Functional Materials 11, No.2 (2004)

Influence of alumina and zirconia nanoparticles on mechanical properties and damping of magnesium

Z.Trojanova, Z.Drozd, P.Lukac, H.Ferkel*, W.Riehemann*

Department of Metal Physics, Charles University, Praha,
Ke Karlovu 5, CZ-121 16 Praha 2, Czech Republic
*Deparment of Materials Engineering and Technology,
Technical University of Clausthal, Agricolastr. 6,
D-38678 Clausthal-Zellerfeld, Germany

Magnesium polycrystals reinforced with 3 % (vol.) ZrO_2 nanoparticles and 3 % (vol.) Al_2O_3 nanoparticles were deformed in tension at a constant strain rate at temperatures between 20 and 300°C. The yield stress of both materials decreases rapidly with temperature. The flow stress of $Mg+Al_2O_3$ is higher than that of $Mg+ZrO_2$. Ductility of the composite with zirconia is substantially higher. The difference in the deformation behaviour of both composites is very probably caused by difference in bonding between matrix and ceramic nanoparticles.

Поликристаллы магния, упрочненные 3% наночастиц ZrO_2 и 3% наночастиц Al_2O_3, были деформированы растяжением при постоянной скорости деформации в температурном интервале 20–300°C. Предел текучести обоих материалов быстро уменьшается с температурой. Напряжение текучести $Mg+Al_2O_3$ выше, чем у $Mg+ZrO_2$. Пластичность композита с цирконием существенно выше. Разница в поведении двух композитов при деформации является скорее всего результатом различия в прочности связи между матрицей и керамическими наночастицами.

Metallic materials with small grains exhibiting a high strength (Hall-Petch strengthening) are interesting from both theoretical and experimental point of view. A further improvement of their mechanical properties is possible using reinforcement with ceramic particles or fibres. Metal matrix composites have been developed to provide engineering materials with combined properties of ceramics and metals. Those exhibit significant advantages as compared to solid materials: higher strength/density and stiffness ratios, enhanced wear resistance and higher upper temperature limit. There is, however, no universal method and scientific proven approach to select the composites and predict their behaviour in actual working conditions. In practice, substantial differences between materials with various ceramic particles may occur. The nature of the interface between the matrix and the reinforcement may influence significantly many properties of metal matrix composites.

The aim of the present work is to study deformation characteristics of microcrystalline Mg reinforced by zirconia and alumina nanoparticles and to try to find an explanation for an pronounced difference in the deformation behaviour of both materials.

Mg with 3 % (vol.) Al_2O_3 (alumina) nanoparticles and Mg with 3 % (vol.) ZrO_2 (zirconia) nanoparticles were used in the experiments. The fine Mg powder having particle diameter of about 20 μm was prepared by gas atomization of a Mg melt with Ar containing 1 % oxygen for powder passivation. Both Al_2O_3 and ZrO_2 powders with a mean particle size of 14 nm were prepared by evaporation using the pulsed radiation of a 1000 W Nd:YAG laser and subsequent condensation of the laser-induced vapor in a controlled gas medium. The preparation method of nanoparticles is described elsewhere [1]. The Mg powder was mixed and milled together with the nanoparticles in an asymmetrically moving mixer for 1 h. The mixture was subsequently pre-compressed followed by hot extrusion at 400°C and 150 MPa pressure. The initially more or less equiaxial grains were changed into elliptical ones with the long axis parallel to the extrusion direction. The grain size was about 3 μm in the cross section and several tens μm in the extrusion direction. Ceramic nanoparticles were located at the grain boundaries, only few particles were found inside of grains.

Tensile tests were performed in an Instron testing machine at temperatures between 20°C and 300°C. The cylindrical specimens used were deformed at an initial strain rate of 6.2×10^{-5} s^{-1}. The specimens for damping measurements were machined as bending beams (81 mm long with a thickness of 4 mm and 10 mm width). The damping measurements were carried out in a vacuum (about 50 mPa) at room temperature. The specimens fixed at one end were excited into resonance (the frequency ranged from 130 to 140 Hz) by a permanent magnet fixed at the free side of the bending beam and a sinusoidal alternating magnetic field. The damping was characterized by the logarithmic decrement δ of the free vibrating beam. The specimens were annealed step by step at temperatures increasing up to 550°C for 0.5 h and quenched into water of ambient temperature after each annealing. The annealing at higher temperatures was performed in argon atmosphere to avoid oxidation. The damping measurements were carried out immediately after heat treatment and quenching at room temperature in vacuum.

The true stress – true strain curves obtained for both materials are shown in Figs. 1a, b. It can be seen that deformation stresses decrease with increasing temperature in both cases. The flow stresses for $Mg+3n\text{–}ZrO_2$ are substantially lower than those for $Mg+3n\text{–}Al_2O_3$ deformed at the same test temperature. Ductility of $Mg+3a\text{–}ZrO_2$ is higher than that of $Mg+3a\text{–}ZrO_2$. Even if both materials were prepared by the same technology, the estimated deformation behaviour is different. The temperature dependences of the yield stress as well as the maximum stress for both materials are presented in Figs. 2 and 3. It can be seen that the differences between the yield stress as well as the maximum stress for both materials deformed at room temperature are about 100 MPa. The stresses decrease with increasing temperature. A similar rapid decrease of the flow stress with increasing temperature was observed in microcrystalline Mg and it was discussed in our previous paper [2].

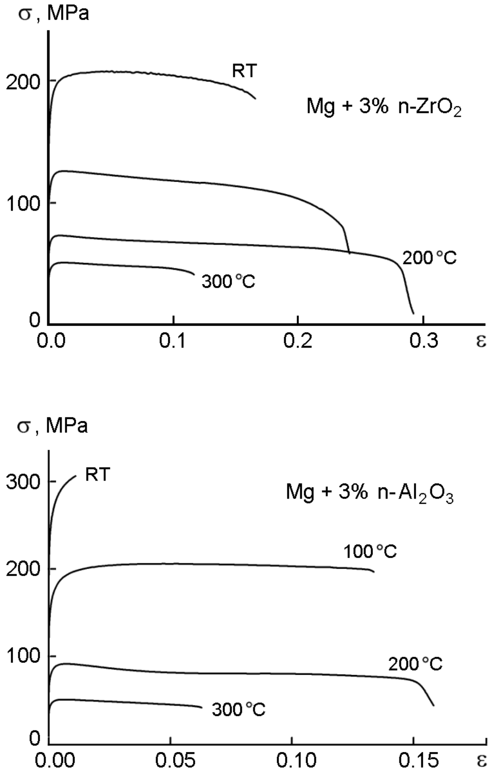

Fig. 1. Stress-strain curves obtained for various temperatures.

The heavy temperature dependence of the yield stress and the maximum stress indicates the occurrence of thermally activated processes during plastic deformation. The activity of non-basal slip systems is required for the grain accommodation in polycrystalline material of hexagonal structure. The glide of dislocations in pyramidal slip systems seems to be very probably the main thermally activated process at higher temperatures. The critical resolved shear stress for non-basal (pyramidal) slip systems decreases rapidly with increasing temperature. This means the activity of pyramidal slip systems to increase with increasing temperature. The density of moving non-basal dislocations may increase [3]. The interaction between the basal dislocations and the non-basal dislocations may result in

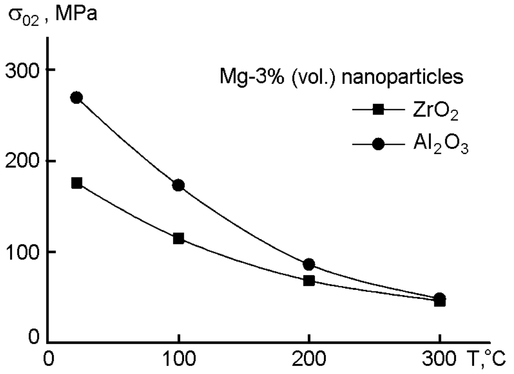

Fig. 2. Temperature dependence of yield stress.

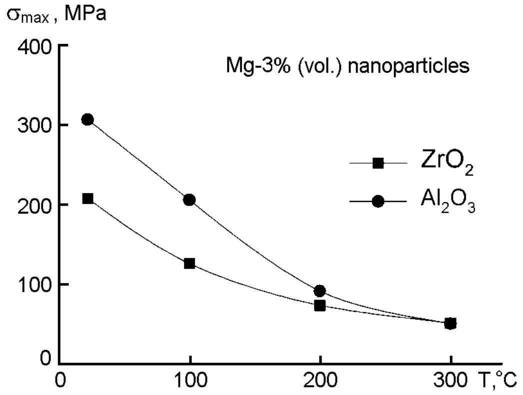

Fig. 3. Temperature dependence of maximum stress.

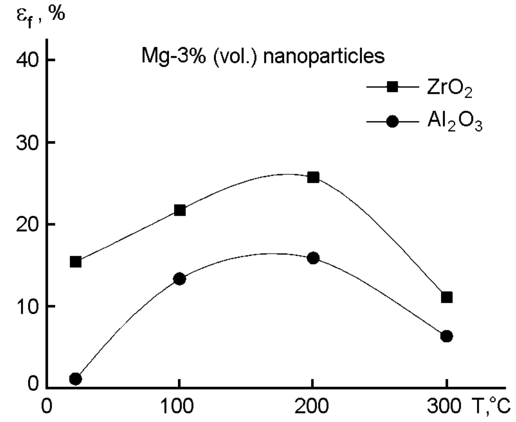

Fig. 4. Temperature dependence of ductility.

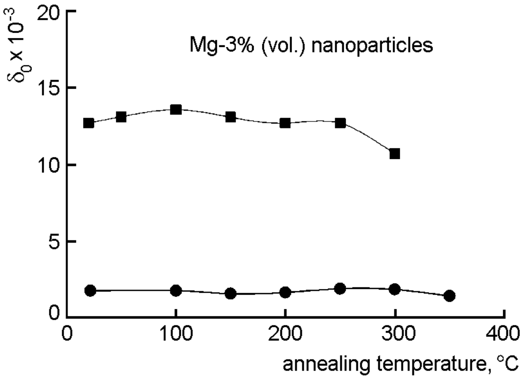

Fig. 5. Amplitude-independent component of logarithmic decrement as a function of annealing temperature.

creation of new basal dislocations according to reaction:

$$\frac{1}{3}[11\bar{2}\,\bar{3}] + \frac{1}{3}[\bar{2}113] \rightarrow \frac{1}{3}[\bar{1}2\bar{1}0]. \qquad (1)$$

The annihilation of dislocations can occur. The interaction probability increases with increasing temperature. The dislocation annihilation causes a decrease in the work hardening. This process can be responsible for softening of both materials observed at temperatures above 100 °C.

There is a difference in the ductility values between both materials, as it can be seen from Fig. 4. Ductility of the composite with zirconia nanoparticles is substantially higher. Local maximum in the temperature dependence of the fracture strain was observed for various magnesium alloys and composites as well as in the case of pure microcrystalline Mg. It was estimated, in all cases, at the test temperatures between 100 and 200°C [3–5].

The different deformation behavior of both materials is very probably also influenced by the bonding between matrix and ceramic particles. Measurements of the logarithmic decrement δ were performed to check this hypothesis. The amplitude independent component of the decrement δ_0 was measured at room temperature after annealing at increasing temperatures. The dependence of the δ_0 component on the annealing temperature for both materials is given in Fig. 5. It is obvious that the δ_0 component for the composite with zirconia particles is substantially higher than that for Mg with alumina particles. The characteristic studied is rather stable against temperature. The δ_0 component is independent of the annealing temperature practically up to about 250°C, then it decreases slightly for Mg+3 % n–ZrO_2.

The difference in logarithmic decrement values for the two composites investigated may be explained under assumption that in the case of Mg+3 % n–ZrO_2,the binding between the matrix and the particles is weak. The effect of weakly bound interface on the damping capacity of particulate reinforced composites has been reported in several papers [6–10]. According to the interface slip model, the interface damping is attributed to the frictional energy loss between particles and metallic matrix under cyclic stress carried by sonic or ultrasonic waves. The logarithmic decrement is expressed as

$$\delta = \frac{3\pi^2}{2}\frac{\kappa\sigma_r}{\sigma_0}V_p, \qquad (2)$$

where V_p is the volume fraction of particles; κ, the friction coefficient between both components of the composite; $\sigma_r r$, the radial stress at the interface corresponding to stress amplitude τ_0 (strain amplitude ε_0). The stress concentration factor $\kappa = \tau_r/\tau_0$ has been reported in literature to be between 1.1 and 1.3 [8]. On the other hand, a perfect binding between the matrix and particles can be a reason for generation of thermal stresses due to temperature changes [11]. Weakly bound grain boundaries in Mg+3 %a–ZrO_2 are the reason why the mechanical properties of this composite are worse.

To conclude, mechanical properties of the microcrystalline Mg reinforced with alumina and zirconia nanoparticles are substantially improved. There is a significant difference between both materials. This difference is due to binding between magnesium matrix and ceramic nanoparticles. Weak binding in the case of Mg+3 %n–ZrO_2 is manifested itself as high values of internal friction. High values of the damping allow to use Mg+3 %a–ZrO_2 as a HIDAMAT (HIgh DAmping Material).

Acknowledgements. The authors acknowledge support from the Grant Agency of the Czech Republic under Grant 205/03/0071.

References

1. H.Ferkel, W.Riehemann, *Nanostruct.Mater.*, **7**, 835 (1996).
2. Z.Trojanova, Z.Drozd, P.Lukac et al., *Scripta Mater.*, **42**, 1095 (2000).
3. Z.Drozd, Z.Trojanova, K.Mathis et al., *Kovove Mater.*, **36**, 3-S 62 (1998).
4. S.Kudela, Z.Trojanova, V.Kolenciak, P.Lukac, in: Proc. of the 3rd Int. Conf. 2000, Bangalore, India, Foundation for Advanced Materials Education, Bangalore (2000), p.679.
5. Z.Trojanova, S.Kudela, P.Lukac et al., *Kovove Mater.*, **39**, 1 (2001).
6. N.N.Kishore, A.Gosh, B.D.Agarwal, *J.Reinforced Plastics and Composites*, **1**, 41 (1982).
7. D.J.Nelson, J.W.Hancock, *Mater.Sci.*, **13**, 2429 (1978).
8. J.Zhang, R.J.Perez, E.J.Lavernia, *Acta Metall.Mater.*, **42**, 385 (1994).
9. J.Zhang, R.J.Perez, C.Wong, E.J.Lavernia, *Mater.Sci.Engn.*, **R13**, 325 (1994).
10. Z.Trojanova, H.Ferkel, P.Lukac, W.Riehemann, *Phys.Stat.Sol.(a)*, **193**, 205 (2002).
11. Z.Trojanova, W.Riehemann, H.Ferkel, P.Lukac, *J.Alloys Comp.*, **310**, 396 (2000).

Вплив наночастинок оксидів алюмінію та цирконію на механічні властивості та демпфірування магнію

З.Троянова, З.Дрозд, П.Лукач, Г.Феркель, В.Рігеман

Полікристали магнію, зміцнені 3 % наночастинок ZrO_2 та 3 % наночастинок Al_2O_3, були деформовані розтягом при постійній швидкості деформації у температурному інтервалі 20–300°C. Границя текучості Mg+Al_2O_3 вища, ніж у Mg+ZrO_2. Пластичність композиту з цирконієм значно вища. Різниця в поведінці обох композитів при деформації є, найімовірніше, наслідком різниці міцності зв’язування матриці з керамічними наночастинками.