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Internal Friction in Vanadium After Heating at Low Air Pressure

Edington and Smallman [1] have studied, using electron microscopy techniques, polycrystalline samples of vanadium after heating at low air pressures. They report the existence of a body-centred tetragonal phase similar to the β-phase with a high concentration of twins. They also observed, on cooling from 1300° C, a martensitic transformation.

During the preparation of polycrystalline vanadium samples with different amounts of oxygen, intended for internal friction studies, we observed the presence of microstructures which are similar to those, reported by Edington and Smallman [1], and which were obtained after heating for 10 to 24 h in an air pressure of 5 × 10^{-3} torr.

Fig. 1 shows the experimental arrangement. A wire, approximately 60 cm in length, was heated by passing an electric current through it. After annealing for 10 to 24 h at 1300 to 1400° C it was quenched by turning off the current. The temperature was measured with an optical pyrometer in the region marked with X in the figure. Then the samples were electropolished with a solution containing 10% perchloric acid and 90% butyl cellosolve with an applied voltage of 20 V and continuous stirring.

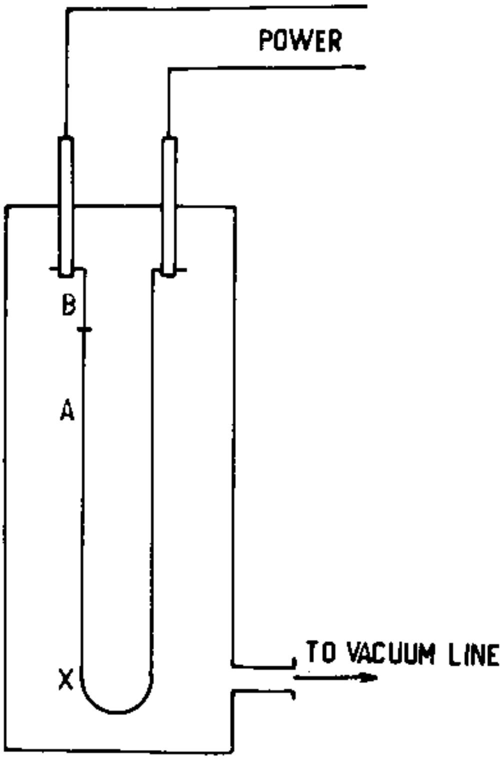

Figure 1 Experimental arrangement for preparation of the samples.

Replicas of the wires were obtained by following the method described by Jacquet [2].

Wires annealed at 900° C show an equiaxed grain structure with precipitates in the matrix (fig. 2). In wires annealed at 1300 to 1400° C two different structures were observed. Fig. 3 corresponds to region A of fig. 1 and is characterised by a high concentration of twins. Fig. 4 shows the region B where martensite-like needles are present. In fig. 5 an intermediate region between A and B is presented showing a mixture of twins and martensite-like needles.

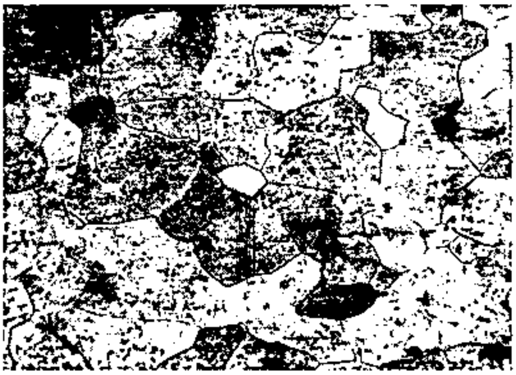

Figure 2 Microstructure of a wire annealed at 900° C showing an equiaxial grain structure with precipitates in the matrix (× 220).

Figure 3 Microstructure of a wire from region A (fig. 1) where a high concentration of twins is observed (× 220).

According to the literature [3, 4] the picture of region A would correspond to the β-phase of the system vanadium oxygen which has an ordered body-centred tetragonal structure and exists, below 1270° C, in the composition range from 15 to 22 at. % oxygen. The bounded structures are characteristic of the lower oxygen limits of the phase field. The observed martensite-like transformation product would support the

Figure 4 Microstructure of a wire from region B (fig. 1) showing the martensitic-like needles (× 110).

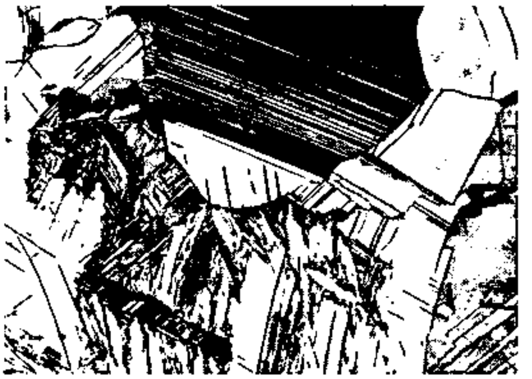

Figure 5 Microstructure of an intermediate region between A and B where both structures are observed (× 220).

conclusion of Seybolt and Sumsion [4] about the existence of a phase stable at high temperature due to the presence of impurities.

The measurements of internal friction and squared frequency (which is proportional to the modulus) between room temperature and 550° C, carried out using an inverted torsion pendulum, show:

(a) In fig. 6 the two internal friction peaks corresponding to oxygen and nitrogen are observed around 170° C and 260° C in the "as received" sample. From the height of the maximum, the concentrations are deduced to be 0.19 at. % and 0.12 at. % for oxygen and nitrogen respectively.

(b) In fig. 7, which corresponds to samples from the A region of fig. 1, a new internal friction peak appears at approximately 390° C for a frequency at room temperature of 0.72 c/s, with the following features:

(i) Its activation energy ΔH, determined by measuring the shift in the temperature of the maximum which accompanies a variation in frequency, is 51000 ± 5000 cal/mol (fig. 8).

(ii) The half-width of the peak does not correspond to a process with a single relaxation time and the observed τ_0 is 4.8×10^{-17} sec.

(iii) The modulus relaxation is about 20% (fig. 7).

In order to explain the observed peak, two possible mechanisms are discussed: (a) stress-induced interstitial ordering, and (b) movement of twin interfaces.

The peak, as mentioned before, does not have a single relaxation time. The measured value for ΔH is too large and it seems unreasonable to associate it with interstitial diffusion. In addition, the modulus relaxation is about three times larger than the value which would correspond to the observed internal friction maximum. For all these reasons we reject the first mechanism.

Internal friction due to stress relaxation at twin boundaries has been observed in pure metals and alloys [5, 6, 7]. Zener [8, 9] has shown how shear stress acting on a twin interface induces an anelastic effect. The twin interface adjusts its position so as to minimise the applied stress. As the stress changes, the continuous rearrangement of the twin interface produces anelastic effects characterised by a large relaxation of the modulus. In our case the ratio of relaxed to unrelaxed modulus is about 0.8, which agrees with values quoted by Zener [8].

The lack of a model able to explain quantitatively this mechanism of movement of twins makes this interpretation appear somehow speculative; however it is the most reasonable if one takes into account the abnormally high modulus relaxation which takes place at the maximum of internal friction.

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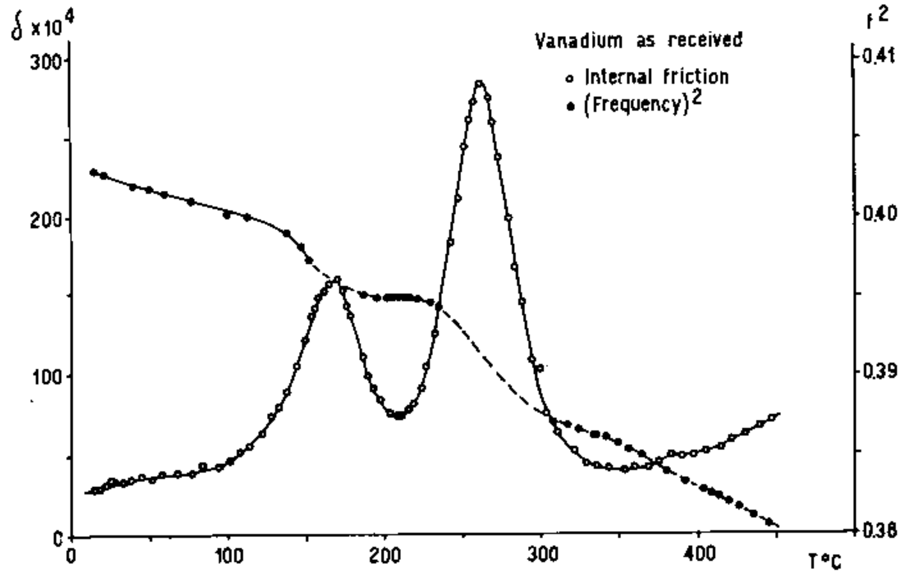

Figure 6 Internal friction and squared frequency of an as-received sample.

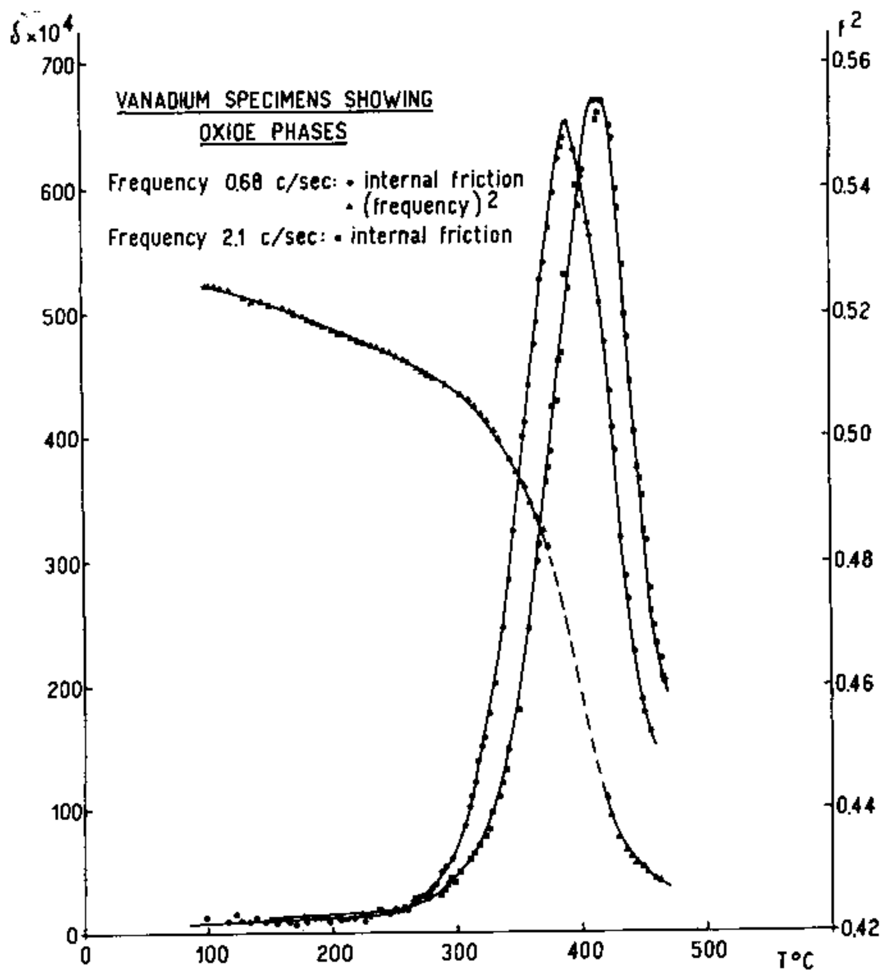

Figure 7 Internal friction and squared frequency of a wire from region A after the thermal treatment. The Snoek peaks due to oxygen and nitrogen have disappeared.

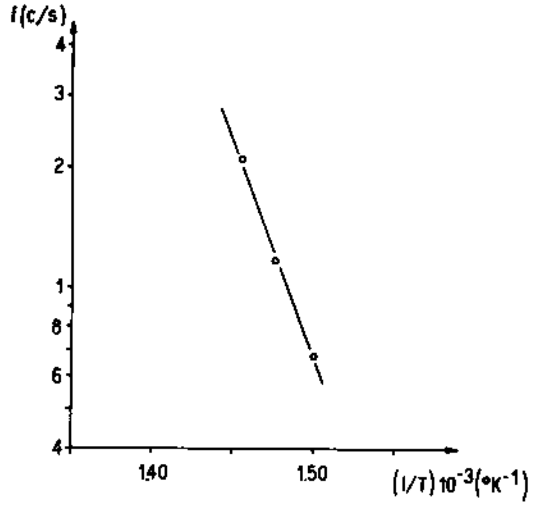

Figure 8 Plot of log f vs $1/T$ from where a value $H = (51\,000 \pm 5000)$ cal/mol is obtained. The quoted values for the frequencies correspond to the frequencies at the maximum of internal friction.

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