iTHE EFFECT OF THERMAL CYCLING ON THE STIFFNESS AND DENSITY OF THE SHORT FIBER AL_2O_3/AZ91 MG ALLOY COMPOSITE

P.Abachi, K.Purazrang and I. Hadi

Materials Sci. and Eng. Dept., Sharif Uni. of Technology, P.O.Box : 11365-9466, Tehran / Iran.

ABSTRACT

Thermal stresses due to the mismatch of the thermal expansion coefficients between metallic matrix and ceramic fibers may restrict the application of the metal matrix composites under temperature fluctuation conditions. At the present work, the possible effects of thermal cycling on microstructural changes, the elastic modulus and density of the 5 vol.% Al_2O_3 short fibers/AZ91 Mg alloy composite were investigated. The examination of specimens by scanning electron microscope revealed the microvoids formation and debonding of the matrix/reinforcement interfaces after thermal cycling. Studies were performed on the specimens with dimension of $10\times10\times50$ mm^3. These specimens were thermally cycled up to 1750 cycles using a fully automated apparatus. Each cycle involved with heating up to 300°C for 15 min. in a tube furnace and cooling down to room temperature during 5 min. via forced air. According to test results, the density and elastic modulus decreased after exposure on thermal cycles. With respect to the mass conversation, the density decrement may be related to microvoids formation. The failure of matrix/reinforcement interfaces may result in deterioration of elastic modulus. The extent of decrements was enhanced with increasing the number of cycles.

Key words: Thermal cycling, Al_2O_3/Mg alloy composite, microstructure, interface, elastic modulus, density

1. INTRODUCTION

The importance of environmental and economical aspects in automotive industry has led to intensive attention to the application of light weight materials. Therefore, in this relation, magnesium alloys could be better candidates than aluminum alloys. Improvements on strength, elastic modulus, wear resistance, lower thermal expansion coefficient, and thereby better performance are also provided by using ceramic reinforcements in such metallic matrixes. Considering safety aspects, all historical and service conditions of structural parts such as temperature changes must be accounted for in part design and material selection. The response of metal matrix composites on changes in temperature, e.g. during cooling from the processing temperature to room temperature, has been investigated mainly on aluminum alloys matrix composites [1-5]. However, some studies have been also provided on microstructural changes and the dislocation generation in thermally cycled magnesium alloys matrix composites using internal friction or acoustic emission measurements [6-8]. At the present work, a fully automated apparatus capable to produce thermal cycles was developed. The effect of thermal cycling on microstructural changes of the 5 vol.% Al_2O_3 short fibers/AZ91 Mg alloy composite was investigated by SEM. The elastic modulus and density of specimens were also measured before and after thermal cycling.

The elastic modulus decrements were also correlated to the density decrements using Mackenzie's equation [9]. The observed differences between experimental and analytical values could be mainly related to the fact that the equation is suggested to monolithic materials rather than composites.

2. EXPERIMENTAL PROCEDURE

The AZ91 magnesium alloy with nominally composition of 10wt.% Al, 0.72% Zn, 0.1% Si, 0.05% Cu, 0.28% Mn and balance Mg, was reinforced by melt infiltration of, 97 wt.% δ-Al_2O_3 and 3% SiO_2 (Saffil), short fibers perform applying squeeze casting method. The short fibers with

diameters of ≅ 3μm and lengths up to a maximum of 25 μm were randomly planar distributed in preform. In addition, the preform contained alumina and starch as binder, SiO_2 as stabilizer. To prevent the premature solidification, the melt overheated up to almost 800°C and the preform heated up to 1000°C. The unreinforced plates were also produced at the same condition for comparison of the microstructures.

For the measurement of density by the 0.001g precision balance and appropriate kit based on Archimedes technique, test specimens with dimension of 10×10×20 mm^3 were machined from the main composite plate and thermally cycled up to 1750 between 300°C and room temperature. The thermal cycling apparatus used in this work is shown in Fig.1. Specimens' heating was conducted by placing them within a tube furnace, via. an automated, digitally controlled piston connected to sample holder. The temperature at the specimen surfaces was determined by means of an iron-constantan thermocouple. The action of the piston to withdraw the specimen from the furnace after a preset time was made by a time controller. Specimens' cooling was realized through a vent blowing the forced air. The number of thermal cycles was also determined by a counter. The temperature vs. time curve used for thermal cycling is shown in Fig.2.

To evaluate the elastic modulus, the original composite plates were firstly machined into tensile testing specimens with a diameter of 5 mm and gauge length of 25.4 mm. The longitudinal specimens' axes were within the plane containing the large axes of the fibers. Tensile tests were carried out on an Instron 6027 universal machine with a cross head speed of 0.5 mm/min. An extensometer was mounted on specimen gauge length to measure the displacement values during the test. Microstructural examinations were performed on polished and Nital etched specimens using the light and scanning electron microscopes.

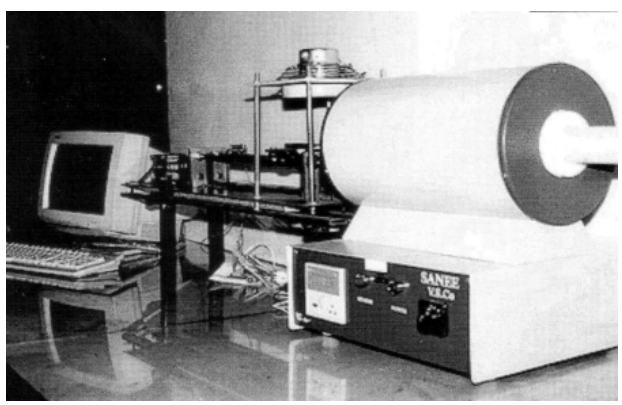


Fig.1. The apparatus used for thermal cycling.

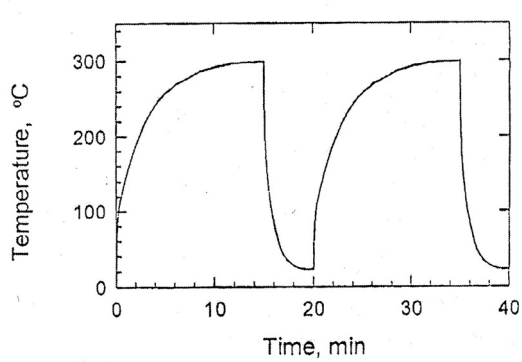
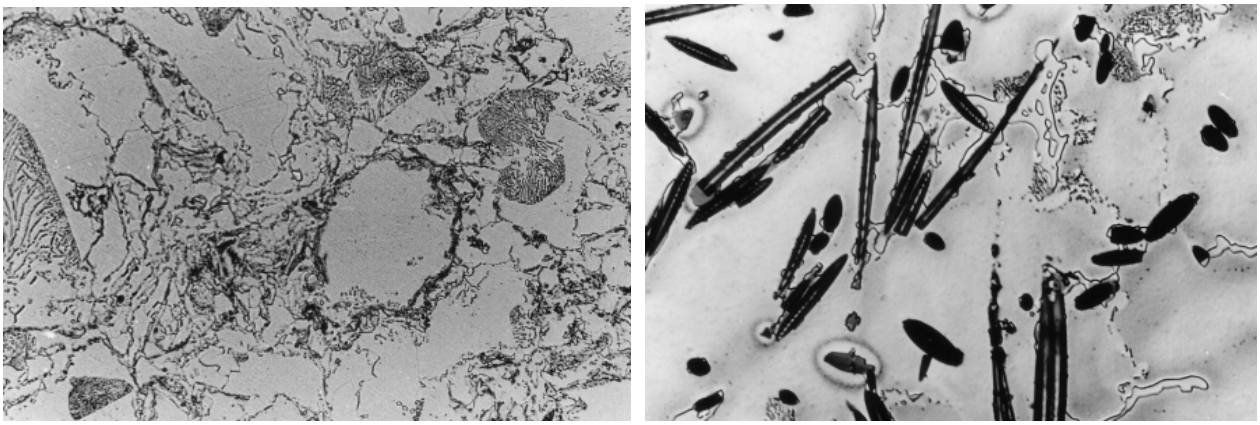


Fig.2. Temperature-time cycles used for thermal cycling.

3. RESULTS and DISCUSSION

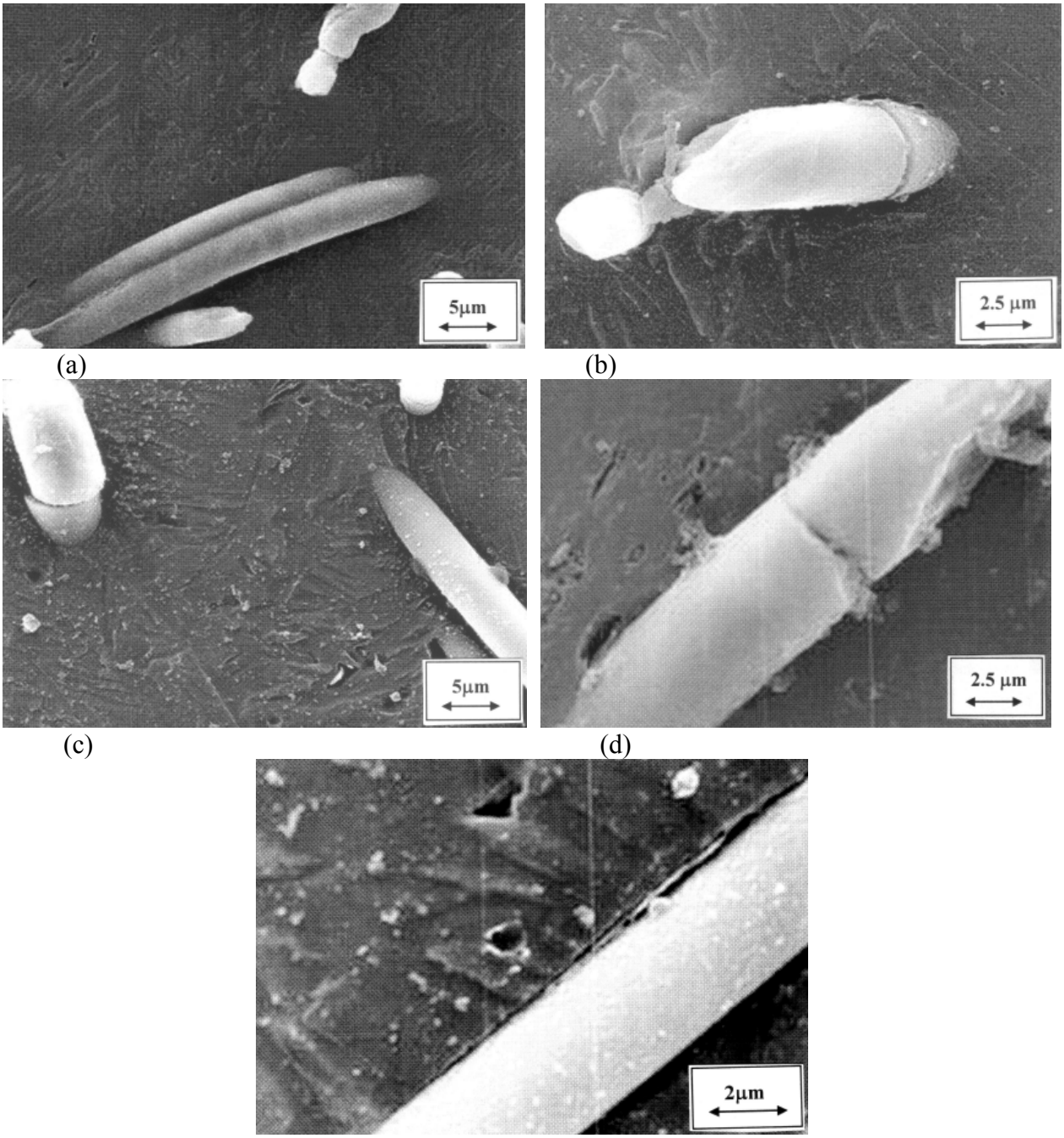
As a result of the slight reaction between the melted matrix alloy and fibers as well as binder, Mg_2Si has been detected at the interface by XRD analysis [10]. In addition, diffractometer phase analysis has shown the evidence of MgO [10]. The optical microscopy examinations on unreinforced specimens indicated on the precipitation of $Mg_{17}Al_{12}$ in both massive and lamellar forms which can be seen in Fig.3a. As shown in figure 3b, the fibers' surfaces may act as proper heterogeneous nucleation sights for the precipitation of $Mg_{17}Al_{12}$ phase. However, according to other works, this may be due to the enrichment of matrix by Al in the vicinity of fibers due to decomposition of inorganic alumina binder [11, 12].



(a) (b)

Fig.3. Showing the $Mg_{17}Al_{12}$ precipitates in, (a) an unreinforced AZ91 alloy in both massive and lamellar forms, (b) the vicinity of fibers, x 500.

The SEM micrographs of the as cast composite and thermally cycled specimens are shown in Figs.4 a-e. According to Fig. 4a, there is a good bonding between fibers and matrix with no evidence of cracks, microvoids or any other failures at the microstructure of the specimen. In Fig. 4b, some failures can be observed. Fig. 4c indicates on the cavitation and cracking of the matrix after 1000 cycles. Fig. 4 d shows some cracks in the matrix and fracture of the fiber at higher magnification. Moreover, the thermal stresses relieving effects as debonding at the fiber/matrix interface can be seen in Fig. 4e.



(a) (b) (c) (d) (e)

Figs.4. Showing SEM micrographs of (a) as cast composite and thermally cycled specimens after, (b) 500 (c) 1000 (d) 1000 and (e) 1750 cycles.

The relative elastic modulus decrement was calculated based on tensile test results for any number of cycles by using Eq.1:

$$D_E = 1-(E_n/E_o) \quad (1)$$

where the original elastic modulus and elastic modulus after n cycles are denoted as E_o and E_n , respectively. Considering Fig. 5, the elastic modulus decrement has increasingly trend up to 1500 cycles. Furthermore, the rate of decrement is high at the intermediate stages but it decreases at

higher number of cycles. In overall, the defects at the fiber/matrix interface due to thermal stresses relieves reduce the interfacial bond strength and, therefore, cause the deterioration of the load transfer from matrix to fiber. These may also reduce the role of the fibers in improving the elastic modulus of the matrix. However, after a certain number of cycles, the high extent of cavitation decreases the interfacial bond strength and, thereby, affects the matrix constraint by reinforcement [9].

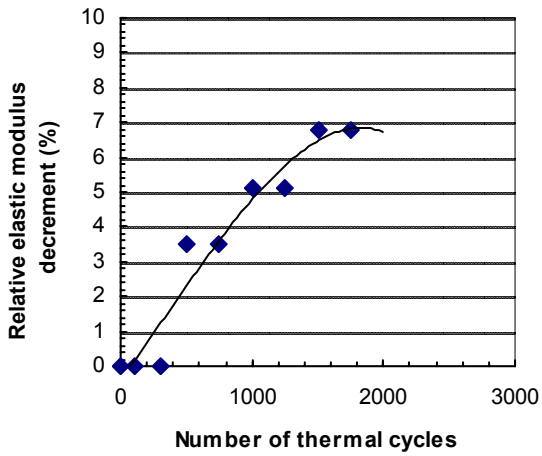


Fig. 5. The relative elastic modulus decrement versus the number of thermal cycles.

In relation to the density of composites, the experimental values indicated on a decrease with increasing the cycle numbers. In Fig. 6, the relative density decrement, calculated from Eq.2, is shown with respect to the number of thermal cycles.

$$D_\rho = 1-(\rho_n/\rho_\circ) \times 100 \qquad (2)$$

where $\rho_\circ$ and ρ_n are the original density of composite and density after n cycles, respectively. Since, no composite weight loss observed after thermal cycling, the density decrease may be associated with microvoids formation and hence with the dimensional changes of the composites.

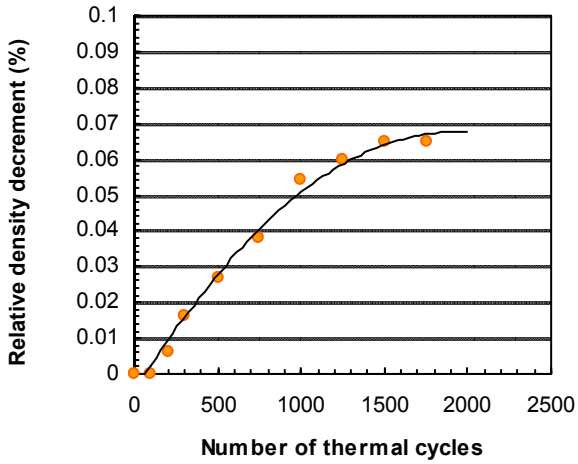


Fig. 6. The relative density decrement versus the number of thermal cycles.

The correlation of elastic modulus decrement to the relative density decrement was provided by Mackenzie's equation, i.e. by using the following equation [9]:

$$D_E = b(\rho_\circ/\rho_m)D_\rho + b_1[(\rho_\circ/\rho_m)\, D_\rho]^2 \qquad (3)$$

where ρ_m and $\rho_\circ$ are the matrix and composite density, based on rule of mixture, before thermal cycling. D_ρ is defined same as Eq.2. The values of b and b_1 are expressed as 1.91 and – 0.91 for materials with Poissons's ratio ν=0.3. The differences between experimental and analytical values, as shown in Fig. 7, could be mainly related to the fact that the Eq.3 is suggested to monolithic materials rather than composites. Furthermore, in this equation, the precise location of voids is not of significance. This means that the Mackenzie's equation treats the material as continuum.

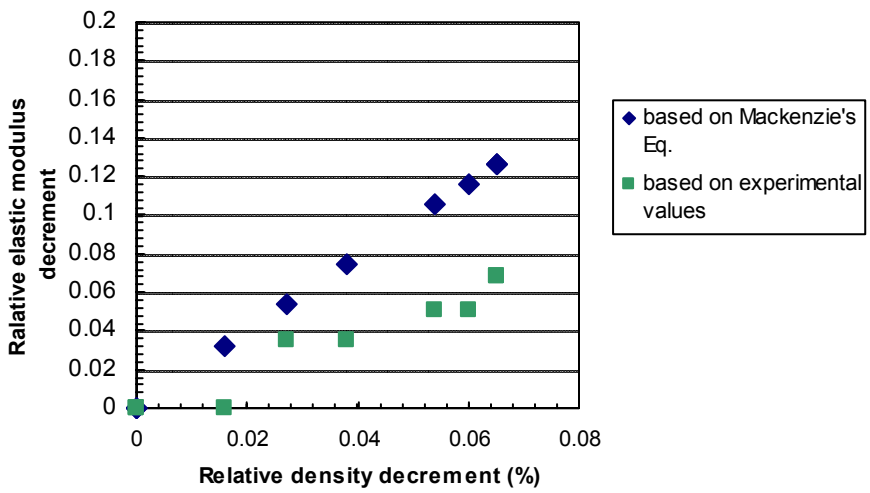


Fig.7. Showing the correlation of relative elastic modulus decrement to the relative density decrement based on experimental and analytical values.

In explanation of the above mentioned concepts, it is noteworthy to pay attention the following descriptions : As a consequence of the short fiber Al_2O_3/AZ91 Mg alloy composites fabrication at elevated temperature by the squeeze casting method, residual thermal stresses due to the mismatch of the thermal expansion coefficients between metallic matrix (with CTE of 26.7×10^{-6} K^{-1}) and ceramic fibers (with CTE of 6.7×10^{-6} K^{-1}) can be generated. In this state, i.e. after cooling, the matrix is under tensile stresses and fibers under compressive stresses. During first cycle of thermal cycling, heating up leads to the reverse signs of the stresses so that during heating the tensile stresses acting on the matrix decreases to zero and then compressive stresses will be revealed. By further heating, these stresses can relax to some extent by deformation. Again, by cooling the composite the internal stresses behave in an opposite manner. At the following, compressive stresses acting on the matrix decreases to zero and then tensile stresses will be revealed. When the internal stresses acting on the matrix exceeds the matrix yield strength, they will be relieved by matrix plastic deformation. Since, the tensile stresses values are more than the compressive stresses [13], the elongation can be observed during initial cycles. Different deformation mechanisms may also operate such as dislocation glide, twinning, diffusional creep and grain boundary sliding. The contribution of these mechanisms is the result of a complex interplay between the temperature dependencies of the thermal stresses and the matrix yield strength and will vary according to the temperature and other parameters of thermal

cycling [13]. Therefore, at higher number of cycles, the matrix will work harden due to the plastic deformation and subsequently its yield strength will increase. Although, aging during heating up of the specimens may act in an opposite manner, so that, it results in further specimen deformation. Finally, the continued thermal cycles will lead to cavitation, cracking of the matrix, fracture of fibers, formation of interfacial microvoids and thereby bond strength decrement at fibers/matrix interfaces, see figs.4 b-e. It seems that, on this way, the constraint imposed on the expansion of the matrix could be reduced. As a result, a decrease on the rate of mechanical properties deteriorations of the MMC's could be observed.

4. SUMMARY and CONCLUSIONS

1. The importance of environmental and economical aspects in automotive industry has led to intensive attention to the application of light weight materials.
2. The improvements on mechanical properties and thereby better performance of the magnesium alloys structural parts are provided by using ceramic reinforcements.
3. After cooling of MMC's from production temperature, the matrix is under tensile stresses and fibers under compressive stresses.
4. As a consequence of thermal cycling, thermal stresses due to the mismatch of the thermal expansion coefficients between metallic matrix and ceramic fibers can be generated.
5. The response of short fiber Al_2O_3/AZ91magnesium alloy matrix composites on changes in temperature was observed as formation of microvoids and debonding of matrix/reinforcement interfaces which result in deterioration in density and elastic modulus.

References

1. **Flom Y.** and **Arsenault R.J., "**Deformation in Al-SiC composites due to thermal Stresses **",** Mat.Sci.&Eng. 75 (1985), 151-167.
2. **Arsenault R.J** and **Shi N., "**Dislocation generation due to differences between the coefficients of **t**hermal expansion**",** Mat.Sci.&Eng. 81 (1986), 175-187.
3. **Arsenault R.J** and **Taya M., "**Thermal residual stress in metal matrix composites**",** Acta Metall. 35/3 (1987), 651-659.
4. **Taya M.**, **Lulay K.E.** and **Lloyd D.J., "**Strengthening of a particulate metal matrix composite by quenching**",** Acta Metall. Mater. 39/1 (1991), 73-87.
5. **Hunt Jr. W.H.**, **Brockenbrough J.R.** and **Magnusen P.E., "**An Al-Si-Mg composites model system : Microstructural effects on deformation and damage evolution**",** Scripta Metallurgica et Materialia, 25 (1991), 15-20.
6. **Chmelik F.**, **Lukac P.**, **Kiehn J.**, **Mordike B.L.**, **Kainer K.U.** and **Langdon T.G., "**Characteristics of thermal cycling in a magnesium alloy composite**",** Mat.Sci.&Eng. A325 (2002), 320-323.
7. **Chmelik F.**, **Lukac P.**, **Janececk M.**, **Moll F.**, **Mordike B.L.**, **Kainer K.U.** and **Langdon T.G., "**An evaluation of creep characteristics of an AZ91 magnesium alloy composite using acoustic emission**"**, Mat.Sci.&Eng. A338 (2002), 1-7.
8. **Wang J.G., Hsiung L.M.**, **Nieh T.G.** and **Mabuchi M., "**Creep of a heat treated Mg-4Y-3RE**",** Mat.Sci.&Eng. A315 (2001), 81-88.
9. **Xu Z.R.**, **Chawla K.K.** , **Wolfenden A.** , **Neumann A.** , **Liggett G.M.** and **Chawla N.**, **"**Stiffness loss and density decrease due to thermal cycling in an alumina fiber/magnesium alloy composite**" ,** Mat.Sci.&Eng. A203 (1995), 75-80.
10. **Rudajevova A.**, **Kiehn J., Lukac P.**, **Kiehn J.**, **Kainer K.U.** and **Mordike B.L., "**Thermal properties of AZ91 and QE22 matrix composites**",** in Magnesium alloys and their applications, ed.: **Mordike B.L.** and **Kainer K.U.,** Proceedings volume, Wolfsburg Germany, Werkstoff-Informationsgesellschaft mbH, 1998, 665-670.

11. **Kiehn J., Kainer K.U.,Trojanova Z., Chemlik F.** and **Lukac P. "**Detection of dislocations in short fiber reinforced magnesium alloys by non-destructive methods**",** in Proceedings of ICCM-11, vol. III, Gold Coast Australia, 1997, 585-996.
12. **Kainer K.U., "**Cast magnesium alloys reinforced by short fibers**",** in Magnesium alloys and their applications, ed.: **Mordike B.L.** and **Hehmann F.,** Proceedings volume, Oberursel Germany, DGM Informationsgesellschaft, 1992, 415-422.
13. **Kiehn J., Kainer K.U., Mordike B.L., Chemlik F.** and **Lukac P. "**Acoustic emission and dilatometry of thermally cycled short fiber reinforced magnesium alloys**",** in Magnesium alloys and their applications, ed.: **Mordike B.L.** and **Kainer K.U.,** Proceedings volume, Wolfsburg Germany, Werkstoff-Informations-gesellschaft mbH, 1998, 671-676.