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Research Paper

Evaluation of Microstructure and Tensile Behavior of Fine-Grained AZ61 Alloy Tube Processed by Severe Plastic Deformation

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ABSTRACT

The aim of this study is to find the effect of the Parallel Tubular Channel Angular Pressing (PTCAP) technic as a Severe Plastic Deformation (SPD) method on microstructure and mechanical properties of as-extruded AZ61 magnesium alloy. The main reason to accomplish this research is to achieve certainty while this process could enhance the mechanical characteristics of magnesium alloy. To this end, the initial material was processed for one, two and three passes at 350 °C. Afterward, the microstructure was studied by optical microscope (OM) and scanning electron microscope (SEM). Next, to verify the mechanical properties alterations, tensile tests were performed for each specimen. Then, in order to investigate stress and strain status during the process, process simulations were fulfilled by employing the software Abaqus. Microstructure investigations revealed the fact that after just one pass, great grain refinement occurred within the material. Al₄Mn as a secondary phase was noted for 1-pass, 2-pass and 3-pass processed specimens via scanning electron microscopy images and Energy Dispersive X-ray Spectroscopy (EDS) patterns. Finite elements method results illustrated the highest value of stress for the second half-cycle of the third pass. The maximum amount of strain tolerated by material belonged to the second half-cycles of the second and third pass. Finally, it could be reasoned that the best properties achieved for the 2-pass processed specimen possessed the best strength and deformability values.

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1. Introduction

In order to have low-weight products, magnesium could be the best material to be used. Hence, nowadays, magnesium has attracted huge attention. This attraction is obviously considerable for transportation vehicles such as automotive factories, airplanes, trains, etc. There are positive aspects that encourage using this material, such as high specific strength, good weldability, and castability. Nevertheless, some negative perspectives restricted using magnesium and its alloys. Principal drawbacks could be summarized as low Young modulus, low strength, and limited corrosion resistance [1]. The alloying element which is employed enormously in magnesium alloys is aluminum. The addition of this element to pure magnesium could result in an increase in strength, hardness, and castability. However, adding more Al would decrease formability [2]. In addition, aluminum and other alloying elements present in the alloy form secondary phases so that affect strength, ductility, corrosion resistance, etc. The most famous magnesium alloys are named AZ and AM. Other elements present in this kind of alloy, besides magnesium and aluminum, are zinc and manganese. AM alloys are mostly employed to use for die-casting applications, while AZ ones are designated for wrought utilizations [3]. The microstructure of alloys, including magnesium alloys, strongly affects mechanical properties. During the last decades, works have been accomplished to demonstrate the fact that by grain refinement process, a UFG microstructure was achieved, which resulted in ameliorated mechanical properties [4, 5]. Nowadays, this field of science has become a matter of interest for researchers due to its positive aspects attributed to UFG materials. This kind of materials with grain size considerably smaller than their CG counterparts exhibit improved mechanical properties, especially strength [6]. Today, many new techniques exist to develop fine-

grained metals from CG ones. Several techniques are available such as the Equal Channel Angular Pressing method (ECAP) [7], High-Pressure Torsion (HPT) [8], Accumulative Roll Bonding (ARB) [9], and Cyclic Extrusion Compression Angular Pressing (CECAP) [10]. There are also techniques to process circular tubes to attain a UFG metal. Tubular Channel Angular Pressing (TCAP) [11] is one of these processes. Afterward, Faraji et al. proposed a new process named Parallel Tubular Channel Angular Pressing (PTCAP) [12] for the fabrication of UFG and nanostructured metals. This new process has some positive aspects, such as lower force needed to accomplish the process and homogeneous strain, which is obviously higher than TCAP [13]. Fig. 1(a) indicates the first half cycle of the PTCAP process where the specimen is constrained between the punch, mandrel, and die wall. The punch used here has a smaller diameter than the other, which is designated for the next pass. In order to accomplish the second pass, a punch having a larger diameter is applied (Fig. 1(b)). Also, Faraji et al. [14, 15] performed plastic deformation analysis in the PTCAP process. They found out that an increase in the channel angles leads to a decrease in the imposed strain on the PTCAP processed tube. A complete study on severe mechanical anisotropy of high-strength UFG Cu-Zn tubes processed by PTCAP was done by Faraji et al. [16] realized that there was a severe anisotropy in UFG samples compared to CG counterparts. The research was accomplished on microstructure and homogeneity of semi-solid 7075 aluminum processed by PTCAP by Faraji et al. [17] observed that as the number of passes was increased, uniform distribution can be achieved, and globalization occurred faster [17]. In another research, mechanical anisotropy in UFG aluminum tubes processed by PTCAP was discussed by Hashemi et al. [18]. As a result, greater strength was obtained along the peripheral direction for PTCAPed specimen.

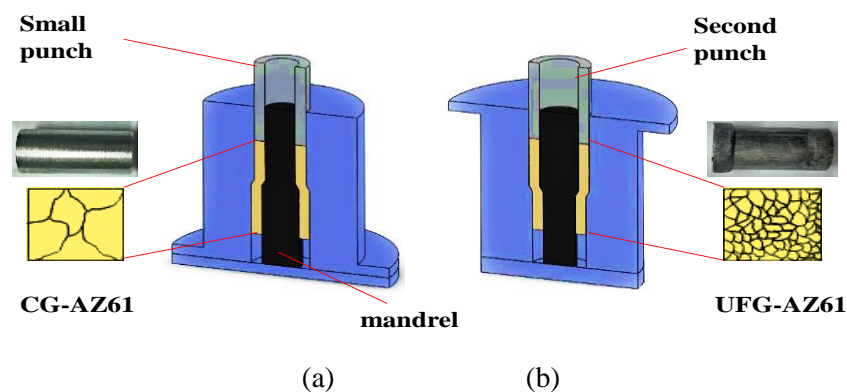


Fig. 1. Schematic of PTCAP process; first half (a) and the second half (b)

In this work, as-extruded tubular AZ61 magnesium alloy was selected as starting material. Afterward, it was PTCAPed via one, two, and three passes. Next, mechanical properties were explored in order to investigate the alterations due to the applied process. Optical microscopy was employed to study the microstructure, and grain refinement appeared after the process. To have a more profound study concerning the microstructure, scanning electron microscopy (SEM) was used for each material.

2. Materials and methods

2.1. PTCAP die

A PTCAP die was designed and manufactured from hot work steel (H13) and hardened up to 50 HRC. The SolidWorks design of this die is illustrated in Fig. 1. All works concerning engineering design, material preparations, and finally forming the die was fulfilled at the college of engineering, University of Tehran.

2.2. Specimens' processing

As-extruded AZ61 magnesium alloy was selected as the primary material (Fig. 1(a)) in which the composition is presented in Table 1. Next, it was machined to a tubular shape having an outer diameter of 20 mm, a thickness of 2.5 mm, and a length of 40 mm. PTCAP process was applied for one, two, and three passes. In fact, the specimen was put into the die containing a mandrel, and a first punch press the

specimen, and shear stress was imposed to the periphery of the tube by the mandrel. So, an enlarged diameter was attained for the specimen. Afterward, the die was placed upside down, and a second punch which was bigger than the former, pushed the specimen through the die, and therefore the specimen was deformed via the mandrel, and the diameter was decreased to the first size. This process was accomplished by an INSTRON press machine at a temperature of 350 °C and at a ram speed of 10 mm/min. It has benefited from Molybdenum disulfide (MoS₂) as a lubricant to reduce the friction between the specimen and die wall and mandrel.

2.3. Microstructure studies

The microstructural evolution of unprocessed and PTCAPed specimens was characterized by an optical microscope. To this end, small samples were cut in the process direction, and they were finished with SiC papers (Nos. 120-2000) under tap water. Next, they were all polished in the polishing machine in order to achieve maximum smoothness. Afterward, to be able to perceive the microstructure, samples were acid-etched in a solution of 4 g picric acid, 10 ml acetic acid, 70 ml ethanol, and 20 ml distilled water. To perform a better study of microstructure, scanning electron microscope and EDAX photos were prepared. For this, an SEM model Hitachi S4160 at a voltage of 20 kV was used.

Table 1 Chemical composition of AZ61

Element	Al	Zn	Mn	Si	Fe	Ca	Mg
Mass(%)	6.2	0.74	0.23	0.04	0.004	0.0013	Bal.

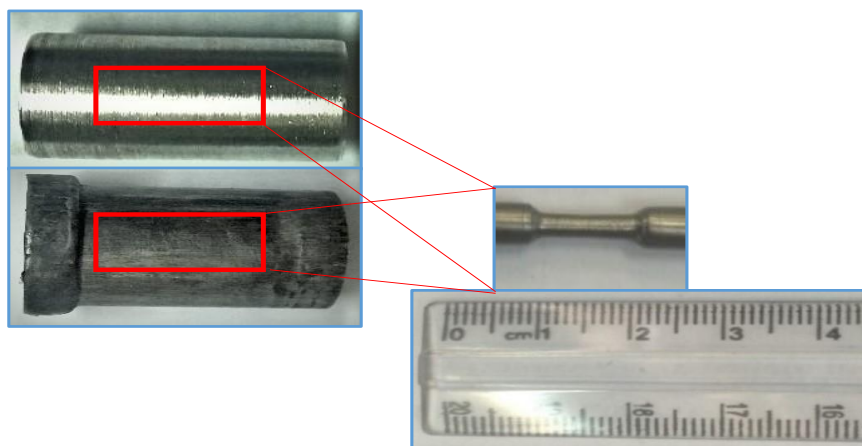


Fig. 2. Tensile sample shape and size

2.4. Mechanical properties

In order to achieve mechanical properties, tensile tests were performed on as-received, one-pass, two-pass, and three-pass PTCAPed specimens. To this end, all materials were sectioned in accordance with ASTM E8 requirements (Fig. 2). Thus, these tensile samples were prepared by electro-discharge machining. For Vickers microhardness, a COOPA model microhardness machine was employed. Tests were performed at a load of 50 g applied for 5 s.

2.5. FEM

In the present study, by using Abaqus software, simulation of different passes for the PTCAP process

of AZ61 magnesium alloy at 350 °C was performed. To this end, the plastic region of true stress- true strain curves offered by the tensile test was used. It should be mentioned that for the rigid PTCAP die, mechanical properties of hot worked steel H13 were intruded as data. The form of meshing was selected as “Element Shape: Hex Dominated”. During the process, because of using molybdenum disulfide, the friction coefficient between a specimen, die wall, and mandrel was selected 0.08.

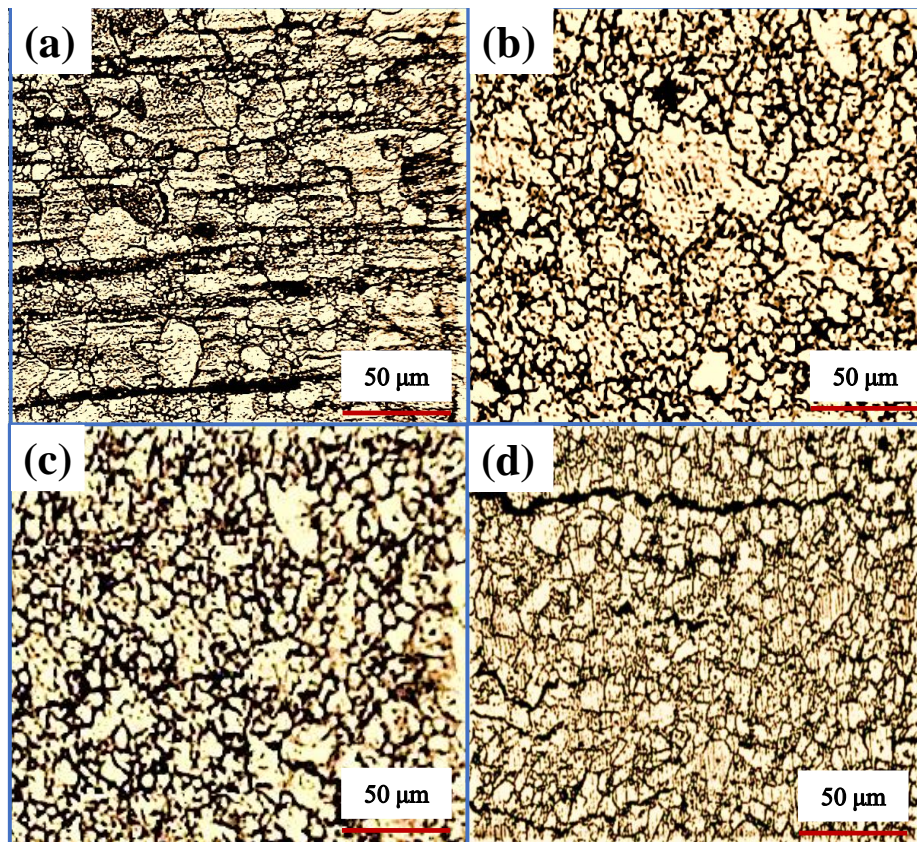


Fig. 3. OM images of as-received (a) and 1-pass (b), 2-pass (c) and 3-pass (d) PTCAPed specimens

3. Results

3.1. Discussion on microstructural evolution

Photos achieved by optical microscope for as-received, 1-pass, 2-pass, and 3-pass PTCAPed specimens are illustrated in Fig 3. As it could be perceived from Fig. 3(a), the number of coarse grains has dominance over the fine ones. As depicted in Fig. 3(b), after only one pass, a considerable grain refinement has occurred in the microstructure so that the grain size decreased from a starting value of $\sim 17 \mu\text{m}$ to $\sim 9 \mu\text{m}$. In fact, new fine grains developed from previous coarse grains by dynamic recrystallization (DRX). Nevertheless, the microstructure includes

fine and coarse grains, which means that the microstructure is bimodal. Afterward, the third pass was applied to the specimen. As it could be figured out (Fig. 3(c)), fine grains are more present in the microstructure, and the intensity of the bimodal microstructure has decreased. Yet, the number of coarse grains is visible, which demonstrates that the DRX process has not been completed. Grain size after the 2-pass PTCAP process decreased to $\sim 6 \mu\text{m}$. Fig. 3(d) illustrates the specimen processed for three passes. The first fact which is obvious in this figure is the homogeneity achieved by applying more plastic strain to the sample. In fact, newly refined

grains have covered all the microstructure, and the primary coarse grains have acted as mother grains to give birth to these recrystallized grains. The second fact is the appearance of intergranular cracks, which could be a result of brittleness that occurred in the third pass. However, the smallest grain size, $\sim 4 \mu\text{m}$, was achieved for this specimen. This result shows the capability of this process for grain refinement. Meanwhile, besides the grain size, another important subject that affects the microstructure is a secondary phase or intermetallic. In order to investigate the probable existence of secondary phases, SEM micrographs and EDS patterns were prepared for each sample. Fig. 4(a) illustrates the presence of a component in the as-received sample, which contains mostly aluminum. This could be $\beta\text{-Mg}_{17}\text{Al}_{12}$ precipitate which its influence on the microstructure of AZ magnesium alloys is investigated by Braszczyńska-Malik [19,20]. However, by increasing the temperature up to $350 \text{ }^\circ\text{C}$ and applying

for the first pass, no $\beta\text{-Mg}$ remained in the microstructure (Fig. 5). This fact could be detected from thermodynamic calculations, which proves the presence of the $\beta\text{-Mg}$ phase up to high temperatures (Fig. 5). As it could be noticed, by increasing the temperature, the $\beta\text{-Mg}$ phase begins to vanish. After the second pass (Fig. 4(c)), the concentration of aluminum in the Al-Mg phase decreased, which could be due to the dissolved Al in the microstructure. What could be seen from Fig. 4(d) for the third pass is a light trace of the precipitate. The reason for this kind of precipitate could be the imposition of 3 successive passes in high temperatures, which forced the precipitate to be dissolved in the microstructure. Fig. 6. presents the amount of precipitates for as-received and also PTCAPed specimens. It should be mentioned that by increasing the number of passes, precipitates content of microstructure decreased.

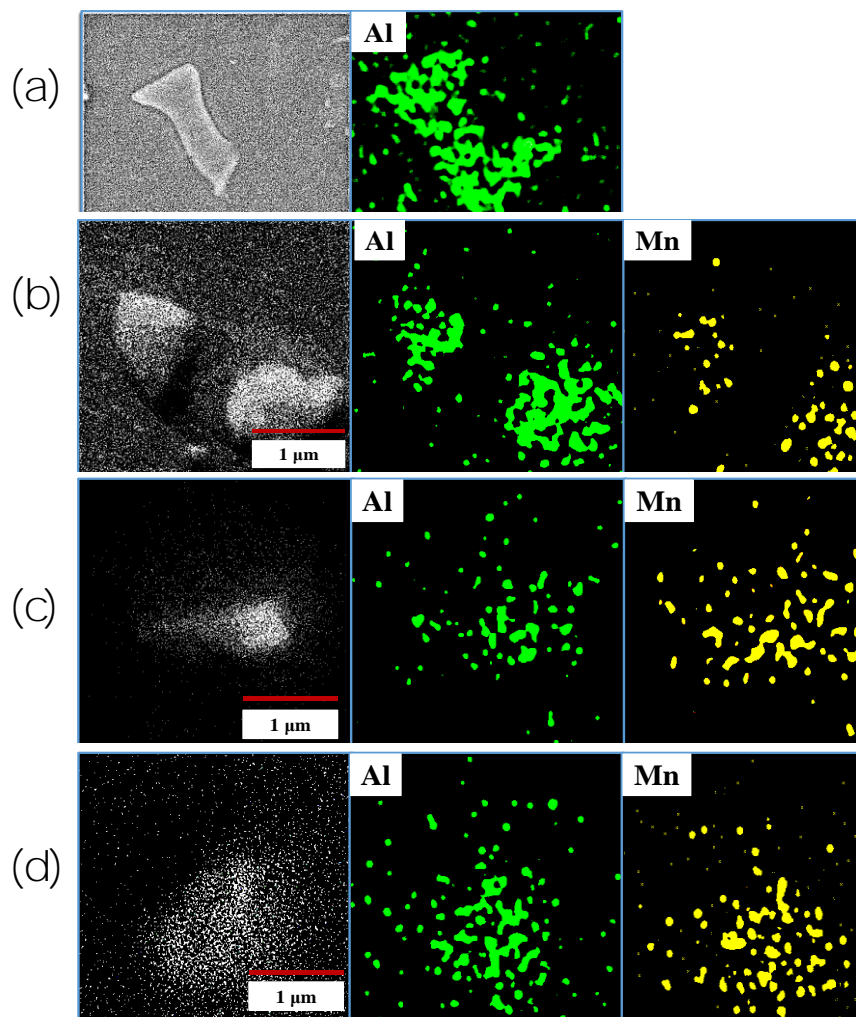


Fig. 4. SEM micrographs and EDS patterns of as-received (a) and 1-pass (b), 2-pass (c) and 3-pass PTCAPed specimens

3.2. Mechanical properties

To study the mechanical properties and to be able to investigate the alterations which could be happened after each pass, the tensile test was performed for all specimens at room temperature. It could be observed that the UTS increased from an initial value of ~100 MPa to ~144 MPa after applying just one pass of the PTCAP process. In fact, this increase might be a result of great grain refinement (17 μm to 9 μm) caused by applying high shear stress to the material. On the other hand, according to EDS patterns (Fig. 4) and thermodynamic calculations (Fig. 5), Al_4Mn as a secondary phase has formed normally in grain boundaries which this phenomenon limited dislocations migration from one grain to another via grain boundaries so that UTS enhancement achieved. Also, according to OM microstructures illustrated in the previous section, unlike the as-received specimen, the microstructure of the 1-pass PTCAPed sample is more homogeneous, which offered an increase in the formability of the material up to ~4.6%, which is two times the formability of as-received sample. In addition, ImageJ software was employed to discuss alterations in precipitation presence applying the PTCAP process. It should be noticed that one pass applied, the percentage of

precipitates decreased (Fig. 6) as a result of $\text{Mg}_{17}\text{Al}_{12}$, the subject that might be interpreted as another reason for formability enhancement. After the second pass, the strength was almost equal to the former pass, but a huge increase in formability was noticed. Based on OM microstructures (Fig. 3), the following explanation is given: a quite homogeneous microstructure was achieved for the second pass, and almost no trace of bimodality was observed. But, it could be seen that the strength of the 2-pass processed sample is approximately equal to the 1-pass one. The reason could be the annihilation of precipitations in the second pass (Fig. 4(c) and Fig.6). In fact, the presence of precipitates in a sample is one of the factors which could ameliorate the strength. As illustrated in Fig. 7, for the three pass processed specimen, mechanical properties deteriorated, and they were even less than those of as-received material. One reason could be the appearance of cracks in the microstructure (Fig. 3(d)). The reason for that should be the high degree of brittleness reached after 3 passes. Another reason is the decreased percentage of the secondary phase due to the dissolution of this phase in microstructure resulted from heating material up to 350 °C three times.

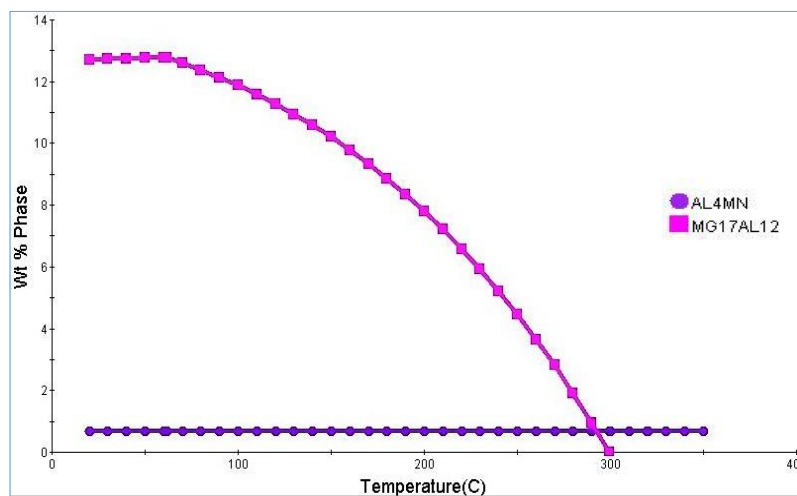


Fig. 5. Thermodynamic calculations

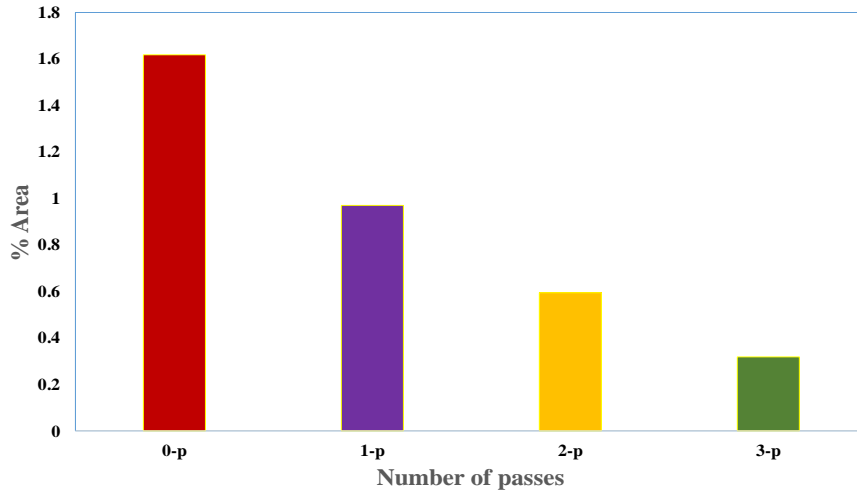


Fig. 6. Percentage of precipitates for each specimen

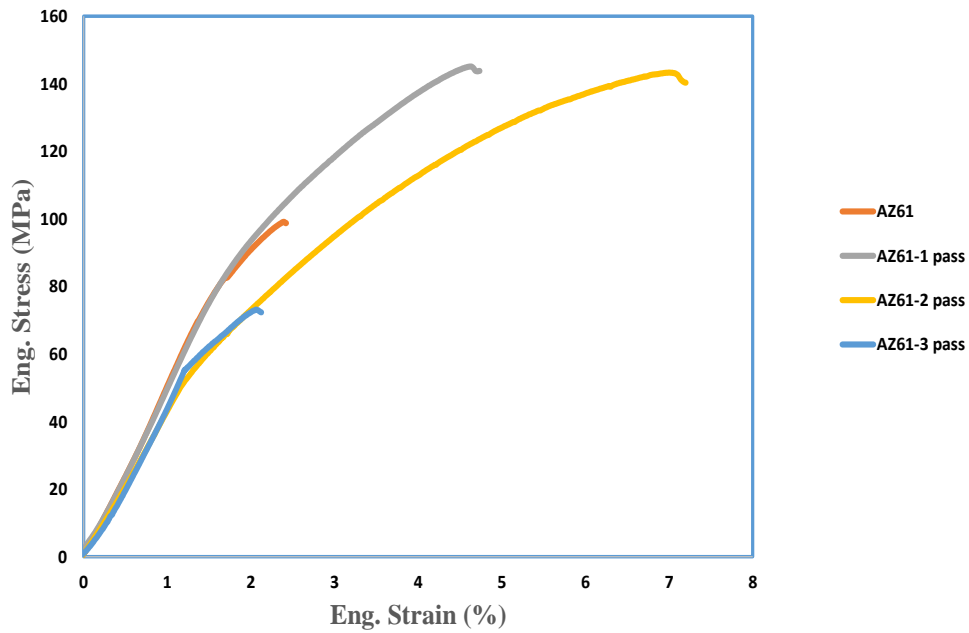
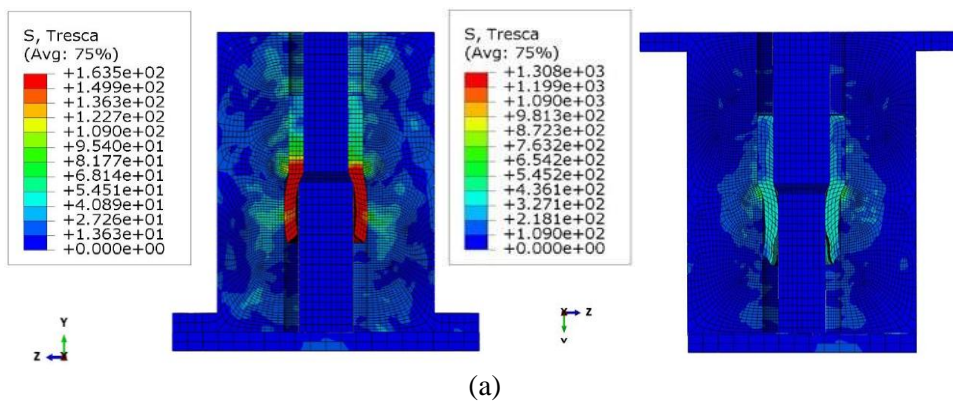


Fig. 7. Stress-strain curves of as-received, 1-pass, 2-pass and 3-pass PTCAPed specimen



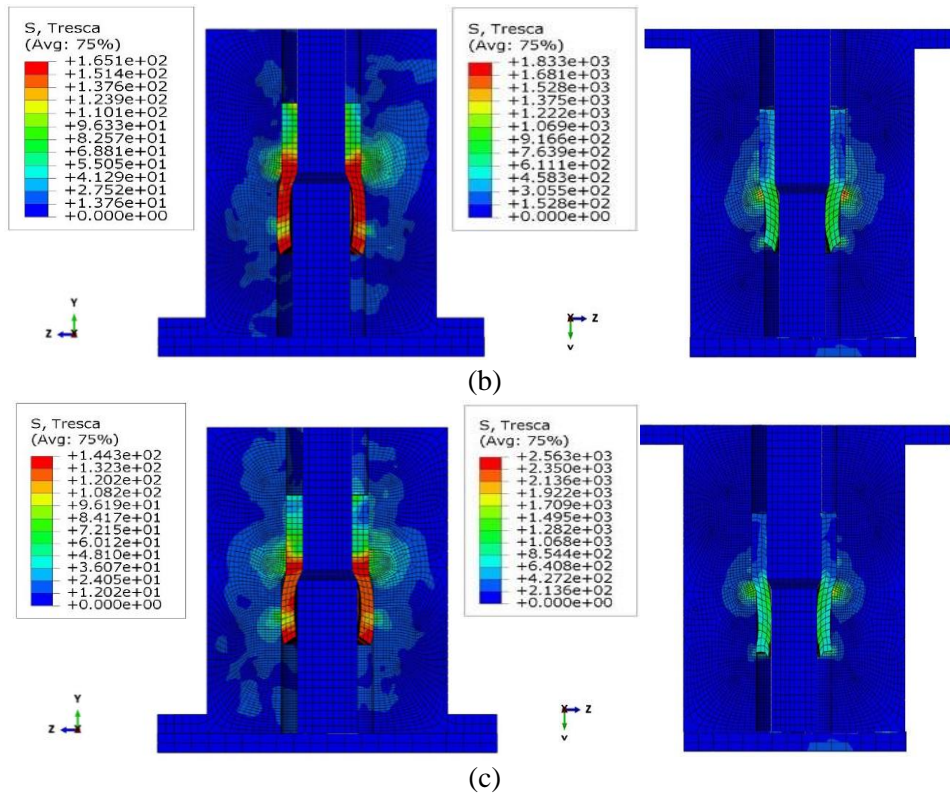
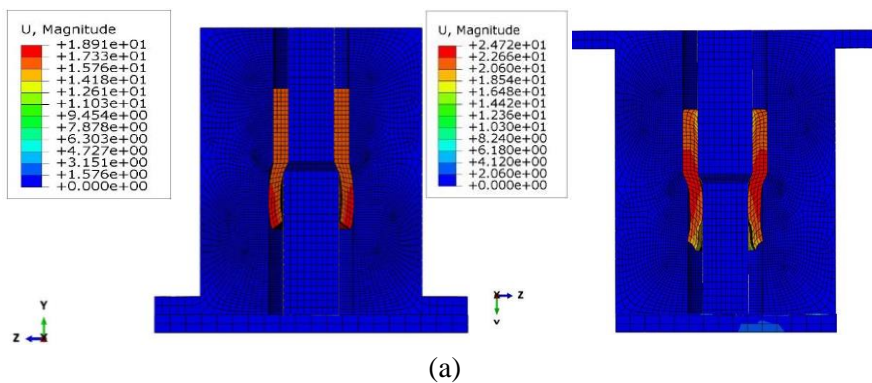


Fig. 8. Stress conditions of samples during the first pass (a), second pass (b) and third pass (c) of PTCAP process

3.3. FEM

According to PTCAP process simulations for different passes of AZ61 magnesium alloy, offered by the software Abaqus, at the first pass, the maximum shear stress belongs to the first half-cycle (Fig. 8(a)). The reason for this could be the first deformation process applied to a material containing coarse grains and a non-homogeneous microstructure. Therefore, the stress decreased for the next half-pass due to reaching a microstructure

being less bimodal. For the second pass, a small increase in shear stress was observed. In fact, it should be explained that by applying for the first pass, the strength of the material improved (Fig. 7), which makes the next deformations more difficult. For the third pass at the second half-cycle, the stress had the maximum value in comparison with other steps. Actually, by imposing the process three times on the material, the brittleness occurred in which the outcome was observed in the simulations (Fig. 8(c)).



(a)

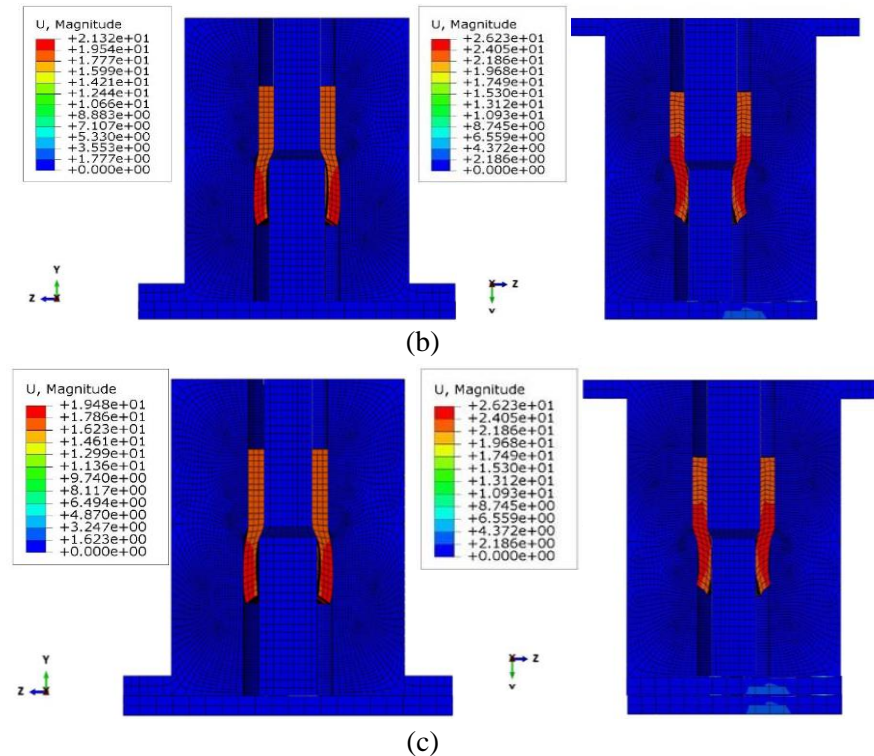


Fig. 9. Strain status of samples during the first pass (a), second pass (b) and third pass (c) of PTCAP process

By considering the simulations accomplished for strain conditions (Fig. 9), for each pass, second half-cycles possessed the biggest strain. By comparing the amounts of strain that belonged to 1-pass and 2-pass samples, by considering the experimental results, it is obvious that the strain was higher for the 2-pass sample, which this subject was clearly presented in the simulations (Fig. 9(b)). Following the process until the third pass, a considerable fall in strain was noted for the first pass, while for the second pass, it was not the same condition. In fact, because of the brittleness that occurred for the third pass, the amount of strain decreased for the first half-cycle. Nevertheless, as a result of the shape of the die, the strain increased in the next half-cycle.

4. Conclusions

- In this experimental and numerical study, the attempt was to process the tubular AZ61 magnesium alloy in order to have a better material possessing enhanced mechanical properties. To this end, the PTCAP process was applied up to 3 passes on as-extruded AZ61 material at 350 °C, and the following results were achieved:

- Starting material contained coarse grains. After just the first pass of the process, great grain refinement occurred from an initial value of 17 μm to 9 μm . However, most refined grains were achieved in the third pass (4 μm). Nevertheless, the homogeneous microstructure belonged to the 2-pass sample.

- Microstructure of the 3-pass specimen contains some cracks which could deteriorate mechanical properties. On the other hand, by considering the images procured by SEM micrographs and EDS patterns, by imposing the process, a new Al4Mn phase was formed, and meanwhile the β -Mg phase diminished as a result of heating applied during the process. Also, the percentage of the secondary phase decreased as a result of both processing and heating the material. In fact, the secondary phase dissolved in the microstructure.

- The first pass applied, the strength of material increased by 44% compared to the unprocessed sample and attained its maximum value of 144 MPa. Also, the formability improved to nearly twice the initial material after the first pass. However, the best elongation was registered for the 2-pass specimen having a value of $\sim 7\%$. By applying the technic up to 3 passes, mechanical properties were damaged. Strength and formability decreased below the values of the initial material. One reason for that was the cracks present across the material. The other reason is the lessened percentage of the second phase for the third pass. The brittleness of material that occurred at this stage could be another proof of the deterioration of mechanical properties.

- FEM results for stress conditions illustrated that the maximum amount of stress tolerated by the

material was for the second half-cycle in the third pass. In fact, because of the brittleness of the material, shear stress increased to a maximum value to be able to deform the material. The highest value of strain bore by the material was also for 2-pass and 3-pass processed samples. In reality, the 2-pass sample had a homogenous microstructure, and the deformation was almost uniform. But it was not the case for the third pass. What happened for the 3-pass specimen was the advent of cracks which this elevated amount of strain could be related to their appearance.

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