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GRAIN GROWTH IN COPPER

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Grain growth in 4N copper has been studied in rods of 1 cm diameter sealed in 13 kPa of hydrogen at 973, 1023 and 1073 K. The data are well encompassed by the relation $D^2 - D_0^2 = Kt \exp(-H/kT)$, with H, the activation energy for grain-boundary self-diffusion, equal to 0.87 eV, and $K = 1.6 \times 10^{-2}$ cm^2 sec^{-1}. The form of the D/t relation and the experimental values of H and K are in good agreement with the requirements of the theory of grain growth. However, the grain size of 5N copper was found to decrease, rather than increase, with increasing time of isothermal annealing, due to hydrogen uptake.

1. Introduction

In a recent paper Butt and Feltham[1)] have shown that isothermal grain-growth in α-brasses containing 10, 15, 20 and 30 at. % zinc at 973—1073 K is well correlated by the equation

$$D^2 - D_0^2 = K\,t \exp(-H/k\,T), \qquad (1)$$

with the stricture that the ratio of the specimen diameter to the final mean grain-diameter attained during isothermal anneal should not exceed about twenty, to avoid the slowing-down of grain-growth due to size-effect. Here D_0 and D are the initial and the instantaneous mean grain-diameters, respectively, t the time of isothermal grain-growth at the temperature T, and H the activation energy for grain-boundary self-diffusion. The parameters K and H were found to be functions of the concentration c of the alloying element in accord with the requirements of the statistical model[2,3] of grain-growth. Theoretical considerations[2] suggest that $K = \lambda V G b^2/8h$, where V is the volume per atom, G the shear modulus, b the lattice parameter, h Planck's constant, and λ is a constant of the order of unity. For copper, with $V = 12 \times 10^{-30}$ m^3, $b = 2.5 \times 10^{-10}$ m, $G = 4.5 \times 10^4$ MNm^{-2} and $\lambda = 1$, one finds $K \approx 7 \times 10^{-2}$ cm^2 sec^{-1}. Also the activation energy for grain boundary self-diffusion, involved in grain growth, would be somewhat less than the activation energy for vacancy migration in copper[3]. The latter, as given in the literature, is close to 1 eV. The main aim of this work was to examine the validity of equation (1) in conjunction with the values of K and H referred to for copper, i. e. to complement the data on Cu-Zn solid-solutions[1] for the case of zero zinc content.

Further, it is evident from the literature, that heat treatment of metals and alloys in a hydrogen atmosphere can change their mechanical response appreciably. Pishko et al.[4] have recently observed that the ultimate tensile strength of various steels was a function of the time of exposure to hydrogen at 723 K and 6.5 MPa. Feltham and Evans[5] found that in 5N copper single crystals creep between 773 and 1143 K was also significantly influenced by hydrogen uptake. To study the effect of hydrogen on grain-growth in copper, with purity as a variable, was another of the present objectives.

2. *Experimental*

The polycrystalline copper of 4N and of 5N purity used was supplied by *Metals Research Limited, Royston, England* in the form of cylindrical rods of 1 cm diameter. Specimens, 2 cm long, were cut from the *as received* rods by means of a fine saw. The 4N copper specimens were grouped into twelve batches, and each batch was sealed into a separate silica tube filled with hydrogen at 13 kPa. They were then annealed for 120 min at 523 K to minimise internal stresses, before the subsequent isothermal grain-growth extending over periods of 15 to 120 min at 973, 1023 and 1073 K. Mean grain-diameters (Fig. 1) were obtained by the linear-intercept method.

Grain-growth was also studied at 1073 K in 5N copper. In this purer metal, the initial mean grain-diameter D_0 was relatively large i. e. 0.9 mm. It was found to decrease, rather than increase, with annealing time t (Fig. 3). This anomaly seems to arise from the high sensitivity of the grain-growth rate in the 5N metal to the presence of hydrogen.

3. *Results and discussion*

The experimental results are shown in Figs. 1—3. A linear relationship between the square of the instantaneous mean grain-diameter, D^2, and the annealing time, t, at each temperature of isothermal grain-growth in 4N copper is evident from

Fig. 1. This is consistent with the functional form of equation (1). The straight line in Fig. 2, derived from the slope of the isotherms (Fig. 1), yields 0.87 eV for the value of the activation energy, H, in excellent agreement with that deduced by Butt and Feltham[1] ($H = 0.87$ eV) and by Feltham and Copley[3] ($H = 0.88$ eV), on extrapolating the experimental H-c data obtained with α-brasses to $c = 0$.

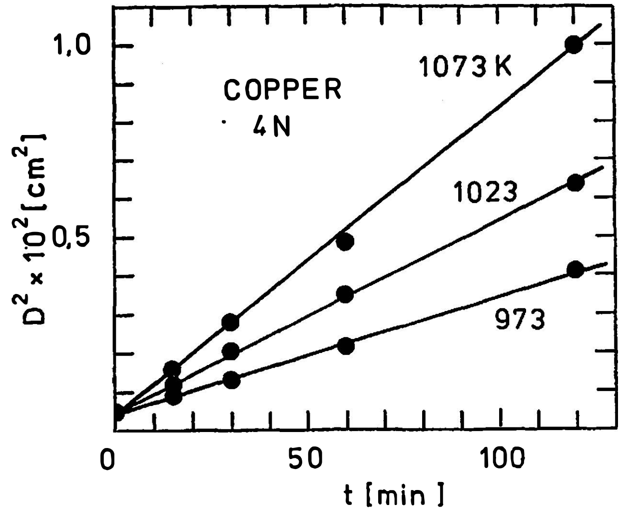

Fig. 1. Grain-growth isotherms of 4N copper.

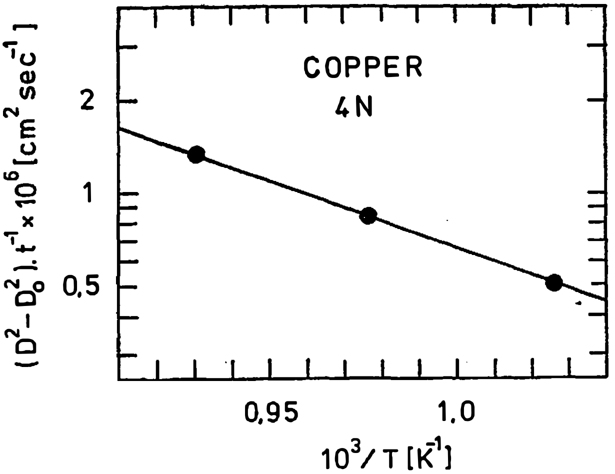

Fig. 2. Temperature dependence of the slope of the grain-growth isotherms of 4N copper referred to in Fig. 1.

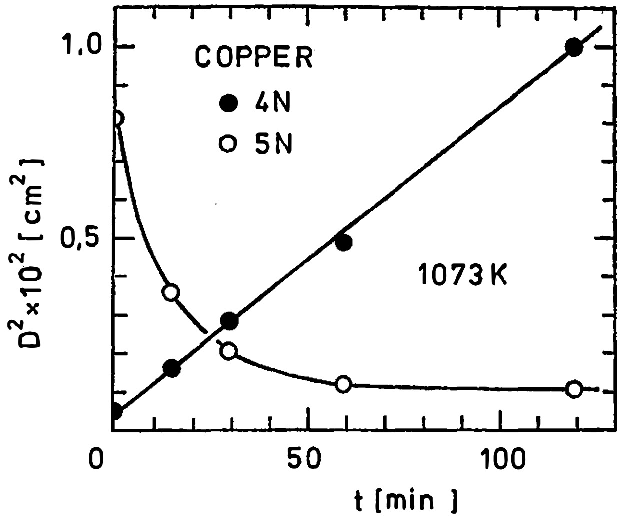

Fig. 3. Grain-growth data appertaining to 4N and 5N copper at 1073 K.

From this, and the data in Fig. 1, one readily finds, by means of equation (1), that $K = 1.6 \times 10^{-2}$ cm^2 sec^{-1}, which is of the right order of magnitude. It is therefore evident from the preceeding discussion that with appropriate values of the parameters K and H, equation (1) describes isothermal grain growth in 4N copper satisfactorily.

Fig. 3 shows the grain-growth data for the 5N copper obtained at 1073 K; those for 4N copper have been reproduced for comparison. The initial mean grain--diameter of the 5N copper ($D_0 = 0.9$ mm) can be seen to decrease, rather than increase, on isothermal annealing. Chemical polishing of some of the 5N copper specimens, for about 4 min at 323—328 K in a solution containing 25% (Vol.) H_3PO_4, 25% acetic acid and 50% conc. HNO_3, revealed numerious *craters* on the surface. This points to the formation of voids on the grain-boundaries which are due to attack by hydrogen[6]. The surface of the 4N copper specimens, which had undergone heat-treatment at 973—1073 K in a hydrogen atmosphere at 13 kPa, and were then chemically polished, was however quite smooth. Thus the anomalous grain-growth behaviour observed in 5N copper at 1073 K may probably be attributed to the diffusion of hydrogen to grain boundaries, which may be but little contaminated in high purity specimens. However, the problem was not investigated further within the framework of the present research.

4. Conclusion

The present results show that, firstly, isothermal grain-growth in 4N copper studied in the temperature range 973—1073 K is well encompassed by equation (1); that K and H have values of the order of magnitude expected and, secondly, that deviation from the normal grain-growth behaviour, observed in 5N copper at 1073 K, seems to arise from hydrogen occluded in the copper of high-purity.

Acknowledgments

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RAST ZRNA U BAKRU

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Istraživan je rast zrna u bakru, tako da su šipke bakra čistoće 4N, promjera 1 cm, zataljene u ampulama napunjenim vodikom, pri tlaku od 13 kPa i temperaturama 973, 1023 i 1073 K. Dobiveni podaci dobro se slažu s relacijom $D^2 - D_0^2 = K t \exp(-H/k T)$, gdje je H aktivacijska energija za samodifuziju u granici zrna jednaka 0,87 eV, a $K = 1,6 \times 10^{-2}$ cm^2 s^{-1}. D/t relacija i eksperimentalne vrijednosti od H i K dobro se slažu sa zahtjevima teorije rasta zrna. Ipak, razvoj zrna bakra čistoće 5N bio je takav da je veličina zrna opadala, umjesto da raste s rastućim vremenom izotermičkog žarenja, što se tumači difuzijom vodika u granice zrna.