-ECAP process.

### 4.2. Materials development

With 4043 aluminium being a commercial alloy with widespread industrial use the analysis

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**Figure 11.** Plot of residuals versus fitted microhardness for 4043 aluminium.

**Figure 12.** Plot of residuals versus temperature (factor A) for 4043 aluminium.
focused on material property enhancements resulting from processing of 4043 discrete variable length rods using IX-ECAP. Figure 17 plots the individual and mean hardness values for samples at both the low and high processing temperatures for each of the number of passes through the system. As illustrated, substantial increases in material hardness were obtained in transitioning through the system using a total of three passes. Standard deviations associated with each of the mean hardness values are tabulated in Table 9.

The final theoretical grain size for the processed samples were obtained from calculated values using the Hall-Petch relation to evaluate system effectiveness and potential in achieving
nanocrystalline or ultrafine grains. Microhardness conversions from Vickers to Brinell were made for the baseline and processed samples using material conversion databases. Brinell hardnesses were then converted to yield strength values for use in the Hall-Petch relation using defined material constants for the 4043 alloy system (Hester, 2015). Manufacturing system performance regarding theoretical grain size reductions was assessed using conversions of mean hardness values to grain sizes based on the number of passes through the system at both high and low temperatures. Table 10 shows the results of performing the conversion analysis for mean hardness values. A scatter plot of the data is presented in Figure 18.

Figure 15. Plot of residuals versus pressing speed (factor D) for 4043 aluminium.

Figure 16. Plot of residuals versus vibration (factor E) for 4043 aluminium.
The discrete variable length rod with the highest mean microhardness, HV = 66.5, was produced at the low temperature factor level and three passes through the system. This value represented an 84.7% increase in microhardness which equated to an increase of 200% in yield strength. The discrete variable length rod with the maximum individual microhardness, HV = 68, was also produced using a low temperature level and three passes through the system. This respective value represented an 88.88% increase in microhardness which equates to an increase of 203.4% in yield strength and a 98.5% reduction in theoretical grain size to a theoretical grain size of 2.5 μm.

Table 8. Levene’s test for equal variances for each factor.

| Process variable (factor) | F-test p-value (normal distribution) | Levene’s-test p-value (any continuous distribution) |
|---------------------------|--------------------------------------|---------------------------------------------------|
| A – Temperature [HTC]     | 0.919                                | 0.421                                             |
| B – No. of passes         | 0.086                                | 0.082                                             |
| C – Back pressure         | 0.680                                | 0.499                                             |
| D – Pressing speed        | 0.870                                | 0.745                                             |
| E – Vibration             | 0.229                                | 0.054                                             |

Table 9. Mean and standard deviation for processed microhardness samples – 4043 alloy.

| Pressing temp | Sample ID | Mean hardness | Standard deviation |
|---------------|-----------|---------------|--------------------|
|               |           | (Vickers)     | (Vickers)          |
| Low           | 1 Pass    | 61.5          | 1.643              |
|               | 3 Passes  | 66.5          | 1.049              |
| High          | 1 Pass    | 60.0          | 1.789              |
|               | 3 Passes  | 62.5          | 3.271              |

Figure 17. Individual value plot of Vickers microhardness versus number of passes at low and high temperatures for 4043 aluminium.
103 nanometers. Additional experimentation with $I_x$-ECAP utilizing revised Hall-Petch relationships incorporating dislocation and silicon particle strengthening terms as well as actual final grain size measurement validations employing scanning electron microscopy (SEM) is warranted to further substantiate process outputs regarding nanocrystalline propensity.

### 5. Summary

Nanotechnology continues to play a pivotal role in providing solutions to legacy engineering problems, offering improvements to existing engineering systems, enabling breakthroughs in new fields of science and technology, and capturing opportunities in promoting sustainable engineering. This emphasis regarding an opportunity in sustainable engineering whereby waste or spent materials and resources from one industry or process provide essential components or resources to additional industries or processes provided the motivation for this research (Huang, 2010).

An opportunity existed to bridge the gap between process potential and actual manufacturing output by developing and building a top down nanomanufacturing prototype system referred to as $I_x$-ECAP. This new process based on an extension of existing ECAP
constructs was successful in transforming traditional bulk solid 4043 aluminium welding rods of discrete variable length into materials with theoretically determined ultrafine and nanostructure grains possessing significant mechanical property enhancements. An extension of the applicability to sustainable engineering involves the capture, transformation, and re-use of spent or scrap welding rod residuals in applications such as composite reinforcements, micro-machine elements, and other materials of construction. I_x-ECAP represents a completely viable top-down nanomanufacturing process for recycling discrete length welding rod residuals into preferential engineering materials with significantly improved performance characteristics.

Disclosure statement
No potential conflict of interest was reported by the authors.

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