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*Received 10 March
and accepted 30 April 1976*

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Influence of vacancies on the precipitation of germanium in an Al–4.0 wt % Ge alloy

Precipitation phenomena in Al–Ge alloys have recently been studied by X-ray, electronmicroscopic and resistivity methods [1–11]. These studies show that the quenched-in vacancies play an important role in the formation of Ge nuclei and the subsequent growth of the Ge precipitates. The present study deals with the influence of quenched-in vacancies on the precipitation of germanium in an Al–4.0 wt % Ge alloy. Measurements were made using X-ray and microhardness methods and the specimens were prepared from super-purity aluminium and germanium (99.999%, Koch Light Laboratories Ltd, UK). The final specimens were homogenized for 24 h at 390, 420, 450, 480 and 510° C, and then quenched in ice-water; the ageing temperature was 160° C. Small-angle X-ray diffraction measurements were performed, using a Kratky X-ray camera in combination with a programmed step scanning device [10]. Some preliminary wide-angle measurements were also carried out by using a Si(Li) semiconductor detector. The microhardness of a specimen was measured using a Reichert apparatus: the surface of a testing specimen was electrolytically polished before the hardness measurements were taken. Each Vickers hardness number in Fig. 1 is the mean value of 20 measurements. All X-ray and hardness measurements were performed at room temperature.

It is known that the age-hardening of an alloy depends on the volume fraction and the mean size of precipitates. Fig. 1 shows that the increase in microhardness of an Al-4.0 wt % Ge alloy with ageing time is greater for higher solution-treatment temperatures. According to our small-angle X-ray measurements, the mean size of small precipitates is practically independent of the solution-treatment temperature, although the total intensity increased with the solution-treatment

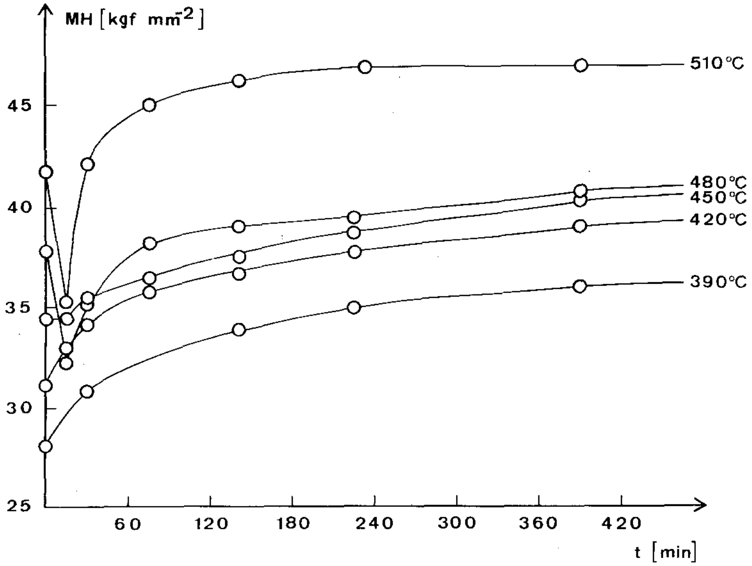

Figure 1 The microhardness of AL–4.0 wt % Ge alloy as a function of ageing time at 160° C. The solution-treatment temperatures are 390, 420, 450, 480 and 510° C.

temperature. Thus, both small-angle X-ray and hardness measurements show that the volume fraction of precipitates increases with the quenched-in vacancies.

When a specimen is ages at 160° C, the hardness depends on solution-treatment temperature. When this temperature is lower than 450° C, the hardness increases with ageing time. Above this temperature, distinct drops in the hardness curves are observed. Thus, part of the Ge precipitates is dissolved at 160° C when the number of quenched-in vacancies is increased. As ageing continues, the hardness increases again with time. The corresponding small-angle X-ray measurements show that the precipitates are now growing.

In a previous paper we have shown that the rod-shaped Ge precipitates develop during isothermal ageing at 160° C [10] and that the growth of these precipitates depends on solution-treatment temperature. The higher the temperature the sooner precipitated particles grow into long rods. With a solution-treatment temperature of 390° C, the small-angle measurements do not obey the rod model. Therefore, this temperature is omitted from Fig. 2, where the mean radius of the cross-sections of the cylindrical rods is a function

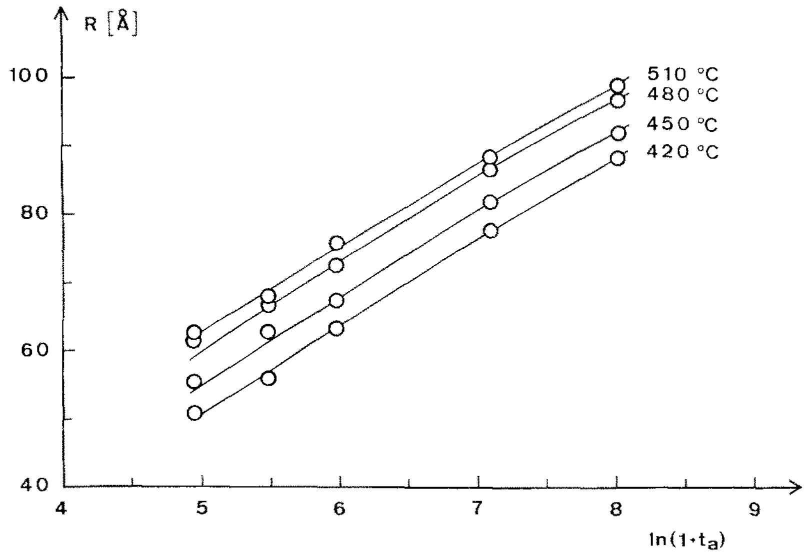

Figure 2 The change in the mean radius of the cross sections of the cylindrical rods during isothermal ageing of 160° C. The solution-treatment temperatures are 420, 450, 480 and 510° C. t_a = ageing time (min).

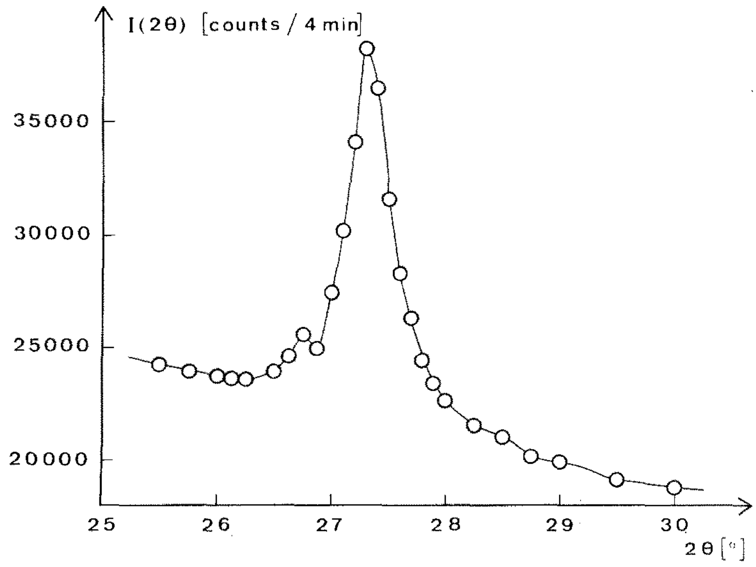

Figure 3 The I I I reflection from Ge precipitates.

of the logarithm of ageing time. The method of calculation is given in the previous paper [10]. During the growth of rod-shaped precipitates the hardness increases relative slowly (Fig. 1).

An example of the preliminary wide-angle measurements is shown in Fig. 3. According to the 1 1 1 reflection, the size of Ge precipitates is about 250 Å, when the specimen was quenched from a temperature of 480° C and aged at 160° C for 3000 min. Side maxima were observed in both the 1 1 1 and 3 1 1 reflections. The side maximum is located at a smaller 2θ value than the corresponding main reflection.

The present X-ray and microhardness studies show that the precipitation of germanium in an Al–4.0 wt % Ge alloy depends upon the content of quenched-in vacancies. After quencing, the number of precipitates increases with quenched-in vacancies, while the mean size of the precipitates is independent of the vacancy concentration. If the vacancy concentration is high enough, a short ageing at 160° C reduces the number of Ge precipitates. When ageing of 160° C is prolonged, rod-shaped Ge precipitates are formed. This growth process also depends on the number of quenched-in vacancies.

Acknowledgement

The grant received from the Finnish State Committee for Natural Science in support of this study is gratefully acknowledged.

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Received 15 March
and accepted 3 May 1976

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Fracture morphology of rigid poly(vinyl chloride)

It is clearly indicated that the cracks in many thermoplastics are generated from the prior formation of crazes [1–5]. In the case of poly(vinyl chloride) (PVC), although thermoplastic, the existence of crazes and their role in the fracture process is still enigmatic, because contradictory results have been published concerning the existence of precrack crazes in PVC. For example, Gotham [6] and Cornes and Haward [7] refer to crazing in rigid PVC, but Kambour [8] found no evidence of precrack craze formation. Because the later stages of fracture in PVC are not clearly known we have studied the nucleation and morphology of fracture in rigid PVC under tensile test conditions.

The material, which was a commercial suspension type PVC (K value of 58), was supplied by Pekema Oy in the form of injection-moulded dumb-bell-shaped test pieces (ASTM D 638). It did not contain plasticizer, but small amounts (< 7 % w/w) of stabilizers and lubricants were added.

Tensile tests were carried out at room temperature (23 ± 1° C). Tests were performed in simple uni-axial tension using an Instron tensile testing machine (floor model) at constant speeds of 0.5, 1.0 and 5.0 mm min^{-1}. The specimens and fracture surfaces were examined using a scanning electron microscope (SEM) (model JEOL JSMU 3), a transmission electron microscope (TEM) (model JEOL 100B) and by light microscopic methods. The SEM samples were coated with carbon and gold. The TEM samples were cast into epoxide polymer and cut using a microtome with a diamond knife (150 to 200 nm thick slices).

When tensile stress was applied to test pieces, stress whitening, necking and extension of the test