Deformation history effect in an AS21 magnesium alloy based composite

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Received 12 February 2006, received in revised form 18 September 2006, accepted 18 September 2006

Abstract

The thermal expansion characteristics of magnesium alloy AS21 reinforced with 25 vol.% of Saffil® short fibres were measured in the temperature range from room temperature to 380 °C. The same measurements were also performed for samples pre-strained in tension as well as in compression. Permanent sample length changes (elongation as well as contraction) were found after the first thermal cycle. The microstructure of samples was examined by transmission electron microscopy (TEM). Stress relaxation tests were used to estimate the internal stress in the alloy as well as in the composite. Residual thermal stresses and different plastic deformation mechanisms in tension as well as in compression are responsible for the thermal expansion behaviour.

Key words: magnesium alloy based composite, thermal stresses, plastic deformation, dislocations, twinning

1. Introduction

Applications of magnesium alloys in industry have increased in recent years because of their low density. Thus in comparison to other conventional alloys, magnesium alloys exhibit a very high specific strength (the ratio of the strength to density). However, the strength of magnesium alloys decreases rapidly with increasing temperature [1–3]. The yield strength of magnesium alloys may be improved by solid solution hardening, precipitation strengthening and grain size refinement [4, 5]. The yield strength increases as the grain size decreases. On the other hand, minimum creep rate in steady-state stage at a given stress increases with decreasing grain size, i.e. the creep resistance may decrease as the grain size decreases.

Further improvement in the mechanical properties of magnesium alloys may be achieved by the addition of reinforcement (fibres or particles) to the matrix. A new group of materials – metal matrix composites is created. Ye and Liu [6] have very recently reviewed recent studies in magnesium matrix composites. The mechanical properties of magnesium-based composites are higher than those of unreinforced alloys (see also e.g. [7]). It is known that in composites, there is a large difference in the coefficients of thermal expansion (CTE) between the matrix and the ceramic reinforcement. When a metal matrix composite (MMC) is cooled from a higher temperature to room temperature, misfit strains occur because of different thermal contraction at the interfaces. These strains induce thermal stresses that may be higher than the yield stress of the matrix. Therefore, the thermal stresses may be sufficient to generate new dislocations at the interfaces between the matrix and the reinforcement. Accordingly, after cooling the composite, the dislocation density in the matrix is higher than in unreinforced matrix. An increase in the dislocation density near reinforcement fibres has been calculated as [8, 9]

$$\Delta\rho = \frac{B\,f\,\Delta\alpha\,\Delta T}{b\,(1-f)\,t}, \qquad (1)$$

where f is the volume fraction of the reinforcement,

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t is its minimum size, b is the magnitude of the Burgers vector of dislocations, B is a geometrical constant, $\Delta\alpha = \alpha_\mathrm{f} - \alpha_\mathrm{m}$ is the difference in the CTEs of the reinforcement and the matrix, and ΔT is the temperature change.

The type, size and distribution of reinforcement influence not only the deformation behaviour of a composite but also its physical properties, such as density, resistivity, thermal conductivity and thermal expansion.

With increasing temperature, the thermal stress (resulting from thermal expansion mismatches) in the metal matrix at the interface may exceed the yield stress of the matrix at a certain temperature, and plastic flow can occur. Permanent elongation or contraction of the sample length during thermal exposition was observed by several authors [10–17]. The samples exhibited residual strain (contraction or elongation) upon cooling to room temperature, if the temperature dependence of the thermal expansion coefficient was measured. The value of the observed permanent (residual) change of the sample length after measurement of thermal expansion depends on the matrix, volume fraction of reinforcement, and the maximum temperature of the thermal cycle. The thermal expansion behaviour may be characterised by the temperature dependence of the thermal strain parameter $\Delta d/\ell_0$ defined by the following equation [13, 18]:

$$\left(\frac{\Delta d}{\ell_0}\right) = \left(\frac{\Delta \ell}{\ell_0}\right)_\mathrm{exp} - \left(\frac{\Delta \ell}{\ell_0}\right)_\mathrm{th}, \tag{2}$$

where $(\Delta\ell/\ell_0)_\mathrm{exp}$ is the measured relative elongation of the composite, $(\Delta\ell/\ell_0)_\mathrm{th}$ is the relative elongation of the composite calculated by the rule of mixtures and l_0 is the original length of the sample. The rule of mixtures is a function of the volume fraction of the reinforcement and the relative elongation of matrix and reinforcement. The parameter $\Delta d/\ell_0$ represents the departure of the composite behaviour from the case where no thermal stresses are present. It should be noted that $(\Delta\ell/\ell_0)_\mathrm{exp}$ is determined by dilatometer in the axial direction of the sample. Experiments focusing on the influence of pre-strain on the thermal expansion behaviour of composites may enable to draw more general conclusions concerning thermal stresses developed in composites.

The aim of this paper is to study thermal expansion of AS21 magnesium alloy reinforced with short Saffil fibres and to reveal a possible influence of the sample pre-straining in tension as well as in compression on the thermal expansion behaviour. Microstructures of samples after pre-straining and after thermal expansion measurements will be examined by transmission electron microscopy (TEM).

2. Experimental procedure

2.1. Thermal measurements

The composite used in this study was produced by squeeze casting. Commercial AS21 alloy (composition in wt.%: 2Al-1Si-0.1Mn, balance Mg) was used as the matrix material. The alloy was reinforced with 25 vol.% of δ-Al_2O_3 short fibres (Saffil®) with a mean diameter of 3 μm and a mean length of about 87 μm (measured after squeeze casting). The volume concentration of the Saffil fibres was determined from the density measurements.

Cylindrical samples for the thermal expansion measurements had a length of 50 mm and a diameter of 6 mm. The linear thermal expansion of samples was measured in argon atmosphere using the Netzsch 402E dilatometer, over a temperature range from room temperature to 380 °C at heating and cooling rates of 5 K/min. The accuracy of the apparatus was controlled by measuring of the coefficient of thermal expansion (CTE) of pure Mg and by comparing it with the literature data. The agreement between measured and tabled data was in the range ± 1 %. The thermal expansion curves of composites were measured with three thermal (heating and cooling) cycles (runs). The planes of planar randomly distributed fibres were parallel to the longitudinal axis of the sample. Results obtained in the third thermal cycle were the same as those in the second cycle. Therefore, dilatational characteristics obtained in the first and second thermal cycles will be presented and discussed.

2.2. Deformation tests

The samples were deformed in an Instron testing machine at a constant crosshead speed giving an initial strain rate of 3×10^{-5} s^{-1}. The samples were deformed in tension or in compression parallel to their longest axis and strain of samples was about 0.5 % or 1 %, respectively. After straining, the linear thermal expansion was measured. Sequential stress relaxation (SR) tests were also performed at increasing stresses along a stress-strain curve during the compression tests. The duration of the SR tests was 300 s. The principle of sequential stress relaxation tests is following: The specimen is deformed to a certain stress σ_0 at which the machine is stopped. The stress is observed to decrease with time and is recorded. Subsequently, the specimen is reloaded to a higher stress (strain) and the test is repeated.

2.3. TEM observations

Thin foils for transmission electron microscopy (TEM) analysis were prepared by cutting slices with diameter of 3 mm and thickness of 0.8 mm. The final step of the specimen preparation was ion milling method with help of PIPS IV3 machine under small angles of incidence of ion beam and voltage between cathodes of 4–5 kV. The specimens were examined using a Philips CM 200 transmission electron microscope operating at 200 kV. The EDX system was used for the composition analysis.

3. Experimental results and discussion

Microstructure of the as-received samples is presented in Fig. 1. From literature [19, 20] it is known that the amount β-$Mg_{17}Al_{12}$ phase in the alloy AS21 is negligible. Silicon is practically insoluble in magnesium and it is bounded in the Mg_2Si particles. In Fig. 1, it can be seen the Mg_2Si phase in two morphologies: characteristic massive particles (a) and a Chinese-script type (b). The Mg_2Si forms a very stable compound. As to Mg_2Si, the microstructure of the composite matrix is very similar to that observed in unreinforced AS21 alloy [20, 21]. TEM of the as-received sample revealed increased dislocation density near the interfaces between the matrix and the Mg_2Si particles, and the Saffil fibres, respectively. TEM micrograph introduced in Fig. 2 documents dislocations that are present in the vicinity of Mg_2Si particle after thermal treatment in the dilatometer. Figure 3 shows the temperature dependence of the relative elongation for AS21 composite without any pre-deformation. Figure 4 shows the temperature dependence of the relative elongation for AS21 composite pre-strained in compression (up to 1 %) at room temperature. The first and second thermal cycles are shown. The third run had the same course as the second one. It can be seen from Fig. 4 that a large permanent elongation of the sample length, about 75 μm, occurs after the first thermal cycle while cooling. This corresponds to the relative elongation of 0.15 %. The temperature dependence of the relative elongation of the deformed composite in the second thermal cycle is the same as that of non-deformed composite in the second run, i.e. no permanent change in the sample length was found. It should be noted that AS21 composite was deformed after two thermal cycles [22].

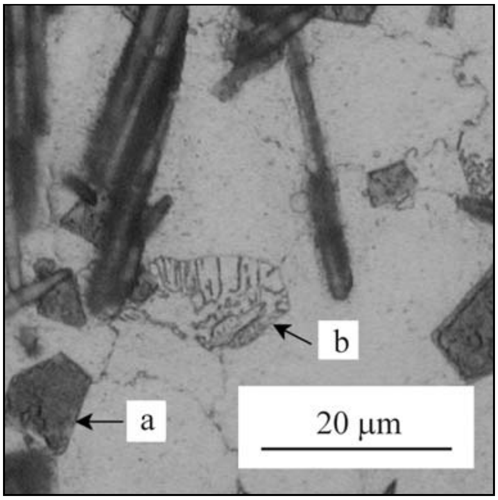

Fig. 1. Microstructure of the as-received composite: (a) massive Mg_2Si particle, (b) Chinese-script type precipitates.

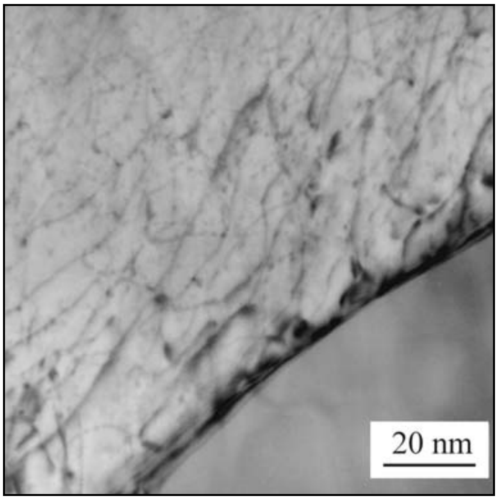

Fig. 2. Dislocations observed in the vicinity of Mg_2Si particle after two thermal cycles in dilatometer.

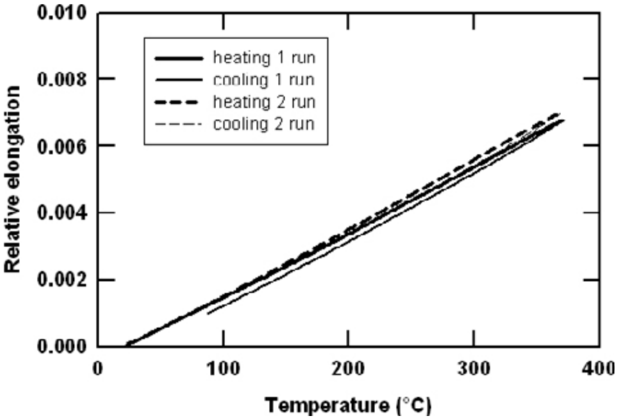

Fig. 3. Relative elongation versus temperature curves for as-received sample.

Figure 5 shows the temperature dependence of the relative elongation for AS21 composite, pre-strained in tension (up to 0.5 %) at room temperature. It can be seen that a significant reduction of the sample length (a permanent contraction), about 46 μm, occurs in the first cooling sub-circuit. Relative elongation versus temperature curve of the deformed sample in the first thermal cycle has the same character as that of the

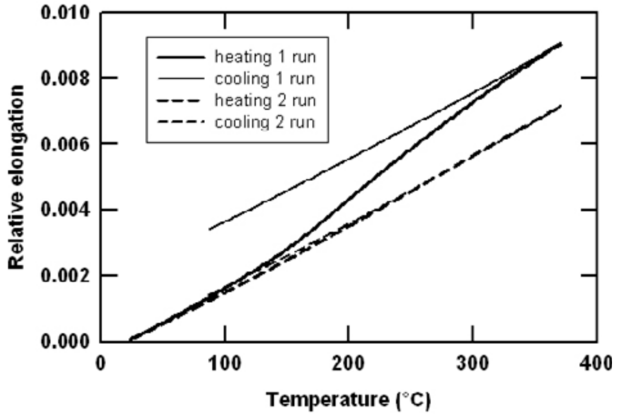

Fig. 4. Relative elongation versus temperature curves for sample pre-strained in compression.

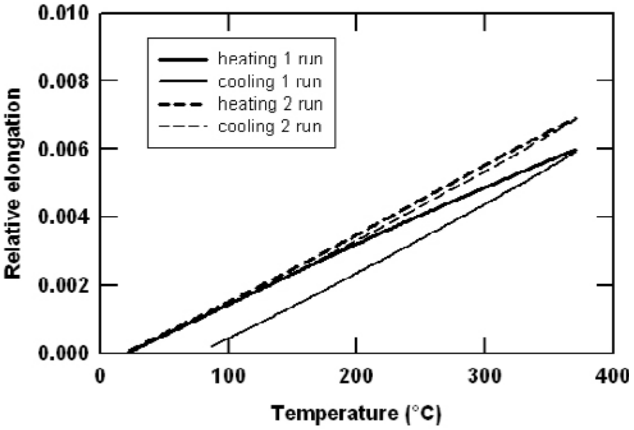

Fig. 5. Relative elongation versus temperature curves for composite pre-strained in tension.

as-prepared sample in the first cycle (see Fig. 3). The results obtained in the second cycle for the composite pre-deformed in tension are the same as those obtained in the second cycle for both as-prepared and compressive pre-strained samples. It should be mentioned that the heating and cooling curves of the composite had different slopes. Also, the plots of the relative elongation of composites against temperature were not linear in the whole temperature range. This behaviour indicates a reduction or an increase in the apparent CTE after reaching a certain temperature while heating or cooling. The permanent changes of the specimen length after the first thermal cycle correspond to the relaxation of thermal stresses.

The pre-deformation of a composite increases the internal stress in the matrix. The internal stress may be estimated using the stress relaxation tests according to the method introduced by Li [23]. The applied stress σ necessary for deformation of polycrystals is usually divided into two components

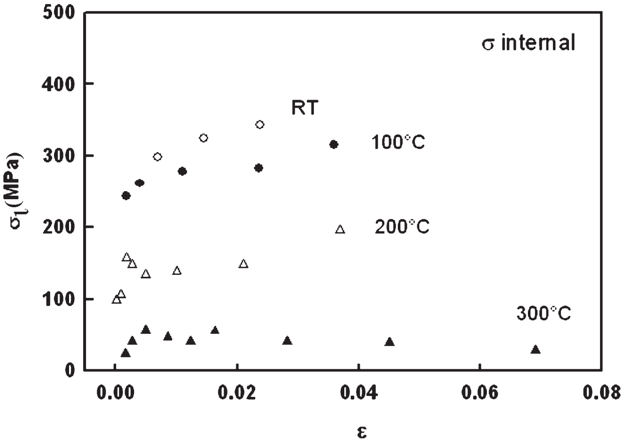

Fig. 6. Strain dependence of the internal stress measured at various temperatures.

$$\sigma = \sigma_{\mathrm{i}} + \sigma^*, \tag{3}$$

where σ_{i} is an athermal component often called internal stress and σ^* is the effective stress that acts on dislocations during their thermally activated motion and decreases with increasing temperature. Stress relaxation tests are very often analysed under the assumption that the stress relaxation rate $\mathrm{d}\sigma/\mathrm{d}t$ is proportional to the strain rate $\mathrm{d}\varepsilon/\mathrm{d}t$ [24], i.e.

$$M(\mathrm{d}\varepsilon/\mathrm{d}t) = -(\mathrm{d}\sigma/\mathrm{d}t), \tag{4}$$

where M is the combined modulus of the specimen – machine set. Starting from the empirical relationship proposed by Johnston and Gilman [25], the dislocation velocity v is proportional to the effective stress σ^*

$$v = \alpha\,(\sigma - \sigma_{\mathrm{i}})^m = (\sigma^*)^m\,, \tag{5}$$

where α and m are constants. In a stress relaxation test, the stress decreases with time, $\mathrm{d}\sigma/\mathrm{d}t$ is proportional to the strain rate and hence to the dislocation velocity v. Then the obtained differential equation

$$\mathrm{d}\sigma/\mathrm{d}t = a\alpha(\sigma - \sigma_{\mathrm{i}})^m \tag{6}$$

can be solved and the following relationship may be written:

$$\sigma - \sigma_{\mathrm{i}} = [a\alpha\,(m-1)]^{1/1-m}\,(t+t_0)^{1/1-m}\,, \tag{7}$$

where t_0 is a time constant and a is a constant determined by the dislocation density, geometry and modulus of elasticity. Equation (7) gives a possibility to obtain the internal stress σ_{i} from the SR experiments. The internal stress σ_{i} values for a sample deformed in compression estimated by fitting of Eq. (7) are introduced as a function of the applied stress in Fig. 6. The pre-straining at room temperature in compression gives an internal stress of ~ 220–230 MPa (at $\varepsilon \sim 0.01$). It should be mentioned that the internal stress of deformed samples depends on the testing temperature and strain.

The expansion curves (the temperature dependences of the relative elongation) for samples pre-strained in compression or tension (Figs. 4 and 5) show the permanent changes of the sample length – elongation or reduction – after the first thermal cycle. After the second and third thermal cycles, no changes in the sample length were found. The relative elongation of composites pre-strained in compression measured in the first thermal cycle is higher than that measured in the second thermal cycle, while the relative elongation values of samples pre-strained in tension measured in the first thermal cycle were lower than those measured in the second thermal cycle. The values of the relative elongation measured in the second thermal cycle are the same for composites pre-strained in both compression and tension as well as for as-prepared sample (without any pre-straining). The effect of pre--deformation on the thermal expansion behaviour may be analysed using Eq. (2) for the temperature dependences of the relative elongation in the first and second thermal cycles. Therefore, it is determined $(\Delta d/\ell_0)_1$ and $(\Delta d/\ell_0)_2$ for the first and second thermal cycle, respectively. The difference of these values, $(\Delta d/\ell_0) = (\Delta d/\ell_0)_1 - (\Delta d/\ell_0)_2$, gives the temperature variation of the residual strain. The second term on the right hand in Eq. (2) corresponds to the relative elongation due to the rule of mixtures is the same for both thermal cycles. The temperature variation of $(\Delta d/\ell_0)$ shown in Fig. 7 characterises the residual strain evolution with temperature. Since the MMC studied was fabricated by squeeze casting at an elevated temperature, the composite contains residual thermal stresses and increased dislocation density due to a large difference in the thermal expansion coefficients between the matrix and the reinforcement [8, 9, 26, 27]. When the MMC is heated, the internal tensile thermal stress acting in the matrix reduces at a certain temperature to zero and, during further heating, compressive stresses are built up. With increasing temperature, if the composite is heated, the compressive thermal stress in the matrix at the interface may exceed the yield stress of the matrix at a certain temperature and plastic deformation occurs in the matrix near the interfaces. This plastic deformation in the matrix relieves the thermal stresses, and the composite contracts at a constant strain rate. This process continues until the sample is cooled to a certain temperature. Cooling below this temperature does not relieve the thermal stresses induced because the yield stress of the matrix is higher than the thermal stresses. These stresses are subsequently frozen in the matrix. In other words, plastic yielding at higher temperatures means an enhanced relaxation of internal thermoelastic field. On the other hand, during cooling, the internal stresses behave in the opposite sense. This plastic deformation may realise as dislocation glide, as twinning or possibly as grain boundary sliding at higher temperatures depending upon the maximum temperature of the thermal cycle and the microstructure of the matrix. In general, it is reasonable to consider that the compressive deformation that appears on heating will give some form of diffusion-controlled high temperature creep. Analogous considerations may be used for explanation of the thermal expansion behaviour of the composite pre-strained in tension. While temperature is increased in the dilatometer, dislocation recovery processes may cause a rapid decrease in the internal stress in the matrix. Then the total stress (a sum of the thermal and dislocation stress) decreases more rapidly than in the case of the undeformed sample and the

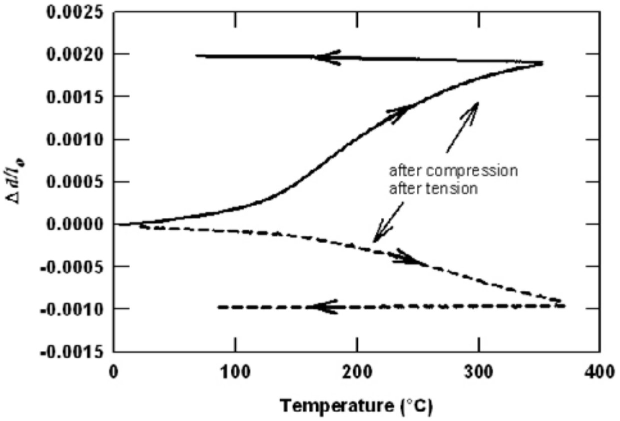

Fig. 7. Temperature dependence of the residual strain $(\Delta d/l_0)$ estimated for both modes of pre-deformation.

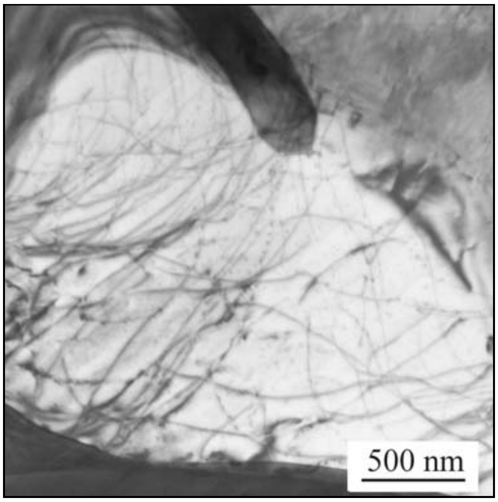

Fig. 8. Dislocations observed in the vicinity of Mg_2Si particle in sample pre-strained in tension.

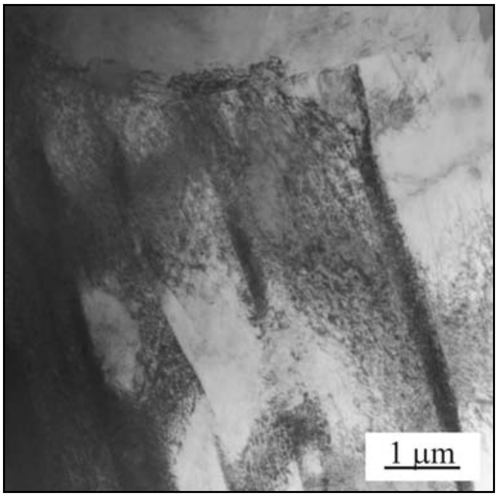

Fig. 9. Twins in the sample pre-strained in compression.

yield stress for compression is achieved at lower temperature. This temperature obtained from the expansion curves is very close to that, at which the yield stress of AS21 alloy and AS21–Saffil composite deformed at constant strain rate decreases rapidly with temperature [28, 29]. It is important to mention that the deformation mechanisms of magnesium alloys deformed at a constant strain rate in tension are different from those of Mg alloys deformed in compression [30, 31]. While the tensile deformation is realised mainly by dislocation motion (see Fig. 8), the compression deformation is realised at low strains in the vicinity of the yield stress almost by the twinning mechanism. TEM analysis showed a high density of twins in the AS21–25 vol.% Saffil after deformation in the compression test (Fig. 9). In this connection it is interesting that reversible movement of twin boundaries may cause a pseudo-elastic behaviour, as observed by Cáceres et al. [32].

4. Conclusions

The thermal expansion behaviour of AS-25 vol.% Saffil studied at temperatures between room temperature and 380 ℃ is influenced by pre-straining of the samples. A strong non-linearity of the temperature dependence of the composite relative elongation was determined in each thermal cycle. A persistent change of the sample length was found after the first thermal cycle. Negligible change in the sample length was observed after the second and third cycles. A residual reduction or elongation of the sample length is found for composites pre-deformed in compression or in tension. The permanent strain and the thermal expansion curves depend on the previous thermal and deformation history of samples. Observed effects can be related to the matrix plastic deformation under mechanical and thermal stresses. Different deformation processes in the matrix acting during tension as well as compression straining may be considered.

Acknowledgements

This work was supported by the Grant Agency of the Czech Republic under Grant 106/06/1354. O. Padalka thanks for supports by the Research Project 1M 2560471601 “Eco-centre for Applied Research of Non-ferrous Metals” that is financed by the Ministry of Education, Youth and Sports of the Czech Republic.

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