Journal of Alloys and Compounds, 211/212 (1994) 169–172
JALCOM 130

Low-temperature thermal cycling of metal-matrix composites studied by the ultrasonic low-frequency stress-coupling technique

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Abstract

The ultrasonic low-frequency stress-coupling technique has been used to study dislocation processes induced by the difference in coefficient of thermal expansion of matrix and particle in an Al/12vol.% SiC composite. The attenuation *vs.* bias stress response was recorded at equally spaced temperatures ranging from 280 K to 80 K, successively on cooling and on heating the specimen. The amplitude of the attenuation change varied strongly with temperature, exhibiting a marked maximum around 240 K. Moreover, whatever the temperature, this amplitude was larger on heating than on cooling. These phenomena are explained in terms of dislocation-point defect breakaway and dislocation movements during the low-temperature thermal cycle.

1. Introduction

In recent years the presence of a high dislocation density around fibres, whiskers or particles in metal-matrix composites (MMCs) has been demonstrated in various ways. Experimentally, this has been revealed in particular by transmission electron microscopy observations [1, 2].

Theoretically, in composites, the generation of dislocations around reinforcements is predicted to occur when they are subjected to temperature changes during thermal cycles. Indeed, during such cycles, internal stresses that result from the difference in thermal coefficient of expansion (ΔCTE) between the matrix and the reinforcement are, at least partially, relaxed by such dislocation processes [3–5].

Since ultrasonic attenuation is very sensitive to mobile dislocations in aluminium [6, 7], such a technique should be able to follow the punching-out of dislocations during thermal cycling of MMCs. This paper presents and discusses the first results that we have obtained during a low-temperature thermal cycle by using the ultrasonic low-frequency stress-coupling technique.

2. Experimental conditions

The experiments were carried out on an Al/12vol.% SiC composite produced by the powder metallurgy route (hot isostatic pressing). The powder aluminium was 99.7% pure. The mean size of the SiC particles was of the order of 10 μm. Samples were cylindrical, with 9 mm diameter for the useful part, whose length was 40 mm. They were terminated at both ends by a screwed head, 12 mm in diameter, for fastening the sample. After machining, the specimens were carefully annealed for 48 h at 473 K and then furnace-cooled. The mean grain size thus obtained for the matrix was about 20 μm.

In order to clarify the role of the SiC particles in the composite, some experiments were also carried out on unreinforced Al specimens prepared according to the above procedure.

The ultrasonic measurements were made by using the coupling technique [6, 7], which consists of submitting the sample simultaneously to a slowly varying bias stress, σ, and a high-frequency stress whose attenuation α is followed during the variation of σ. The measuring device has been described elsewhere [8]. The ultrasonic pulsed compressional wave, whose frequency was about 15 MHz, was directed parallel to the mechanical tension axis. Its emission and reception were ensured by two polyvinylidene fluoride transducers. The state of the sample before each test was taken as a reference state for computing the attenuation change $\Delta\alpha$.

After 30 min of rest in isothermal conditions, $\Delta\alpha$ was measured during the slow, linear vs time, tension loading and unloading of the sample. The measurements were repeated at equally spaced temperatures ranging from 280 K to 80 K, successively on cooling and on heating the specimen. The maximal value σ_0 of the bias stress was chosen such that no microyielding was observed, with a microplastic proof strain of 10^{-6},

SSDI 0925-8388(94)00130-5

during a loading–unloading cycle performed at 320 K prior to the low-temperature thermal cycling.

3. Results

For the composite, in the temperature range 280–200 K, the shape of the $\Delta\alpha$–σ curves looks like a Γ at increasing bias stress. Such curves are reported in Fig. 1 for $T=240$ K. Note the Γ shape is similar to previous observations for predeformed unreinforced Al, which were interpreted in terms of dislocation-point defect breakaway [9]. However, two important features of the $\Delta\alpha$–σ response have to be noticed for the Al/SiC composite:

(1) $\Delta\alpha$ is much more important, at a given temperature, for an experiment performed during the heating stage (curve b, Fig. 1) than for one performed during the cooling stage (curve a, Fig. 1).

(2) The loading–unloading evolution of $\Delta\alpha$ appears to be strongly hysteretic. In fact, this phenomenon seems to be linked with the appearance of some microplasticity that occurs in the specimen, in spite of the criterion used at 320 K to choose the value of σ_0.

In the low-temperature range (160 K–80 K), the attenuation change becomes very small, as in unreinforced Al [9]. This behaviour can be seen in Fig. 2, in which $\Delta\alpha$ induced by a given applied stress ($\sigma_0=6$ MPa) has been plotted *vs.* T for the cooling and heating stages, for both the composite and the unreinforced Al.

For the composite, in addition to the marked difference between the cooling and heating stages, Fig. 2 (curve a) shows that $\Delta\alpha(\sigma_0)$ exhibits a maximum centred around 240 K for both the cooling and heating stages. Furthermore, the above-mentioned microplasticity phenomenon has been characterized by the mi-

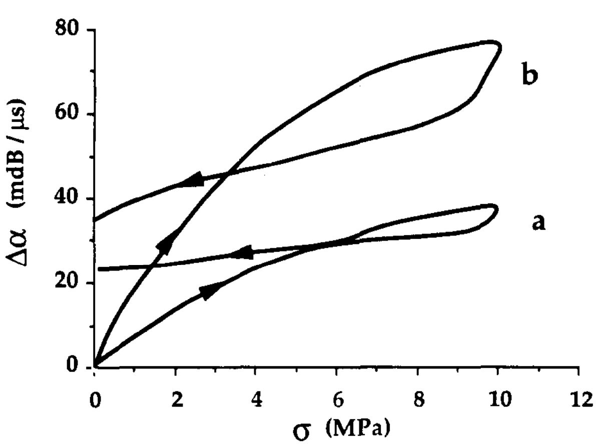

Fig. 1. Attenuation variation $\Delta\alpha$ *vs.* bias stress σ in isothermal conditions at 240 K for the Al/12vol.% SiC composite: (a) during the cooling stage; (b) during the heating stage.

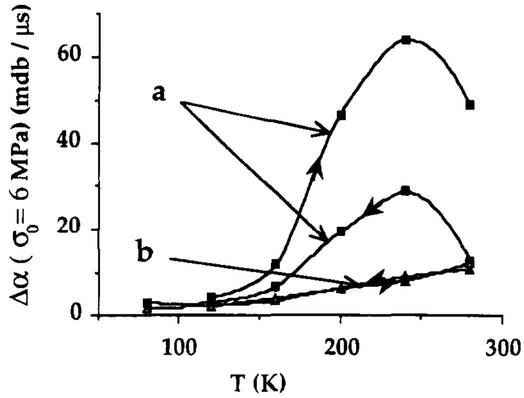

Fig. 2. Change in attenuation $\Delta\alpha(\sigma_0)$ *vs.* temperature T: (a) Al/12vol.% SiC composite; (b) unreinforced Al.

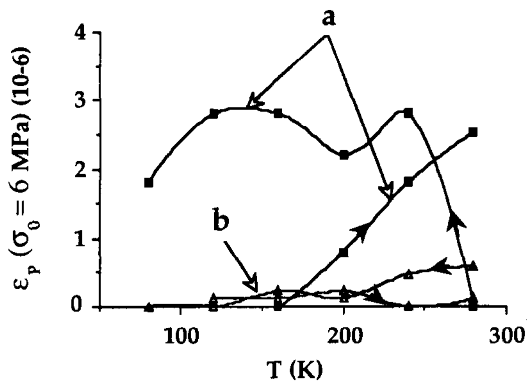

Fig. 3. Microplastic strain at $\sigma_0=6$ MPa *vs.* T: (a) Al/12vol.% SiC composite; (b) reinforced Al.

croplastic strain ϵ_p detected at $\sigma_0=6$ MPa. The evolutions of $\epsilon_p(\sigma_0)$ *vs.* T are reported in Fig. 3. For the composite (curve a), it can be noted that, as for $\Delta\alpha(\sigma_0)$, there is a strong difference between the cooling and heating stages. Moreover, there is no microyielding at all during the beginning of the heating stage (range 80–160 K).

In the case of the unreinforced material (see curves b in Figs. 2 and 3), it appears that:

(1) the maximum in $\Delta\alpha$ *vs.* T is not present;

(2) $\Delta\alpha(\sigma_0)$ is less important than for the composite;

(3) there is no difference between the cooling and heating stages;

(4) there is no microplasticity at all.

Finally, in order to help towards explaining the appearance of a maximum of $\Delta\alpha$ ($\sigma_0=6$ MPa) vs. T for the composite, the influence of the ageing time and temperature, which are known to play an important role in the segregation of point defects about dislocations, have been studied in the following way. The unreinforced Al has been plastically deformed by 0.15% at 240 K, and then $\Delta\alpha(\sigma_0)$ has been determined at this temperature after various ageing times at 240 K and at 280 K. The results, presented in Table 1, show that

TABLE 1. Change in attenuation, $\Delta\alpha(\sigma_0)$, when the bias stress is applied at 240 K, for various ageing times and temperatures following a plastic strain of 0.15% at 240 K. Unreinforced Al

$\Delta\alpha(\sigma_0)$ (mdB μs^{-1})	71	95	127	66	50	43
Ageing conditions	3 min at 240 K	10 min at 240 K	30 min at 240 K	30 min at 280 K	2 h at 280 K	12 h at 280 K

at first the attenuation change increases with increasing ageing time at 240 K, then it goes through a maximal value, and finally it decreases with increasing ageing time at 280 K.

4. Discussion

The role of the ΔCTE between the matrix and the reinforcement in the mechanical properties of MMCs has already been pointed out by many authors [1–5, 10–12]. As the presented results show that a specific attenuation behaviour during low-temperature thermal cycling is linked with the presence of particles, we first present briefly the internal stresses and dislocation phenomena expected from the ΔCTE. Secondly, we recall how the attenuation changes caused by the application of this bias stress are linked with the dislocation breakaway process. Then, on this basis, we propose an interpretation of the observed phenomena.

4.1. Internal stresses and dislocation punching out from particles

Various approaches have been proposed for modelling the plastic relaxation of thermal stresses around particles, which are induced by the ΔCTE on cooling reinforced metals. Among these, Hamann [3] have developed a model for dislocation punching-out from a single spherical particle on the following basis. When the local stress reaches a critical level, dislocation interstitial loops are emitted from the particle–matrix interface and glide away from the interface as long as the driving shear stress remains greater than the critical shear stress τ_c to move dislocations in the matrix. Note that, in this model, it is assumed that dislocations move in their glide plane and no annihilation of dislocations occurs.

In the case of our Al/SiC system, the numerical simulations made with this model, indicate the above-mentioned processes are expected to occur as soon as the sample has been cooled by about 20 K from the stress-free state. So thermally induced emission and movement of dislocations should be involved in the observed attenuation phenomena. Furthermore, the microplasticity detected on the stress–strain response of the Al composite during the cooling stage (Fig. 3, curve a) is presumably linked also with the thermal internal stress field. Indeed, according to the above model, at a given temperature, dislocations around particles are in positions such that the internal driving shear stress equals τ_c. Therefore, when mechanical stress is applied, in all regions where the applied and internal stresses act in the same sense, τ_c is exceeded, thus leading to the observed microplasticity. In contrast, in the first stage of the heating procedure (80–160 K), the thermal stress field is quasi-elastically reduced, so that local stresses remain lower than τ_c.

4.2. Dislocation breakaway

The dislocation-point defect breakaway process has been widely studied during the past 20 years, especially by means of ultrasonic waves, because their attenuation is very sensitive to mobile dislocations:

$$\alpha = \beta\Lambda/K^2 \tag{1}$$

where β is a damping factor, Λ is the density of mobile dislocations, and K is a restoring force which varies as $1/L^2$ (L is the freely vibrating loop length) in the case of the string model.

By using the coupling technique, on increasing σ, the depinning of dislocations from their Cottrell cloud occurs, thus leading to an increase of the free loop length L. This results in an increase of α (Fig. 1), which can be written simply in the case of the string model:

$$\Delta\alpha = \beta\Lambda(L^4(\sigma) - L_0{}^4) \tag{2}$$

where L_0 and $L(\sigma)$ are the effective free loop lengths before and after the application of σ, respectively.

The influence of ageing time and temperature on $\Delta\alpha(\sigma_0)$ (Table 1) can be readily understood from the combined evolutions of $L(\sigma_0)$ and L_0. At 240 K the migration of point defects to dislocations freshly created by plastic deformation is slow. Therefore the Cottrell atmosphere that is formed at this temperature remains rather dilute. Thus the breakaway is easy, but the corresponding attenuation change is small. This situation is schematized in Fig. 4(a), (a′). With increasing ageing time, L_0 decreases, but in the initial stage the breakaway process is still complete under the application of σ_0. Therefore, under these conditions the difference $(L^4(\sigma_0) - L^4{}_0)$ is expected to increase with increasing ageing time (Fig. 4(b), (b′)). At 280 K, the diffusion of point defects towards dislocations is enhanced. Consequently, dislocations are more and more strongly anchored by their Cottrell cloud, so that $L(\sigma_0)$ is no longer equal to the network loop length. Hence $\Delta\alpha(\sigma_0)$ decreases with increasing ageing time at this temperature (Fig. 4(c), (c′)).

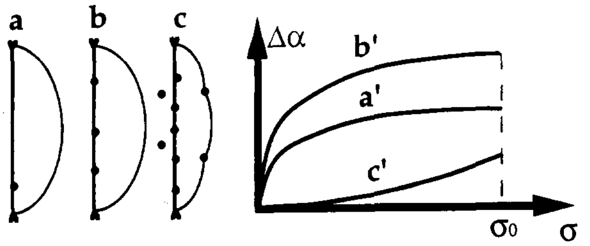

Fig. 4. Schematic of the breakaway process with various densities of the pinning atmosphere (a, b, c), and the corresponding $\Delta\alpha$ *vs.* σ responses (a′, b′, c′).

4.3. Thermal cycling

First, we comment on the evolution of $\Delta\alpha$ vs. T for the unreinforced Al. As the Cottrell cloud has been formed during an annealing at high temperature, it is expected to be quite stable. Therefore the monotonic decrease of $\Delta\alpha(\sigma_0)$ observed with decreasing test temperature (Fig. 2, curve b) is explained by the decrease of the damping factor and the increase of the breakaway stress. Moreover, owing to the above-mentioned stability, no difference is observed between the cooling and heating stages.

However, on cooling the reinforced Al, as explained previously, on the one hand fresh dislocations are emitted from particles, and on the other hand existing dislocations are submitted to an increasing thermal stress, so that they are set free from point defects. Therefore after each temperature change a new Cottrell cloud has to be formed on both types of dislocation during the isothermal rest time preceding each ultrasonic measurement. Then, since the mobility of point defects decreases exponentially with decreasing temperature, the atmosphere is expected to be more and more dilute with decreasing T. Therefore it is not surprising to observe a marked maximum of $\Delta\alpha(\sigma_0)$ *vs.* T, around 240 K, because, as explained for the plastically deformed aluminium, such an effect is expected to occur when the density of the Cottrell cloud varies monotonically. In fact, this cloud density effect is superimposed on the other effects linked with temperature, which acts on the damping factor, the thermal activation of breakaway and the punched-out dislocation density.

Finally, the marked excess of $\Delta\alpha(\sigma_0)$ that is observed for the heating stage with respect to the cooling one can be explained as follows. Owing to the evolution of thermal stresses on cooling the sample down to 80 K: (i) existing dislocations that have been firmly pinned by point defects when the sample was staying at room temperature should be displaced away from their atmosphere; (ii) as predicted by Hamann's model, the dislocation density Λ around particles should have been increased during this thermal cycle.

5. Conclusion

During a low-temperature thermal cycle, an original behaviour of the $\Delta\alpha(\sigma)$ response has been observed for Al/12 vol.% SiC composite. The evolutions of $\Delta\alpha(\sigma_0)$ can be explained only by taking into account the emission and the movements of dislocations induced by thermal stresses.

Acknowledgments

This work was supported by the Aérospatiale Society (Suresnes Laboratory), which is gratefully acknowledged. We are also very grateful to P. Fondères (Villetaneuse University) for elaborating the Al/SiC composite.

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