Performance of Thermo-Mechanically Processed AA7075 Alloy at Elevated Temperatures—From Microstructure to Mechanical Properties

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Abstract: This study focuses on the high temperature characteristics of thermo-mechanically processed AA7075 alloy. An integrated die forming process that combines solution heat treatment and hot forming at different temperatures was employed to process the AA7075 alloy. Low die temperature resulted in the fabrication of parts with higher strength, similar to that of T6 condition, while forming this alloy in the hot die led to the fabrication of more ductile parts. Isothermal uniaxial tensile tests in the temperature range of 200–400 °C and at strain rates ranging from 0.001–0.1 s \(^{-1}\) were performed on the as-received material, and on both the solution heat-treated and the thermo-mechanically processed parts to explore the impacts of deformation parameters on the mechanical behavior at elevated temperatures. Flow stress levels of AA7075 alloy in all processing states were shown to be strongly temperature- and strain-rate dependent. Results imply that thermo-mechanical parameters are very influential on the mechanical properties of the AA7075 alloy formed at elevated temperatures. Microstructural studies were conducted by utilizing optical microscopy and a scanning electron microscope to reveal the dominant softening mechanism and the level of grain growth at elevated temperatures.

Keywords: high strength aluminum; thermo-mechanical processing; microstructure; high temperature deformation

1. Introduction

Lightweight components are widely being utilized in the aerospace and aviation since decades [1]. Among a broad range of metallic materials, high strength aluminum alloys are promising candidates to meet the requirements of manufacturing lightweight parts with acceptable strength [2,3]. For example, high strength Al-Zn-Mg-Cu alloys have been extensively used not only in the aerospace industry but also in automobile and transport industries because of their outstanding properties [4–7]. These series of precipitation hardenable aluminum alloys exhibit high strength-to-weight ratios, excellent corrosion resistance, good fatigue properties, high fracture toughness and ductility [2,3,8–10]. Since the mechanical properties of the 7000 series of aluminum can be modified via heat treatment or thermo-mechanical processing, these alloys are employed for a variety of engineering applications [1,8,11].

Recently, researchers and engineers found out that the employment of different thermo-mechanical process strategies can result in the development of components with locally varied mechanical properties [12–15]. The impact of process parameters during a novel hot stamping technique was
studied attesting the improvement of mechanical properties of a high strength aluminum alloy as compared to the as-received T4 condition [13]. Forming limit curves (FLCs) for the hot forming of an aluminum alloy were also obtained in another study by designing a novel test device based on a commercial Gleeble machine [14]. The results of this preliminary research awaked the interest in hot stamping and thermo-mechanical processing of AA7075 alloy and entailed a high number of scientific and industrial studies [5, 6, 16–18]. It was shown that hot stamping of AA7075 alloy, followed by an adequate artificial aging treatment, resulted in the production of components with mechanical properties similar to the T6 condition [6].

The demand for reliable data to be used for calculation of the required forces applied during warm and hot working of AA7075 alloy has shaped interest toward high-temperature characteristics of this alloy [11, 13, 19–27]. These can be affected by various heat treatment conditions, i.e., solution heat treating, T6, and over-aging [13, 22, 25]. It was found that over-aged AA7075 alloy has the highest stability in maintaining mechanical properties at elevated temperatures, whereas, solution heat-treated and T6 conditions showed instability at relatively high temperatures [22]. Coarsening of fine and dispersed precipitates in the material in T6 condition and the occurrence of dynamic precipitation in the solution heat-treated samples were found to be the main reasons for thermal instability and increase of flow stress levels during hot deformation. Because of the importance of predicting hot deformation behavior of AA7075 alloy, some attempts were made to model flow stress curves of this material at high temperatures [11, 19, 26]. The agreement between experimental results and utilized models implied that constitutive equations are applicable for predicting flow stress curves for deformation conditions outside the experimental window as well. However, not only the precipitation distribution and sizes but also the effect of pre-plastic deformation should be taken into account [20]. For example, prior cold rolling improved the strength of this alloy at deformation temperatures up to 250 °C, while above this temperature level, the previous cold deformation enabled higher total elongation reaching over 50%.

Elevated temperature response of aluminum alloys is of interest for estimation of critical force levels for any application-oriented loading event as well as for metal forming at elevated temperatures [21, 22, 28–33]. Many researchers utilized hot compression, tension, and torsion experiments to obtain flow curves at elevated temperatures [21, 22, 28–33]. Instability phenomena during tensile testing are a barrier toward constitutive analysis. Both compressive and tensile loadings stimulate a relatively higher number of slip systems as compared to the shear loading condition [33, 34]. It is also worth noting that under the hot tensile mode fracture mechanisms can be explored in parallel and, hence, process-microstructure-property-damage relationships can be established.

As elaborated above, despite the numerous research studies the elevated temperature behavior of thermo-mechanically processed AA7075 alloy has not been studied in sufficient depth yet. Therefore, the purpose of this research work is to provide key insights into the mechanical and microstructural changes in the course of hot deformation of a thermo-mechanically processed high strength aluminum alloy at a wide range of temperatures and strain rates. Many engineering applications demand components with the ability to work at elevated temperatures and, hence, high temperature behavior of thermo-mechanically processed AA7075 alloy has to be well-understood. In this regard, two different die temperatures (24 °C and 350 °C) were used to produce hot formed samples. Subsequently, elevated temperature tensile tests at temperatures ranging from ambient to 400 °C conducted at various strain rates were performed on the as-received T6, solution heat-treated, and the thermo-mechanically processed samples. Analysis of mechanical properties is supported by microstructural analysis to allow for a better understanding of the underlying elementary mechanisms. The results achieved open up new pathways for manufacturing lightweight components with satisfactory strength in a wide range of loading scenarios in different conditions. Results will be the basis for safe and reliable use of microstructurally graded parts, i.e., parts with locally designed mechanical properties in order to meet current challenging demands of several branches such as the mobility sector. Such parts will be realized and thoroughly characterized in follow-up studies.
2. Materials and Methods

Sheets of AA7075 alloy with a thickness of 1.5 mm were received with the chemical composition listed in Table 1. The condition of as-received material was T6, i.e., solution heat-treated, quenched, and artificially aged. For the forming and heat treatment experiments, the sheet material was cut into blanks of 250 mm × 140 mm × 1.5 mm.

| Element | Si   | Fe  | Cu | Mn | Mg  | Cr | Zn | Ti | Zr | Al  |
|---------|------|-----|----|----|-----|----|----|----|----|-----|
| AA7075 (Wt. %) | 0.10 | 0.11 | 1.49 | 0.03 | 2.38 | 0.20 | 5.57 | 0.03 | 0.04 | Balanced |

The experimental set-up consisting of a roller hearth furnace, a hydraulic press with a hat-shaped die, and a furnace for the aging treatment is shown in Figure 1. Thermo-mechanical processes and routes utilized in the present study were chosen according to a previous paper published by some of the authors of the present manuscript [35]. To evaluate the influence of die temperatures on the elevated temperature behavior and microstructural evolution of AA7075 alloy, two thermo-mechanical processing routes were used (Figure 1c). In this study, the sheets of AA7075 alloy were heated up to the solution heat treatment temperature (480 °C) and soaked for 15 min to dissolve previously formed precipitates being present in the as-received condition. After the solution annealing, the heated blanks were transferred, within a period of 6 to 8 s, to the forming dies. They were then formed and quenched in dies with temperatures of 24 °C and 350 °C. All formed samples were subsequently aged at a temperature of 120 °C for 20 h. The solution heat treatment and aging temperatures were chosen according to the previous study [35].

The test samples were electro-discharge machined with their loading axis parallel to the rolling direction and a gauge length of 40 mm. Those tensile samples of thermo-mechanically processed material were cut from the top hat sections (Figure 1d). To characterize the mechanical properties of AA7075 alloy in different conditions at room temperature, tensile tests were performed isothermally at strain rates of 0.001, 0.01, and 0.1 s⁻¹ and at temperatures of 200, 250, 300, 350, and 400 °C. Samples were ground and polished before tensile testing. All samples were heated up to the deformation temperature and subsequently deformed in a single loading step. The sample temperature was monitored during the tests using thermocouples. The strain was measured utilizing an extensometer directly attached to the sample surfaces. Three tensile tests for each condition were carried out and the average values are reported. Standard polishing procedures were employed to prepare samples for microstructural analysis. Optical microscopy and a scanning electron microscope (SEM) equipped with an electron
backscatter diffraction (EBSD) unit operating at a nominal voltage of 20 kV were used to characterize microstructural evolution and fracture surfaces of the samples in different conditions. For the EBSD examination, the samples were prepared by 24 h of vibro-polishing in a colloidal silica solution (OPS).

3. Results and Discussion

3.1. Room Temperature Mechanical Properties and Microstructure of Thermo-Mechanically Processed AA7075 Alloy

Room temperature mechanical properties of as-received, solution heat-treated, and the thermo-mechanically processed AA7075 alloy are summarized in Table 2. From this table, it can be deduced that the processing of AA7075 alloy in the hot and cold dies results in significantly different mechanical properties. The sample processed in the cold die shows a strength level similar to that of the T6 as-received condition, while the sample formed and cooled in the hot die exhibits lower yield and ultimate tensile strength.

| Conditions                  | Yield Strength (MPa) | Ultimate Tensile Strength (MPa) | Elongation (%) |
|-----------------------------|----------------------|--------------------------------|----------------|
| As-received (T6)            | $470 \pm 4$          | $580 \pm 5$                    | $14 \pm 1$     |
| Solution heat-treated       | $171 \pm 6$          | $393 \pm 7$                    | $22 \pm 3$     |
| Die temperature 24°C        | $489 \pm 8$          | $576 \pm 6$                    | $10 \pm 2$     |
| Die temperature 350°C       | $164 \pm 8$          | $338 \pm 9$                    | $14 \pm 3$     |

The EBSD micrographs (image quality map) of AA7075 before and after thermo-mechanical processing are illustrated in Figure 2. The average grain sizes were determined by the linear intercept method. The average grain sizes of as-received and the thermo-mechanically processed samples (in the dies with temperatures of 24 °C and 350 °C) were found to be 24, 25, and 22 µm, respectively. Thus, grain size remains relatively constant in the utilized thermo-mechanical processes as already reported before for two different aluminum alloys [18]. The same finding, i.e., thermo-mechanical processing has minimal impact on the grain size of this alloy, was reported by Harrison et al. in [6]. SEM micrographs obtained using back-scattered electron (BSE) contrast are also displayed in Figure 2. Evidently, die temperatures affect the morphology and size of precipitates introduced in the AA7075 alloy. Coarse precipitates were formed and segregated along the grain boundaries in the sample processed in the hot die, whereas, fine and dispersed precipitates, similar to that of T6 counterpart, were introduced in the sample processed in the cold die. Lower cooling rates after solution treatment favor the evolution of relatively large precipitates alongside the grain boundaries eventually leading to a reduction of precipitate forming elements in the surrounding areas [10,36–38]. Therefore, the high strength of AA7075 alloy formed and cooled in the cold die can be attributed to the formation of fine and dispersed precipitates after the aging treatment [17,18].
precipitation (DPN) in such an unstable condition [22,40]. As such, DPN can be favored at a high level of supersaturation while deformation takes place at moderate temperatures.

The elevated temperature mechanical properties of AA7075 alloy processed in the cold die, results in virtually identical mechanical properties not only at room but also at elevated temperatures.

Figure 2. Electron backscatter diffraction (EBSD) micrographs (image quality maps) and corresponding BSE images of AA7075; (a) as-received, (b) processed in a die with temperature of 24 °C, and (c) processed in a die with temperature of 350 °C.

3.2. Elevated Temperature Flow Behavior of Thermo-Mechanically Processed AA7075 Alloy

The flow curves of AA7075 alloy at elevated temperatures and different strain rates are presented in Figure 3. The flow behavior of AA7075 alloy in the T6 condition exhibits a strong dependence on the deformation temperature and rate. With increasing the deformation temperature and decreasing strain rate, the strength of the as-received T6 material decreases substantially, attesting the activation of softening mechanisms i.e., dynamic recovery (DRV) and dynamic recrystallization (DRX) at such high temperatures [20,26]. For in-depth evaluation of the behavior in these temperature ranges, tensile strength in many studies is considered, e.g., [31,39]. At lower deformation temperatures (200, 250, and 300 °C), the flow stress curves show a softening after reaching tensile stress without ever reaching the steady-state region (Figure 3). However, samples deformed at temperatures above 300 °C display a steady-state region and considerable ductility after reaching tensile stress.

The flow curves of the solution heat-treated material at elevated temperatures and various strain rates are also illustrated in Figure 3. Results reveal that strength and ductility of the solution heat-treated AA7075 alloy are sensitive to both temperature and strain rate. Higher deformation temperature and/or lower strain rate lead to a significant reduction in tensile strength of the solution heat-treated samples. It is also worth noting that at deformation temperatures of 200 and 250 °C, the solution heat-treated AA7075 alloy shows a remarkable work hardening (WH) before reaching tensile strength except at 250 °C and 0.001 s⁻¹. This behavior can be linked to the occurrence of dynamic precipitation (DPN) in such an unstable condition [22,40]. As such, DPN can be favored at a high level of supersaturation while deformation takes place at moderate temperatures.

The elevated temperature mechanical properties of AA7075 alloy processed in the cold die are similar to those of as-received T6 counterparts. The reduction in tensile strength values and enhancement of ductility with the increase of deformation temperature are also evident. Similar to the as-received material, this condition also exhibits a steady-state region at or above deformation temperatures of 300 °C. The very similar microstructural state, i.e., almost equal grain sizes and same morphology and sizes of precipitates in both as-received and thermo-mechanically processed materials...
(in the cold die), results in virtually identical mechanical properties not only at room but also at elevated temperatures.

The flow curves of AA7075 alloy processed in die with a temperature of 350 °C can also be seen in Figure 3. At 200 and 250 °C, an obvious reduction in strength can be seen for this condition as compared to other conditions. Low flow stress levels at moderate deformation temperature in this condition can be attributed to the formation of large precipitates already during the thermo-mechanical process in the hot die as shown in Figure 2c. It is well-known that large precipitates lead to lower strength during plastic deformation. Forming in a heated die leads to precipitations during cooling before intentional aging. Thus, the resulting state before aging is not supersaturated to the same level as after forming in a cold die. Consequently, the existing precipitates coarsen and less new precipitates are formed during subsequent aging, resulting in this reduced strength [22].

**Figure 3.** Elevated temperature flow behavior of AA 7075 at various strain rates and deformation temperature of (a) 200 °C, (b) 250 °C, (c) 300 °C, (d) 350 °C and (e) 400 °C.

The flow curves of AA7075 alloy processed in die with a temperature of 350 °C can also be seen in Figure 3. At 200 and 250 °C, an obvious reduction in strength can be seen for this condition as compared to other conditions. Low flow stress levels at moderate deformation temperature in this condition can be attributed to the formation of large precipitates already during the thermo-mechanical process in the hot die as shown in Figure 2c. It is well-known that large precipitates lead to lower strength during plastic deformation. Forming in a heated die leads to precipitations during cooling before intentional aging. Thus, the resulting state before aging is not supersaturated to the same level as after forming in a cold die. Consequently, the existing precipitates coarsen and less new precipitates are formed during subsequent aging, resulting in this reduced strength [22].

### 3.3. Effects of Temperature and Deformation Rate on the Microstructural Evolution of Thermo-Mechanically Processed AA7075 Alloy

The effect of deformation temperature on microstructure evolution of AA7075 alloy in different processing states is depicted in Figure 4. The examined regions were prepared from the deformed part of the sample. Grains of AA7075 alloy in all processed states are elongated alongside the tensile direction (being parallel to rolling direction). It can also be seen that the increase of deformation temperature to 400 °C led to the growth of grains in all conditions. Obviously, there is sufficient thermal
energy to promote nucleation and growth of dynamically recrystallized grains. Stored energy induced by deformation of material during elevated temperature tensile tests as well as the previous rolling process brings about a considerable driving force for the recrystallization phenomenon. The occurrence of DRX and growth of recrystallized grains at high deformation temperatures were previously reported for cold rolled and solution heat-treated AA7075 alloy [20,24]. It should also be noted that the influence of different thermo-mechanical process routes employed in the present study seems to be of minor importance with respect to the final grain sizes of samples upon hot tensile tests.

The influence of the deformation rate on the microstructure of AA7075 alloy in different processing states was additionally studied and results are shown in Figure 5. From this figure, it can be deduced that the lower deformation rate results in a more pronounced growth of recrystallized grains. This observation can be imputed to the sufficiency of time for the growth of recrystallized grains after nucleation [20]. It was previously reported that in case of hot working of metallic materials, flow stress levels depend on the change in dislocation densities linked to deformation and annihilation contributing to the general softening mechanisms, i.e., DRV and DRX, and, eventually, to the grain size of recrystallized grains [41–43].

The effect of tensile deformation at temperatures of 200 and 400 °C for samples processed in the hot die was further analyzed via EBSD as displayed in Figure 6. Low-angle grain boundaries (i.e., boundaries characterized by misorientation angles of 2 to 15 degree) and high-angle grain boundaries (i.e., boundaries characterized by misorientation angles of > 15 degree) are marked by white and black lines, respectively. The sample deformed at 200 °C consists of a higher fraction of low-angle grain boundaries as compared to that upon deformation at 400 °C. The presence of substructures and a high fraction of low-angle grain boundaries, respectively, for the sample deformed at 200 °C indicates that the dominant softening mechanism is DRV at this deformation temperature. However, high-angle grain boundaries dominate upon deformation at a temperature of 400 °C confirming the occurrence of DRX at this temperature. It was already reported that the onset of DRX generally can take place at temperatures above 300 °C in elevated temperature deformation of AA7075 alloy [26].
were analyzed. The impact of deformation temperature on the fracture surfaces is presented in Figure 7. All fracture surfaces show a considerable number of equiaxed dimples and microvoids confirming the presence of ductile fracture mechanisms [44]. Deformation of the AA7075 alloy at higher deformation temperatures increased the sizes of dimples and microvoids. Obviously, the diffusion rate as the responsible mechanism for the coalescence of microvoids is increased at higher deformation temperatures.

Figure 5. Optical micrographs of (a) as-received (AA 7075-T6 aluminum alloy), (b) the solution heat-treated material, (c) sample processed in a die with a temperature of 24 °C, and (d) sample processed in a die with a temperature of 350 °C. All micrographs highlight microstructure evolution after tensile tests at the temperature of 350 °C and strain rates of 0.001 s⁻¹ (upper row) and 0.1 s⁻¹ (lower row).

Figure 6. EBSD inverse pole figure (IPF) maps of samples processed in a die with a temperature of 350 °C upon hot tensile tests at the strain rate of 0.01 s⁻¹ and temperature of (a) 200 °C and (b) 400 °C.

3.4. Fractography

To characterize the failure mechanisms of AA7075 alloy in various conditions, the fracture surfaces were analyzed. The impact of deformation temperature on the fracture surfaces is presented in Figure 7. All fracture surfaces show a considerable number of equiaxed dimples and microvoids confirming the presence of ductile fracture mechanisms [44]. Deformation of the AA7075 alloy at higher temperatures increased the sizes of dimples and microvoids. Obviously, the diffusion rate as the responsible mechanism for the coalescence of microvoids is increased at higher deformation
temperature [45]. Hence, larger dimples and microvoids can be expected for the samples deformed at higher deformation temperatures.

Besides the effect of deformation temperature on the fracture mechanisms of AA7075 alloy in different states, the influence of the deformation rate on the fracture surfaces was analyzed as illustrated in Figure 8. Surfaces of the samples deformed at a lower strain rate are characterized by slightly larger dimples as compared to those deformed at a higher rate. It was already mentioned in literature that in the high temperature regime materials deformed at a lower rate have sufficient time for growth and coalescence of microvoids [44].

3.5. Process-Microstructure-Property-Damage Relationships

The effect of thermo-mechanical processing on the mechanical properties, microstructural evolution, and damage mechanisms of AA7075 alloy at elevated temperature is discussed in this section to highlight elementary mechanisms and eventually establish process-microstructure-property-damage relationships.
In this regard, tensile properties, the corresponding grain sizes and dimple sizes (with their standard deviations) in various deformation conditions are listed in Table 3. It should be noted that samples deformed at various conditions were ruptured at different strains. Although this fact may have an effect on the accuracy of any comparisons based on the grain size measurements, such an analysis was still found to be appropriate for capturing microstructural evolution and underlying mechanisms [20,29,46,47]. From Table 3, it can be deduced that grain sizes of samples deformed at 200 °C under a strain rate of 0.01 s⁻¹ are similar to those reported before tension, while samples deformed at 400 °C under the strain rate of 0.01 s⁻¹ are characterized by coarser grains and larger dimples. As discussed earlier, the growth of dynamically recrystallized grains and coalescence of microvoids at a higher deformation temperature is thought to be the main reason for such an observation [20,44]. Considering grain sizes of samples deformed at 350 °C under various strain rates, and microstructures seen in Figure 5, at the same time, it can be concluded that deformation of this alloy at higher rate seems to result in heterogeneous recrystallization [48]. Relatively higher standard deviations in grain sizes of samples deformed at 350 °C with a strain rate of 0.1 s⁻¹ compared with other deformation conditions also confirm the occurrence of non-homogeneous DRX. During heterogeneous recrystallization, softening does not occur at the same time for all grains (lower row in Figure 5). It is also worth noting that with the increase in grain size at 400 °C, tensile strength drops. An inverse power-law relationship between the strength of metals and recrystallized grain size was already reported [42,43].

Table 3. Summary of the tensile properties and the corresponding grain and dimple sizes of thermo-mechanically processed AA7075 in various deformation conditions; standard deviations with 80% confidence interval are provided in the table.

| Condition                                    | Grain Size (µm) | Dimple Size (µm) | Tensile Strength (MPa) | Elongation (%) |
|----------------------------------------------|-----------------|------------------|------------------------|---------------|
| As-received (AA7075-T6 aluminum alloy)       | 24 ± 8          | -                | -                      | -             |
| Solution heat-treated material               | 25 ± 7          | -                | -                      | -             |
| Sample processed in die with a temperature of 24 °C | 25 ± 10        | -                | -                      | -             |
| Sample processed in die with a temperature of 350 °C | 22 ± 8         | -                | -                      | -             |
| As-received tensioned at 200 °C under 0.01 s⁻¹ | 26 ± 10         | 12 ± 3           | 344 ± 9                | 14 ± 2        |
| As-received tensioned at 350 °C under 0.1 s⁻¹ | 24 ± 15         | 14 ± 3           | 54 ± 5                 | 25 ± 3        |
| As-received tensioned at 350 °C under 0.01 s⁻¹ | 30 ± 9          | 15 ± 4           | 35 ± 3                 | 22 ± 2        |
| As-received tensioned at 400 °C under 0.1 s⁻¹ | 34 ± 8          | 20 ± 5           | 28 ± 3                 | 12 ± 1        |
| Solution heat-treated material tensioned at 200 °C under 0.01 s⁻¹ | 26 ± 9          | 11 ± 4           | 321 ± 8                | 27 ± 2        |
| Solution heat-treated material tensioned at 350 °C under 0.1 s⁻¹ | 26 ± 11         | 12 ± 4           | 66 ± 6                 | 33 ± 4        |
| Solution heat-treated material tensioned at 350 °C under 0.01 s⁻¹ | 31 ± 10         | 15 ± 5           | 39 ± 4                 | 25 ± 3        |
| Solution heat-treated material tensioned at 400 °C under 0.01 s⁻¹ | 37 ± 9          | 18 ± 7           | 37 ± 3                 | 24 ± 3        |
| Sample processed in die with a temperature of 24 °C tensioned at 200 °C under 0.01 s⁻¹ | 25 ± 9          | 13 ± 3           | 353 ± 8                | 13 ± 2        |
| Sample processed in die with a temperature of 24 °C tensioned at 350 °C under 0.1 s⁻¹ | 23 ± 12         | 14 ± 5           | 81 ± 6                 | 26 ± 2        |
| Sample processed in die with a temperature of 24 °C tensioned at 350 °C under 0.01 s⁻¹ | 29 ± 9          | 17 ± 7           | 47 ± 5                 | 24 ± 3        |
| Sample processed in die with a temperature of 24 °C tensioned at 400 °C under 0.01 s⁻¹ | 32 ± 9          | 19 ± 6           | 42 ± 3                 | 27 ± 4        |
| Sample processed in die with a temperature of 350 °C tensioned at 200 °C under 0.01 s⁻¹ | 24 ± 8          | 21 ± 8           | 199 ± 7                | 25 ± 2        |
| Sample processed in die with a temperature of 350 °C tensioned at 350 °C under 0.1 s⁻¹ | 23 ± 14         | 14 ± 5           | 64 ± 4                 | 38 ± 3        |
| Sample processed in die with a temperature of 350 °C tensioned at 350 °C under 0.001 s⁻¹ | 33 ± 8          | 16 ± 5           | 37 ± 2                 | 33 ± 4        |
| Sample processed in die with a temperature of 350 °C tensioned at 400 °C under 0.01 s⁻¹ | 33 ± 9          | 22 ± 7           | 39 ± 3                 | 30 ± 2        |
Process-microstructure-property relationships are highlighted schematically in Figure 9. The schematic detailing microstructural evolution upon thermo-mechanical treatment as well as elevated temperature tensile tests depicts every single step. As detailed earlier, hot forming and quenching within forming dies at various temperatures led to the different cooling rates and eventually changes in the supersaturation level. As a result, precipitates formed in the following aging process differ in morphology and size. During the hot forming process, dislocation density is expected to increase as shown schematically in Figure 9. This behavior could accelerate and influence the precipitation kinetics during aging treatment. At the lowest tensile temperature of 200 °C, the main softening mechanism in thermo-mechanically processed AA7075 alloy was found to be DRV leading to the formation of substructures. At 400 °C, however, deformation temperature is sufficiently high for the occurrence of DRX and the subsequent growth of recrystallized grains. It should also be noted that the coarsening of fine precipitates can take place because of the further heating of the sample processed in the cold die during elevated temperature tensile tests. Further in-depth microstructural analysis needs to be carried out by high-resolution transmission electron microscopy to experimentally establish the mechanisms and relationships explained in this section. However, such analysis is beyond the scope of present work and, hence, will be subject of follow-up studies.

**Figure 9.** Schematic illustration highlighting the microstructural evolution induced by the thermomechanical process as well as tensile tests at elevated temperatures.

4. Conclusions

High temperature characteristics of thermo-mechanically processed AA7075 alloy were explored in the temperature range of 200–400 °C and the strain rate range of 0.001–0.1 s⁻¹. The following conclusion can be drawn:

(i) Room temperature tensile tests and microstructural analysis reveal that the die temperature in the forming process of AA7075 alloy substantially affects the strength and ductility because of the formation of precipitates with different sizes and morphologies. Higher die temperatures resulted in the formation and segregation of coarse precipitates alongside the grain boundaries and, hence, precipitates formed were found not to be suitable for strengthening of the material. On the contrary, the material formed in a cold die showed mechanical properties similar to those of the T6 condition due to the formation of fine and well-dispersed precipitates in the subsequent aging treatment.
(ii) The flow behavior of AA7075 alloy in all processing states was significantly affected by the deformation temperature and strain rate. Higher deformation temperatures and lower strain rates led to the drop of tensile strength values, attesting the occurrence of softening mechanisms at elevated temperature.

(iii) OM and SEM analysis of specimens revealed that DRV is the dominant softening mechanism at lower deformation temperature, while DRX took place in case of samples deformed at 400 °C. Besides, a higher deformation temperature promotes the growth of dynamically recrystallized grains. Fracture analysis also showed that the sizes of microvoids and dimples are enlarged with the increase in deformation temperature and decrease in deformation rate.

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References
1. Starke, E.; Staley, J. Application of Modern Aluminum Alloys to Aircraft. Prog. Aerosp. Sci. 1996, 32, 131–172. [CrossRef]
2. Jayaganthan, R.; Brokmeier, H.-G.; Schwebke, B.; Panigrahi, S. Microstructure and texture evolution in cryorolled Al 7075 alloy. J. Alloys Compd. 2010, 496, 183–188. [CrossRef]
3. Cepeda-Jiménez, C.; García-Infanta, J.; Ruano, O.A.; Carreño, F. Mechanical properties at room temperature of an Al–Zn–Mg–Cu alloy processed by equal channel angular pressing. J. Alloys Compd. 2011, 509, 8649–8656. [CrossRef]
4. Tisza, M.; Czinege, I. Comparative study of the application of steels and aluminium in lightweight production of automotive parts. Int. J. Light. Mater. Manuf. 2018, 1, 229–238. [CrossRef]
5. Liu, Y.; Zhu, Z.; Wang, Z.; Zhu, B.; Wang, Y.; Zhang, Y. Formability and lubrication of a B-pillar in hot stamping with 6061 and 7075 aluminium alloy sheets. Procedia Eng. 2017, 207, 723–728. [CrossRef]
6. Harrison, N.R.; Luckey, S.G. Hot Stamping of a B-Pillar Outer from High Strength Aluminum Sheet AA7075. SAE Int. J. Mater. Manuf. 2014, 7, 567–573. [CrossRef]
7. Poznak, A.; Freiberg, D.; Sanders, P. Automotive Wrought Aluminium Alloys. Fundam. Alum. Metall. 2018, 333–386. [CrossRef]
8. Li, X.-M.; Starink, M. Effect of compositional variations on characteristics of coarse intermetallic particles in overaged 7000 aluminium alloys. Mater. Sci. Technol. 2001, 17, 1324–1328. [CrossRef]
9. Feng, A.; Chen, D.; Ma, Z. Microstructure and Cyclic Deformation Behavior of a Friction-Stir-Welded 7075 Al Alloy. Met. Mater. Trans. A 2010, 41, 957–971. [CrossRef]
10. Kumar, P.V.; Gankidi, M.R.; Rao, K.S. Microstructure, mechanical and corrosion behavior of high strength AA7075 aluminium alloy friction stir welds – Effect of post weld heat treatment. Def. Technol. 2015, 11, 362–369. [CrossRef]
11. Senthil, K.; Iqbal, M.; Chandel, P.; Gupta, N. Study of the constitutive behavior of 7075-T651 aluminum alloy. Int. J. Impact Eng. 2017, 108, 171–190. [CrossRef]
12. Maikranz-Valentin, M.; Weidig, U.; Schoof, U.; Becker, H.-H.; Steinhoff, K. Components with Optimised Properties due to Advanced Thermo-mechanical Process Strategies in Hot Sheet Metal Forming. Steel Res. Int. 2008, 79, 92–97. [CrossRef]
13. Ma, W.-Y.; Wang, B.; Lin, J.; Tang, X.-F. Influence of process parameters on properties of AA6082 in hot forming process. Trans. Nonferr. Met. Soc. China 2017, 27, 2454–2463. [CrossRef]
14. Shao, Z.T.; Bai, Q.; Lin, J.G. A Novel Experimental Design to Obtain Forming Limit Diagram of Aluminium Alloys for Solution Heat Treatment, Forming and In-Die Quenching Process. Key Eng. Mater. 2014, 622, 241–248. [CrossRef]
15. Shao, Z.; Jiang, J.; Lin, J. Feasibility study on direct flame impingement heating applied for the solution heat treatment, forming and cold die quenching technique. *J. Manuf. Process.* 2018, 36, 398–404. [CrossRef]

16. Xiao, W.; Wang, B.; Zheng, K.; Zhou, J.; Lin, J. A study of interfacial heat transfer and its effect on quenching when hot stamping AA7075. *Arch. Civ. Mech. Eng.* 2018, 18, 723–730. [CrossRef]

17. Scharififar, E.; Sajadifar, S.V.; Moeini, G.; Weidig, U.; Böhm, S.; Niendorf, T.; Steinhoff, K. Dynamic Tensile Deformation of High Strength Aluminum Alloys Processed Following Novel Thermomechanical Treatment Strategies. *Adv. Eng. Mater.* 2020. [CrossRef]

18. Sajadifar, S.V.; Scharififar, E.; Weidig, U.; Steinhoff, K.; Niendorf, T. Effect of Tool Temperature on Mechanical Properties and Microstructure of Thermo-Mechanically Processed AA6082 and AA7075 Aluminum Alloys. *HTM J. Heat Treat. Mater.* 2020, 75, 177–191. [CrossRef]

19. Li, L.-T.; Lin, Y.; Zhou, H.-M.; Jiang, Y.-Q. Modeling the high-temperature creep behaviors of 7075 and 2124 aluminum alloys by continuum damage mechanics model. *Comput. Mater. Sci.* 2013, 73, 72–78. [CrossRef]

20. Shojaei, K.; Sajadifar, S.V.; Yapici, G. On the mechanical behavior of cold deformed aluminum 7075 alloy at elevated temperatures. *Mater. Sci. Eng. A* 2016, 670, 81–89. [CrossRef]

21. Lu, J.; Song, Y.; Hua, L.; Zheng, K.; Dai, D. Thermal deformation behavior and processing maps of 7075 aluminum alloy sheet based on isothermal uniaxial tensile tests. *J. Alloys Compd.* 2018, 767, 856–869. [CrossRef]

22. Cerri, E.; Evangelista, E.; Forcellese, A.; McQueen, H. Comparative hot workability of 7012 and 7075 alloys after different pretreatments. *Mater. Sci. Eng. A* 1995, 197, 181–198. [CrossRef]

23. Gupta, R.K.; Kumar, V.A.; Krishnan, A.S.; Niteshraj, J. Hot Deformation Behavior of Aluminum Alloys AA7010 and AA7075. *J. Mater. Eng. Perform.* 2019, 28, 5021–5036. [CrossRef]

24. Lin, Y.; Li, L.-T.; Fu, Y.-X.; Jiang, Y.-Q. Hot compressive deformation behavior of 7075 Al alloy under elevated temperature. *J. Mater. Sci.* 2011, 47, 1306–1318. [CrossRef]

25. McQueen, H.; Ryan, N. Constitutive analysis in hot working. *Mater. Sci. Eng. A* 2002, 322, 43–63. [CrossRef]

26. Xiao, W.; Wang, B.; Wu, Y.; Yang, X. Constitutive modeling of flow behavior and microstructure evolution of AA7075 in hot tensile deformation. *Mater. Sci. Eng. A* 2018, 712, 704–713. [CrossRef]

27. Zhu, D.Y.; Zhen, L.; Lin, C.; Shao, W.-Z. High Temperature Deformation Mechanism of 7075 Aluminum Alloy. *Key Eng. Mater.* 2007, 353, 691–694. [CrossRef]

28. Prasad, Y.V.R.K.; Rao, K.P.; Sasdihara, S. *Hot Working Guide: A Compendium of Processing Maps*, 2nd ed.; ASM International: Geauga County, OH, USA, 2015; ISBN 978-1-62708-091-0.

29. Khamei, A.A.; Dehghani, K. Effects of strain rate and temperature on hot tensile deformation of severe plastic deformed 6061 aluminum alloy. *Mater. Sci. Eng. A* 2015, 627, 1–9. [CrossRef]

30. Khamei, A.A.; Dehghani, K. Hot Ductility of Severe Plastic Deformed AA6061 Aluminum Alloy. *Acta Met. Sin. Engl. Lett.* 2015, 28, 322–330. [CrossRef]

31. Khamei, A.A.; Dehghani, K.; Mahmudi, R. Modeling the Hot Ductility of AA6061 Aluminum Alloy After Severe Plastic Deformation. *JOM* 2015, 67, 966–972. [CrossRef]

32. Leo, P.; Cerri, E.; McQueen, H. Hot Tensile Behaviour and Constitutive Analysis of Al-5.5Zn-1.2Mg/Zr Alloys. In *Supplemental Proceedings*; John Wiley & Sons, Inc.: Hoboken, NJ, USA, 2011; Volume 2, pp. 157–165.

33. Sang, D.; Fu, R.; Li, Y. The Hot Deformation Activation Energy of 7050 Aluminum Alloy under Three Different Deformation Modes. *Metals* 2016, 6, 49. [CrossRef]

34. Miller, M.; McDowell, D. Modeling large strain multiaxial effects in FCC polycrystals. *Int. J. Plast.* 1996, 12, 875–902. [CrossRef]

35. Scharififar, E.; Knoth, R.; Weidig, U. Thermo-mechanical forming procedure of high strength Aluminum sheet with improved mechanical properties and process efficiency. *Procedia Manuf.* 2019, 29, 481–489. [CrossRef]

36. Sajadifar, S.V.; Moeini, G.; Scharififar, E.; Lauhoff, C.; Böhm, S.; Niendorf, T. On the Effect of Quenching on Postweld Heat Treatment of Friction-Stir-Welded Aluminum 7075 Alloy. *J. Mater. Eng. Perform.* 2019, 28, 5255–5265. [CrossRef]

37. Starink, M. Reduced fracturing of intermetallic particles during crack propagation in age hardening Al-based alloys due to PFZs. *Mater. Sci. Eng. A* 2005, 390, 260–264. [CrossRef]

38. Rhodes, C.; Mahoney, M.; Bingel, W.; Spurling, R.; Bampton, C. Effects of friction stir welding on microstructure of 7075 aluminum. *Scr. Mater.* 1997, 36, 69–75. [CrossRef]
39. Leo, P.; McQueen, H.J.; Cerri, E.; Spigarelli, S. Properties, Microstructure and Hot Deformation Behaviour of Different Al-Zn-Mg (Zr) Alloys. In ICAA13 Pittsburgh; Springer Science and Business Media LLC: Berlin/Heidelberg, Germany, 2012; pp. 1635–1641.

40. Lin, Y.; Yang, H.; He, D.-G.; Chen, J. A Physically-Based Model Considering Dislocation–Solute Atom Dynamic Interactions for A Nickel-Based Superalloy at Intermediate Temperatures. Mater. Des. 2019, 183, 108122. [CrossRef]

41. Nourani, M.; Sajadifar, S.V.; Ketabchi, M.; Milani, A.S.; Yannacopoulos, S. On the microstructural evolution of 4130 steel during hot compression. Recent Patents Mater. Sci. 2012, 5, 74–83.

42. Sakai, T.; Jonas, J. Overview no. 35 Dynamic recrystallization: Mechanical and microstructural considerations. Acta Met. 1984, 32, 189–209. [CrossRef]

43. Sakai, T. Dynamic recrystallization microstructures under hot working conditions. J. Mater. Process. Technol. 1995, 53, 349–361. [CrossRef]

44. Zhou, M.; Lin, Y.; Deng, J.; Jiang, Y.-Q. Hot tensile deformation behaviors and constitutive model of an Al–Zn–Mg–Cu alloy. Mater. Des. 2014, 59, 141–150. [CrossRef]

45. Morin, L.; Leblond, J.-B.; Benzerga, A.A. Coalescence of voids by internal necking: Theoretical Estimates and numerical results. J. Mech. Phys. Solids 2015, 75, 140–158. [CrossRef]

46. Zhang, W.-P.; Li, H.-H.; Hu, Z.-L.; Hua, L. Investigation on the deformation behavior and post-formed microstructure/properties of AA7075-T6 alloy under pre-hardened hot forming process. Mater. Sci. Eng. A 2020, 139749. [CrossRef]

47. Taheri-Mandarjani, M.; Zarei-Hanzaki, A.; Abedi, H. Hot ductility behavior of an extruded 7075 aluminum alloy. Mater. Sci. Eng. A 2015, 637, 107–122. [CrossRef]

48. Humphreys, F. The nucleation of recrystallization at second phase particles in deformed aluminium. Acta Met. 1977, 25, 1323–1344. [CrossRef]

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