# Dilatation study of the recovery processes in AX41-12 vol.% Saffil fibre composites

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**Abstract**

The dilatation characteristics of the pre-deformed AX41 magnesium alloy and AX41-12 vol.% Saffil fibre composite were determined in the temperature range of 20–380 °C. Two orientations of the fibres in the composites were investigated: with the planes of planar randomly fibres parallel and perpendicular to the longest axis of sample. During the first thermal cycle after compression deformation, the recovery of the alloy and composite occurred in two steps. We observed the recovery of twins at low temperatures, and the recovery of the dislocation slip at higher temperatures. The released plastic deformation was the largest in composites with the fibres perpendicular to the sample axis, and was the lowest for the alloy.

Key words: MMC, fibres, recovery, plastic deformation

## 1. Introduction

Magnesium alloys (the lightest structural metallic materials) have found many applications in industry in recent years because of their specific strength and high damping capacity. Industrial applications are often restricted due to the loss of mechanical strength at higher temperatures, poor creep resistance, and low corrosion resistance. These disadvantages may be overcome by the change of chemical composition, refinement of grain size, and by adding reinforcement. Recently, a series of Mg-Al-Ca alloys was developed for elevated temperature applications [1]. The alloys, in some cases containing small (microalloying) additions of strontium, exhibit an improvement of high temperature mechanical properties and excellent creep resistance [2]. Further improvement in the mechanical properties of the alloys may be caused also by reinforcement.

In magnesium crystals, strain along the c-axis can only be accommodated by $\langle c + a \rangle$ slip or twinning. The most detailed evidence of the deformation mechanism in magnesium has been obtained in single-crystals studies (e.g. [3]) and has been effectively extrapolated to explain the behaviour of polycrystalline materials. Twinning is an important deformation mode of the plastic deformation of magnesium and magnesium alloys [4–7]. The dependence of twinning of Mg alloys on the texture development was studied in the work of Jiang et al. [4]. In this work, it is shown that the effect of twinning on the flow stress is more significant than the effects of grain size. Mechanical twinning influences the deformation behaviour in Mg and Mg-alloys, as has been demonstrated in many studies [8, 9].

The effect of twinning on the bulk deformation behaviour was studied by various methods [10–12], but a dilatation study has not yet been used. The dilatation characteristics give information not only about thermal vibrations in the lattice, but also about all deformations occurring in the material during thermal loading. A release of plastic deformation is a thermally activated process connected with the macroscopic length changes of the sample. Therefore, dilatometry is a non-destructive method that can provide important information about the evolution of twinning and

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slip strain during thermal loading. The aim of this work is to investigate the dilatation characteristics of the pre-deformed AX41 alloy and composites where this alloy was reinforced with 12 vol.% of Saffil fibres. The dilatation characteristics are measured and analysed in the temperature range from room temperature up to 380 °C.

## 2. Experimental details

The materials in the present study were magnesium composites based on AX41 (Mg-4Al-1Ca) alloy that was reinforced with 12 vol.% Saffil fibres. The composites were supplied by the Centre of Advanced Materials, Clausthal, and were prepared by squeeze casting. The molten alloy (700 °C) was inserted into a preheated die (350 °C) with Saffil preform. The two-stage application of pressure was used (40 MPa for 15 s followed by 70 MPa for 90 s). The ingot dimensions were 100 × 100 × 30 mm.

The short fibres in the composite kept their original arrangement (i.e., its preform arrangement); they were planar randomly arranged, i.e., axes of fibres lay randomly in parallel planes. The length of the fibres was on the order of tens of μm, and their diameter (thickness) was approximately 3 μm. Particles of secondary phases, namely $CaAl_2$, were randomly distributed in the structure. From the composite part of the ingot, two kinds of parallelepipeds were cut: a) samples where the angle between the longest sample axis and the planes of randomly distributed fibres was 0° and b) when the angle was 90°. Cylindrical specimens of 6 mm in diameter and 25 mm or 20 mm long were prepared for dilatation measurements. The composite samples with angle $\alpha = 0°$ and 90° are further called C-0 and C-90.

The linear thermal expansion of the composite specimens was measured in a helium atmosphere using a Netzsch 402 C/4/G dilatometer from room temperature to 380 °C for heating and cooling rates of $2\,°C\,min^{-1}$. The thermal expansion curves for composites were measured during three consequent heating and cooling cycles on the pre-deformed samples. It is important to note that samples before deformation were subjected to three thermal cycles (at $2\,°C\,min^{-1}$) in order to remove thermal strains generated during fabrication of the composite. The first thermal cycle was measured on the samples compression pre-deformed in INSTRON testing machine at room temperature and at a constant speed of crosshead giving an initial strain rate $\dot{\varepsilon} = 10^{-4}\,s^{-1}$ up to 1.5 %. Compression deformation was always made in direction of the longitudinal sample axis. The results obtained on the samples before deformation are also presented, and are marked as 0 run.

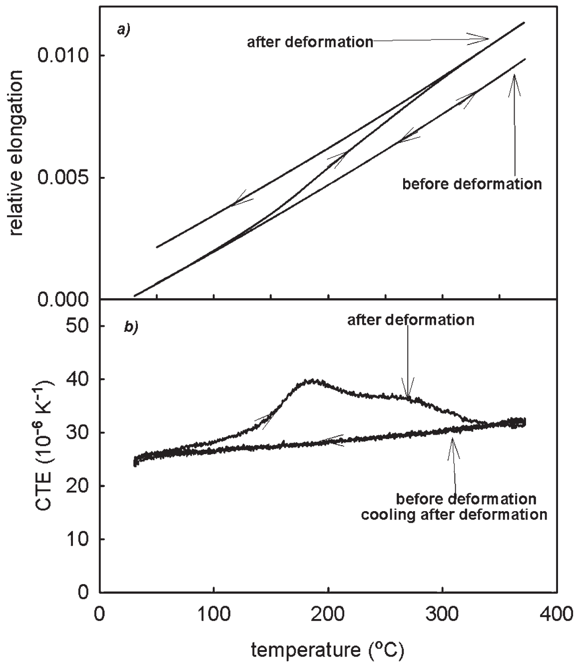


Fig. 1. Temperature dependence of a) the relative elongation and b) the CTE for AX41 alloy.

## 3. Results

Figure 1 shows the temperature dependence of the relative elongation and the coefficient of thermal expansion (CTE) for AX41 alloy in a pre-deformed and non-deformed state. The temperature dependence of the CTE shows two temperature regions with two maxima. After the first thermal cycle, a permanent elongation of the sample was found. The relative elongation and the CTE in the second and third thermal cycle in the pre-deformed alloy were the same as the dilatation characteristics of the non-deformed alloy. Figure 2 displays three figures of the microstructure of the alloy: before deformation (a), after deformation (b), and after thermal cycle (c) with a maximum temperature of 200 °C.

Dilatometry measures all deformation changes occurring in composites during thermal cycling. These deformation changes are additive, so the measured relative elongation can be divided into several contributions. Measured relative elongation, $(\Delta l/l_o)_{meas}$ can be expressed as

$$\left(\frac{\Delta l}{l_o}\right)_{meas} = \frac{\Delta l}{l_o} + \sum_i \left(\frac{\Delta l}{l_o}\right)_i, \qquad (1)$$

where $\frac{\Delta l}{l_o}$ is the relative elongation caused by thermal vibrations, and the second part of Eq. (1) is a sum of all deformations occurring during the heating load.

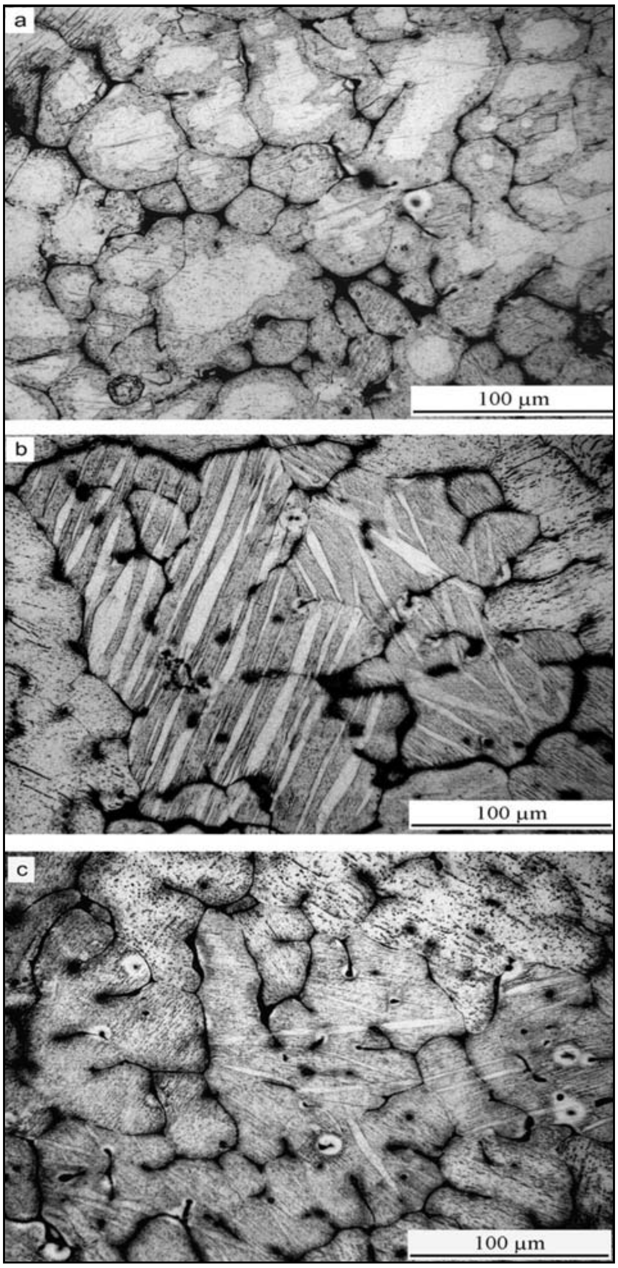


Fig. 2. Microstructure of AX41 alloy a) before deformation, b) after deformation and c) after heating up to 200 ℃.

Convenient processing of the experimental data can provide information about the processes occurring in the material. Relation (1) was first used for obtaining information about the first recovery process in the alloy after pre-deformation. The pre-deformed alloy was heated up to 200 ℃ and the relative elongation was measured. The difference between the values of the relative elongation obtained in the thermal cycle before deformation and the obtained values of the relative elongation of pre-deformed samples, $\varepsilon_{\mathrm{recover}}$, was estimated at each temperature. Thus the values of $\mathrm{d}\varepsilon_{\mathrm{recover}}/\mathrm{d}T$ were calculated. The logarithm of these values was plotted against $1/T$ (see Fig. 3).

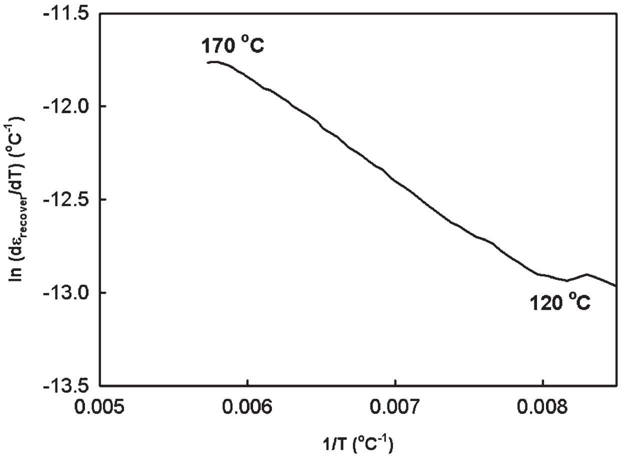


Fig. 3. Dependence of $\ln(\mathrm{d}\varepsilon_{\mathrm{recover}}/\mathrm{d}T)$ on $1/T$ ($\varepsilon_{\mathrm{recover}}$ is a deformation released during heating).

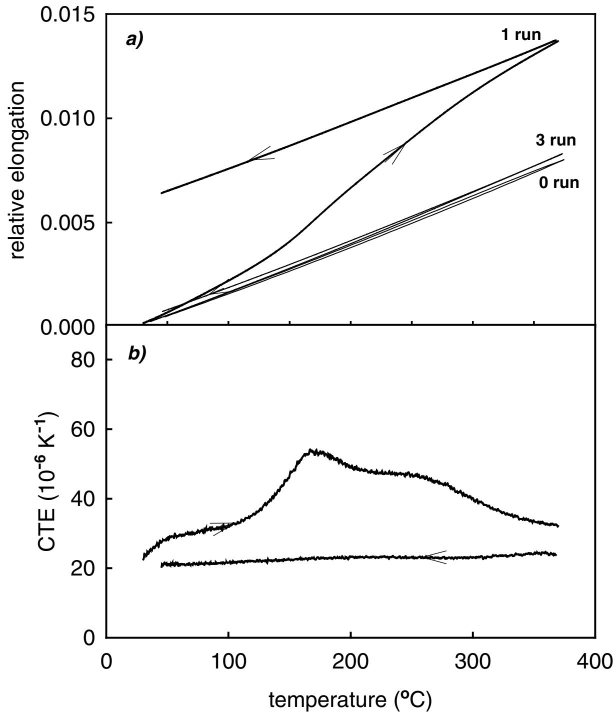


Fig. 4. Temperature dependence of a) the relative elongation and b) the CTE for C-0 composite.

The temperature dependence of the relative elongation and the CTE for pre-deformed C-0 composite is revealed in Fig. 4. The character of both temperature dependences, the relative elongation and the CTE, is similar as that for the pure alloy. Two temperature regions are perceptible in the temperature dependence of the CTE. While the dilatation characteristics in the

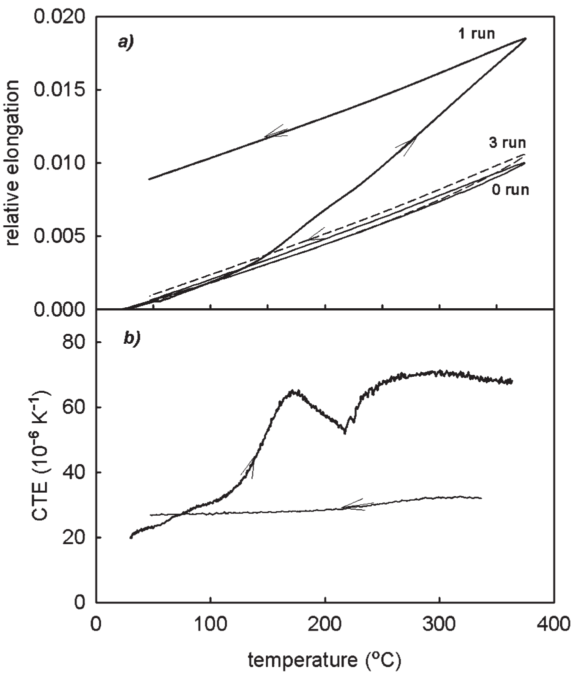


Fig. 5. Temperature dependence of a) the relative elongation and b) the CTE for C-90 composite.

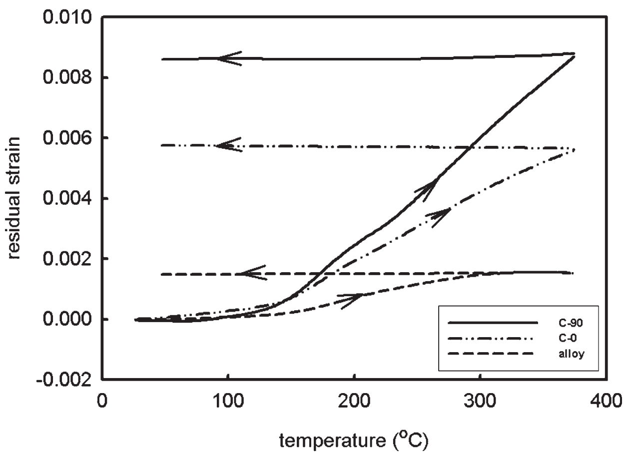


Fig. 6. Temperature dependence of the residual strain of AX41 alloy and C-0 and C-90 composites.

second and third thermal cycles of the pre-deformed alloy were the same as for the non-deformed alloy, the dilatation characteristics of the non-deformed C-0 composite and pre-deformed composite in the second and the third thermal cycles differed. A small part of the permanent elongation was found even after the third thermal cycle. Similar dilatation characteristics were obtained also for the C-90 composite. The measured results are shown in Fig. 5.

Residual strain is the strain that can be removed during thermal cycle. It can be obtained as the dif-

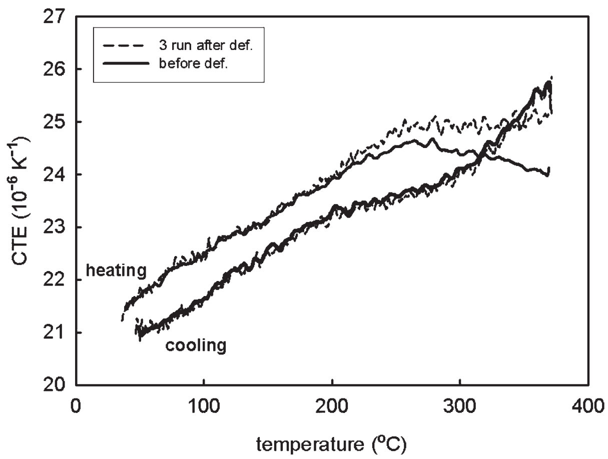


Fig. 7. Temperature dependence of the CTE in the third thermal cycle after deformation and in the thermal cycle before deformation for C-0 composite.

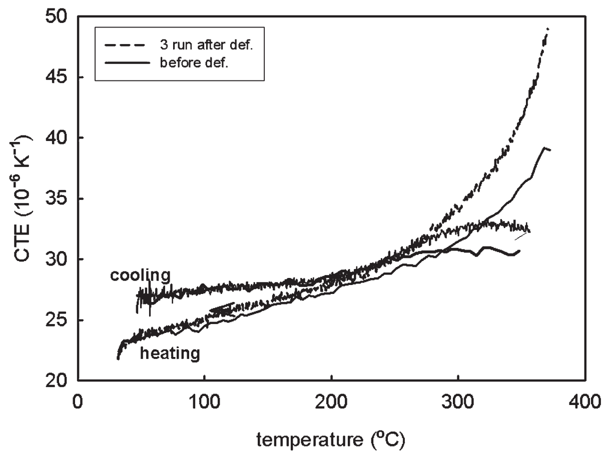


Fig. 8. Temperature dependence of the CTE in the third thermal cycle after deformation and in the thermal cycle before deformation for C-90 composite.

ference between the relative elongations of two runs. The temperature dependences of the residual strain, obtained as the difference of the relative elongations between the first and second thermal cycle, for both kinds of composite and the alloy are shown in Fig. 6. They give information about deformation release during the thermal cycle. It can be seen that no release of plastic deformation occurs up to 120 °C during heating and during cooling. Table 1 then shows the evaluation of the release of plastic deformation after three thermal cycles.

Figures 7 and 8 show the temperature dependence of the CTE before deformation and after deformation in the third cycle for C-0 and C-90 composites, respectively. The influence of the plastic deformation on the CTE in the third thermal cycle after pre-deformation is perceptible only at higher temperatures.

Table 1. The removed part (%) of the deformation introduced by pre-straining in compression

| | Removed part (%) in | | |
|---|---|---|---|
| | 1 run | 2 run | 3 run |
| Alloy | 12 | – | – |
| C-0 | 37 | 5 | 5 |
| C-90 | 58 | 5 | 5 |

## 4. Discussion

Figure 1 shows that the CTE of the alloy compression pre-deformed is higher than the non-deformed alloy. The deformation energy is released during heating, and the sample length increases. Release of the deformation energy occurs in two steps, in two temperature ranges that overlap. The boundary between two ranges is at about 230 ℃. There are two deformation mechanisms that we can assume to exist in the pre-deformed alloy at room temperature. As was mentioned in the introduction, the main deformation modes in magnesium and magnesium alloys are twinning and basal slip. To define the first low temperature process, the microstructure of a non-deformed, pre-deformed and heated sample was investigated (but only up to 200 ℃). The results are shown in Fig. 2. It can be seen that twins are nearly absent in the sample before deformation. After compression deformation, large amounts of twins were found in the sample (Fig. 2b). These twins were nearly removed by a heating cycle with a maximum temperature of 200 ℃. The removal of twins is a thermal activation process; it takes place only during heating. The rate of removal is exponentially dependent on $1/T$ (Fig. 3).

Twinning results in new orientations of the lattice with respect to the deformation axis, creating the possibility of activating additional slip systems. Backx et al. [13] investigated the influence of annealing on twins in magnesium alloy AZ31. The samples were annealed for various time periods at 350 ℃. In the un-annealed sample, almost all grains were twinned; in the sample that was annealed for 60 minutes, no twins were found in the optical microscope. They showed that the extent and character of the twinning depends on the history of the material as well as on the amount of deformation and annealing time.

Analysis of the deformation mechanisms and the observed microstructures [7] shows that compressive deformation starts by extensive twinning at low strains. The slip and slip-twinning interaction occur when the twinning capacity is exhausted due to the formation of basal texture. During recovery of the compressive plastic deformation in the second temperature range, recovery of the deformation-induced dislocations occurs after removal of twins. This second process takes place at temperatures above 230 ℃. This recovery process is also thermally activated, but the character of the increase of the recovery rate cannot be estimated due to the overlap of the temperature range of both processes. After the removing of the twins in the first thermal cycle, the alloy stays macroscopically deformed because only part of the macroscopic deformation was removed (Table 1). The deformed alloy has the same dilatation characteristics as the non--deformed alloy because the temperature dependences of the expansion characteristics are the same as in the thermal cycle before deformation. It is assumed that no dynamic recrystallization occurs during compression pre-deformation of the alloy up to 1.5 %.

A similar character of the temperature dependences of the dilatation characteristics as in the alloy was found for both composites C-0 and C-90 (Figs. 4 and 5). Two types of plastic strain are released during heating in the first thermal cycle. However, this effect is significantly stronger in the composites (as can be seen in Fig. 6), where the temperature dependence of the residual strain of all studied materials is compared. The highest part of the compressive plastic strain can be removed from the composite C-90, the lowest from the pure alloy (Table 1). To reach a deformation 1.5 % in the studied samples, the lowest stress was used for the alloy, and the highest for C-90 composite.

In order to understand the difference between C-90 on one side and C-0 and alloy on the other side, we can imagine the situation where the strained fibres in the C-90 pre-deformed composite caused a compressive stress state (this case is qualitatively different from the case of alloy and C-0 composite). First, the stress from the fibres can confine the movement of twin boundaries that were observed to be partially reversible in magnesium alloys [14], thus leading to a higher volume of twins in the pre-deformed sample in this case. This would explain the substantially higher maximum of the first peak on the CTE curve that is connected with the twins. Second, the strains in fibres are finally relieved when the temperature is increased enough for the dislocation mechanisms to come into operation, leading to the second peak on the CTE curve. These assumptions regarding the effect of strained fibres are confirmed by the fact that the CTE curves for C-0 composite and the alloy are similar, which differs from the C-90 composite by a different course of the release of the dislocation slip. It should be mentioned that at higher temperatures (above about 230 ℃) the activity of non-basal, pyramidal slip systems occurs [15, 16].

In our previous work [17], we have studied the dilatation characteristics of AX41 Saffil fibre composites in various fibre directions relative to the longitudinal sample axis (the dilatation characteristics were measured in this direction). We have found that the composite with fibres in the direction of the longest sample axis is in tension state (C-0). If fibres were perpendicular to the sample axis then the composites were in compression state in the sample axis direction (C-90). This anisotropy can be seen also from Figs. 7 and 8 where the temperature dependences of the CTE in the third cycle before and after deformation are revealed. The CTE values are higher than those before deformation above 200 ℃. This means that any recovery (departure in the CTE of non-deformed and pre-deformed samples that is perceptible at high temperatures) takes place even in later thermal cycles (in contrast to the alloy where all recovery process took place in the first thermal cycle).

The anisotropy of mechanical properties of a composite may also influence thermophysical properties of the composite as shown for copper matrix composites reinforced with carbon fibres [18]. The thermal expansion behaviour of composites and the variation of the CTE with temperature may be affected by precipitation [19]. However, it should be stressed that the deformation temperature influences the microstructure of the composites and hence their properties [16, 20, 21].

## 5. Conclusions

Analysis of the compression pre-deformed AX41 magnesium alloy and composites AX41 12 vol.% Saffil fibres up to 1.5 % shows that heating of these materials leads to recovery process in two steps. In the first step, recovery of the twins was observed in the temperature range up to 230 ℃. The highest rate of this process occurred at 180 ℃ for the pure alloy and 170 ℃ for both composites. Recovery of the dislocation slip takes place with a maximum rate at about 230 ℃. The temperature ranges of both processes overlap. The deformation released during both recovery processes was larger for composites than for the alloy. The thermal strain that strongly influences the dilatation characteristics of the non-deformed composites practically does not influence any recovery processes.

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