

averaged 1.53 MN m^{-2} . This is in satisfactory agreement with $\tau = 1.72 \text{ MN m}^{-2}$ for a specimen with $\theta = 12^\circ$ [3].

The appearance of specimens after testing was consistent with the above description. For example, in a specimen with $\theta = 5.4^\circ$ the zone of deformation is readily detected (Fig. 3); scribes on the gauge section are characteristically curved at the edges of the slipped zone. Elongation, such as that shown in Fig. 3, took place entirely by thickness reduction, with no change in specimen width. Such behaviour is consistent with the attribution of deformation to basal plane shear processes. A specimen with $\theta = 7.1^\circ$ was pulled to failure, which as expected occurred by shearing off of basal layers in the deformation zone. True strain at failure was 210%.

In summary, the technique of cutting tensile specimens at an angle to the basal planes produces extensive basal shear in pyrolytic graphite at 3035 K. For the specimen geometry described, the angle θ must exceed about 7° for this observation to be made. The resolved shear stress for basal flow was observed to be about 1.5 MN m^{-2} , and the evidence of Figs. 2 and 3 would be consistent with the suggestion [3, 4, 7] that this shear occurs by glide of basal dislocations, assisted by diffusional processes.

Acknowledgements

This work was performed while the author was

associated with the Jet Propulsion Laboratory, California Institute of Technology, Pasadena, California, under the auspices of the National Aeronautics and Space Administration, Contract NAS7-100. Experimental assistance of L. Gnewuch, and helpful discussions with D. B. Fischbach, W. V. Kotlensky and H. E. Martens, are gratefully acknowledged.

References

1. O. J. GUENTERT, *J. Chem. Phys.* **37** (1962) 884.
2. E. R. STOVER, "High Temperature Materials II" (Interscience, New York, 1963) p. 437.
3. D. B. FISCHBACH and W. V. KOTLENSKY, *Electrochem. Tech.* **5** (1967) 207.
4. D. B. FISCHBACH, *Phil. Mag.* **21** (1970) 1.
5. W. V. KOTLENSKY and H. E. MARTENS, "Proceedings of the 5th Conference on Carbon" (Pergamon, New York, 1963) p. 625.
6. *Idem*, *J. Amer. Ceram. Soc.* **48** (1965) 135.
7. W. V. KOTLENSKY, A. W. THOMPSON and K. H. TITUS, presented at the 6th Conference on Carbon, Pittsburgh, Pennsylvania, June 1963.
8. W. V. KOTLENSKY and H. E. MARTENS, "High Temperature Materials II" (Interscience, New York, 1963) p. 403.

Received 24 May
and accepted 3 June 1974

ANTHONY W. THOMPSON
Science Center, Rockwell International,
Thousands Oaks, California, USA

Contribution of dissolved or precipitated oxygen to the electrical resistivity of vanadium

It is widely said that the so-called stage III "recovery" of body-centred cubic metals originates in the existence of impurity interstitials [1-5], although some investigators have pointed out the importance of the role of intrinsic defects produced by irradiation or cold-work [5, 6]. Recently, for neutron-irradiated vanadium, Stanley *et al.* [4] have concluded that the decrease in resistivity of $3.2 \mu\Omega\text{-cm (at. \%)}^{-1}$ after annealing results from the precipitation of impurity interstitials at the radiation-produced defects. In their experiments, this value was about 36% of the resistivity increase caused by the dissolution of 1 at. % of impurity interstitials. From this fact, it is expected that in the case of cold-worked specimen, the decrease in resistivity due to ageing or annealing treatments

must also considerably differ in amount from the increase due to dissolution of the oxygen atom. The purpose of this paper is to investigate the difference in the resistivity changes caused by dissolved and strain-aged oxygen in vanadium.

The starting material was electron-beam melted polycrystalline vanadium. Chemical analysis showed that gas impurity content of the material was as follows: oxygen 310 to 2000; nitrogen 15; carbon 130; hydrogen 5 (ppm by weight). Wire specimens, 0.5 to 0.8 mm in diameter, were prepared using grooved rolls and a drawing machine. All the specimens were annealed at 900°C for 2 h in a vacuum of 2×10^{-6} mm Hg. Electrical resistivity was measured at liquid nitrogen temperature using a potentiometer. A measurement of the Snoek damping was made in a torsion pendulum at about 1 Hz with a strain amplitude of about 7×10^{-5} . Defects were introduced by cold-drawing for the ageing experiments.

In the present study, the relationship between the height of the oxygen Snoek damping, Q_{\max}^{-1} and the results of chemical analysis, C , was represented by the equation,

$$Q_{\max}^{-1} = 2.11 \times 10^{-1} C(\text{wt. } \%) \quad (1)$$

This is in good agreement with the results obtained by former investigators [4, 7-9], but not those of Powers and Doyle [10]. Plots of the specific resistivity, ρ , at liquid nitrogen temperature, versus Q_{\max}^{-1} gave the equation,

$$\rho(\mu\Omega\text{-cm}) = 2.90 + 1.00 \times 10^2 Q_{\max}^{-1} \quad (2)$$

From Equations 1 and 2,

$$\rho = 2.90 + 2.09 \times 10C \quad (3)$$

Equation 3 shows that the increase in resistivity due to dissolution of 1 at. % of oxygen is 6.42 $\mu\Omega\text{-cm}$. This is reasonable in view of the results obtained by other workers [4, 7].

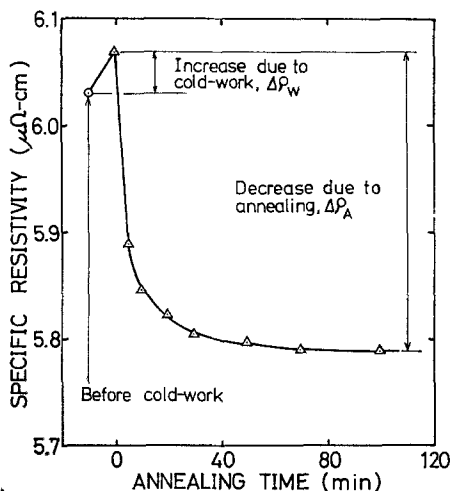


Figure 1 An example of resistivity changes caused by cold-drawing and subsequent annealing at 230°C.

An example of the resistivity increase due to cold-drawing, $\Delta\rho_w$ and the decrease due to subsequent annealing at 230°C, $\Delta\rho_A$ is shown in Fig. 1, where the reduction in area of the specimen was about 16%. Plots of $\Delta\rho_A$ versus the decrease in the oxygen Snoek damping due to annealing at 230°C for 100 min are shown in Fig. 2, where the values of $\Delta\rho_A - \Delta\rho_w$ are also plotted in relation to the change in the oxygen Snoek damping. From the results shown in this figure, we obtain

$$\Delta\rho_A(\mu\Omega\text{-cm}) = 2.19 \times 10\Delta Q_{\max}^{-1} \quad (4)$$

with the assumption that the decrease in resistivity is determined only by the amount of oxygen atoms which go out of solution.

It is believed, from a comparison of Equation

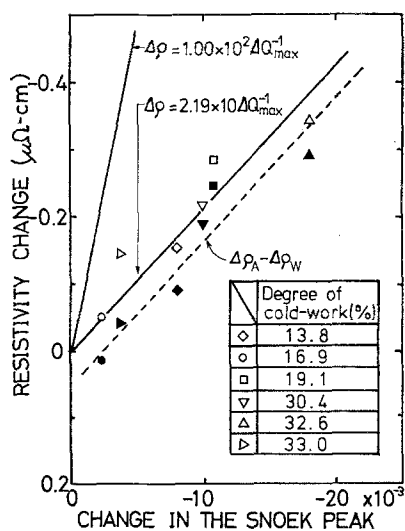


Figure 2 Plots of the change in resistivity versus that in the oxygen Snoek damping caused by annealing at 230°C for 100 min.

3 with Equation 4, that the magnitude of resistivity decrease due to precipitation of oxygen to, probably, dislocations [5] is about 22% of the amount of increase due to dissolution of oxygen. Since $\Delta\rho_A$ may include the change due to annealing of defects produced by cold-work, the magnitude of the decrease in resistivity due to precipitation of oxygen is possibly smaller than that represented by Equation 4. The dotted line in Fig. 2 which is fitted to the points, $\Delta\rho_A - \Delta\rho_w$, appears to show that the value $d(\Delta\rho_A - \Delta\rho_w)/d(\Delta Q_{\max}^{-1})$ is almost the same as $d(\Delta\rho_A)/d(\Delta Q_{\max}^{-1})$ which can be calculated from Equation 4. Thus, the magnitude of the decrease in resistivity due to the precipitation of oxygen is believed to be about one-fifth of the increase due to dissolution. This suggests that any recovery of defects produced by cold-work is not large. In fact, high-purity specimens have shown almost no recovery at annealing temperatures of 100 to 300°C [3, 4]. In the present experiments, the effect of precipitation of carbon, nitrogen and hydrogen is neglected, but if such an effect is taken into account, the resistivity decrease per at. % of oxygen precipitated would be smaller than that calculated from Equation 4.

Thus, it is concluded that the resistivity decrease due to precipitation of oxygen at dislocations is about 1.43 $\mu\Omega\text{-cm}(\text{at. } \%)^{-1}$, which is smaller than that of the increase caused by dissolved oxygen.

References

1. J. M. WILLIAMS, J. T. STANLEY and W. E. BRUNDAGE, USAEC Report, ORNL-4097 (1967).
2. A. KÖTHE and F. SCHLÄT, *Phys. Stat. Sol.* **21** (1967) K73.
3. *Idem*, *J. Mater. Sci.* **2** (1967) 201.
4. J. T. STANLEY, J. M. WILLIAMS, W. E. BRUNDAGE and M. S. WECHSLER, *Acta Met.* **20** (1972) 191.
5. M. ETO, *J. Mater. Sci.* **7** (1972) 567.
6. J. H. PEREPEZKO, R. F. MURPHY and A. A. JOHNSON, *Phil. Mag.* **19** (1969) 1.
7. G. HÖRZ, *Z. Metallkde* **61** (1970) 371.
8. G. H. KEITH and H. G. IVERSIN, US Bureau of Mines RI 7222 (1969).
9. D. F. HASSON and R. J. ARSENAULT, *Scripta Met.* **5** (1971) 75.
10. R. W. POWERS and M. V. DOYLE, *J. Appl. Phys.* **30** (1959) 514.

Received 18 June
and accepted 2 July 1974

MOTOKUNI ETO
Japan Atomic Energy Research Institute,
Tokai-mura, Ibaraki-ken 319-11,
Japan
TETSU NARUTANI
Department of Nuclear Engineering,
University of Tokyo, Tokyo 113,
Japan

V_3Ga growth kinetics in vanadium/gallium-copper composites

V_3Ga is a β -W type superconducting compound with a high transition temperature, a high upper critical field and capable of carrying a high current density. Consequently, its production and properties have received considerable study during the past few years [1-5]. An important manufacturing process relies on the selective diffusion of gallium into vanadium from a gallium-copper alloy. In the process, a composite of vanadium and gallium-copper is fabricated as a tape or filamentary wire. The composite is mechanically deformed to the desired final size. A heat-treatment in the temperature range 575 to 800°C produces a V_3Ga layer at the interface between the vanadium and the gallium-copper. The purpose of the present paper is to show that a single equation can reproduce the growth rate of the V_3Ga layer at different reaction temperatures and for different compositions of the gallium-copper alloy.

The experimental data used for the basis of the analysis are taken from: Suenaga and Sampson [1] who used three vanadium rods embedded in a nominal 15 at. % gallium-copper matrix; Tachikawa *et al.* [2, 3] whose samples were strips of vanadium rolled in a 20 at. % gallium-copper sheath; Howe *et al.* [4] using a single vanadium filament in a 15.4 at. % gallium-copper sleeve; and Critchlow *et al.* [5] whose sample was a multifilamentary vanadium composite having a 14 at. % gallium-copper matrix. The growth rate data given by Suenaga and Sampson agree very closely to those of Critchlow *et al.*, and in the analysis it has been assumed that both sets of samples had the same composition, namely 14 at. % gallium.

In order to make comparisons between the superconducting properties of the V_3Ga layers, as produced by different workers, it has been customary to use data for a 1 μ m thickness. The reaction time, at different temperatures, required to produce this thickness is frequently reported. In the case of Tachikawa *et al.* [2], however, it was necessary to extrapolate the data given for the production of thicker layers to determine the time to grow a 1 μ m layer.

The basic equations governing the rate of growth of a diffusion layer are:

$$x^2 = kt \quad (1)$$

where x is the layer thickness, t the reaction time and k is proportional to the diffusion coefficient [6], and

$$k = k_0 \exp(-Q/RT) \quad (2)$$

where Q is the activation energy, R the gas constant and T the absolute temperature. Equation 1 is usually derived for a planar system. Its applicability to cylindrical geometries where the V_3Ga layer is small has been verified [4, 7]. In some of the cases where the equation fails to hold, there is a significant reduction of gallium concentration as the reaction proceeds. Equation 2 is the well-known Arrhenius equation.

Substituting Equation 1 into Equation 2, we get for the time to grow a 1 μ m thick V_3Ga layer

$$t_1 = 10^{-8} k_0^{-1} \exp(Q/RT). \quad (3)$$

Plots of $\log t$ versus $1/T$ (see Fig. 1) show that the data under study satisfy Equation 3 reasonably well. The value of the activation energy is, however, dependent on the concentration of gallium available for diffusion. Critchlow *et al.* have shown that t_1 is, in fact, an exponential