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EFFECTS OF PRIOR ANNEALING ON THE MECHANICAL PROPERTIES OF A TWIST-EXTRUDED AA 7075 ALUMINUM ALLOY

VPLIV PREDHODNEGA ŽARJENJA NA MEHANSKE LASTNOSTI ZVOJNO EKSTRUDIRANE ALUMINIJEVE ZLITINE AA 7075

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This paper discusses the mechanical properties and grain refinement of an AA 7075 aluminum alloy twist-extruded at the cold-working temperature, and variations in the properties caused by prior annealing and as a function of the mechanical properties. Post-processing properties at room temperature, namely, the tensile strength, micro-hardness and microstructure were determined. An increase in the micro-hardness and tensile strength after three passes of twist extrusion (TE) was seen when compared with the starting annealed state. When prior annealing was done at 300 °C, the increase in the micro-hardness was 37 % and the increase in the tensile strength was 5 %. The corresponding figures for prior annealing at 400 °C were 34 % and 4 %, respectively, while for the 500 °C prior annealing, the figures were 31 % and 3 %, respectively. However, the rate of increase in the micro-hardness and tensile strength of a sample in the annealed state was higher at 300 °C than at 400 °C or 500 °C. In addition, a very fine structure of the AA 7075 aluminum alloy developed during the three TE passes as observed with a scanning electron microscope.

Keywords: twist extrusion, grain refinement, AA 7075 aluminum alloy, micro-hardness, tensile strength

V pričujočem članku avtorji opisujejo študijo sprememb mehanskih lastnosti in udrobljenje kristalnih zrn Al zlitine AA 7075, ki je bila izpostavljena hladni zvojni oz. torzijski ekstruziji (iztiskavanju). Pri tem je bilo izhodno stanje zlitine različno zaradi predhodne toplotne obdelave izvedene pri različnih temperaturah. Po izvedbi postopka hladnega iztiskavanja so pri sobni temperaturi določili natezno trdnost in mikrotrdoto zlitine ter analizirali njeno mikrostrukturo. Po treh prehodih skozi orodje za hladno zvojno ekstruzijo (TE), so avtorji študije ugotovili, da sta se povečali mikrotrdota in natezna trdnost zlitine v primerjavi z izhodnim žarjenim stanjem. Po predhodnem žarjenju pri 300 °C in iztiskavanju je mikrotrdota narasla za 37 % in natezna trdnost za 5 %, pri 400 °C predhodnem žarjenju in iztiskavanju je mikrotrdota narasla za 34 % in natezna trdnost za 4 %, pri 500 °C predhodnem žarjenju in iztiskavanju pa je mikrotrdota narasla za 31 % in natezna trdnost za 3 %. Vendar pa avtorji ugotavljajo, da je bil največji prirastek mikrotrdote in natezne trdnosti izmerjen na preizkušancih, ki so bili predhodno žarjeni na 300 °C in nato zvojno ekstrudirani. Analiza mikrostrukture na vrstičnem elektronskem mikroskopu (SEM) je pokazala, da je bila v vseh primerih dosežena zelo fina oz. drobnozrnata mikrostruktura Al zlitine AA 7075 po treh prehodih skozi orodje za hladno pregibno ekstruzijo.

Ključne besede: zvojna ekstruzija, udrobljenje kristalnih zrn, Al zlitina AA 7075, mikrotrdota, natezna trdnost

1 INTRODUCTION

Severe-plastic-deformation (SPD) methods, applied to aluminum alloys, gained importance in advanced metal forming for producing ultrafine-grained and nano-grained materials.[1] However, the high cost associated with these techniques is a matter of serious concern.[2] Many SPD techniques are available. These include planar TE,[3] high-pressure torsion (HPT),[4] twist-channel angular pressing (TCAP),[5] equal-channel angular extrusion,[6] equal-channel angular pressing (ECAP),[7] cold-rolled sheets,[8] pressing of formed castings (piston, plunger, piston plunger and through-gate runners),[9] friction-stir back extrusion (FSBE),[10] simple shear extrusion,[11] torsion and annealing,[12] torsion,[13] extrusion,[14] and TE.[15] A TE operation involves a hydrostatic load acting on a billet pressed by a plunger and pushed through a twisted channel with a β angle of slope of 36° and an θ angle for the a rotation of 90°.[16] The specimen shape remains unchanged after TE.

The effect of multiple passes on the strain suffered by a material was already studied.[17,18] M. Iqbal et al.[19] established grain refinement in the AA 7075-T6 aluminum alloy and its mechanical properties showed an improvement due to TE involving multiple passes. S. R. Bahadori et al.[7] examined the grain size and micro-hardness variations in pure aluminum using different SPD techniques of equal-channel angular pressing (ECAP) and TE with cold rolling (CR) as the post process. R. Kulagin et al.[20] carried out experiments that showed the cross-sectional flow in multilayer TE as a result of severe mixing of the material. This novel solution[4]

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ensured a more efficient grain refinement with fewer passes of TE, which made the rolling after TE easier. M. Tajally et al.[21] emphasized that the annealing temperature has a very significant effect on the recrystallization kinetics and its effect on the softening of aluminum alloy AA 7075. Moreover, selected TE specimens were made of the AA 7075 aluminum alloy due to its low density and high strength-to-weight ratio. Such alloys are used in various components of aerospace applications.[22,23] However, a lack of examination and improvement during the TE experiments of aluminum alloys is observed. This study aims at examining the effect of the annealing temperature on the TE behavior of aluminum alloy AA 7075, as indicated by the post-processing mechanical properties, including the tensile strength, micro-hardness and microstructural characteristics.

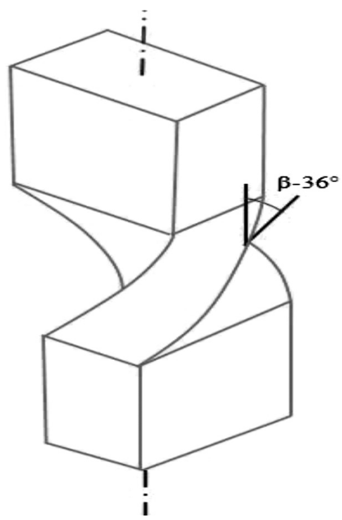

Figure 2: Schematic representation of the TE process

2 EXPERIMENTAL PART

The TE experiment was performed on a four-column vertical hydraulic press with the maximum plunger load of 100 tones as shown in **Figure 1**. Plates of the AA 7075 aluminum alloy were fabricated into billets with a rectangular cross-section of 28 mm × 18 mm and a length of 100 mm. Then, the billets were annealed at (300, 400 and 500) °C for 90 min, and cooled in a muffle furnace.

In order to develop ductility without a significant reduction in the strength of the particle structure, annealing was chosen as the most promising way to achieve a good combination of ductility and strength. A schematic representation of the TE process is shown in **Figure 2**. The experiments of TE were performed at a cold-working temperature (T_m of 0.4 °C where T_m is the melting temperature on the absolute scale) after three passes at the prior-annealing temperatures (at 300, 400 and 500 °C). The die had a twist channel of a slope line angle of 36° and a rotational angle of 90°. The samples were machined to the optimal size of 28 mm × 18 mm × 100 mm with a vertical milling machine. The ram velocity during TE was 2 mm s^{-1} and a uniform force was applied with the hydraulic press. In the present experiments, two input parameters, namely the prior-annealing temperature and the number of extrusion passes were used, each of which was varied at three levels. An orthogonal array of L_9 was chosen for the TE experiments. **Table 1** provides the details of the Taguchi experimental design for the L_9 orthogonal array. All these investigational conditions were applied at the prior-annealing temperature of the AA 7075 aluminum-alloy material.

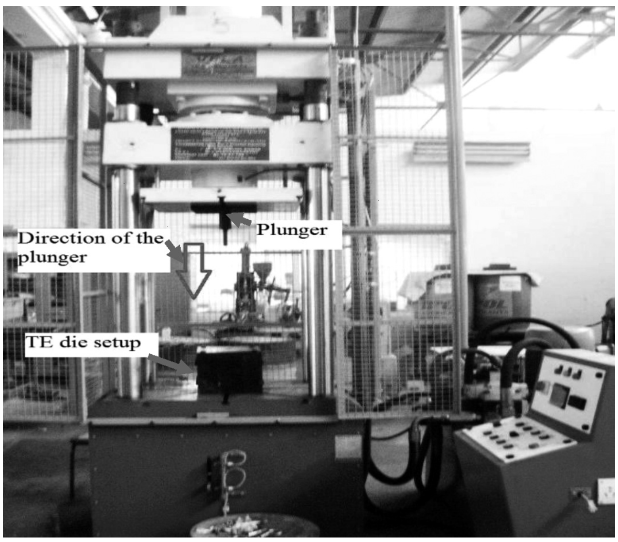

Figure 1: Four-column vertical hydraulic press for the TE forming process

Table 1: Experimental design for the TE process

S. No.	Prior-annealing temperature (°C)	Number of passes
1	300	1
2	300	2
3	300	3
4	400	1
5	400	2
6	400	3
7	500	1
8	500	2
9	500	3

The twist-extruded samples were tested for the room-temperature tensile strength and Vickers micro-hardness (a Wolpert device) by applying a load of 500 g (HV 0.5 kg) and a dwell time of 10 s. The tensile samples were prepared according to ASTM-B557M-10, a standard with a fillet radius of 6.5 mm, gauge thickness of 6 mm and gauge length of 28 mm. For the microstructure investigation, the pieces were polished and etched with Keller's reagent. Microstructure and elemental-composition studies were conducted using a Hitachi S-3400 N-type scanning electron microscope (SEM) with energy-dispersive X-ray spectroscopy (EDS). The

Table 2: Experimental results for the micro-hardness and tensile strength of the AA 7075 aluminum alloy subjected to TE

S. No.	Sample state	Tensile strength (MPa)	Percentage increase in the tensile strength (%)	Micro- hardness (HV)	Percentage increase in the micro-hardness (%)	
1	500 °C	As-annealed	396.5	-	87	
2		One pass	400.6	1	107	**23**
3		Two passes	409.4	3	115	**32**
4		Three passes	411.1	3	118	**35**
5	400 °C	As-annealed	408.3	-	92	**-**
6		One pass	419.5	2	111	**21**
7		Two passes	424.5	3	120	**30**
8		Three passes	426.4	4	126	**36**
9	300 °C	As-annealed	415.0	-	98	**-**
10		One pass	423.3	2	117	**19**
11		Two passes	428.4	3	125	**27**
12		Three passes	436.2	5	139	**42**

grain-size distribution was obtained using the ImageJ software.

3 RESULTS AND DISCUSSION

3.1 Tensile strength

The TE experiments conducted on the AA 7075 aluminum alloy were subjected to different annealing temperatures and several passes during the TE process. The three annealing temperatures were (300, 400 and 500) °C. **Figure 3** shows the tensile-strength specimens prepared after different TE experiments. The tensile strength was measured after each pass of the extrusion. The variations in the tensile strength of the specimens in the as-annealed state and after one, two or three TE passes are shown in **Table 2**. The percentage increase as a result of each of these changes is also included in **Table 2**. The room-temperature tensile strength of the samples annealed at (300, 400 and 500) °C increased from 415 MPa to 436.2 MPa, from 408.3 MPa to 426.4 MPa and from 396.5 MPa to 411.1 MPa, respectively, after three passes of TE. The increase in the tensile strength of the specimens in the annealed state, at (300, 400 and 500) °C, and TE-processed during three passes were about 5 %, 4 % and 3 %, respectively.

Therefore, the TE processing at 300 °C was seen as a more effective grain refinement compared with the processing at a higher temperature. After three passes of the TE experiments, the tensile-strength rate resulted in the minimum rate of enhancement in the samples prior-annealed at 400 °C and 500 °C. An improvement in the TE samples[12,24] was expected to occur at a lower prior-annealing temperature and an increased number of passes.

Figure 3: Samples before the tensile-strength testing and after annealing at different temperatures (500, 400 and 300) °C and then subjected to different TE steps

Figure 4 shows the tensile-strength values corresponding to different annealing treatments with various passes of TE. The prior-annealing temperature of 300 °C and the increasing number of passes during TE clearly cause an increase in the tensile strength.

3.2 Micro-hardness

Table 2 summarizes the variations in the micro-hardness before and after TE with 1, 2 and 3 passes. Micro-hardness was measured at three locations for each sample and the average value was reported. After three passes of TE, the micro-hardness of the samples annealed at (500, 400 and 300) °C showed increases from 87 HV to 118 HV (a nearly 35-% increase), from 92 HV to 126 HV (a nearly 36-% increase) and from 98 HV to 139 HV (a nearly 42-% increase), respectively, as shown in **Table 2**. A comparison of these three annealed states led to a conclusion that the micro-hardness after the annealing at 300 °C and after three passes of TE was

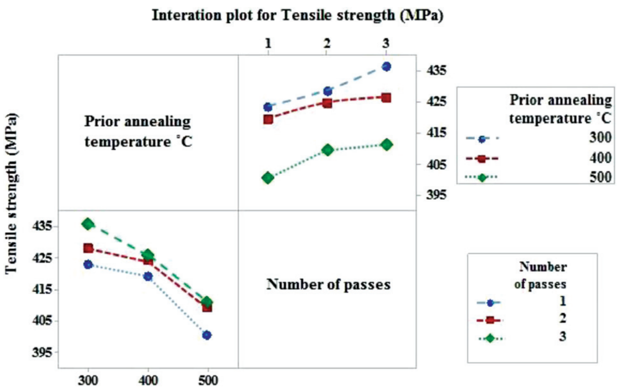

Figure 4: Tensile-strength values corresponding to different treatments

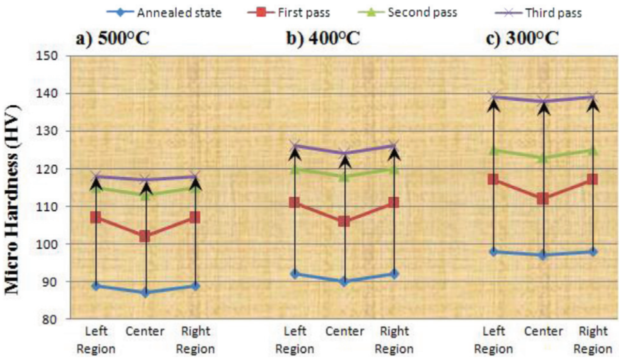

Figure 5: Micro-hardness at different locations before and after TE for the billets of the AA 7075 aluminum alloy annealed at different temperatures: a) 500 °C, b) 400 °C and c) 300 °C

higher than after the annealing at 400 °C or 500 °C as the formations of $MgZn_2$ and MgZn were observed. The creation of an enhanced cyclic-torsion effect in the billet, due to TE with an increased number of passes, was found. This effect led to a finer grain structure and improved mechanical properties. Besides, the micro-hardness was also measured along the transverse section (the left side, center and right side) on the cross-section at 90° to the TE direction and the results are presented in **Figure 5**.

The hardness of the billets was found to be 117 HV and 118 HV in the middle and the peripheral region on the transverse section (a nearly 1-% increase), respectively, after three TE passes for the material annealed at 500 °C. For the samples TE-processed after prior annealing at 400 °C and 300 °C, the micro-hardness in the middle and in the peripheral regions of the billets was 124 HV and 126 HV (a nearly 1-% increase) and 138 HV and 139 HV (a nearly 1-% increase), respectively. However, the conclusion was that the percentage variation was of a very minimum value in the TE experimental investigation. A larger strain was achieved at the corner location than at the center region as shown by the micro-hardness results. The billet peripheral areas suffered plastic deformation during TE due to the contact between the billet and the die and the shear distortion in those regions compared with the central region of the billet. This observation was consistent with the earlier results.[16,25] This variation in the strain distribution was reduced by the performance of the increasing TE passes.[26] The TE experiments made the micro-hardness more homogeneous compared to the other severe plastic-deformation techniques. **Figure 6** shows the micro-hardness values corresponding to different annealing treatments with various TE passes. It is observed that the prior-annealing temperature of 300 °C and the increased number of passes during TE tend to increase the micro-hardness. However, the specimen that underwent prior annealing at 300 °C exhibited a higher hardness than those annealed at 400 °C and 500 °C, due to the formations of $MgZn_2$ and MgZn and the particle refinement.

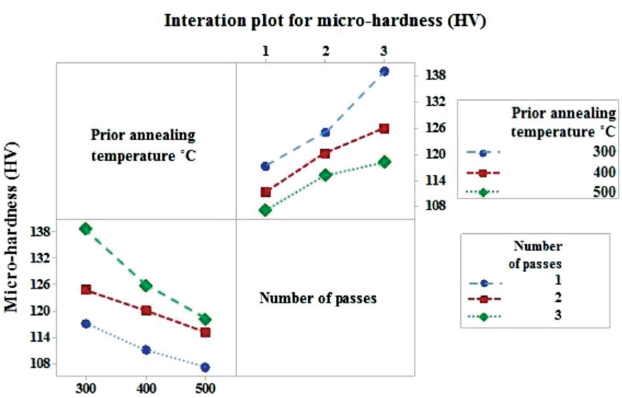

Figure 6: Micro-hardness values for the AA 7075 aluminum alloy corresponding to different treatments

3.3 Microstructure

A TE billet was characterized using a SEM (**Figure 7**). A microstructural investigation of the structural changes led to the observation of the formation of various precipitates formed (**Figure 7a** to **7c**) in the AA 7075 aluminum alloy when annealed at various temperatures for a constant annealing time.

Phase precipitates of MgZn, $MgZn_2$ (at the prior-annealing temperature of 300 °C and 400 °C), Al_2Cu and AlCuMg (at the prior-annealing temperature of 300 °C and 500 °C) were formed from the supersaturated solid solution, which was confirmed through an

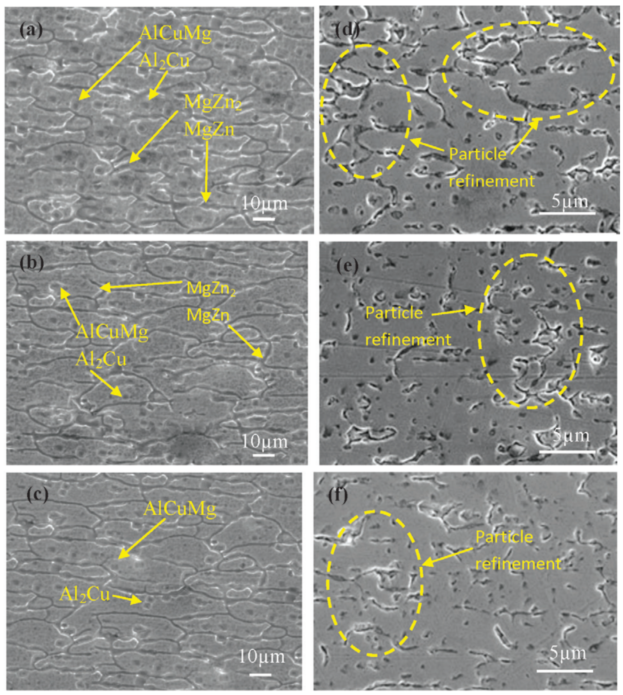

Figure 7: SEM surface morphologies of the AA 7075 aluminum alloy in the starting condition prior to TE and after three TE passes: annealed at: a) 300 °C, b) 400 °C, c) 500 °C; and after TE: d) three passes after annealing at 300 °C, e) three passes after annealing at 400 °C, f) three passes after annealing at 500 °C

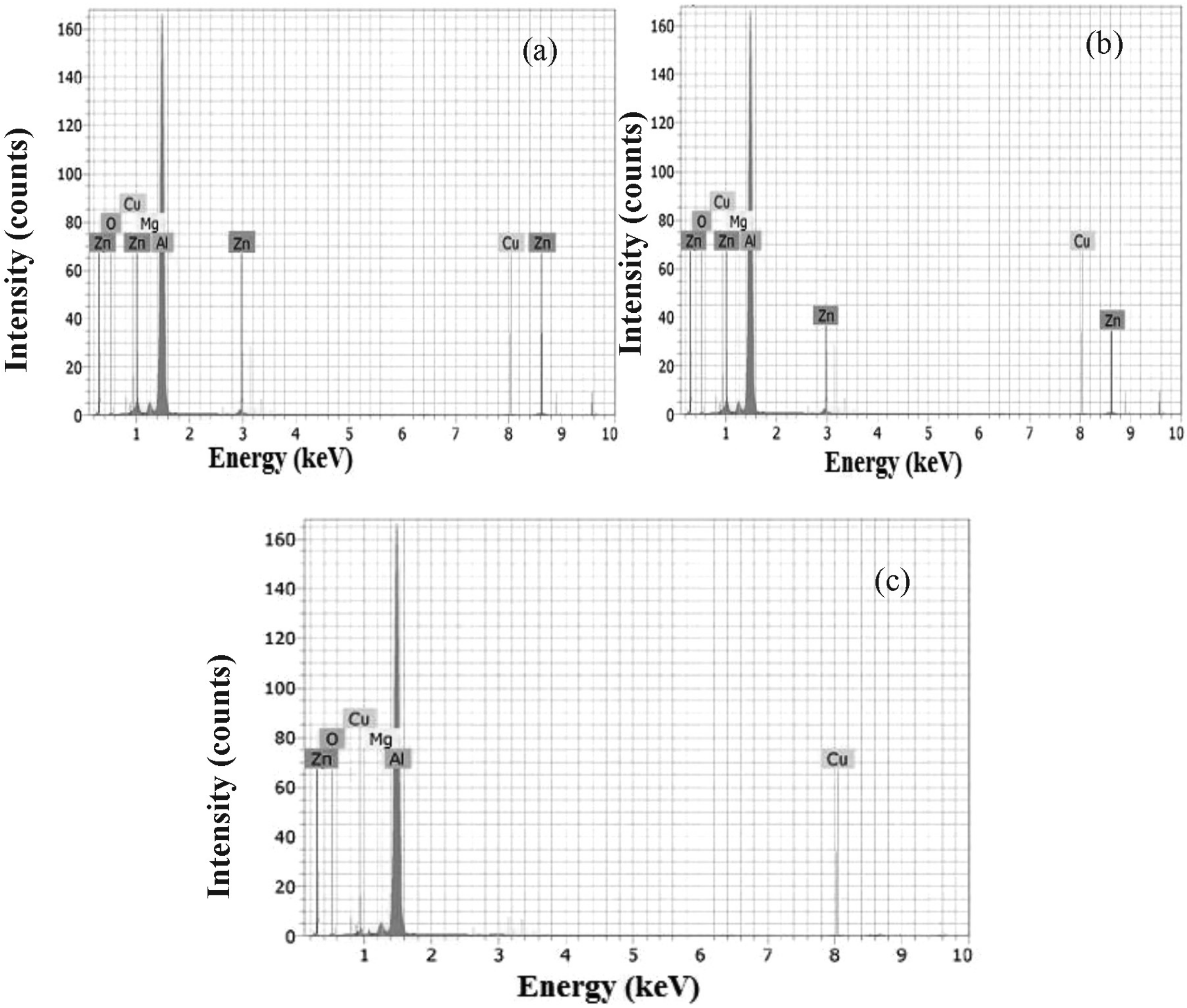

Figure 8: EDX analysis after prior annealing at: a) 300 °C, b) 400 °C, c) 500 °C

EDS analysis as shown in **Figure 8a** to **8c**. The annealed state suggests a reduction of the intermetallic precipitates and dissolution of the Mg, Zn and Cu grains in the Al matrix. The same results were reported for the isothermal annealing process.[27] The examination showed minimum dislocations within the grains after the first pass and a significant increase in the dislocations after three TE passes. **Figure 7d** to **7f** shows a significant change in the particle structure observed in a deformed TE billet.[22]

To depict the grain refinement, one can utilize the model proposed by H. Zendehdel et al.[25] based on the idea that once a plastic deformation starts, dislocation density increases bringing about the development of sub-boundaries that block separation developments. Progressively, due to the aggregation of forced plastic strains and misorientation of the adjoining grains, the stretched grains tend to split into smaller grains forming an equiaxed microstructure. This is a phenomenon similar to the one seen in SPD procedures, called a unique continuous recrystallization. New grains are formed with the increased number of TE passes.[28] The plastic flow during TE is defined by two principal processes: a) A vortex-like billet that flows with the strain gradient stretches across one path of the material elements and their mixture. The grain elongation and bending in successive TE passes are seen. b) A constant lamellar-flow pattern is noticeable with the increased number of TE processes.[29] The net effect is the accumulation of the micro-strains within the grains.[30] With the increased number of TE passes, the formation of fine, new grains with small-angle boundaries, which, with further strain, become converted into grains of high-angle boundaries, is in place during the TE processes. The grain size becomes finer and the microstructure becomes more homogeneous. This is confirmed with the ImageJ software.[31,32]

4 CONCLUSIONS

The effects of prior annealing and the TE process with various passes on the mechanical characteristics of

AA 7075 aluminum-alloy samples were studied. The following conclusions are drawn from the results of the investigation.

The two major factors that influence a billet's tensile strength and micro-hardness are the prior-annealing temperature and the increased number of TE passes. The TE experiments show an enhancement in the billet micro-hardness and tensile strength by 35–42 % and 3–5 %, respectively.

According to the investigation, there was an increase in the micro-hardness and tensile strength of a specimen with a higher number of passes after prior annealing at 300 °C due to the development of strengthening phases ($MgZn_2$, MgZn) and a particle refinement. However, the rates of increase in the micro-hardness and tensile strength were higher for the sample annealed at 300 °C than for the samples annealed at 400 °C and 500 °C.

The micro-hardness of the billets annealed before TE was found to be (87, 92 and 98) HV for the material annealed at the temperatures of (500, 400 and 300) °C, respectively, and the micro-hardness of these specimens increased to (118, 126 and 139) HV after three passes of TE. The tensile strength and micro-hardness found for the sample annealed at 300 °C were higher by 1.5 % and 6 % than those for the sample annealed at 500 °C.

Several categories of precipitants including MgZn and $MgZn_2$ (at prior-annealing temperatures of 300 °C and 400 °C), AlCuMg and Al_2Cu (at prior-annealing temperatures of 300 °C and 500 °C) were formed. The TE effect on the microstructural changes of the AA 7075 aluminum alloy was examined. Moreover, the AA 7075 aluminum alloy achieved a very fine structure after three passes of the TE treatment.

The investigation shows that the strain of the material becomes higher between the initial pass and the final pass (the third pass), indicating an increase in the homogeneity of the strain distribution due to a higher number of passes.

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