

Aging of WE43 magnesium alloy after mechanical crushing and subsequent high pressure torsion

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Present work aims at investigation of the consequences of mechanical crushing prior to high pressure torsion (HPT) of the Mg-Y-Nd-Zr (WE43) alloy. Specifically the presence and size of the effect on the aging properties compared to the initially solid state and subsequent HPT are studied. For this, the WE43 alloy was mechanically crushed into particles of 0.5–1 mm size. Then the obtained powder was formed to pellets and deformed at a pressure of 6 GPa for 10 revolutions with 1 rpm rotation speed. Thermal stability of the HPT processed alloy microstructure was studied by monitoring its microhardness and aging. Mechanical crushing and subsequent HPT processing at room temperature results in significant strengthening of magnesium alloy WE43. It was found that strengthening induced by HPT sustained to 200°C. The strength of the HPT processed alloy was additionally improved by subsequent aging. Extraordinarily high maximum value of microhardness of 1557 ± 25 MPa was reached. We suppose that crushing prior to high pressure torsion creates additional defects induced by the surfaces of individual powder particles during HPT. Additionally the surfaces of individual powder particles can act as segregation centers for rare earth elements. That decreases electrical resistivity due to lower precipitate dissolution and lower solid solution supersaturation.

Keywords: nanocrystalline metals, magnesium alloys, severe plastic deformation, high pressure torsion.

УДК: 539.8

Старение магниевого сплава WE43 после механического измельчения и последующего кручения под высоким давлением

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Целью представленной работы является исследование наличия и степени влияния механического измельчения сплава Mg-Y-Nd-Zr (WE43) перед деформацией, путем кручения под высоким давлением (КВД), на процесс старения по сравнению с литым состоянием до КВД. Для этого сплав WE43 механически измельчали на частицы размером 0.5–1 мм. После этого полученный порошок компактировался в таблетки и деформировался путем КВД под давлением

6 ГПа на 10 оборотов со скоростью 1 об/мин. Термическая стабильность микроструктуры сплава, подвергнутого КВД, была исследована путем наблюдения за его микротвердостью и электрическим сопротивлением. Механическое измельчение и последующая обработка путем КВД при комнатной температуре вызвала существенное упрочнение измельченного сплава WE43. Было обнаружено, что упрочнение, вызванное КВД, сохранялось до 200°C. Прочность подвергнутого КВД сплава WE43 была дополнительно повышена путем последующего старения. Было получено экстраординарно высокое значение микротвердости в 1557 ± 25 МПа. Предположительно измельчение перед КВД создает дополнительные дефекты на границах отдельных частиц порошка во время КВД. Также поверхности отдельных частиц порошка выступают в качестве сегрегационных центров для редкоземельных элементов. Это снижает электрическое сопротивление из-за меньшего растворения частиц второй фазы и меньшего перенасыщения твердого раствора.

Ключевые слова: нанокристаллические металлы, магниевые сплавы, интенсивная пластическая деформация, кручение под высоким давлением.

1. Introduction

Properties of nanocrystalline metallic materials, such as mechanical characteristics, corrosion rate and others, came to the front in the last few decades. For example, it has been shown that classical methods for increasing the strength and yield stress of polycrystalline materials are applicable when the size of grains in a polycrystal is reduced only to a grain size of several tens of nanometers. Nanocrystalline materials showed unusual functional properties since they contain an extremely high proportion of atoms located at the grain boundaries and other defects in the crystal structure. These defects can change the phase composition of nanocrystalline materials from the equilibrium phase composition. Among all the methods of obtaining nanocrystalline alloys, high pressure torsion (HPT) holds a specific place. It belongs to the severe plastic deformation (SPD) methods and demonstrates a number of advantages compared to its competitors. For example, it makes production of nanocrystalline materials in relatively large volumes possible from both pure metals and alloys.

The WE43 is a commercially available bioresorbable magnesium alloy Mg-3.56%Y-2.20%Nd-0.47%Zr, wt.%. The WE43 alloy containing rare earth metals (REM) is one of the most popular medical magnesium alloys [1]. Doping with REM improves the corrosion resistance of magnesium [2], and also increases its strength. Another method of improving the strength characteristics is SPD, which leads to the formation of ultrafine-grained structure (UFG) in magnesium and its alloys [3]. This structure provides significant hardening [3–6], and also leads to an increase in corrosion resistance and a decrease in gas evolution.

The goal of present work is to investigate the consequences of mechanical crushing prior to high pressure torsion (HPT) of WE43 alloy. Specifically the presence and size of the effect on aging properties compared to the initially solid state and subsequent HPT are studied. Previously our colleagues performed a work, where aging of an as-cast WE43 magnesium alloy processed by high pressure torsion was investigated [3]. That gives us the opportunity to compare the data for cast and crushed alloy prior to HPT.

The need for such investigation was explained in our previous work. It demonstrated the possibility of producing a metal-ceramic composite by introducing oxide particles of various metals into the WE43 alloy and processing it by HPT [7]. This production involved crushing the alloy into particles of 0.5–1 mm size. Then thin films of nanocrystalline oxides were deposited onto WE43 particles using pyrolysis

of organometallic compounds containing salts of metals and carbon acids [8–14]. After applying an oxide film on the surface of the magnesium alloy particles, the powder was subjected to SPD by HPT. For that reason distinguishing the strengthening effect of crushing the alloy and oxide particles introduction is needed for deeper understanding of our work.

2. Experimental

The WE43 alloy was solution treated (ST) at 525°C for 8 h. After annealing the samples were quenched in air at room temperature. Following the homogenization the alloy was mechanically crushed into particles of 0.5–1 mm size. The powder was poured into the cavity in the lower anvil and compressed in order to obtain a solid pellet. Then another portion of powder was poured onto the first produced pellet. Four portions of powder in total were used for each sample. After pouring the last portion, the sample was pressed to the deformation pressure and the deformation started. By this mean we provided enough material to form a solid sample without significant pores. The excess material was pressed out of deformation cavity during the deformation process. Samples were deformed at a pressure of 6 GPa for 10 revolutions with 1 rpm rotation speed. The cavity in the lower anvil was of 0.2 mm depth.

To examine thermal stability of the HPT strengthening the samples were heated at temperatures of 100–400°C in increments of 50°C for 1 hour at each temperature. Several samples were also isothermally aged at 200°C for up to 128 h to verify the ability of the alloy to additional hardening due to precipitation process.

Vickers microhardness was measured using a 402 MVD Instron Wolpert Wilson Instruments tester with a load of 50 g and 10 s holding time. Electrical resistivity was measured with a BSZ-010-2 microohmmeter on the specimens with a size of $10 \times 4 \times 0.2$ mm. Since the response of electrical resistivity to phase transformation is stronger compared to defects annihilation and recrystallization, the electrical resistivity measurements were used mainly to trace the REM precipitation from the solid magnesium on heating. The decrease of electrical resistivity indicates depletion of the magnesium solid solution by REM whereas its increase is a result of the solid solution enrichment by dissolving RE metals therein. Transmission electron microscopy (TEM) was performed on the FEI Titan 80–300 Transmission Electron Microscope with a high-angle annular dark field scanning transmission electron microscopy (HAADF-STEM)

and EDAX S-UTW energy dispersive X-ray analysis (EDX) detectors. The samples for the TEM were prepared first by precision grinding using a Gatan Model 656 Dimple Grinder and final ion polishing by a Gatan 691 PIPS with additional low-voltage argon ion polishing system and cryo cooling.

3. Results and discussion

As shown in Fig. 1 nano-sized grains are formed similarly to the grains in non-crushed HPT processed alloy. The micrographs of bright field Fig. 1a and dark field Fig. 1b show some nano-sized grains with high-angle boundaries. The ring-like electron diffraction patterns with a small number of point reflexes suggest a high ratio of low-angle boundaries.

Microhardness of the samples after the HPT was measured on the half radius of the sample. The microhardness measurements show that HPT results in significant strengthening of the WE43 alloy compared to the initial ST condition. The average microhardness of the ST sample is 774 ± 50 MPa, while microhardness level after crushing and HPT at room temperature is 1331 ± 49 MPa. Evidently, the strengthening induced by HPT is mainly caused by grain refinement.

The dependence of thermal stability of microhardness and electrical resistivity of an ST sample and samples processed by crushing and HPT on annealing temperature is presented in Fig. 2. Microhardness of the WE43 alloy after crushing and HPT is much higher at all temperatures compared to ST sample (Fig. 2 a). The dependence of microhardness of the HPT and ST samples is non-monotonic and possesses a maximum. Microhardness increases with an increase in the annealing temperatures up to 200°C and 250°C for HPT samples and ST sample, respectively. The increase in microhardness in a range of aging temperatures (where the sample strengthens) is evidently associated with precipitation of reinforcing metastable phases. This is supported by a simultaneous decrease in the electrical resistivity (Fig. 2 b). Microhardness of ST sample decreases gradually after reaching the maximum, whereby microhardness of the crushed and HPT sample dramatically drops during annealing at temperatures above 200°C. At higher temperatures microhardness of the ST and HPT samples strives to a similar level at 400°C. Softening of the alloy with increasing annealing temperature is explained by grain growth and precipitation, with subsequent partial dissolution of precipitates. The supersaturation of magnesium solid solution with RE metals is in charge of the rise of electrical resistivity at high annealing temperatures.

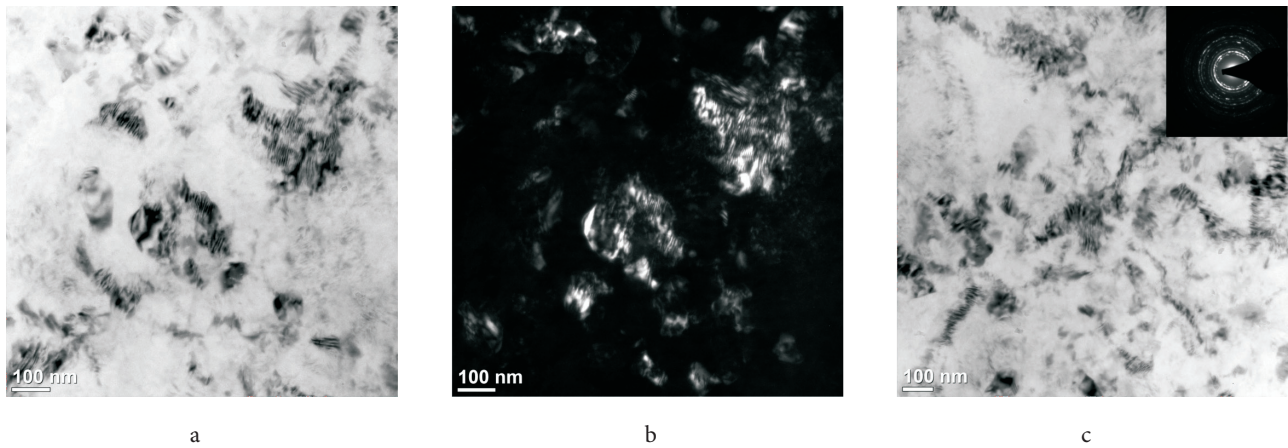


Fig. 1. TEM micrograph of bright field (a) dark field (b), and bright field with corresponding electron diffraction pattern (c) of the crushed and HPT processed WE43.

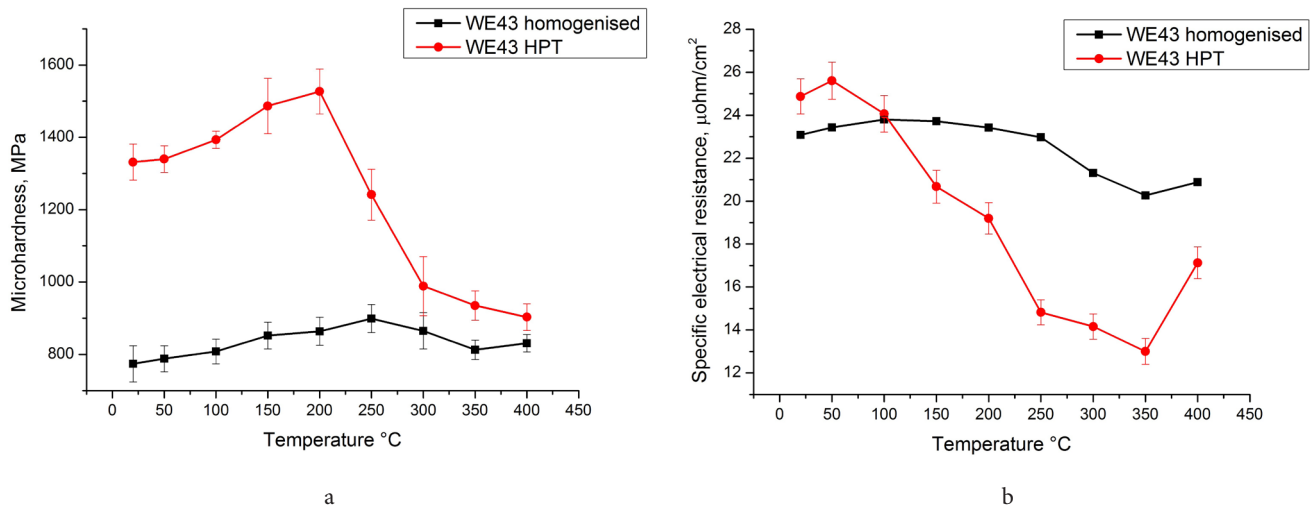


Fig. 2. (Color online) The dependence of microhardness (a) and electrical resistivity (b) of the WE43 alloy on the annealing temperature. The annealing was performed subsequently on one sample, 1 h for each temperature.

The behavior of microhardness and electrical resistivity of the sample processed by crushing and HPT and the ST sample during isothermal aging at 200°C is shown in Fig. 3. An increase in microhardness with increasing aging time occurs not only for the ST sample but also for the HPT processed ones (Fig. 3a). All microhardness curves possess a peak in the aging time dependence. Nevertheless, the crushed and HPT processed sample reaches the microhardness peak much earlier than the ST sample. Position of the microhardness peaks for the HPT processed sample corresponds to about 1 h. In the same way, electrical resistivity decreasing during aging process (Fig. 3b) is explained by precipitation of RE atoms from solid solution in magnesium. Electrical resistivity drops more rapidly for the sample processed by HPT than for the ST sample. This indicates that HPT enhances the precipitation rate and shortens aging time to achieve the microhardness maximum. The fast aging kinetics of the WE43 alloy processed by HPT can be associated with high dislocation density and a large area of grain boundaries, which promote nucleation and growth of precipitates during aging.

In the work [3] our colleagues obtained similar microhardness and electrical resistivity curves for solid WE43 processed by HPT followed by annealing at rising temperature or isothermal aging. Nevertheless, all levels of microhardness for the initially solid sample are lower, than for the initially crushed ones. After the HPT the initially solid sample microhardness is 1189 ± 33 MPa and the crushed ones — 1331 ± 49 MPa. The final level of microhardness for the solid sample reaches the level of the ST sample. But the crushed sample does not soften to such level. During aging record microhardness for initially solid state sample reaches 1411 ± 40 MPa. Crushing the alloy prior to the HPT we exceeded that level and reached 1557 ± 25 MPa.

Hot extrusion of WE43 alloy and subsequent aging provided microhardness of 1020 MPa and 1050 MPa, annealing, and full aging (T6 treatment) — 1100 MPa [15]. Forging and additional aging resulted in microhardness of 950 MPa and 1150 MPa [16]. Microhardness values of 810 MPa and 970 MPa were reached in [17] after hot rolling and additional aging respectively. The WE43 alloy processed by ECAP has 1020 MPa in microhardness according to [18].

The final electrical resistivity during the investigation of the thermal stability shows the rate of the dissolution of precipitates and supersaturation of magnesium solid solution with RE metals. For initially solid samples electrical resistivity reaches values similar to the ST sample, 20.5 ± 0.25 $\mu\text{ohm}/\text{cm}^2$. But the initially crushed samples resistivity does not reach that level and stays at 17.17 ± 0.74 $\mu\text{ohm}/\text{cm}^2$. That shows lower precipitates dissolution and solid solution supersaturation. Additionally electrical resistivity during aging for the crushed and HPT processed sample reached a lower value, 11.73 ± 0.52 $\mu\text{ohm}/\text{cm}^2$ than that of a solid and HPT processed sample 14.39 ± 0.77 $\mu\text{ohm}/\text{cm}^2$ [3].

Higher levels of microhardness can be explained by the additional second phase precipitation. That precipitation is conditioned by additional defects induced by the surfaces of individual particles during the HPT which act as precipitation centers. The surfaces of individual particles can also induce strengthening magnesium oxide particles into the sample. Additionally, lower electrical resistivity points out a lower precipitate dissolution and solid solution supersaturation. That can be explained by RE segregation on the surfaces of individual powder particles.

4. Conclusion

1. Mechanical crushing and subsequent high pressure torsion at room temperature results in significant strengthening of magnesium alloy WE43. The strength of the HPT processed alloy is additionally improved by aging. Extraordinarily high maximum value of microhardness, in excess of 1557 ± 25 MPa was reached. Such microhardness level was not yet reached by other methods.

2. Additionally, electrical resistivity for the crushed and HPT processed sample reached a lower value, 11.73 ± 0.52 $\mu\text{ohm}/\text{cm}^2$ than that of a solid HPT processed sample.

3. Crushing prior to high pressure torsion creates additional defects induced by the surfaces of individual powder particles during HPT. Additionally the surfaces of individual powder particles can act as segregation centers for rare earth elements. That decreases the electrical resistivity due to lower precipitate dissolution and lower solid solution supersaturation.

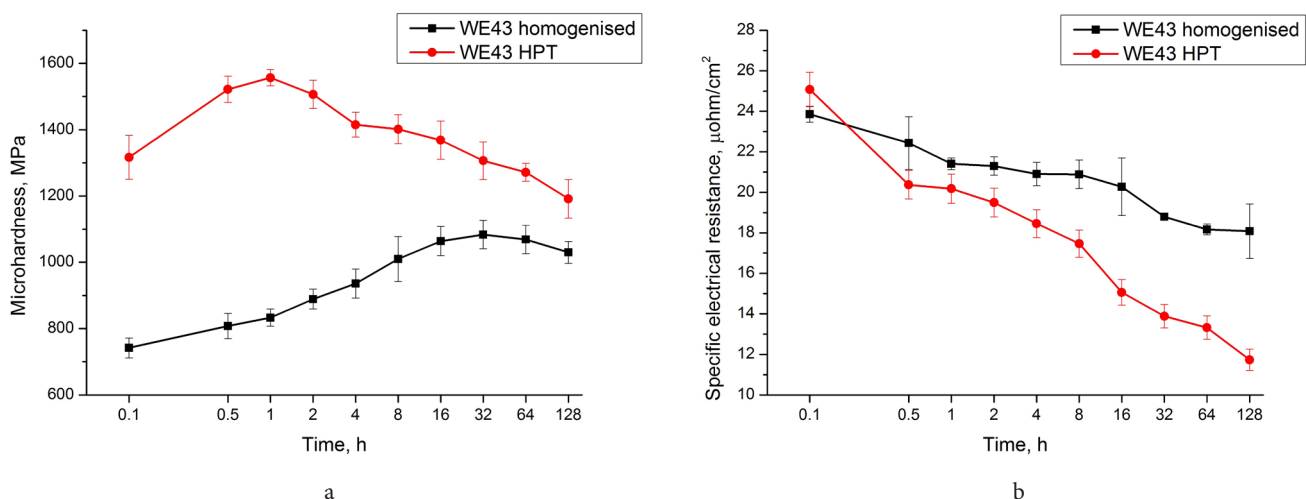


Fig. 3. (Color online) The dependence of microhardness (a) and electrical resistivity (b) of the WE43 alloy on the aging time during isothermal aging at 200°C.

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