

## X-RAY STUDY OF DEFECTS IN RAPIDLY QUENCHED ALUMINIUM

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Introduction

Defects in aluminium were examined using the small-angle X-ray scattering. Rapidly-quenched from the liquid, and cold-worked samples of pure aluminium were used. Absolute intensity measurements were carried out with a home-made Levelut-Guinier chamber (1), the axial symmetrical construction of which offers a great advantage in comparison with other small-angle scattering chambers because of its great sensibility. Some of the measurements were repeated with a Kratky chamber, where the intensity was measured only relatively, applying the film method.

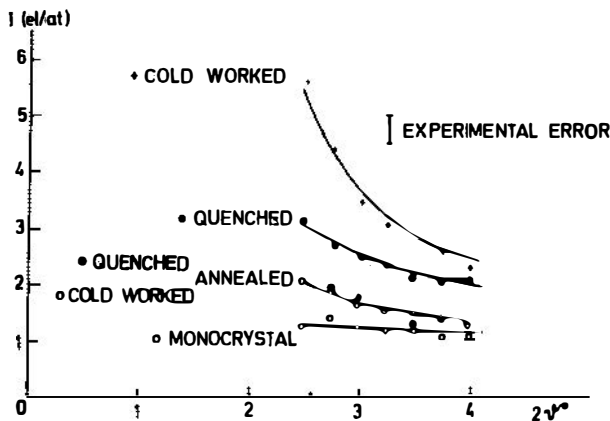
Experimental Procedure and Results

FIG. 1

The scattered intensities are given in Fig. 1. The aluminium samples were 99,997% pure and approximately  $55 \mu\text{m}$  thick. In order to check whether the results are reproducible, the measurements were carried out both on different and on identical samples. The results obtained over a period of several months

did not show any change. Samples of approximately equal quenching velocity were used. Corrections, which are necessary because of the unequal sensibility of the counter at different areas of scintillation crystal, were made. After annealing, the intensities of both the cold-worked and quenched samples decreased. However, in comparison with the scattering from a single crystal, they were still larger by the amount of double Bragg scattering. This was to be expected, because the double Bragg contribution can be avoided when a single crystal is used. An annealing of 15 minutes at a temperature of 200°C is sufficient to obtain the intensity difference between annealed and non-annealed samples.

### Discussion

The measured intensities include thermal, Compton and double Bragg scatterings. For the theoretical calculations of these the formulas given by Warren (2) and Walker (3) were used. But unfortunately, all these calculations are approximative only. However the uncertainties are of no importance when the measured intensities are strong, for instance in the case when alloys are used. But in the case of pure metals, the situation becomes complicated, because the total intensity is of the same order of magnitude as the sum of the parasitic ones. This is why only scattering exceeding that caused by the annealed samples was taken to be due to cold working and quenching. Fig. 1 shows that the intensities left are not independent from the scattering angle. This means that single vacancies cluster together in both treatment processes if the scattering was caused by them. It is not easy to determine the number of clustering vacancies, because the measuring interval used is not large enough. The Guinier formula

$$\log (I/I_1) = f(s^2)$$

was used, where  $I$  is the measured intensity,  $I_1$  the intensity of the same number of single vacancies (if the clusters were dispersed), and  $s$  the scattering vector. The value  $I_1$  was determined from the integral intensity (4). In such a representation the intersections of the intensity curves with the ordinate give directly the number of vacancies in clusters. In this way it was found that clusters contain from about ten to about a hundred single vacancies, a more exact number cannot be given because the Guinier approximation is valid for equal particles only.

Acknowledgements

This work was sponsored by the Federal Fund for Scientific Research, Belgrade, and by the Republican Fund for Scientific Research, Zagreb, Yugoslavia.

References

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2. B.E. Warren, Acta Cryst., 12, 837 (1959)
3. C.B. Walker, Phys.Rev., 103, 547 (1956)
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## DISCUSSION

- A. Guinier : It is not sure that double Bragg diffraction is the same in annealed and cold worked metal. The neutron diffraction is the best way, to avoid any double diffraction which in any case is difficult to calculate.
- E. Lang : May I ask at which temperature you performed your cold work experiments ?
- A. Prodan : At room temperature.
- E. Lang : Then I wonder if you can expect vacancies to be retained by this deformation at all. According to my knowledge the work by Lücke et al, Turnbull, Federighi Ceresara etc.  
(W. De Sorbo and J. Turnbull, Phys.Rev. 115 (1959) 560;  
H.D. Mengelberg, M. Meixner and K. Lücke, Acta Met. 13, (1965) 835  
T. Federighi in "Lattice Defects in Quenched Metals",  
p. 217 ed. by Cottrell et al. Acad. Press, N.Y. 1965;  
S. Ceresara, Phil. Mag. 17, (1968) 1299  
A.v.d. Benkel in "Vacancies and Interstitials in Metals"  
p. 427  
e.d. by Seeger et al., North Holland Publishing 1970.)  
suggests that the vacancies in pure Aluminium migrate already below about  $-50^{\circ}\text{C}$ .
- G.W. Lorimer : The inconsistent results which are being reported concerning the observation of vacancy loops in splat-cooled metal foils might be attributed to the same factors which affect the density and distribution of loops and voids in pure aluminium, where the purity of the metal and the constitution of the atmosphere in which the metal is heated are important parameters.
- P. Furrer : In our electron-microscopic examination of rapidly quenched pure Al samples, we observed vacancy loops in a rather dense distribution. No loops could be detected if the grain size was very small or if dislocations introduced during the formation of the splat worked as vacancy sinks. We did not make any attempt to measure the vacancy concentration. But I would like to point out that McComb et al. (J.A. McComb, S. Nenno, M. Meshii, J.Phys.Spc. Japan 19, (1964) 1891;  
G.Thomas, R.H. Willens, Acta Met. 12, (1964) 191,  
Acta Met. 13, (1965) 139, Acta Met. 14, (1966) 1385,

have found that the vacancy concentration in splat-cooled Al foils corresponds roughly to the equilibrium vacancy concentration at the melting point, in contradiction to Thomas and Willens.