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Neutron Diffraction Study and Deformation Behavior of a Composite Based Mg Alloy Reinforced by Short Saffil Fibers

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In the present work neutron diffraction has been applied for *ex situ* investigation of residual stresses in Mg–4%Al–1%Ca (AX41) magnesium alloy reinforced with short Saffil fibers. Samples were deformed in compression at room temperature. Two types of fiber arrangement were investigated. In both samples the fibers were homogeneously distributed and arranged in parallel planes with a random fiber orientation. In the first sample these planes were parallel with the loading axis and in the second one they were perpendicular to the loading direction. Significant dependence of both the mechanical properties and residual strains on the fibers orientation was observed. Sample with parallel fiber arrangement showed higher hardness and lower ductility. Further the increment of residual tensile lattice strain in the matrix with a macroscopic deformation is much higher than in the other case. It was found that the residual strain evolution strongly depends on the orientation of grains in the matrix.

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1. Introduction

Magnesium alloys belong to the lightest structural materials and therefore they are attractive candidates for many industrial applications. Nevertheless, some of their properties, as e.g. the low creep resistance or the observed degradation of the mechanical properties at elevated temperatures, limit their application potential. These disadvantages can be considerably eliminated by an adding of ceramic reinforcement phases, as e.g. SiC or Al_2O_3, to the matrix alloy [1–4].

The investigation of the deformation mechanisms of fibers reinforced magnesium-based composites is a challenging task. Beside the deformation mechanisms operating in the matrix (basal and non-basal slip, twinning) [4], further reinforcement effects (e.g. load transfer from the matrix to fibers; enhanced dislocation density due to geometrical and thermal mismatch between matrix and fibers) have to be taken into account. Thus, resultant mechanical properties of a particular composite are influenced by several factors such as size and volume fraction of fibers and their orientation with respect to the acting load [5] and existing residual internal stresses. Understanding the role of these parameters in the plastic deformation process is crucial from the point of view of the possible production of high-performance composites.

The neutron diffraction method has been found as an effective experimental technique for the investigation of the metal matrix composites [6]. Owing to the large penetration depth of the thermal neutrons, the results characterize a large sample volume. Since the diffraction pattern is sensitive to the changes of the lattice parameter, the lattice strain and the residual stress distribution can be evaluated.

The present work focuses on the investigation of the evaluation of the residual strains in the deformed AX41 magnesium alloy reinforced with short Saffil fibers. The influence of the fibers orientation with respect to the loading axis is discussed in detail.

2. Experimental

AX41 magnesium alloy (nominal composition Mg–4 wt% Al–1 wt% Ca) reinforced with 20% of short Saffil fibers (δ-A_2O_3) was investigated in this study. The material was prepared by the squeeze casting technique. The fibers exhibited the mean length of 80 μm and a diameter of 3–5 μm. Specimens with the size $20 \times 10 \times 10$ mm^3 were fabricated from ingots with two orientations of the fibers plane: respectively parallel and perpendicular to the stress axis. Within a given fibers plane, the fibers had random orientation (see Fig. 1).

Neutron diffraction measurements were performed in a high resolution ($\Delta d/d \approx 2$–3×10^{-3}) biaxial diffractometer TKSN-400 optimized for the study of internal stresses in polycrystalline materials. Diffracted neutrons exhibited the wavelength of $\lambda = 2.3$ Å, the neutron flux was approximately 10^5 n per cm^2/s. The diffractometer was equipped with two-dimensional ^{3}He positive sensitive detector with 220×220 mm^2 active window and space resolution of 0.8×0.8 mm^2. The detector position was fixed during the whole experiment. Duration of each measurement was 4 h. The gauge volume was situated inside the sample with two 5.5×5.5 mm^2 slits.

The tests took place *ex-situ*, i.e. the specimens were pre-deformed at room temperature in compression with an initial strain rate of 10^{-3} s^{-1} to predetermined strain values. The lattice strain of the magnesium matrix was

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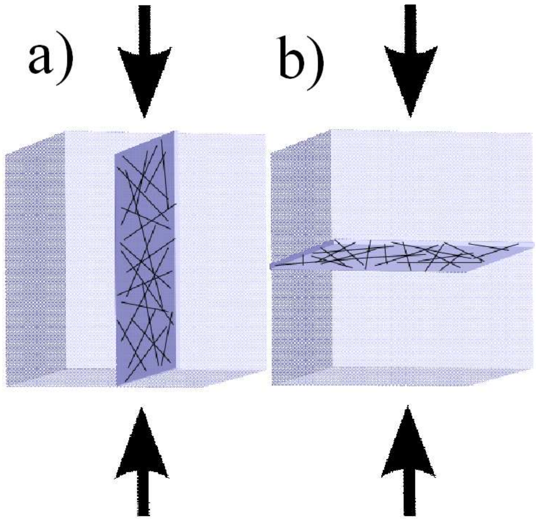

Fig. 1. Scheme of the fibers orientation in the specimens (a) fibers plane parallel to the load axis (parallel orientation), (b) fiber plane perpendicular to the load axis (perpendicular orientation).

evaluated as a function of the macroscopic stress level. Lattice strains were measured in both axial and radial geometry, i.e. in parallel and perpendicular orientation of the diffraction vector with respect to the loading and fibers plane directions. Since the diffraction spots from individual grains were visible in the diffraction ring, the sample was rotated during measurement in order to increase a number of diffracting grains in the irradiated gauge volume and improve the averaging of the measured lattice strains.

3. Results and discussion

As it is obvious from Fig. 2, the mechanical properties show a strong influence of the fibers orientation with respect to the loading axis. In the case of the parallel orientation, the sample exhibit significantly higher yield and tensile strength. As it was shown by several authors [7, 8], the largest contribution to the strengthening of fibers reinforced composites is given by the load transfer from the matrix to the fibers. The observed behavior is in agreement with the shear lag model [9], which determines the stress necessary for deformation of a composite due to the load transfer as follows [10]:

$$\sigma_{\rm LT} = \sigma_m \left(1 + \frac{(L+t)A}{4L}\right) f + \sigma_m (1-f), \tag{1}$$

where σ_m is the yield stress in the matrix, L is the fiber size in the direction of the applied stress, t is the fiber size in the perpendicular direction, A is the fiber aspect ratio (L/t) and f is the volume fraction of the fibers. Using the above listed values determined from experimental data, the ratio between the $\sigma_{\rm LT}^{\parallel}$ and $\sigma_{\rm LT}^{\perp}$ is approximately 2, which corresponds (within the experimental error) to the ratio between yield stresses measured in parallel ($\sigma_{02}^{\parallel}$ =287 MPa) and perpendicular ($\sigma_{02}^{\perp}$ =150 MPa) direction, respectively.

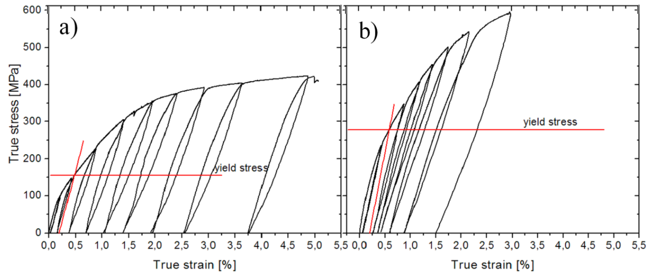

Fig. 2. True stress-true strain curve of AX41 composite (a) with perpendicular fibers plane orientation, (b) with parallel fibers plane orientation.

The residual lattice strain was calculated using the Bragg law, which gives the relation between the scattering angle (θ) and lattice spacing (d) as

$$2d \sin\theta = \lambda. \tag{2}$$

The strain (e) is given by differentiating the Bragg equation

$$e = \frac{d-d_0}{d_0} = -(\cot\theta)(\theta-\theta_0). \tag{3}$$

The angle θ_0 was measured for each diffraction peak on the reference sample, which was the unreinforced AX41 magnesium alloy which was considered to be in the stress-free state. Four peaks were measured with the 2D detector: (00.2), (10.0), (11.0) and (10.3).

The residual strain is plotted as a function the applied stress for both types of composites in Figs. 3 and 4. In the composite with the parallel fiber plane orientation the residual strain on all four planes has a tensile character already in the initial state. This feature can be explained by the different thermal expansion coefficient (CTE) of the matrix and alumina fibers (Mg: 27×10^{-6} K^{-1}, Al_2O_3: 7.2×10^{-6} K^{-1}) [11]. Owing to the larger CTE of magnesium, the hydrostatic tensile thermal strain arises during the solidification process [12] and following cooling to ambient temperature. This effect is not present in the axial direction for the perpendicular fiber plane orientation, most probably due to the high aspect ratio of the fibers (Fig. 4a). On the other hand, high tensile strain appears in the radial direction (Fig. 4b).

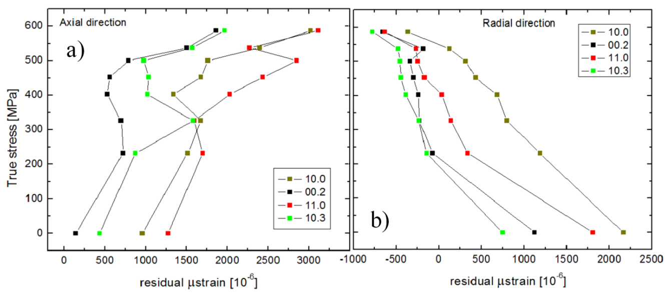

Fig. 3. Residual strain evolution in AX41 composite with parallel fiber plane orientation (a) in axial direction, (b) in radial direction.

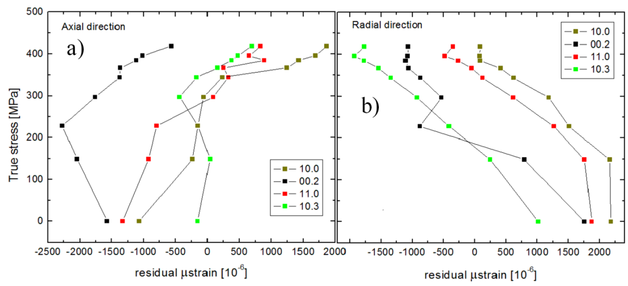

Fig. 4. As in Fig. 3, but for perpendicular fiber plane orientation.

An interesting effect can be observed at both composites in the axial direction (Figs. 3a and 4a). The lattice strain evolution shows an inverse behavior, i.e. the compressive deformation increases the residual tensile strain in the matrix. In the case of the parallel fiber orientation this effect can be explained as follows: the reinforcement phase during macroscopic loading deforms elastically and during unloading returns to its initial state. The matrix, owing to the previous compressive deformation, needs to elongate, which creates tensile strain in it [13]. In the case of perpendicular fiber orientation the appearance of the tensile strain is rather unexpected. Its presence can be attributed to the not perfectly planar distribution of the reinforcement, i.e. the longitudinal axis of the majority of the fibers is not quite perpendicular to the loading direction (Fig. 5).

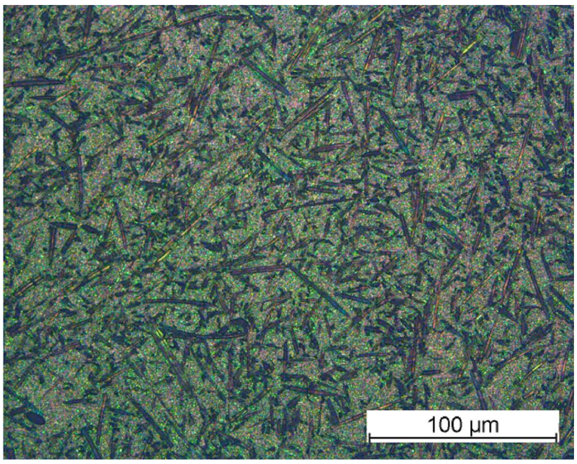

Fig. 5. AX41 composite with fiber plane perpendicular to the load axis.

In the radial direction, the initial tensile strain decreases for both fiber orientations (Figs. 3b and 4b). This effect is caused by the balancing Poisson reaction to the axial tensile stress that increases with increasing differential flow of the matrix relative to the fibers.

Based on the lattice strain results two further comments can be proposed:

First, in the axial direction the lattice strain in the matrix is larger for the parallel fiber plane orientation, which indicates a more effective fibers effect of the fibers. Second, in the radial direction the compressive lattice strain reached a higher level for the perpendicular fiber orientation. As it can be seen in Fig. 1a, the perpendicular specimen is more ductile. It means that the matrix was more deformed and during the unloading the larger compressive strains arose owing to the different response of the matrix and fibers on this process.

It is obvious that the evolution of the residual lattice strains with the applied stress is different for the particular planes. Similarly to the observations in the literature [14-15] "soft-orientations" and "hard-orientations" were observed. The extensive twinning on the $\{11\bar{2}0\}$ planes belongs to the most probable deformation mode in magnesium along the basal slip [16]. The measured {00.2} and {10.3} planes represent parent orientation with respect to the axial detector in the given experimental geometry. Figures 3a and 4a show a relaxation of the {00.2} and {10.3} lattice strains at the stress levels close to the experimental yield stress, which emphasizes the important role of the $\{11\bar{2}0\}$ twinning mode in the strain accommodation. Consistently, the daughter {10.0} and {11.0} orientations have to accumulate larger portion of load, since the twinning process reorients them out from position favorable for both basal slip and extension twinning. Therefore their deviation has an opposite sign than that of their counterparts.

4. Conclusions

In this work AX41 composite was investigated with *ex situ* neutron diffraction method. The following conclusions may be drawn:

- Deformation curves show significant differences between the two types of composite samples. Parallel fiber plane orientation significantly improves the yield stress and strength. On the other hand, the ductility decreases in comparison to the perpendicular fiber plane arrangement.
- Neutron diffraction measurements support the mechanical test results. The residual lattice strain in the matrix in axial direction, is higher for parallel fiber plane arrangement, which indicates a more effective reinforcement effect of the fibers.
- For the perpendicular fiber orientation, the compressive lattice strain in the matrix achieved a higher level (measured in the radial direction), which is caused by the higher plastic deformation of the matrix.

Acknowledgments

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References

[1] H. Ferkel, B.L. Mordike, *Mater. Sci. Eng. A* **298**, 193 (2001).

[2] K.B. Nie, X.J. Wang, X.S. Hu, L. Xu, K. Wu, M.Y. Zheng, *Mater. Sci. Eng. A* **528**, 5278 (2011).

[3] Z. Trojanova, V. Gartnerova, A. Jager, A. Namesny, M. Chalupova, P. Palcek, P. Lukac, *Compos. Sci. Technol.* **69**, 2256 (2009).

[4] R. Mises, *Z. Angew. Math. Mech.* **8**, 161 (1928).

[5] M. Knezevic, A. Levinson, R. Harris, R.K. Mishra, R.D. Doherty, S.R. Kalidindi, *Acta Mater.* **58**, 6230 (2010).

[6] M.R. Daymond, C. Lund, M.A.M. Bourke, D.C. Dunand, *Metall. Mater. Trans. A* **30**, 2989 (1999).

[7] Z. Trojanova, Z. Szaraz, F. Chmelik, P. Lukac, *Mater. Sci. Eng. A* **528**, 2479 (2011).

[8] Z. Trojanova, Z. Szaraz, J. Labar, P. Lukac, *J. Mater. Process. Technol.* **162-163**, 131 (2005).

[9] M. Taya, R.J. Arsenault, *Scr. Metall.* **21**, 349 (1987).

[10] Z. Trojanova, K. Mathis, P. Lukac, M. Janecek, G. Farkas, *Metall. Mater. Trans. A* **45(1)**, 29 (2014).

[11] S.-H. Ryu, J.-H. Park, C.S. Lee, S.-H. Ahn, S.-T. Oh, *Mater. Trans.* **50**, 1553 (2009).

[12] M.T. Hutchings, P.J. Withers, T.M. Holden, T. Lorentzen, in: *Comprehensive Materials Processing*, Eds. T.R. Watkins, G.S. Schajer, M.J. Lance, Taylor & Francis, Boca Raton 2012.

[13] G. Farkas, K. Máthis, P. Lukáš, J. Pilch, M. Vrána, M. Janeček, Z. Trojanová, *Mater. Sci. Forum* **777**, 92 (2014).

[14] G. Garces, G. Bruno, *Compos. Sci. Technol.* **66**, 2664 (2006).

[15] P. Van Houtte, L. De Buyser, *Acta Metall. Mater.* **41**, 323 (1993).

[16] S.R. Agnew, D.W. Brown, C.N. Tomé, *Acta Mater.* **54**, 4841 (2006).