

## Low temperature anelasticity in Ti6Al4V alloy and Ti6Al4V–SiC<sub>f</sub> composite

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### ABSTRACT

Internal friction and dynamic Young modulus measurements have been performed in the temperature range 80–350 K on the Ti6Al4V alloy and a Ti6Al4V–SiC<sub>f</sub> composite reinforced by unidirectional SiC fibers (SCS-6). A vibrating reed apparatus operating at low strain amplitude ( $<10^{-5}$ ) in the frequency range  $10^2$ – $10^4$  Hz was employed. Two anelastic relaxation peaks: P<sub>1</sub>, P<sub>2</sub> have been observed in both materials. The peak temperatures at 1 kHz are respectively 120 K and 250 K. The fiber reinforced composite in the same temperature range shows a higher background damping and enhanced relaxation strength for the P<sub>1</sub> peak. The activation energy and frequency factors evaluated from the peak temperature shift with frequency are respectively  $H_1 = 0.21 \pm 0.02$  eV,  $H_2 = 0.50 \pm 0.03$  eV and  $\tau_{01}^{-1} = 10^{15} \text{ s}^{-1}$ ,  $\tau_{02}^{-1} = 10^{12} \text{ s}^{-1}$ . Both peaks position and relaxation strength depend on hydrogen content and the relative amount of defects in  $\alpha$  and  $\beta$  phases.

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### 1. Introduction

Titanium fiber reinforced composites are promising materials for aircraft industry [1,2]. Gaseous impurities such as hydrogen nitrogen and oxygen absorbed during manufacturing processes and in operating conditions can strongly modify their mechanical behaviour. In these composites further structural instability phenomena may occur at the fiber–matrix interfaces determined by diffusion processes and thermally driven chemical reactions [3–5].

The Ti6Al4V alloy, consists of an ( $\alpha + \beta$ ) two phase structure. The solubility of hydrogen in the bcc  $\beta$  phase is much higher than in the hcp  $\alpha$  phase, the activation energy reported for hydrogen diffusion in the  $\beta$  phase is much lower than in the  $\alpha$  phase [6]. Anelasticity measurements through mechanical spectroscopy have been extensively employed to understand details of point defects dynamics in Ti alloys [7] and to investigate transient or permanent damping effects depending on the evolution of thermal stresses at the fiber–matrix interfaces in composites [8]. This paper companion of another one [9] aims at a comparative investigation of the anelastic behaviour at low temperatures of the Ti6Al4V–SiC<sub>f</sub> composite and the corresponding monolithic matrix alloy, to understand the role of the microstructure, defects content on anelasticity phenomena depending on hydrogen.

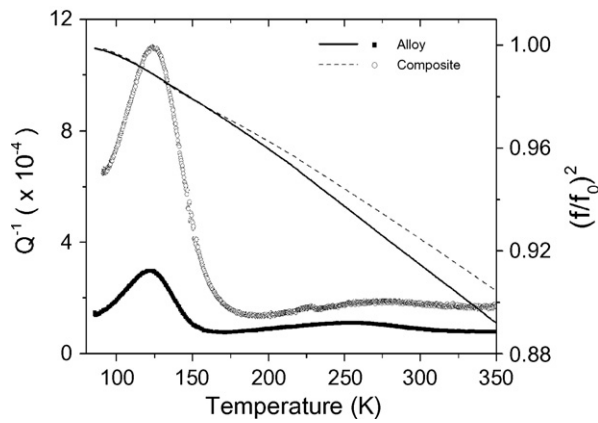
### 2. Experimental

The Ti6Al4V–SiC<sub>f</sub> composite was produced at Centro Sviluppo Materiali (CSM) by HIP (Hot Isostatic Pressing) in a multistep process, starting from layers of unidirectional SCS-6 fibers in alternate sequence with Ti6Al4V sheets. Details of the processing steps are reported elsewhere [9]. The microstructure of the composite matrix mainly consisted of  $\alpha$  phase with about 4%  $\beta$  phase. The monolithic alloy displays a similar duplex ( $\alpha + \beta$ ) microstructure.

The inverse mechanical quality factor (internal friction) and the resonance frequency, proportional to the square root of the dynamic young modulus at constant density and shape, have been measured by a completely automated vibrating reed analyzer VRA-1604 (CANTIL Srl). The apparatus employs electrostatic excitation and frequency modulation detection of the flexural vibration modes of cantilevered mounted reed samples with typical dimensions: 6 cm length, 0.5 cm width, 0.08 cm thickness. All measurements of the internal friction ( $Q^{-1}$ ) and of the resonance frequency ( $f$ ), were performed in the 0.6–3.5 kHz frequency range at a strain amplitude  $\varepsilon < 10^{-5}$  and a pressure in the vacuum chamber  $10^{-4}$  Pa to  $10^{-3}$  Pa. Before each measurement run, in the 80–350 K range at a constant heating rate of 1 K/min, all samples were thermally equilibrated at the lower temperature for 30 min. All ageing treatments at temperatures in the 600–900 K range have been performed in a vacuum of  $10^{-4}$  Pa. To gain a clearer understanding of some specific features of the internal friction spectra, some measurements were performed also on a commercial  $\beta$  rich  $\alpha + \beta$  titanium alloy SP-700 (Ti–4.5Al–3V–2Mo–2Fe).

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**Fig. 1.** Internal friction and normalized squared resonance frequency of the monolithic alloy and the composite as prepared.  $f_0$  = resonance frequency at the lowest temperature at the start of the heating run:  $\sim 600$  Hz for Ti6Al4V and  $\sim 900$  Hz for the Ti6Al4V + SiC composite.

### 3. Results

Fig. 1 shows the internal friction and the squared resonance frequency (normalized to the value at the lower temperature at the start of measurement:  $(f/f_0)^2$ ), versus temperature for the monolithic alloy and the as prepared fiber reinforced composite. A prominent peak thereafter indicated  $P_1$ , is observed in both materials at approximately 120 K (1 kHz), the relaxation strength is significantly higher in the composite in comparison with the monolithic alloy. A second very broad peak  $P_2$  centered at 250 K in the alloy and 270 K in the composite spectra has been observed. The background damping in the whole temperature range investigated appears higher in the composite.

The relaxational origin of both peaks was confirmed by the shift of the peaks temperature with frequency. The values obtained for the activation energy and frequency factors are reported in Table 1. After thermal ageing up to 900 K in a  $10^{-4}$  Pa vacuum, a significant reduction of the  $P_1$  peak relaxation strength both in the monolithic alloy and the composite, up to 50%, is experienced, as can be seen in Fig. 2.

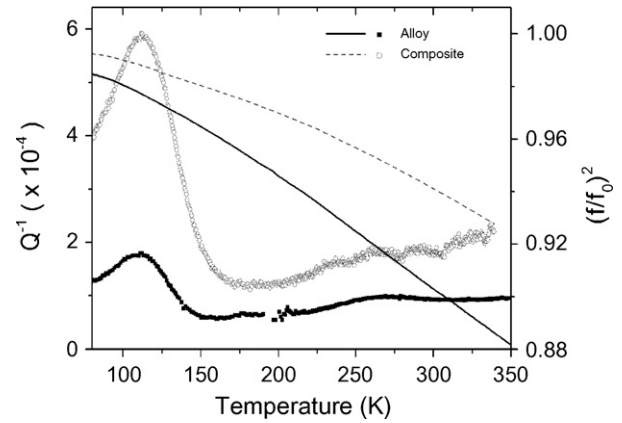
In the composite the background damping with respect to the as received samples (Fig. 1) increases slightly with temperature, moreover a small reduction of the modulus (frequency) occurs with reference to the values experienced in as fabricated condition (Fig. 1). The  $P_1$  peak with a relaxation strength similar to that of the composite and significantly higher than that of the monolithic matrix alloy is observed also on the SP-700 alloy (Fig. 3).

### 4. Discussion

On the basis of the internal friction spectra presented above, the  $P_1$  peak can be attributed to hydrogen. The activation energy obtained from the peak shift (Table 1) is slightly lower than that reported for hydrogen diffusion in the  $\beta$  phase of vanadium free titanium [6], but is slightly higher than those obtained by NMR in titanium with vanadium additions [10]. A value comparable with our results, within the uncertainty of measurements, was obtained by internal friction measurements on the Ti6Al4V alloy [11]. In the

**Table 1**  
Anelastic relaxation peaks parameters.

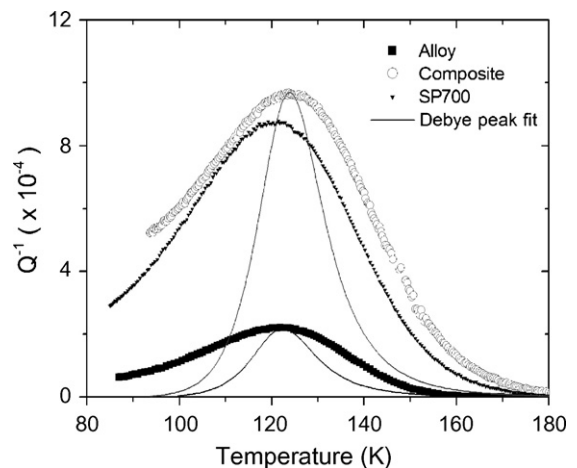
Peak	$P_1$	$P_2$
Temperature at 1 kHz frequency (K)	120	250–270
Activation energy (eV)	$0.21 \pm 0.02$	$0.50 \pm 0.03$
Inverse frequency factors ( $s^{-1}$ )	$10^{15}$	$10^{12}$



**Fig. 2.** Internal friction and resonance frequency of the same samples as in Fig. 1 after ageing at 900 K in vacuum.  $f_0$  resonance frequency of Fig. 1.

SP-700 alloy (Fig. 3), a well resolved peak having roughly the same activation energy of that in the Ti6Al4V alloy is present. Measurements performed at low frequency [12] yield similar activation energy. The prominent  $P_1$  peak can be therefore attributed to a Snoek type relaxation caused by interstitials hydrogen atoms in the  $\beta$  phase.

A significant difference in the  $P_1$  relaxation strength is yet observed (Fig. 3) between the matrix alloy and the composite. Moreover the peak relaxation strength in the SP-700, mainly  $\beta$  phase alloy, is similar to that of the composite (Fig. 3). The overall hydrogen content, measured by means of chemical analysis, was respectively: 116 ppm in the monolithic Ti6Al4V alloy, 187 ppm in the composite and 97 ppm in the SP-700 alloy. These values are within the low concentration solubility limit where a linear dependence between the relaxation strength and the amount of interstitial hydrogen responsible for Snoek relaxation in the  $\beta$  phase is observed [12]. It is well known that in two phase ( $\alpha + \beta$ ) titanium alloys hydrogen mainly accumulates in  $\beta$  phase; the relative amount of hydrogen in  $\alpha/\beta$  interfaces and in  $\alpha$  phase is very small [13,14] so the concentrations in the  $\beta$  phase are close to the overall contents reported above. Therefore, the larger relaxation strength in SP700, with lower hydrogen content, than in Ti6Al4V appears surprising. Moreover, the ratio between relaxation strengths of Ti6Al4V and composite does not correspond to that between their hydrogen contents. The apparent inconsistency of hydrogen contents and relaxation strengths can be explained by assuming that not all the



**Fig. 3.** Comparison of the internal friction spectra in the temperature range of the  $P_1$  peak for the composite, the alloy and SP-700. Background damping has been subtracted. The  $P_1$  peak is broader with respect to that resulting by assuming a Debye relaxation.

hydrogen atoms present in the  $\beta$  phase participate to relaxation processes because a part of them are trapped by lattice defects. This hypothesis is supported by the values of dislocation densities determined by XRD which are  $5.5 \times 10^{10} \text{ cm}^{-2}$ ,  $6.1 \times 10^9 \text{ cm}^{-2}$  [9] and  $2.5 \times 10^9 \text{ cm}^{-2}$  respectively for Ti6Al4V, composite and SP700.

The relaxation strength reduction after ageing, which is of the same order (50%) in the Ti6Al4V alloy and in the composite for similar ageing treatments (Figs. 1 and 2), is consistent with a similar mechanism of hydrogen occupancy reduction of the interstitial sites in the  $\beta$  phase, responsible for the  $P_1$  Snoek type relaxation. No appreciable defect recovery was observed after long-term treatments at 873 K, temperature close to that of ageing [15,16].

Present measurements show that the  $P_1$  peak is much broader than a Debye peak (Fig. 3). The broadening effect is larger in the composite. A possible effect of relaxation time distribution connected to microstrain and lattice distortion is under investigation.

The  $P_2$  peak observed at around 250 K, has an activation energy, Table 1, corresponding within the measurement uncertainty, to that reported for hydrogen diffusion in the  $\alpha$  phase [6]. A peak with similar activation energy was observed in Ti6Al4V [11] and ascribed to hydrogen in  $\alpha$  phase. The mechanism proposed is a stress-induced reorientation of interstitial-substitutional pairs in the  $\alpha$  phase according to the model proposed by Gupta and Weining [17] for hcp metals.

## 5. Summary

Comparative measurements at low temperatures on the Ti6Al4V–SiC<sub>f</sub> composite and the corresponding monolithic alloy showed two anelastic relaxation peaks,  $P_1$ ,  $P_2$ :

$P_1$  results from a Snoek type relaxation due to interstitial hydrogen in the minority bcc  $\beta$  phase. Relaxation strengths are affected by the higher dislocation density in alloy than in composite

$P_2$  has been ascribed to stress-induced reorientation of interstitial-substitutional pairs in the hcp  $\alpha$  phase.

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