Thermal Stress Relaxation in Al-Al₂O₃ (f) Composites During Thermal Cycling

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Abstract. The thermal stress relaxation mechanisms in Al 99.99% reinforced with SAFFIL alumina short fibers have been investigated by mechanical spectroscopy during long period and low temperature thermal cycles ($T = 2$ K/min, 100 K - 450 K). During these cycles large internal stresses are built in the specimen due to the difference in thermal expansion coefficient between the matrix and the reinforcement. It has been found that the internal friction, the elastic modulus and the zero point drift behaviours are extremely sensitive to these internal stresses and specially to the relaxation mechanism operating: dislocation generation or dislocation creep. The internal friction shows a broad non anelastic maximum during cooling which is attributed to dislocation movement in a continuously developing plastic zone nearby the fibers. The elastic modulus behaviour as a function of temperature leads to define two particular temperatures: T_{start} related to the initial growth of plastic zones nearby the fibers and T_{end} related to their later overlapping. The zero point coordinate describes during each thermal cycle a characteristic trajectory which may be analyzed in terms of a thermal fatigue loop. The study is performed in composites with different volumetric fraction of fibers and matrix strength.

1. INTRODUCTION

Metal matrix composites (MMCs) are light, stiff and strong materials characterized by high performance mechanical properties. On the other hand, they present the disadvantage of thermal fatigue originated near the interfaces during thermal cycling [1]. This aspect of the composites is the interest of this paper. Thermal cycling generates thermal misfit strains to accommodate the thermal expansion mismatch between matrix and fibers. The resulting high internal stresses built up at the interfaces may be relaxed by different mechanisms [2] comprising microplasticity of the metal matrix and damage accumulation by fiber-matrix debonding. Mechanical Spectroscopy is a useful tool to study these relaxation mechanisms at low temperatures.

2. EXPERIMENTAL PROCEDURE

Composites samples with Al 99.99% matrix containing 10, 15, 20 and 30 % vol. of Al₂O₃ SAFFIL fibers were produced by squeeze casting by Alusuisse-Lonza Ltd. The average fiber length was about 12 μm and the diameter 1.9 μm . Samples of 1.8 mm diameter and 100 mm in effective length were obtained by machining. Specimens were treated 3 hs at 600 K and slowly cooled to room temperature inside the furnace in order to relax internal stresses and defects built during machining. Mechanical Spectroscopy measurements were performed in the forced torsion pendulum Charango II [3]. The mechanical loss angle $\text{tg}(\phi)$ and the elastic shear modulus (arbitrary units) were measured with forced oscillations during

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controlled heating and cooling cycles between 100 K and 450 K. Typical strain amplitudes of 7×10^{-6} and temperature change rates of 2 K/min were used. The deformation at zero applied stress (zero point drift) was simultaneously measured. The influence of the matrix strength was studied measuring a composite Al7Si4Cu - 15%SAFFIL treated 3 hs at 600 K and then water quenched and aged 5hs at 460K in order to precipitate the intermediate phase θ' Al₂Cu.

3. RESULTS

Mechanical spectroscopy during thermal cycling: Figure 1a shows that the mechanical loss angle has a large and broad maximum during cooling from the high annealing temperature (30min at 450K). During subsequent heating, after annealing 15 min at 100K another maximum appears smaller in height and located at a higher temperature. It is worth to note the strong evolution of internal friction during annealing at both extreme temperatures, leading to the large hysteresis observed. The whole damping spectrum is reproducible during thermal cycling. The increasing background level at high temperature may be attributed to a relaxation peak located at 450K (1Hz) [4].

The elastic modulus also exhibits a large hysteresis which is reproducible during thermal cycling; this indicates that the internal damage accumulation as the number of cycles increases is not severe (Figure 1b). During cooling the modulus shows a plateau between the temperatures T_{start} and T_{end} .

The zero point drift (Figure 1c) shows that the displacement reverses when the sign of $\dot{T}$ changes, drawing an almost closed loop. Two characteristic deformation values are defined: γ_m the maximal value attained in a cycle and γ_p the permanent deformation after annealing at high temperature.

Effect of measurement conditions: Figures 2a, b, c shows the influence of the temperature change rate $\dot{T}$, the oscillation frequency ω , and the measurement strain amplitude ϵ on the IF spectrum during cooling for the composite containing 20% fibers. Peak height increases if the cooling rate is increased. The peak height strongly decreases with frequency and also hysteresis reduces to become almost null at 7Hz. The maximum temperature is not frequency sensitive so that the relaxation is not originated in anelastic mechanisms. Finally, the maximum level decreases as the measurement strain amplitude increases. No definite trend may be established for the maximum temperature.

Effect of fiber content: All the composites exhibit the same qualitative behaviour showed in Figures 1a-c, with the exception of the material with 10% content of fibers. In this case it is not possible to resolve T_{end} and the zero point drift shows an irreversible evolution during cycling.

Figure 1a, b, c: Internal friction, elastic modulus and zero point drift for SAFFIL during thermal cycling. $\dot{T} = 2\text{K/min}$, $\omega = 0.1\text{ Hz}$, $\epsilon = 7 \times 10^{-6}$

A more detailed analysis of the T_{start} and T_{end} dependence with f is presented in the paper of Urreta et al. [5]. Concerning the zero point, the maximum reversible strain achieved γ_m increases and the permanent strain γ_p decreases with f (Figure 3).

The effect of the matrix strength: Three principal differences between Al4N-15% and Al7Si4Cu-15% behaviours can be observed. The first one is that in the alloyed composite internal friction maxima is lower (Figure 4), displaced to lower temperatures and progressively reduces during cycling. The second one is that elastic modulus shows an almost linear behaviour with temperature. The third difference is that no zero point close loops are present, instead, an irreversible increase in the displacement takes place during cooling and heatings.

4. DISCUSSION AND CONCLUSIONS

Internal damping spectrum: The transitory $\dot{T}$ effect showed in Fig. 2a is associated with the evolution of the dislocation microstructure during the measurements. It exhibits the same characteristics as those reported in AlSi containing particles [6] and in Al alloys based MMCs [7]. This effect has been attributed to interfacial dislocation movements induced by thermal stresses due to the thermal expansion mismatch between matrix and reinforcements [8]. Zhou et al. [6], assume that the dislocation velocity near the interfaces can be expressed as:

$$v = v_0(1 + \zeta \sin \omega t)$$

where v_0 is the mean velocity due to the continuous evolution of thermal stresses and ζ is a modulation factor induced by the pendulum oscillation. Taking v_0 proportional to $\dot{T}$ and ζ proportional to the strain amplitude, they obtain for the additional contribution to the internal friction due to the $\dot{T}$ effect:

$$IF_{\text{add}} \propto \frac{\dot{T}}{\omega} F(\tau_M, \rho, M)$$

where F is a function of the thermal internal stresses τ_M , the dislocation density ρ and the dislocation mobility M . This expression accounts for the dependence of IF on the cooling rate and oscillation frequency but not on the strain amplitude. The initial increase in IF during cooling is associated with an increase in the dislocation density and mobility nearby the fibers. The subsequent IF decrease can be explained by the hardening in the plastic zones and the reduction of the intrinsic mobility of dislocations at low temperature.

As a phenomenological approach, it can be considered that the modulation factor ζ is weakly dependent on the strain amplitude ε by a power n less than 1. It results the follow expression for internal friction:

Figure 2: Internal friction dependence with the cooling rate (a), oscillation frequency (b) and measurement strain amplitude (c) for a composite Al-20%SAFFIL.

$$IF_m \propto \frac{\dot{T}}{\omega \epsilon^{1-n}}$$

with $n = 0.7$ calculated from the Figure 2c.

Elastic Shear Modulus: the plateau observed during cooling shows the apparition of plasticity between the T_{start} and T_{end} temperatures. At T_{start} the plastic zone begins to develop around the fibers and dislocations are gradually introduced in the neighbouring matrix. The plastic zones continue to grow until they reach a critical size at the temperature T_{end} . At lower temperatures the dislocation mobility drastically reduces producing the elastic modulus recovery.

Zero point drift: the origin of the reversible strain observed in the Figure 1c can be attributed to the elastic recoverable deformation energy stored in the fibers. Figure 3 shows that increasing the fiber content, the mechanical stability during thermal cycling is increased. Thermal stresses at interfaces are proportional to the variation in temperature from a free-stress state at high temperature. Therefore, the zero point trajectory in the (γ, T) space during a thermal cycle may be related to a given trajectory in a (γ, σ) space. The enclosed area is proportional to the energy dissipated in plastic deformation and the loop could be analyzed in terms of a fatigue cycle. As the internal friction is also proportional to the dissipated energy both magnitudes must be correlated. This correlation was observed: large internal friction effects are linked with large loop areas in the (γ, T) plot.

Influence of the matrix strength: The changes observed are due to the influence of the precipitates that reduce the growth of the plastic zone around the fibers. The interaction between precipitates and dislocations, as was expected, plays an important role in the dissipation process near the interfaces.

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Figure 3: Dependence of γ_m and γ_p on the fiber content

Figure 4: Comparative internal friction of Al-15% SAFFIL and Al7Si4Cu-15% SAFFIL. $\dot{T} = 2K/min$, $\omega = 0.1$ Hz, $\epsilon = 7 \times 10^{-6}$