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Anelastic behaviour in Nb–Ti alloys containing interstitial elements

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Abstract

Internal friction measurements as a function of temperature were performed between 250 and 700 K in an Nb–48wt.%Ti alloy heat treated under various conditions, using a torsion pendulum of the inverted Kê type with an oscillation frequency of about 3.5 Hz. The internal friction data were used to obtain spectra of multiple anelastic relaxation as a function of temperature, which were resolved into elemental interactions. The following metal–interstitial interactions were identified: Nb–O (430 K), Nb–N (541 K), Ti–O (467 K), Ti–O–O (495 K), Nb–O–O (443 K) and Ti–O–O–O (523 K). The anelastic relaxation parameters, height of the internal friction peak, peak temperatures and activation energy were calculated for each of the processes using Debye's elemental peaks.

1. Introduction

Metals containing solute atoms dissolved interstitially often show anelastic behaviour due to a process known as stress-induced ordering. One manifestation of this anelastic behaviour is the internal friction, which was originally observed by Snoek [1] in Fe containing carbon and nitrogen as interstitial solutes. The anelasticity is caused by stress-induced migration of interstitial atoms in octahedral positions of b.c.c. lattices.

Several techniques can be used to measure the internal friction [2–4]; of these the torsion pendulum is best suited to the study of metal–interstitial solute interactions (such as carbon, nitrogen or oxygen). At low interstitial solute concentrations, the relaxation strength of the internal friction at a given temperature is a function of the nature, position and concentration of the interstitial atoms.

In the lattice of an Nb–Ti alloy (b.c.c.), oxygen and nitrogen atoms are expected to occupy positions similar to those of carbon and nitrogen in the Fe lattice [1]. The positions are octahedral of the types $(\frac{1}{2}, 0, 0)$ and $(\frac{1}{2}, \frac{1}{2}, 0)$. The ensuing strain tensor has local tetragonal symmetry with one of the principal axes coinciding with one of the three main directions $\langle 100 \rangle$ of the b.c.c. matrix [5].

In the present work, the internal friction was measured as a function of temperature in an Nb–48wt.%Ti alloy in two different conditions: (A) annealed and (B) annealed with subsequent treatment in a nitrogen atmosphere (obtained from gas with a purity of 99.999% containing a few parts per million by weight of impurities such as H_2O, O_2, CO_2 and CO).

2. Experimental procedure

The Nb–48wt.%Ti alloy was obtained by electron-beam zone melting and was supplied in the form of swaged rods of 8 mm in diameter. Part of each rod was milled to a rectangular bar of 0.1 mm thickness. The samples used in this work were obtained from the rectangular portions. Sample A was annealed by direct heating at 1070 K for 150 min in a vacuum at a pressure of about 1×10^{-7} mbar. Sample B, in addition to the latter treatment, was heated to 1070 K for 20 min in nitrogen at a partial pressure of 1×10^{-5} mbar. Samples (length, 40 mm), which had been chemically polished to 1.2 mm thickness in a mixture of nitric and fluoric acids, were used for the internal friction measurements and also for lattice parameter determination by X-ray diffraction (powder method).

The internal friction values were obtained in the temperature range 250–700 K, using a torsion pendulum of the inverted Kê type [6], with a heating rate of 1 K min^{-1}, a pressure near 10^{-3} mbar and an oscillation frequency of about 3.5 Hz. The data of the oscillation decay were collected automatically by two phototransistors connected to a computer. A laser beam was deflected by a mirror on the pendulum bar, and the phototransistors were placed side-by-side in the path

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TABLE 1. Chemical analysis of the impurities present in the Nb–48wt.%Ti alloys in the as-received condition

Impurity	Ta	Fe	Si	Ni	Cu	Al	O	N	C
Content (wt.-ppm)	<500	67	65	93	6	29	500	126	44

TABLE 2. Density, titanium content estimated by the density and lattice parameters of Nb–Ti alloys in the annealed (A) and nitrogenated (B) conditions

Parameter	Sample A	Sample B
Density (g cm^{-3})	5.98	
Ti content (wt.%)	48	
Lattice parameter (Å)	3.291	3.293

of the deflected beam near the centre of oscillation so that the beam transit time was inversely proportional to the amplitude of oscillation. The internal friction was obtained from the amplitude decay which was determined by the ratio of the two measured transit times [7].

The internal friction curves as a function of temperature were resolved into elemental Debye peaks [5] using the method of successive subtraction. The anelastic relaxation processes were identified, and the relevant parameters of each of the processes were obtained.

Table 1 shows the chemical analysis of the impurities present in the Nb–48wt.%Ti alloys in the as-received condition.

Table 2 shows the density, the amount of titanium estimated by the density [8] and the lattice parameters of the two samples used. The results indicate that there was no loss of Ti during heat treatment.

3. Results and discussion

X-Ray diffraction revealed only peaks related to the cubic structure in the thermally treated sample. However, in the sample treated in the nitrogen atmosphere, in addition to the cubic peaks, some low-intensity peaks were associated with a hexagonal structure. Unfortunately, it was not possible to calculate the lattice parameter of this phase.

Figure 1 shows the internal friction as a function of temperature for samples A and B.

The experimental relaxation spectra observed for samples A (annealed) and B (nitrogenated) were resolved into a series of constituent Debye peaks corresponding to different interactions. The decomposition of the spectra was made purely on a mathematical basis, but allowed the presence of interstitial residual gas to be detected and the effect of oxygen absorption

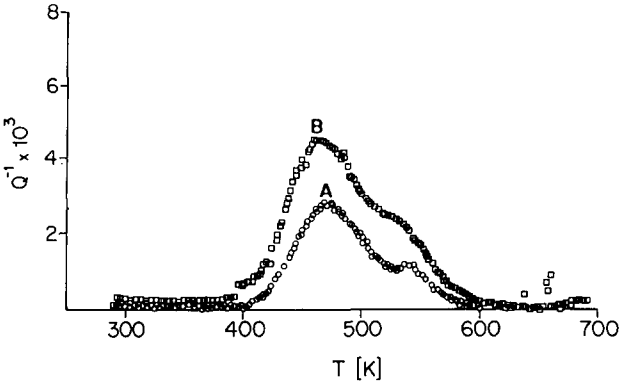

Fig. 1. Internal friction measurements as a function of temperature for samples A (annealed) and B (annealed and nitrogenated).

during nitrogen treatment to be determined. For each relaxation process, it was possible to obtain the activation energy E, the height Q_{max}^{-1} and the peak temperature T_p. It is evident from Table 3 that the four peaks for the annealed sample A (Fig. 2) confirm earlier investigations carried out with O and N in unalloyed Nb [9, 10] and in low-alloy Nb and Ti [11], although a polycrystalline Nb alloy containing 48 wt.% Ti was used in the present investigation. On the basis of this comparison, the four peaks in the resolved spectrum of specimen A were attributed to the interactions Nb–O, Ti–O, Ti–O–O and Nb–N.

The resolved relaxation spectrum for the nitrogenated sample B (Fig. 3) revealed six peaks. Here, the relaxation parameters were calculated using a successive subtraction method. The broken curve is the sum of the individual Debye curves and coincides well with the experimental data.

The various matrix–interstitial interactions which appear in specimens A and B, *i.e.* Nb–O, Nb–N, Ti–O and Ti–O–O, and the interaction Nb–O–O in sample B have identical peak temperatures, and their activation energies are very close to those presented in the literature. The basic difference between equivalent peaks lies in the peak heights, caused by the differences in O and N contents (Table 3).

The existence of the single additional peak in specimen B has not been observed previously. This peak was generated by computational analysis of the experimentally determined relaxation spectrum for specimen B. It is suggested that this peak is due to the Ti–O–O–O matrix–interstitial interaction for which the internal friction parameters are given in Table 3.

4. Conclusions

(1) Internal friction measurements as a function of temperature were performed for Nb–48wt.%Ti alloys containing different amounts of oxygen and nitrogen.

TABLE 3. Relaxation parameters for metal–interstitial interactions

Sample	Interaction	$Q_{max}^{-1} \times 10^3$	T_p (K)	E (kcal mol^{-1})	Composition
A	Nb–O	0.414	430	26.534	Nb–48wt.%Ti
	Nb–N	0.754	541	35.110	
	Ti–O	0.975	467	28.535	
	Ti–O–O	1.246	495	29.898	
B	Nb–O	1.123	430	26.503	Nb–48wt.%Ti
	Nb–O–O	1.017	443	27.930	
	Nb–N	0.868	541	35.110	
	Ti–O	2.933	467	28.420	
	Ti–O–O	1.397	495	29.860	
	Ti–O–O–O	1.033	523	32.710	
Ref. 9	Nb–O		428	26.800	Nb
	Nb–O–O		457	28.600	
	Nb–O–O–O		486	29.300	
Ref. 10	Nb–O		430	26.500	Nb
	Nb–O–O		443	28.000	
	Nb–N		562	34.900	
Ref. 11	Nb–O	1.3	421		Nb–2.7wt.%Ti
	Ti–O	3.8	463		
	Ti–O–O	2.7	500		

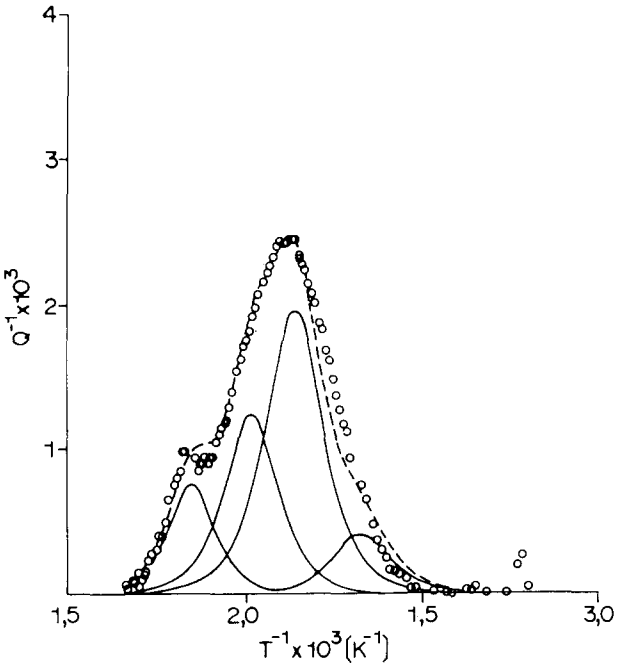

Fig. 2. Internal friction measurements as a function of temperature for sample A (annealed) of an Nb–48wt.%Ti alloy. The figure reveals four elemental Debye peaks attributed to the interstitial–metal interactions listed in Table 3.

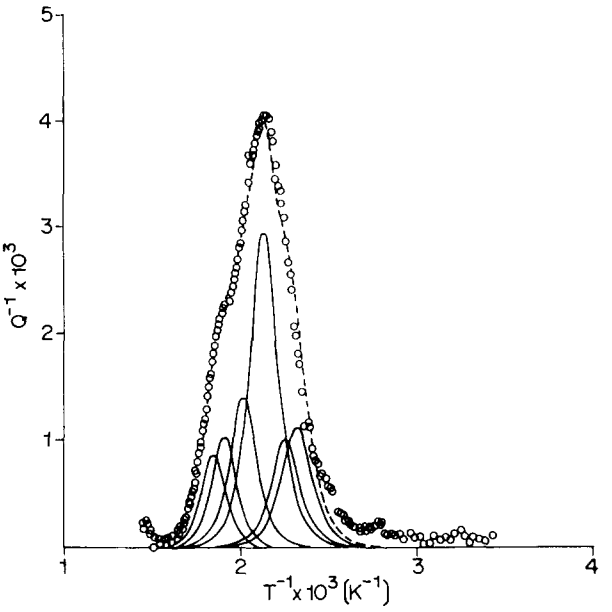

Fig. 3. Internal friction measurements as a function of temperature for sample B (annealed and nitrogenated) of an Nb–48wt.%Ti alloy. The figure reveals six elemental Debye peaks attributed to the interstitial–metal interactions listed in Table 3 and discussed in the text.

(2) The spectra of multiple anelastic relaxation were resolved into elemental interactions and the following metal–interstitial interactions were identified: Nb–O (430 K), Nb–O–O (443 K), Nb–N (541 K), Ti–O (467 K), Ti–O–O (495 K) and Ti–O–O–O (523 K).

(3) The existence of the Ti–O–O–O peak has not been observed previously. However, the peaks corresponding to the other interactions are in good agreement with the literature in terms of the anelastic relaxation parameter, temperature and activation energy.

(4) The different heights of the internal friction peaks of equivalent interaction processes are caused by different amounts of either oxygen or nitrogen dissolved interstitially in the metallic matrix.

(5) The results of this work demonstrate that the spectrum of multiple anelastic relaxation can be resolved into Debye's elemental peaks for an alloy.

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