

TABLE II Analysis of the first five reflections of the alloy.

Reflection	$\beta = B - \frac{b^2}{B}$ rad. $\times 10^3$	$\eta = \frac{\lambda}{\beta \cos \theta}$ $\text{Å}$	$\epsilon = \frac{\beta}{4 \tan \theta}$ $\times 10^3$	Devia- tion from mean value of $\eta$ %	Devia- tion from mean value of $\epsilon$ %	$\beta' = B - b_0^2/B$ rad. $\times 10^3$	$\eta' = \frac{\lambda}{\beta' \cos \theta}$ $\text{Å}$	$\epsilon' = \frac{\beta'}{4 \tan \theta}$ $\times 10^3$	Devia- tion from mean value of $\eta'$ %	Devia- tion from mean value of $\epsilon'$ %
(111)	12.05	138	7.61	50	9	10.40	159	6.57	54	10
(200)	23.76	72	12.65	22	52	17.79	96	9.47	7	29
(220)	23.58	82	7.83	11	6	22.73	85	7.55	18	3
(311)	33.11	66	8.32	28	0	32.54	67	8.18	35	12
(222)	22.61	101	5.19	10	38	21.11	108	4.84	5	34
Mean value	—	92	8.32	—	—	—	103	7.32	—	—
Mean deviation from mean value	—	—	—	24	21	—	—	—	24	18

apparent domain size ( $\eta$ ,  $\eta'$ ) and apparent strain ( $\epsilon$ ,  $\epsilon'$ ) are calculated, assuming the entire deformation-broadening to be caused solely by either one of these factors. The mean deviation from the mean value is then calculated for both. If either one of these factors were to be preponderantly responsible for the broadening then the mean deviation from the mean value would be much less for this one than for the other [4]. In the present case it is seen that the mean deviation from the mean value is large in both cases and no appreciable variation is observed even after correcting for faults. This seems to indicate that both of these factors contribute in comparable amounts to the line-broadening.

Before concluding it may be worthwhile to compare our results with those of Welch and Otte [5]. The values quoted by these authors,  $\alpha = 0.025$ , domain sizes and strains along  $\langle 111 \rangle$  and  $\langle 100 \rangle = 142 \text{ Å}$ ,  $92 \text{ Å}$ ,  $0.003$  and  $0.006$  respectively, without correcting for stacking fault-broadening, agree reasonably well with our own values of  $\alpha = 0.022$ ,  $163 \text{ Å}$ ,  $105 \text{ Å}$ ,  $0.004$  and  $0.009$  respectively, for work of this nature.

**Acknowledgement**

The author is thankful to the Alexander von

Humboldt Stiftung for the grant of a research fellowship.

**References**

1. M. S. PATERSON, *J. Appl. Phys.* **23** (1952) 805.
2. J. B. COHEN and C. N. J. WAGNER, *ibid* **33** (1962) 2073.
3. B. E. WARREN and B. L. AVERBACH, *ibid* **21** (1950) 595.
4. P. RAMA RAO and T. R. ANANTHARAMAN, *Z. Metallk.* **54** (1963) 658.
5. D. O. WELCH and H. M. OTTE, "Advances in X-ray Analysis", Vol. 6, edited by Mueller, Mallett and Fay (Plenum Press, New York, 1964) p. 96.
6. T. R. ANANTHARAMAN and J. W. CHRISTIAN, *Acta Cryst.* **9** (1956) 479.
7. T. R. ANANTHARAMAN, "Golden Jubilee Research Volume" (Indian Institute of Science, Bangalore, 1959) p. 280.

25 August 1969

R. VASUDEVAN  
*Institut für Metallkunde  
 Max-Planck-Institut für  
 Metallforschung  
 Stuttgart, W. Germany*

*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  $\beta$ -phase with a high concentration of twins. They also observed, on cooling from  $1300^\circ \text{C}$ , 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  $5 \times 10^{-3}$  torr.

Fig. 1 shows the experimental arrangement. A wire, approximately 60 cm in length, was heated by passing an electric current through it. After

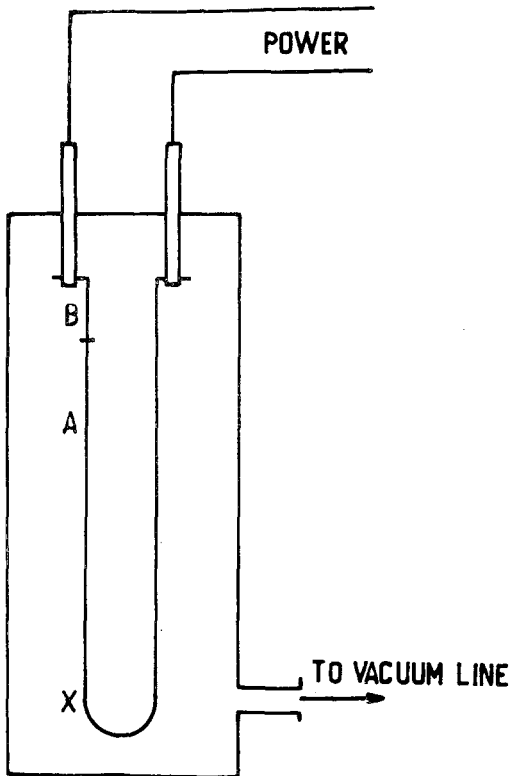


Figure 1 Experimental arrangement for preparation of the samples.

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.

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



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



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

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.

According to the literature [3, 4] the picture of region A would correspond to the  $\beta$ -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 ( $\times 110$ ).

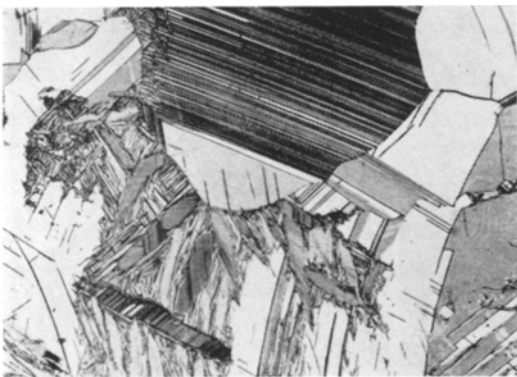


Figure 5 Microstructure of an intermediate region between A and B where both structures are observed ( $\times 220$ ).

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  $550^{\circ}\text{C}$ , 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  $170^{\circ}\text{C}$  and  $260^{\circ}\text{C}$  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  $390^{\circ}\text{C}$  for a frequency

at room temperature of 0.72 c/s, with the following features:

(i) Its activation energy  $\Delta H$ , determined by measuring the shift in the temperature of the maximum which accompanies a variation in frequency, is  $51000 \pm 5000$  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  $\tau_0$  is  $4.8 \times 10^{-17}$  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  $\Delta H$  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.

## References

1. J. W. EDINGTON and R. E. SMALLMAN, *Acta Met.* **13** (1955) 155.
2. JACQUET, *Rev. de Met.* **55** (1958) 531.
3. W. ROSTOKER, "The Metallurgy of Vanadium" (J. Wiley Publ., New York); W. ROSTOKER and A. S. YAMAMOTO, *Trans. AIME* **47** (1955) 1002.
4. A. U. SEYBOLT and H. T. SUMSION, *ibid* **5** (1953) 292.
5. L. A. CHIRKINA, *Sov. Phys. Solid State* **10** (1968) 735.
6. R. J. GOODWIN, *Met. Sci. J.* **2** (1968) 121.

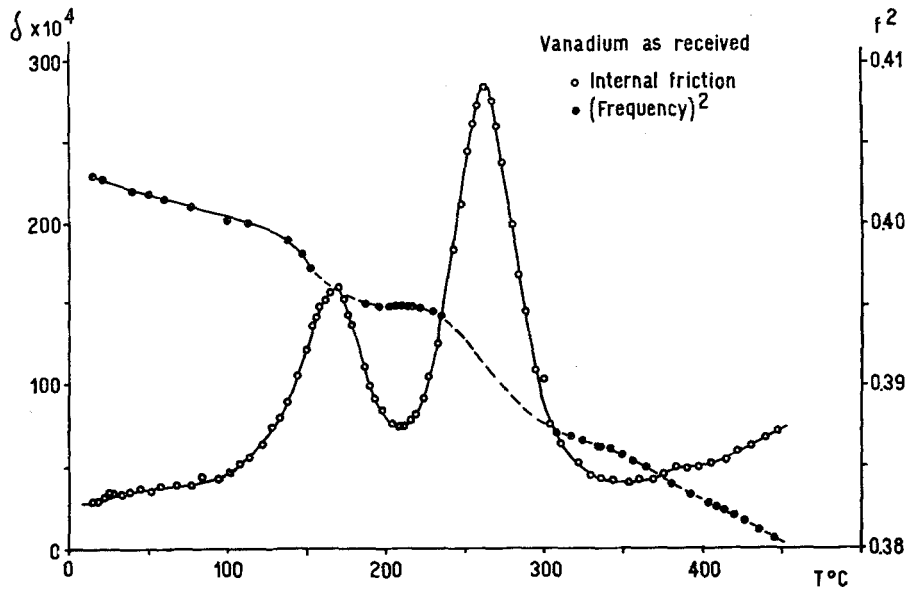


Figure 6 Internal friction and squared frequency of an as-received sample.

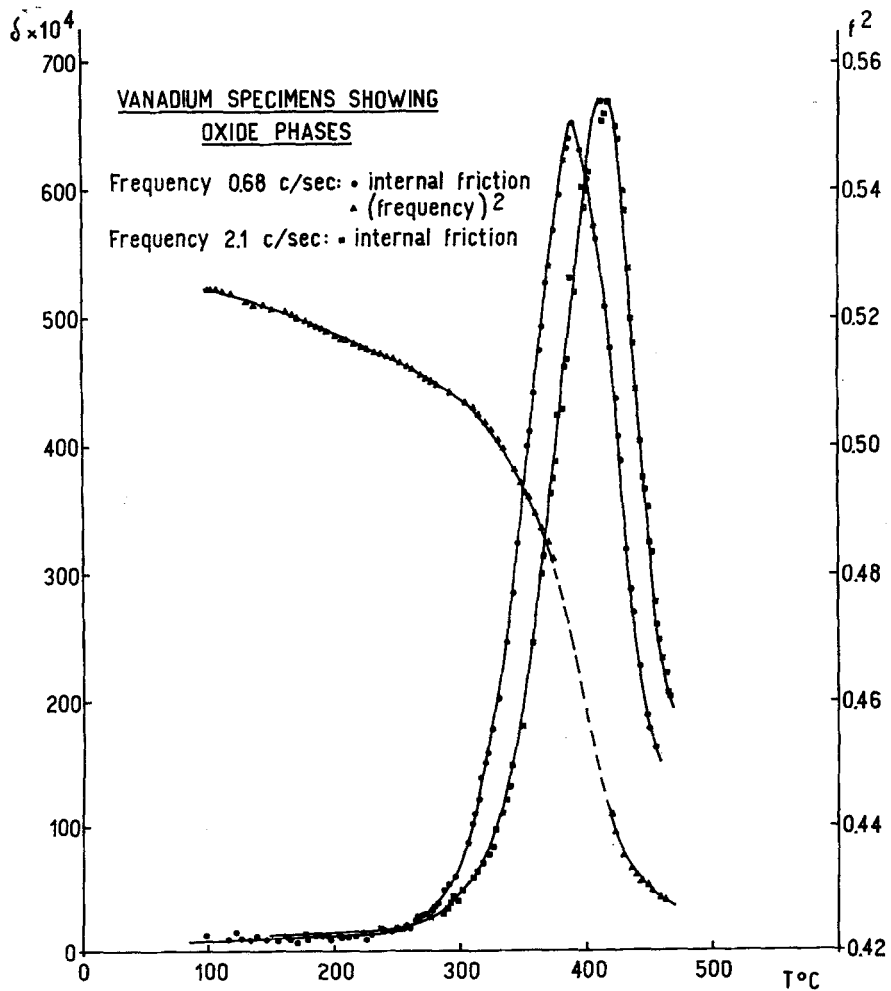


Figure 7 Internal friction and squared frequency of a wire from region A after the thermal treatment. The Snoek peaks due to oxygen and nitrogen have disappeared.

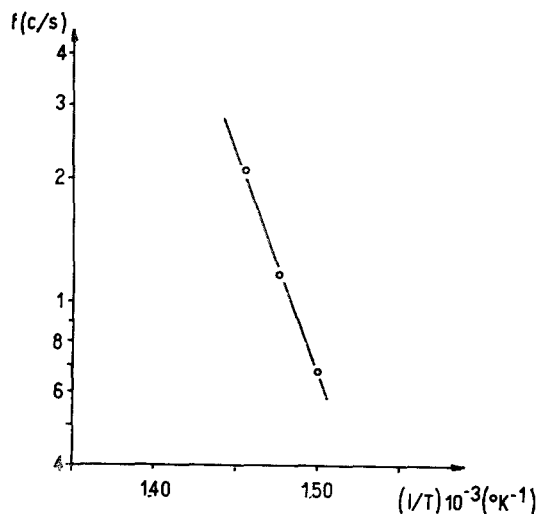


Figure 8 Plot of  $\log f$  vs  $1/T$  from where a value  $H = (51\ 000 \pm 5000)$  cal/mol is obtained. The quoted values for the frequencies correspond to the frequencies at the maximum of internal friction.

\*Departamento de Metalurgia, Comisión Nacional de Energía Atómica, Buenos Aires, Argentina.

7. F. R. WORRELL and A. V. SEIFERT, *J. Appl. Phys.* **22** (1951) 1257.
8. C. ZENER, "Elasticity and Anelasticity of Metals" (University of Chicago Press, 1948).
9. *Idem*, *Nuovo Cimento Supp.* Vol. VII (1958).

30 April 1969

M. A. MONDINO  
D. VASSALLO\*

M. C. DE ACHTERBERG  
*Comisión Nacional de Energía Atómica*  
*Universidad Nacional de Cuyo*  
*San Carlos de Bariloche - Río Negro*  
*Argentina*

## Book Review

### Proceedings of the British Ceramic Society No. 12, Fabrication Science, 2

Pp. 255 (Published by the Society, 1969) 90s

The No. 12 Proceedings of the British Ceramic Society comprises 20 papers which were presented at a meeting of the Basic Science Section of the Society held at the University of Leeds on 18-20 September 1967. The first paper points out how much is still to be learned about fabrication processes for ceramics and emphasises that it is impossible to design and produce a ceramic body without resorting to a considerable amount of empirical data. The remaining papers emphasise the validity of this viewpoint. The papers are almost entirely concerned with reporting the results of practical experiments and very little theoretical work is directly included. The fabrication techniques which receive most attention are those of sintering and hot-pressing but other

techniques such as chemical bonding, vibration pressing, electrophoretic deposition and vapour deposition are also described. The majority of the work described has been on pure oxide materials e.g.  $Al_2O_3$ , MgO, BeO, ZnO etc. but reference is also made to sulphide, carbide, halide and phosphide materials.

Although individual papers are only concerned with work along narrow lines, the overall effect of the book is to give a fairly good picture of the type of work being carried out at present on ceramic fabrication techniques and the problems facing ceramic technologists in developing satisfactory bodies. In addition the majority of the papers give numerous references to previous work on the various topics and this in itself is very useful.

In general the book represents a useful addition to the literature available to workers wrestling with these ceramic fabrication processes.

G. PARTRIDGE