Damage mechanism of AZ61-F Mg alloy with nano- Al_2O_3 particles

M. Besterci¹, J. Ivan², S.-J. Huang³, O. Velgosová^{4*}, B. Z. Lin³, P. Hvizdoš¹

¹Institute of Materials Research, Slovak Academy of Sciences, Watsonova 47, 043 53 Košice, Slovak Republic

²Institute of Materials and Machine Mechanics, Slovak Academy of Sciences,

Račianska 75, 831 02 Bratislava, Slovak Republic

³Department of Mechanical Engineering, National Taiwan University of Science and Technology, 43, Sec. 4,

Keelung Rd., Taipei, 106, Taiwan, R.O.C.

⁴Department of Materials Science, Faculty of Metallurgy, Technical University in Košice,

Park Komenského 11, 042 00 Košice, Slovak Republic

Received 8 August 2011, received in revised form 2 November 2011, accepted 10 November 2011

Abstract

Deformation of magnesium alloy AZ61-F with 1 wt.% of Al₂O₃ was analysed by "in-situ tensile test in SEM" and a model of fracture mechanism was suggested. Microstructure of the experimental materials is heterogeneous with grain size of 50 μ m. It has been shown that during tensile deformation the failure of Mg₁₇Al₁₂ particles appeared and at the same time decohesion of the matrix-Al₂O₃ particles occurred as a result of different physical properties. The increase of stress caused the cracks propagation into the specimen and simultaneously the large damaged Mg₁₇Al₁₂ particles contributed to the fracture process. Fracture surface had transcrystalline ductile character.

Key words: AZ61-F Mg-Al₂O₃, in-situ tensile test in SEM, model of fracture mechanism

1. Introduction

Damage mechanisms of composite materials are interesting from theoretical as well as practical point of view. Usually, the micromechanisms of fracture of broken specimens after mechanical tests are described. Method of "in-situ tensile test in SEM" enables to record and study the deformation process directly [1, 2], which means that the initial and developing stages of the plastic deformation and fracture can be characterised more precisely.

In our previous works [3–9] we used this method to analyse deformation of various types of Cu, Al-based composite materials with constant or variable volume fraction of the secondary phase grains. These studies resulted in various models of damage in which physical parameters of the matrix and dispersed particles, as well as their spatial distribution, size and morphology were taken into account.

Magnesium alloys are gaining more recognition as the lightest structural materials for lightweight applications, due to their low density and high stiffness-to-weight ratio. Even so, Mg alloys have not been used for critical performance applications because of their inferior mechanical properties, compared to other engineering materials. Hence, many researchers attempt to fabricate Mg-based metal-matrix composites (Mg MMCs) by different methods in order to obtain lightweight materials with excellent mechanical properties [10-15]. Magnesium based nanocomposite (AZ91D--Al₂O₃) was studied in [16], where the effect of nanoalumina particles and heat treatment on microstructure, mechanical properties and failure mechanism were investigated.

The aim of this work is to describe damage mechanism of AZ61-F Mg alloy with nominal volume fraction 1 wt.% of Al_2O_3 nano-particle reinforcement.

2. Materials and methods

The matrix used in this work is magnesium alloy AZ 61-F with 5.95 % aluminium made by Metaltech Industrial Co, LTD, Taiwan. Its chemical composi-

^{*}Corresponding author: tel.: +421 55 602 2427; fax: +421 95 633 7048; e-mail address: oksana.velgosova@tuke.sk



Fig. 1. The melt-stirring scheme used to fabricate the Mg MMCs.

tion is shown in Table 1. Al₂O₃ particles with volume fraction of 1 wt.% within MMCs are used as the reinforcement phase. The commercially available Al₂O₃ powder with a particle diameter about 20 nm with the purity \geq 99.8 % is added into AZ 61-F to form Mg-based metal-matrix composites.

The melt-stirring technique was used to fabricate the present Mg MMC. The AZ61-F and Al₂O₃ particles were initially placed inside a graphite crucible and heated to 400 °C in a resistance-heated furnace, Fig. 1; then a stirring vane functioned; meanwhile, CO₂ and SF₆ gushed from gas tank into the crucible to help the mixture of melt. CO₂ and SF₆ also can prevent the melt from oxidation. The crucible was continuously heated up to 700 °C, and then the molten alloy was stirred with a vane operated at 350 rev min⁻¹ for 3 min. Finally, the composite melt was poured into a metallic mould. The Mg MMC containing 1 wt.% of Al₂O_{3p} was prepared for further mechanical testing.

For the purposes of investigation very small flat tensile test pieces with 0.15 mm thickness were prepared by electroerosive machining, keeping the loading direction identical to the direction of extrusion. The specimens were ground and polished down to the thickness of approximately 0.1 mm. Finally, the specimens were polished on both sides by ion gunning. The test pieces were fitted into special deformation grips in the electron microscope JEM 100 C, which enables direct observation and measurement of the deformation by the option instrumentation ASID-4D. On the





Fig. 2. Fracturing (a) and diffractogram (b) of Mg₁₇Al₁₂ particles.

basis of direct observation both the deformation and failure processes of the test pieces it was possible to detect the first initiation of crack and its development.

3. Results and discussion

Microstructure parameters of the system, namely the matrix grain size, mean size of the dispersed particles and their average distance, were evaluated.

Microstructure of the experimental materials is heterogeneous with grain size of 50 μ m. According to works [17, 18] the heat deformation process of the systems, beside formation of incorporated Al₂O₃ phase, leads also to creation of intermetallic compound Mg₁₇Al₁₂. Deformation is realised through slip systems, which mutually interact by forming microcracks. On Fig. 2a also fracturing Mg₁₇Al₁₂ particles are visible. They can be the source of cracks in the material microstructure.

Transmission electron microscopy and selective electron diffraction showed presence of Mg₁₇Al₁₂ phase in the microstructure. This phase has a body centered cubic structure with the lattice parameter a = 10.569 Å, Fig. 2b. It has spherical morphology; grain size between 0.2–0.8 µm and it is coherent with



Fig. 3. The specimen before deformation (a), growth of the cracks (b), the final shape of fracture (c).

the matrix. Fine Al_2O_3 particles were distributed randomly as well as in clusters.

The samples of experimental materials, Fig. 3a,



Fig. 4. Final fracture surface.

were deformed at 20 °C at a rate of 6.6×10^{-4} s⁻¹ in elastic region. Because the materials had low plasticity due to the high volume fraction Mg₁₇Al₁₂ and Al₂O₃ phases, the damage and cracking of test piece was very fast. Cracks were initiated on the surface and propagated approximately perpendicularly to the direction of the tensile load. Overall growth of the cracks is illustrated in Fig. 3b which shows the specimen surface initiation of cavities at the matrix-particle interphase boundaries. Coalescence of the final fracture progressed along densely populated rows parallel to the load direction. The fracture surface formed by the crack growth was inclined by 45° with respect to the specimen principal axis, Fig. 3c.

Detailed study of the deformation changes showed that the crack initiation was caused by decohesion of Al₂O₃ particles and occasionally also by rupture of large Mg₁₇Al₁₂ particles. Decohesion is a result of different physical properties of both phases of the system. The Mg matrix has significantly higher thermal expansion coefficient and lower elastic modulus (26.5×10^{-6} K⁻¹ and 44.7 GPa) than Al₂O₃ (8.3×10^{-6} K⁻¹ and 393 GPa) [19]. In the system Al--Mg₁₇Al₁₂ as in the system Mg-Mg₁₇Al₁₂ there is good relationship between matrix and reinforcement phase. Mg₁₇Al₁₂ has very good bond with the matrix so that during deformation rather than particle/matrix interphase decohesion the particle failure occurs.

Large differences in the thermal expansion coefficients α result in high stress gradients, which arise on the interphase boundaries during the preparation of system. Since $\alpha_{\text{matrix}} > \alpha_{\text{particle}}$, high compressive stresses can be expected. However, because the stress gradients arise as an effect of temperature changes, during cooling (which results in increase of the stress peaks) their partial relaxation can occur. Superposition of the external load and the internal stresses can



Fig. 5a,b,c. Model of the fracture mechanism.

initiate cracking at interphase boundary. The fractures of the studied materials started at the side edges of the deformed samples [20].

On the fracture surface, the strengthening Al_2O_3 particles were observed (data not shown). The particles were distributed irregularly, individually or in clusters. During deformation, decohesion between matrix and Al_2O_3 particles takes place that is reflected in the ductile fracture with dimples. Overall, the fracture has transcrystalline ductile character, Fig. 4.

Based on the microstructure changes observed in the process of deformation, the following fracture damage mechanism has been proposed, Fig. 5:

a) Fine Al_2O_3 particles are incorporated in the microstructure. During material preparation intermetallic $Mg_{17}Al_{12}$ particles with roughly Poisson distribution are created.

b) During the deformation, failure of $Mg_{17}Al_{12}$

particles occurs and at the same time decohesion of Al_2O_3 dispersoids takes place. Fracture is initiated at the sample surface.

c) Final fracture results from coalescence of fractured $Mg_{17}Al_{12}$ particles and decohesively dislodged dispersed Al_2O_3 particles. The fracture propagates at approximately 45° angle. The secondary phases lead to transcrystalline dimple fracture of the system.

4. Conclusions

Using the "in-situ tensile test in SEM" a model of damage mechanism of the AZ61-F alloy with 1 wt.% of Al_2O_3 particle reinforced MMCs has been proposed.

It had been shown that the initial cracks were formed on the side of the specimen surfaces and by failure of $Mg_{17}Al_{12}$ particles together with decohesion of the matrix- Al_2O_3 particle boundary in Al_2O_3 particle clusters. Further increase of load led to the development of fracture towards the sample centre with contribution of damaged large $Mg_{17}Al_{12}$ particles. The final rupture took place in direction along the 45° angle with respect to the loading. The fracture was transcrystalline ductile.

Acknowledgement

The work was supported by the grant VEGA 2/0025/11.

References

- VELÍSEK, R.—IVAN, J.: Kovove Mater., 32, 1994, p. 531.
- [2] BROUTMAN, L. V.—KROCK, R. H.: Compos. Mater., 5, 1974, p. 27.
- [3] BESTERCI, M.—IVAN, J.: J. of Mater. Sci. Lett., 15, 1996, p. 2071.
- [4] BESTERCI, M.—IVAN, J.: J. of Mater. Sci. Lett., 17, 1998, p. 773.

http://dx.doi.org/10.1023/A:1006639700906

[5] BESTERCI, M.—IVAN, J.—KOVÁČ, L.—WEISS-GAERBER, T.—SAUER, C.: Mat. Letters, 38, 1999, p. 270.

http://dx.doi.org/10.1016/S0167-577X(98)00171-2

- [6] BESTERCI, M.—IVAN, J.—KOVÁČ, L.—WEISS-GAERBER, T.—SAUER, C.: Kovove Mater., 36, 1998, p. 239.
- [7] IVAN, J.—KOVÁČ, L.: Kovove Mater., 38, 2000, p. 21.
- [8] BESTERCI, M.—VELGOSOVÁ, O.—IVAN, J.— HVIZDOŠ, P.—KULU, P.: Kovove Mater., 47, 2009, p. 221.
- [9] BESTERCI, M.—VELGOSOVÁ, O.—IVAN, J.—
 KVAČKAJ, T.: J. of Mat. Sci., 45, 2010, p. 4073.
 <u>http://dx.doi.org/10.1007/s10853-010-4493-5</u>

- [10] ROGATHI, P. K.: ASM Handbook-Castings, 15, 1992, p. 840.
- [11] CAO, G.—CHOI, H.—OPORTUS, J.—KONISHI, H.—LI, X.: Materials Science and Engineering A, 494, 2008, p. 127.
- http://dx.doi.org/10.1016/j.msea.2008.04.070
- [12] UGANDHAR, S.—GUPTA, M.—SINHA, S. K.: Composite Structures, 72, 2006, p. 266. <u>http://dx.doi.org/10.1016/j.compstruct.2004.11.010</u>
- [13] HASSAN, S. F.—GUPTA, M.: J. of Alloys and Compounds, 419, 2006, p. 84. <u>http://dx.doi.org/10.1016/j.jallcom.2005.10.005</u>
- [14] HASSAN, S. F.—GUPTA, M.: Materials Science and Engineering A, 392, 2005, p. 163. <u>http://dx.doi.org/10.1016/j.msea.2004.09.047</u>

- [15] MISHNAEVSKY, L. L.—LIPPMANNA, N.— SCHMAUDERB, S.—GUMBSCH, P.: Engineering Fracture Mechanics, 63, 1999, p. 395.
- [16] SHANTHI, M.—TUN, K. S.—PANDEY, R. S.— GUPTA, M.: Kovove Mater., 49, 2011, p. 197.
- [17] PO-CHOU LIN—SONG-JENG HUANG—PIN-SEN HONG: Acta Metallurgica Slovaca, 16, 2010, p. 237.
- [18] EL-MORSY, A.—ISMAIL, A.—WALY, M.: Materials Science and Engineering: A, 486, 2008, p. 528.
- [19] BRANDES, E. A.—BROOK, G. B.: Smithells Metals Reference Book. 7th Ed. Butterworth-Heinemann Ltd. 1998.
- [20] LUKÁČ, P.—TROJANOVÁ, Z.: Kovove Mater., 44, 2006, p. 243.