Contents lists available at ScienceDirect

Materials Science and Engineering A

High temperature damping behaviour of Ti6Al4V–SiC$_f$ composite

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ARTICLE INFO

ABSTRACT

The Ti6Al4V–SiC$_f$ composite, reinforced by unidirectional SiC fibres (SCS-6), has been investigated by internal friction (IF) and dynamic modulus measurements in the temperature range 300–1173 K. Experiments have been carried out by using a vibrating reed technique with electrostatic excitation and frequency modulation detection of flexural vibrations in the frequency range from 600 to 1800 Hz.

For comparison the monolithic Ti6Al4V alloy has been submitted to the same experiments.

The IF spectrum of the composite exhibits a relaxation peak at about 870 K superimposed to an exponentially increasing background. The peak, not present in the spectrum of matrix alloy, has activation energy $H = 186 \, kJ \, mol^{-1}$ and relaxation time $\tau_0 = 2.3 \times 10^{-15}$ s.

The peak has been explained by an interstitial-substitutional pair reorientation mechanism in the α phase of Ti6Al4V matrix around the fibres.

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1. Introduction

Composites of Ti-alloys reinforced with fibres are materials of great interest for aeronautical applications [1–4]. Mechanical behaviour is governed by the fibre–matrix interface where diffusion of atomic species and chemical reactions may occur leading to structural instability.

The Ti6Al4V–SiC$_f$ composite was produced at Centro Sviluppo Materiali (CSM). Fig. 1a shows the detail of the matrix around a single fibre; the SCS-6 fibres (Fig. 1b) are coated by a carbon layer separating SiC and metal matrix.

In the past the material has been investigated by the authors in as-fabricated condition and after long-term heat treatments (up to 1000 h) at 873 K, i.e. the temperature foreseen for its application in aeronautical engines [5–9]. Tensile, hardness and fatigue tests showed that the mechanical characteristics are preserved after heat treatments because the interface is substantially stable. The growth of a thin layer of TiC between carbon coating and matrix during fabrication process slows down carbon diffusion towards the matrix and determines the structural stability of the composite. It has been observed that prolonged heat treatments at 873 K produce only small modifications of the interface.

The diffusion of carbon is a two-step process: (1) diffusion of carbon through the TiC layer, (2) diffusion of carbon in the Ti6Al4V alloy. The structural stability depends on the slow kinetics of the first step.

Anelastic phenomena could play an important role on the mechanical behaviour of fibre-reinforced composites. For instance, Schaller [10] showed that thermal stress relaxation at the interface gives rise to transient mechanical loss in Mg composites reinforced by C fibres due to dislocation motion.

Results of IF experiments on monolithic Ti6Al4V alloy are reviewed in the book of Blanter et al. [11]. The elastic properties of a unidirectional Ti6Al4V–SiC$_f$ composite have been characterized by Ogi et al. [12] using the mode-selective contactless resonance ultrasound spectroscopy.

The anelastic behaviour of composite and matrix alloy has been investigated by the authors in the temperature range 100–1173 K. Present paper reports data from 300 to 1173 K while a companion paper presented at this conference [13] concerns data at lower temperature.

2. Material and experimental

The composite has been fabricated by hot isostatic pressing (HIP) in a multi-step process. Preforms (450 mm × 200 mm) made of four layers of unidirectional SCS-6 fibres ($\Phi = 0.14$ mm) alternated with five Ti6Al4V sheets (thickness of 0.6 mm) have been prepared. They were then put inside a AISI 304 steel die, which was then evacuated (3×10^{-6} mbar) and sealed. HIP cycle has been realized by a hot isostatic press ASEA-QH21, which permits to control independently temperature and pressure. The material was kept at 1163 K for 1.8×10^3 s under a pressure of 1200 bar; after cooling to room temperature the composite sheets were extracted from the die.

The matrix of the composite is mainly made of the hcp α phase with a minor amount (~4%) of the bcc β phase, the same structure of the monolithic Ti6Al4V alloy.

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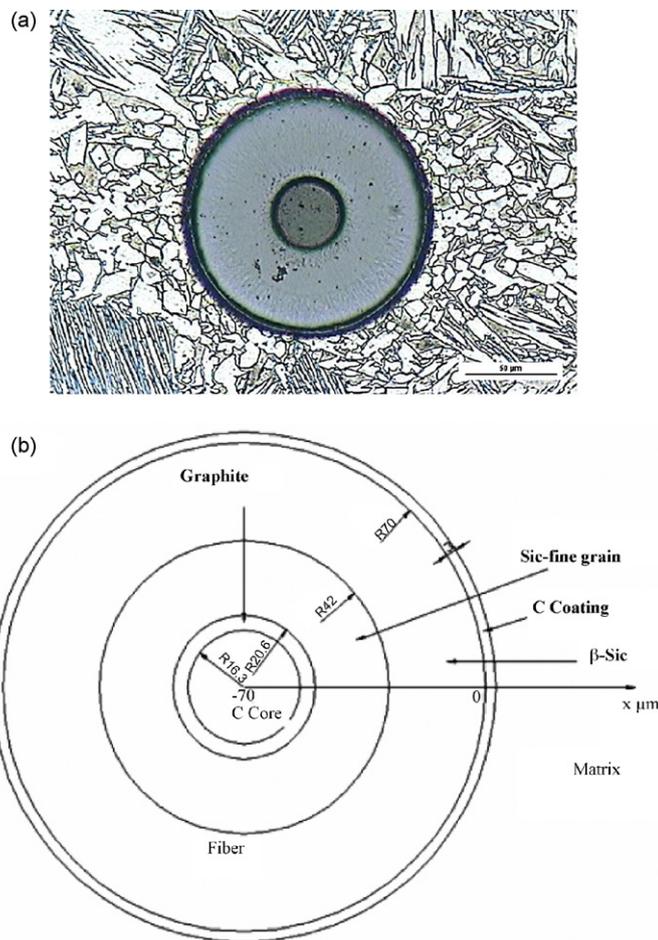 *E-mail address:* roberto.montanari@uniroma2.it (R. Montanari).

Fig. 1. (a) Section of the composite. (b) Sketch of the internal structure of a SCS-6 fibre.

IF and dynamic modulus measurements have been carried out on bar-shaped samples using the method of frequency modulation. The VRA 1604 apparatus used in the experiments has been described in detail in [14]. The resonance frequencies were in the range 600–1800 Hz. The samples have been heated from room temperature to 1150 K with a rate of 1.7×10^{-2} Ks^{-1}. Strain amplitude was kept lower than 1×10^{-5}.

X-ray diffraction (XRD) measurements have been carried out by using Co Kα radiation (λ = 0.1789 nm). Precision peak profiles of the most intense XRD reflections have been recorded with angular 2Θ steps of 0.005° and counting time of 20 s per step. The half-height line widths β have been determined and corrected from instrumental broadening.

For comparison all the experiments have been performed on both the composite and the monolithic Ti6Al4V alloy.

3. Results

At room temperature the Young's modulus of the composite is 138 GPa, ~20% higher than that of the monolithic alloy (114 GPa).

Fig. 2 shows Q^{-1} and $(f/f_0)^2$ vs. T in the composite. The Q^{-1} curve exhibits a peak at about 870 K superimposed on an exponentially increasing background, in correspondence of the peak the modulus exhibits a change. The peak position depends on the frequency; from the Arrhenius plot, which is not shown, the activation energy $H = 186$ kJ mol^{-1} and the relaxation time $\tau_0 = 2.3 \times 10^{-15}$ s have been obtained.

Repeated tests on the same sample do not induce permanent changes of resonance frequency f_0 and Q^{-1} at room temperature.

After heat treatments of 1.8×10^4 s at 1073 K the IF peak is still present without remarkable intensity variation.

For comparison the Q^{-1} curve of the monolithic Ti6Al4V alloy is also displayed in Fig. 2. The alloy does not show the IF peak but only the exponential background, higher than that of composite.

Both composite and monolithic alloy have been examined by XRD. Peak profiles of the alloy are quite broader than those of the composite. For example, Fig. 3 displays the {1 0 0} reflections whose intensities have been normalized for making the comparison easier.

For each XRD reflection the total line broadening β_T, corrected from instrumental broadening, is basically due to two contributions, the size of coherently diffracting domains (β_D) and the micro-strains (β_ε). β_T can be written as:

$$\beta_T = \beta_D + \beta_\varepsilon = \frac{K\lambda}{D\cos\vartheta} + 2\varepsilon\tan\vartheta \tag{1}$$

where D is the domain size, ε the average micro-strain, ϑ the Bragg angle, λ the X-ray wavelength and K a constant (=0.89). In the case of Ti and Ti alloys the coherently diffracting domains are the grains which from metallographic observations result to be near of the same mean size ($D \approx 30$ μm) in alloy and composite. Owing to the very large grain size , the β_D term in Eq. (1) can be neglected:

$$\beta_T \cong \beta_\varepsilon = 2\varepsilon\tan\vartheta \tag{2}$$

Introducing the β_T values of the main XRD reflections into Eq. (2), the micro-strain ε has been determined. Finally, the dislocation density ρ was calculated by means of the Williamson–Smallman
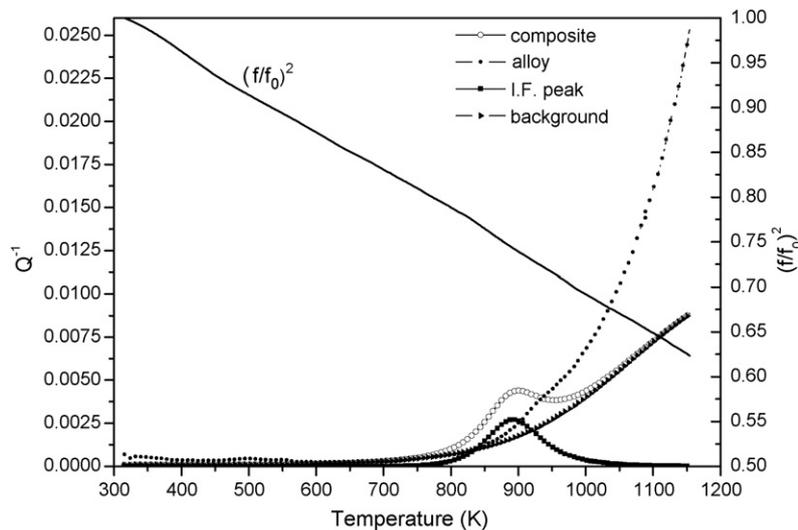
Fig. 2. Q^{-1} and $(f/f_0)^2$ vs. T trends of the composite ($f_0 = 898$ Hz). The Q^{-1} curve is the superposition of a Debye peak and an exponential background. The IF curve of the monolithic Ti6Al4V alloy is displayed for comparison.

relationship [15]:

$$\rho = \frac{\mathit{\Xi}\varepsilon^2}{k_0 b^2} \tag{3}$$

where $\mathit{\Xi} = 16$ is a constant, b is the modulus of Burgers vector and $k_0 \cong 1$ is a factor depending on dislocation interaction. From this calculation the values of $\rho = 6.1 \times 10^9$ cm^{-2} for the composite and $\rho = 5.5 \times 10^{10}$ cm^{-2} for the monolithic alloy have been obtained. This result, which could appear surprising, can be understood if one considers that a partial recovery of defective structures takes place in composite matrix during the slow cooling to room temperature following the isostatic pressing at 1163 K.

4. Discussion

IF spectra of composite and alloy show a background which exponentially increases with temperature. The background, which is strongly structure-sensitive [16], is higher for the alloy than for the composite. The result can be explained by considering a different contribution to background from the dislocation damping in the two materials: the grain size is near the same in both of them while they have a different dislocation density, which is about one order of magnitude higher in the alloy.

The IF spectrum of the composite shows a Debye peak which is not observed in the Ti6Al4V monolithic alloy. Since the peak has

Fig. 3. Precision {1 0 0} XRD peak profiles of alloy and composite.

been observed also after a pronged heat treatment at 1073 K, it cannot be related to a possible transformation of unstable β phase. Its origin is clearly connected to the presence of the fibres and their effects on the surrounding matrix; for giving an explanation some hypothesis have been considered.

Different phenomena giving rise to energy dissipation may occur at the fibre–matrix interface when composites are subjected to thermal and/or mechanical stresses. At high stresses plastic flow and interface de-bonding may occur, at low stresses matrix and fibres undergo only elastic distortions.

The effects of plastic flow and fibre–matrix de-bonding on modulus and damping have been described and discussed by Schaller [10]. As a consequence of the different values of coefficient of thermal expansion between matrix and fibres internal stresses arise along the production process during cooling from HIP temperature (1163 K) to room temperature. However, a simple calculation shows that the stresses are not sufficiently high to induce the formation of dislocations in the matrix near the fibres. The mean stress α_M in the matrix arising from cooling from HIP temperature (T_{HIP}) to room temperature (T_R) can be expressed by:

$$\sigma_M = \frac{E_F E_M}{(E_F \phi + E_M (1 - \phi))} \phi (\alpha_M - \alpha_F)(T_{HIP} - T_R) \tag{4}$$

where E_F, α_F and E_M, α_M are the Young's modulus and the coefficient of thermal expansion of fibre and matrix respectively, ϕ the volume fraction of fibres. Introducing in Eq. (4) the values $E_F = 400$ GPa, $E_M = 114$ GPa, $\alpha_M = 9.6 \times 10^{-6}$ K^{-1}, $\alpha_F = 4.1 \times 10^{-6}$ K^{-1} and $\phi = 0.3$, $\sigma_M = 350$ MPa has been obtained. This value is much lower than the matrix yield stress $\sigma_Y = 850$ MPa.

Furthermore, dynamic modulus and Q^{-1} at room temperature do not change after repeated test runs on the same sample. Therefore, plastic flow and de-bonding at the fibre–matrix interface have been ruled out as a possible cause of the anelastic phenomena observed by the authors and the attention has been focused on elastic strains at the interface.

This condition has been analysed by He and Lim [17] on the basis of the interfacial diffusion mechanism [18]. When a shear stress is applied, the interface, which is not perfectly planar on a microscopic scale, is under tension at some locations and under compression at some others. The stress gradient induces atom diffusion along the interface causing anelastic behaviour. However, the activation energy determined from our experiments, $H = 186$ kJ mol^{-1}, is quite different from those controlling the atomic diffusion of Ti, Al and V
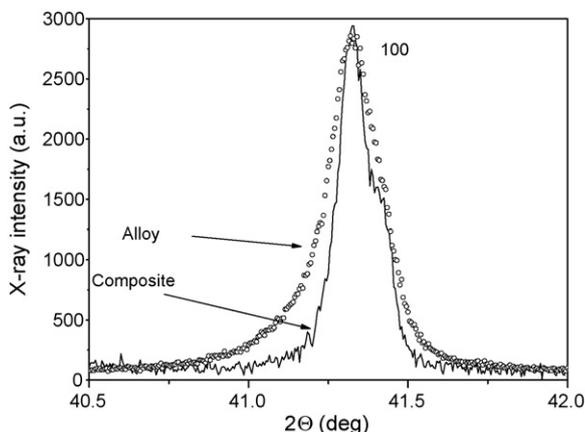
in both α and β phases present in the matrix thus the IF peak can not be ascribed to such mechanism. Also diffusion processes inside the fibres, which have a stratified axial-symmetric structure (Fig. 1b), are not compatible with the peak activation energy because the diffusion energies of C [19] and Si [20] in SiC, respectively 318 and 912 kJ mol^{-1}, are much larger.

As shown in Fig. 1, the SiC fibres are coated by a carbon layer (thickness ≈3 μm) which separates SiC from Ti6Al4V matrix. During the process of composite fabrication, carried out at high temperature (1163 K), carbon reacts with titanium forming a thin layer (few nanometers thick) of titanium carbide (TiC) [8]. TiC thickness grows when the material is heat treated but the kinetics is very slow. The activation energy for TiC growth, independently determined by Naka et al. [21], is 194 kJ mol^{-1}, close to that of IF peak. Therefore, the peak seems somehow connected to the growth of the TiC layer between fibre carbon coating and the matrix. Furthermore, one has to consider that its activation energy is in fact very close to that of carbon diffusion in the α-Ti ($H^* = 182$ kJ mol^{-1}). On these grounds, it is believed that the peak is due to stress induced reorientation of interstitial-substitutional (i-s) pairs (C–Al and C–V) in the hcp α phase of the matrix near the fibres where the formation of TiC produces a zone richer in substitutional atoms than the remaining matrix. The mechanism has been discussed by Gupta and Weining [22] and Povolo and Bisogni [23] for hcp metals.

5. Conclusions

The composite Ti6Al4V + SiC$_f$ has been investigated by IF and dynamic modulus measurements in the temperature range from room temperature to 1173 K. For comparison the same experiments have been performed on the corresponding monolithic alloy.

The IF spectrum of the composite exhibits a relaxation peak ($H = 186$ kJ mol^{-1} and $\tau_0 = 2.3 \times 10^{-15}$ s) which has been tentatively explained as due to C–Al and C–V pairs reorientation in the α phase of Ti6Al4V matrix around the fibres.

Both composite and alloy show a background which exponentially increases with temperature. Since the alloy has a higher dislocation density than the composite, dislocation damping is larger and background more intense.

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