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Microstructural and mechanical properties evolution of magnesium AZ61 alloy processed through a combination of extrusion and thermomechanical processes

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Abstract

In this work, the microstructures and tensile properties of a commercial magnesium alloy "AZ61" processed by a combination of hot extrusion and thermomechanical processing (TMP) were investigated. The TMP was consisting of two or three hot rolling steps with large reductions per pass, thus allowing significant grain refinement. The microstructural evolution has been studied by means of optical and scanning electron microscopes, as well as X-ray diffraction analysis. The as-cast material is extruded in the form of a cylinder with initial diameter of 250 mm to a final diameter of 110 mm (80% reduction in cross-sectional area). Then hot rolling regimes were performed at 300 °C with different percentage of strain per pass. Tensile and hardness tests were performed in the samples (as-cast, extruded, and rolled) at room temperature in order to evaluate the mechanical properties of the material. The results of experiments demonstrated that fine grain size might be achieved in magnesium alloy AZ61 by using a two-step processing route involving an initial extrusion step followed by thermomechanical processing with large reduction in thickness per pass. This two-step process, designed to achieve average grain sizes of ∼10–20 μm.

Keywords: Grain refining; Mg alloy "AZ61"; Large-strain deformation; Hot rolling; Scanning electron microscope; Recrystallization; Microstructure

1. Introduction

In recent years, research and development studies of magnesium have greatly expanded with regard to weight reduction, energy-saving and environmental protection. Magnesium also offers other attractive properties: such as high damping capacity, dimensional stability, good machinability and recyclability [1–3].

As a relatively new structural material, magnesium and its alloys have demonstrated a significant potential for applications in many industries: including automobile [4,5], transportation, power-tools/equipment and new energy sources. However, the use of magnesium and its alloys has been limited to narrow range because of the limited ductility and poor formability [2,4,6,7]. Limited ductility of Mg alloys may be due to difficulty of the dislocation activity in non-basal slip system [8].

It is tempting to use the refined grain structure of the magnesium alloy in order to enhance their mechanical properties including ductility and strength [9]. Besides, fine-grained Mg alloy possesses better ductility, as well as, lower ductile brittle transition temperature (DBTT), thus its formability at room temperature can be improved [10]. A fine-grained material is harder and stronger than coarsely grained material because it has a greater total grain boundary area to impede dislocation motion.

Several methods have been developed for grain refining in Mg alloy with even submicron or nano-crystalline grain size such as, hot and warm rolling [4,10,11], hot extrusion at high reduction ratio [12,13], and severe plastic deformation process such as equal channel angular pressing (ECAP) [14–21]. Although submicron grain sizes can readily be achieved in many alloys by ECAP, this technique is capable of producing only small amount of material and cannot be scaled up for commercial processing. Among these methods, rolling is the most probable technique to be scaled for fabricating large bulk sheet or plate samples [11].

The present investigation was initiated to evaluate the optional for achieving an ultrafine grain size in a commercial

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Table 1
Chemical composition of the material used

Al	Zn	Si	Mn	Fe	Mg
6.81	1.5	0.047	0.017	0.001	Balance

Table 2
Extrusion conditions of AZ61

Initial diameter of sample	250 mm
Final diameter of sample	110 mm
Extrusion temperature	300 °C
Extrusion speed	4 m/min
Environmental condition	Pressure atmosphere

magnesium alloy AZ61 by using a two-step processing route involving an initial extrusion step followed by thermomechanical processing consisting of two to three hot rolling steps with large reductions per pass. Both optical microscope (OM) and scanning electron microscopy (SEM) were used to investigate the microstructures.

2. Experiments

2.1. Material

The samples for all tests were taken from one heat of 120 kg magnesium alloy AZ61A. This alloy was prepared by melting of high purity Mg ingots in a heat resistance furnace, with cast iron crucible of 120 kg Mg capacity. Commercial pure aluminum and aluminum 90% manganese alloy, in addition to electrolytic zinc, were used in order to adjust the chemical composition of the produced alloy. The melting process was carried out under a flow of argon gas (as inert gas) covering the charging materials during heating and melting process to prevent any reaction that could be occurred with oxygen or water vapor. Molten metal was poured into cast iron molds of 250 mm inner diameter and 600 mm long, after melting and adjusting the chemical composition to reach the composition shown in Table 1. The cast iron molds were coated with alcoholic base graphite coating material, then dried and dusting with sulfer powder before pouring.

2.2. Forward extrusion process

After solidification of the mold, the bottom and top parts were removed, and then homogenized at 380 °C for 10 h. After that, the homogenized billets were extruded at 300 °C with a rate of 4 m/min to give solid bars with diameters of 110 mm corresponding to a reduction ratio in area of 80%. The extraction conditions are listed in Table 2.

2.3. Thermomechanical process

Thermomechanical processing consisting of two or three hot rolling regimes with large reduction in thickness per pass was conducted with the aim of obtaining fine-grained microstructure capable of undergoing superplastic deformation. The extruded bars were sectioned into suitable slides for rolling process in parallel to the extrusion direction. The hot rolling regimes were carried out at 300 °C on a laboratory mill with a work roll diameter of 100 mm. The rolled pieces were heat treated for 5 min at 300 °C between passes to stabilize the rolling temperature. The starting piece of 10 mm was reduced down to compressive deformation ratio of 20, 40, 60, and 80%. The details of the different hot rolling processing routes are listed in Fig. 1. Rolling direction was made parallel to the previous extrusion one.

2.4. Microstructure investigations

The microstructure of the as-cast, the extruded and the hot rolled samples was examined by optical, scanning electron microscopy, and X-ray diffraction analysis. The grain structure was revealed by normal preparing grinding and polishing processes subsequent by etching using a solution of 100 mL ethanol (95%l), 6 g picric acid, 5 mL acetic acid and 10 mL water. The polishing process was carried out using diamond paste with oil base lubricant in order to avoid oxidation of Mg. Grain size measurements were done by the linear intercept method on the optical micrographs. Tensile and hardness tests were performed

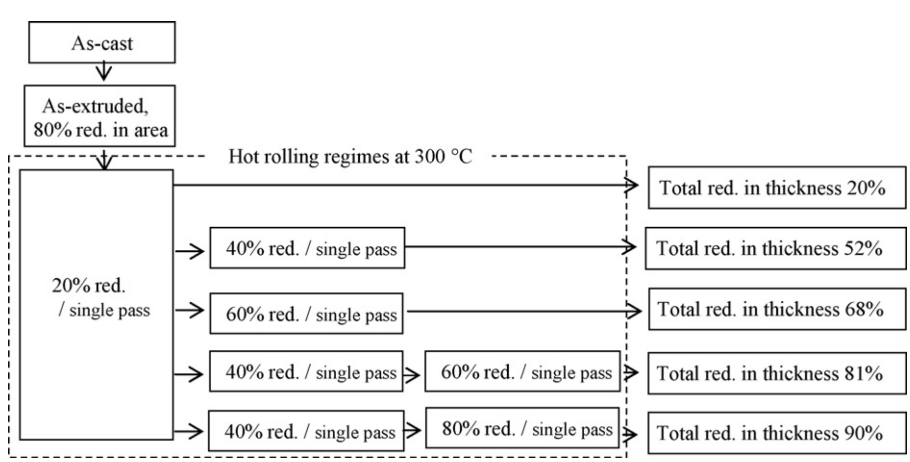

Fig. 1. Schematic diagram of thermomechanical processing with different routs.

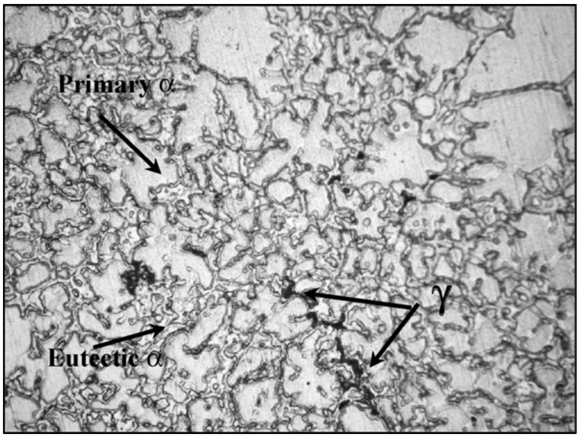

Fig. 2. Optical Micrograph of the as-cast AZ61, X200.

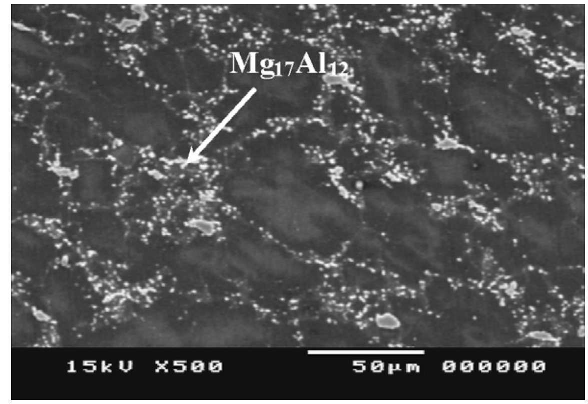

Fig. 4. SEM micrograph after extrusion of AZ61.

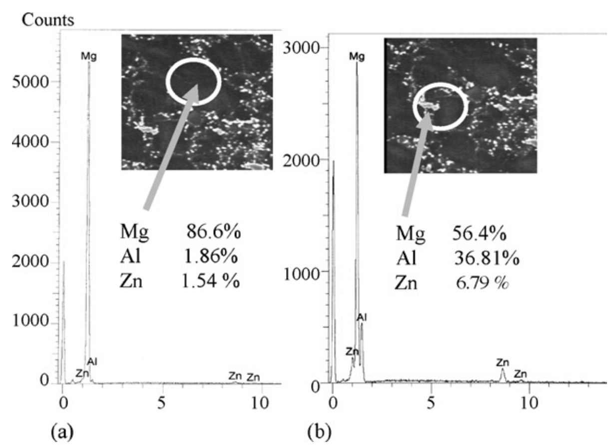

Fig. 5. SEM-EDS of extruded AZ61. (a) EDS inside the grain; (b) EDS on the grain boundary.

at room temperature in order to evaluate the mechanical properties of the material.

3. Results and discussion

3.1. Microstructure evolution

The optical micrograph of the as-cast magnesium AZ61 alloy is shown in Fig. 2. The microstructure consists of second phases α-Mg and β (precipitation of the $Mg_{17}Al_{12}$ phase). This second phases forms along the grain boundaries.

Fig. 3 shows the optical microstructure of longitudinal sections of the extruded bar. It is apparent from the micrograph of the extruded sample, that still there are second phase precipitate ($Mg_{17}Al_{12}$) along the grain boundaries of the grains although the reduction ratio is so high (80%).

Fig. 4 shows the SEM micrograph after forward extrusion. The microstructure shows the presence of the second phase precipitate ($Mg_{17}Al_{12}$) along the grain boundaries. The presence of the precipitate $Mg_{17}Al_{12}$ is also confirmed by the EDS and by the XRD spectrum shown in Figs. 5 and 6, respectively. The average grain size after forward extrusion condition was measured as ~33 μm.

Fig. 6 confirms the presence of grain boundaries having high angle of misorientation of $Mg_{17}Al_{12}$ after extrusion. The previous works [22] shown that the $Mg_{17}Al_{12}$ particles lie preferentially on the grain boundaries in magnesium alloy AZ61 processed by extrusion and it is reasonable anticipate these particles are beneficial in inhibiting grain growth.

Attempts to perform large strain hot rolling process on the cast Mg alloy at temperature 300 °C were unsuccessful and the sample was broken in the first pass with 20% reduction in thickness. By contrast, the extruded samples were easily rolled through two passes regimes (with total reduction ranging from 20 to 68%)

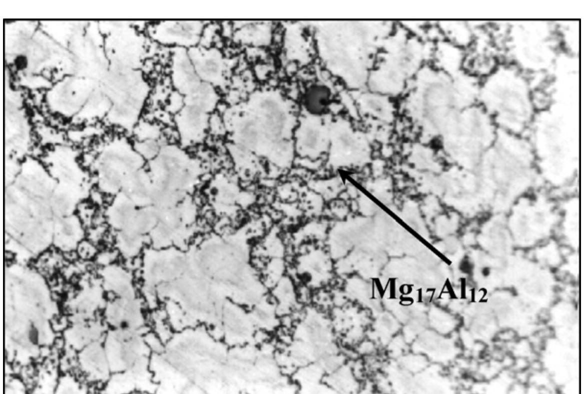

Fig. 3. Optical micrograph after extrusion of AZ61.

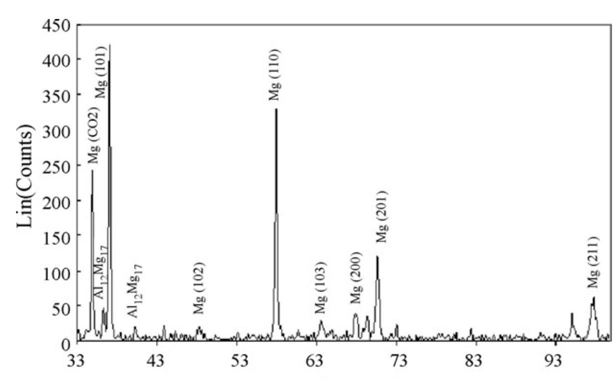

Fig. 6. XRD spectrum of extruded specimen.

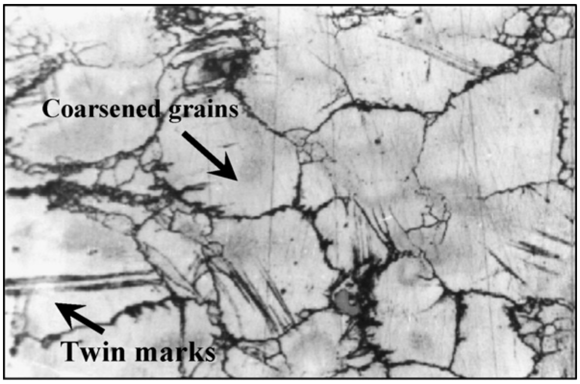

Fig. 7. Optical micrograph of AZ61 after hot rolling regime 1 [20%].

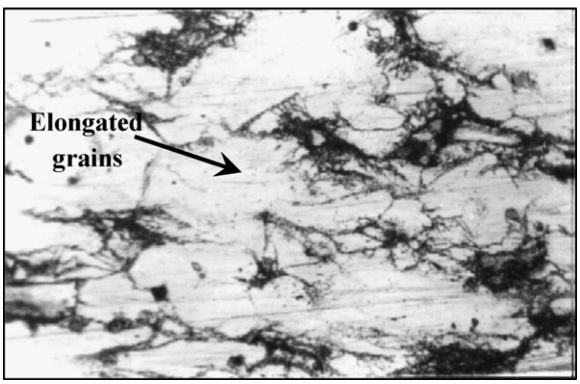

Fig. 8. Optical micrograph of AZ61 after hot rolling regime 2 [20 + 40%].

however, there was some limited surface cracking after the third pass (three passes regimes). The results of the hot rolling process indicate that there is significant difference when the large reduction hot rolling processes conducted using as-cast Mg alloy and that conducted using extruded ones. The same results were observed by Matsubara et al. [14] when ECAP was conducted using as-cast and extruded Mg alloy.

The optical micrographs of the hot rolled AZ61 are shown in Figs. 6–10. After the first rolling pass of 20% reduction in thickness, the microstructure shows coarsen grains due to the coalescence of similar texture grains and the disappearance of the precipitation of the $Mg_{17}Al_{12}$ particles, which are observed on the grain boundaries in the extrusion process, as shown in the optical micrograph of Fig. 7. It can be observed that the grain size distribution tends to be rather heterogeneous; i.e. coarse grains are surrounded by finer grains and some twining is generated in the microstructure. The average grain size is near 48 μm. Valle et al. [7] noticed that reductions larger than 30% in the first pass can lead to fracture along the easy slip bands (ductile zones).

After the first pass, the samples were heat treated at 300 °C for 5 min to stabilize the deformation temperature before the second pass. Further thickness reduction 40% in the second pass (52% total thickness reduction) resulted to elongated grains along the rolling direction as shown in Fig. 8 and dislocation density increases. Valle et al. [7] reported that during the second pass, the applied rolling strain could be accommodated along the paths of easy slip bands (ductile zones) formed by the small recrystallized grains, thus much larger reduction per pass could be attained.

Fig. 9 shows the micrograph of the samples after further thickness reduction 60% in the second pass (68% total reduction in thickness). The figure shows that the grains are more elongated (giving pancaked structure) and there is slight decrease in the grain size. With the aim of refining, further the grain size and avoiding edge cracks, which can be observed with high reduction in thickness as large as 85% in the second pass [7] an alternative rolling regime with a third reduction of 60% (81% total thickness reduction) is investigated (rolling regime 4). Fig. 10 shows the micrograph of the alloy after deformation using three rolling steps (20 + 40 + 60%). In this figure, new small recrystallized grains (sub-grains) are observed and probably formed by dynamic recovery. As the strain increased, new grains generated and dynamic restoration during thermomechanical processing resulted partially recrystallized microstructure (i.e. recovered and recrystallized grains).

After extremely higher strain (rolling regime 5 with 90% total reduction in thickness), the microstructure becomes more homogenous and the equiaxed grains are observed due to fully dynamic recrystallization as shown in Fig. 11. The average grain size decreases down to ∼15 μm. Fig. 12 shows the measured average grain size of the alloy as a function of the total reduction percentage in thickness. The grain size of the different specimens is measured using Synder–Graff intercepted method [23].

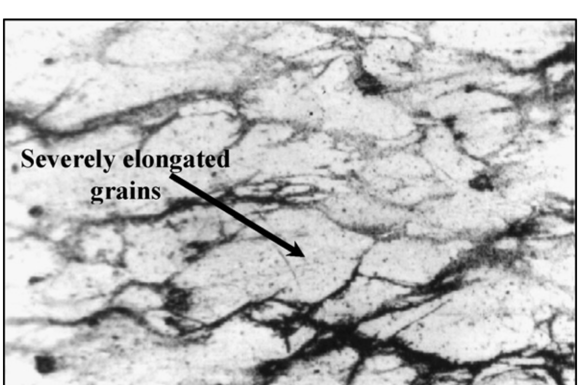

Fig. 9. Optical micrograph of AZ61 after hot rolling regime 3 [20 + 60%].

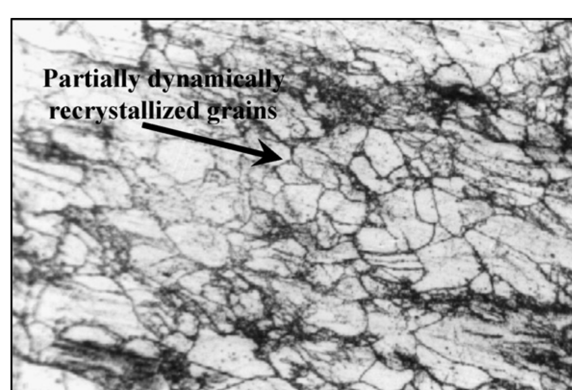

Fig. 10. Optical micrograph of AZ61 after hot rolling regime 4 [20 + 40 + 60%].

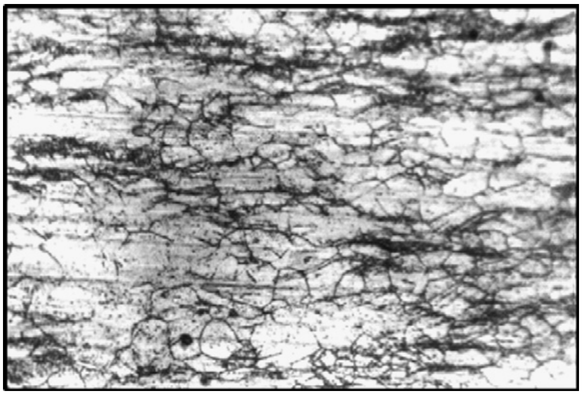

Fig. 11. Optical micrograph of AZ61 after hot rolling regime 5 [20 + 40 + 80%].

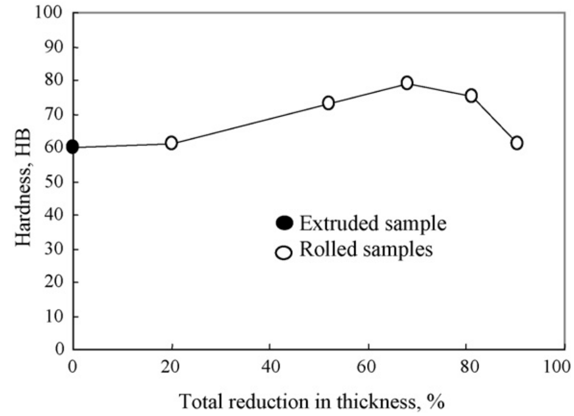

Fig. 13. Average Brinell hardness measured vs. total reduction in thickness.

3.2. Mechanical properties

Fig. 13 shows the average value of Brinell hardness measured on the edges of the extruded and the rolled samples in the transverse direction as well. It is clear that hardness gradually increases as the amount of reduction increases until critical rolling percent. This increase of hardness is due to that the first steps of rolling which are responsible for strain and high dislocation density accumulation until critical rolling percent where the microstructure has a lot of substructure. After this critical rolling percent, the hardness values decreases as the hot rolling percent increases due to microstructural changes where the dislocations annihilate each other and the microstructure recover and new grains form on the substructure. Therefore, the hardness value decreases with increasing rolling percent and dynamic recrystallization producing new fine grains.

Fig. 14 shows the behavior of material strength after different deformation processes. The cast sample provides 135 MPa ultimate strength while after extrusion process the strength increases up to 280 MPa (i.e. twice). This increase is due to the effect of the extrusion process, where it pushes both precipitate and grains towards each other producing a strong texture in AZ61 magnesium alloy with a general alignment of the basal planes [22]. After applying 42% total reduction in thickness, the material strength decreases to 230 MPa. This can be attributed to the elongated and coarsened grains. By further deformation reached up to 81% the strength slightly enhances to be 240 MPa due to dynamically recovered and recrystallized grains. On the other hand, severe deformation up to 90% total reduction allows the microstructure to form newly dynamically recrystallized grains and enhances gradually the strength to be 257 MPa.

For coarse-grained materials, plastic deformation is mainly realized by the motion of dislocations, which pile up near the grain boundaries. Based on the dislocation pileup mechanism, grain refinement generally makes the materials stronger. The yield strength as a function of grain size can be described by a Hall–Petch relation. When the grain size is reduced to ultrafine

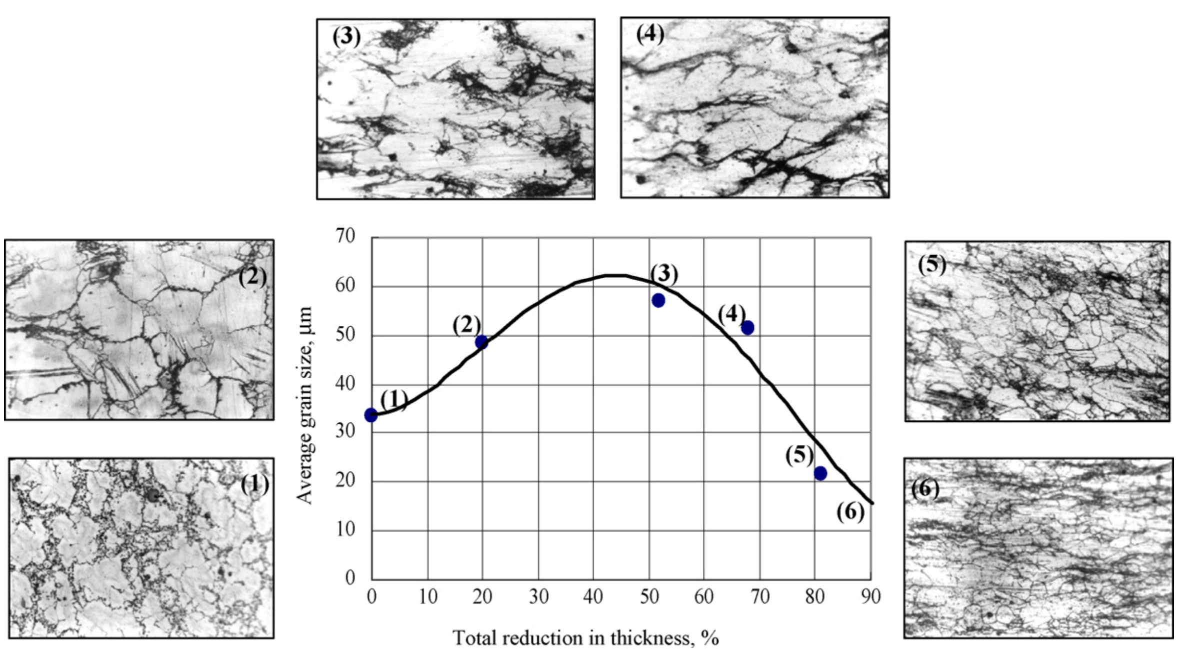

Fig. 12. Effect of the total reduction in thickness on the average grain size of rolled AZ61.

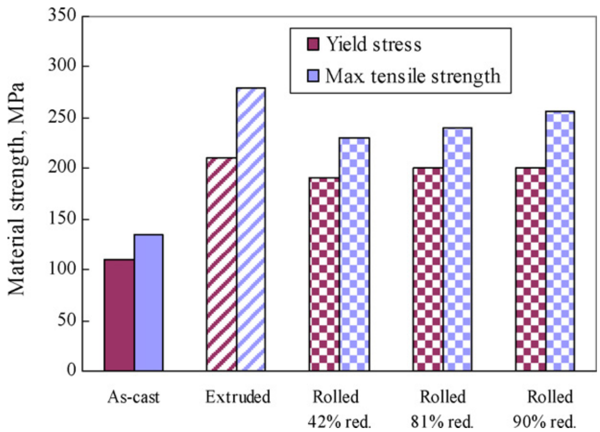

Fig. 14. Effect of deformation process on the tensile strength for AZ61.

or to nanometer scales, deformation mechanisms other than the dislocation pileup mechanism may be responsible for the plastic deformation. The experimental evidences suggest that the strength starts to deviate from the Hall–Petch relation with a reduced *k* value, when the grain is reduced to the ultrafine-scale region. Below a certain critical grain size, an inverse Hall–Petch relationship is observed. The deviation of the Hall–Petch relation may be due to dislocation motion different from the dislocation pileups at the grain boundaries. The observed inverse Hall–Petch relation may be attributed to the grain boundary activities responsible for the plastic deformation of fine-grained materials.

4. Conclusion

The microstructure and tensile properties of magnesium alloy "AZ61" processed by a combination of hot extrusion and thermomechanical processing (TMP), consisting of two to three hot rolling steps with large reduction per pass, were evaluated. The microstructure investigations were carried out by means of optical, scanning electron microscope and X-ray diffraction (XRD) as well. The conclusion of this work could be summarized as follow:

- Application of high strain during hot rolling process on the as cast magnesium alloy AZ61 at 300 °C will lead to damage of the rolled piece.
- A fine grain size might be achieved in magnesium alloy AZ61 by using a combination of two-steps processing routs such as extrusion and thermomechanical process. This combination leads to grain refining effect and produced an average grain size of 10–20 μm.
- After the first pass of 20% reduction in thickness, the microstructure showed a coarser grain that the extruded one. This may be due to coalescence of similar texture grains and disappearance of the precipitation of $Mg_{17}Al_{12}$ particles, which are observed on the grain boundaries of the extruded samples.
- Appling of large strain (rolling regime no. 5 with 90%total reduction percent) produced a more homogenous and equiaxed grains. Thus, due to successive breaking-up of coarse grains into finer grains.
- The hardness values slightly increased as reduction percent increased up to certain reduction percent called critical rolling percent. After that the hardness values decreased because of annihilation of dislocation, recovery process and generation of new grains on the substructure (recrystallization).

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