

MICROHARDNESS AND SMALL-ANGLE X-RAY SCATTERING STUDY
OF AN Al-16 wt.% Ag ALLOY QUENCHED FROM THE LIQUID STATEK. Kranjc and M. Stubičar
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The purpose of this study was to examine the behaviour of microhardness and the growth of Guinier Preston zones during isochronal annealing of an Al-16 wt.% Ag (4.5 at.% Ag) alloy quenched both from the solid and from the liquid state. The behaviour of specimens quenched from the solid state (SQ-specimens) is known from previous works. According to Köster et al. (1), there are two stages of hardening, a cold and a warm one. The isochronal microhardness curves constructed from the isothermal curves have shown the cold and the warm hardening stages clearly separated from each other. Much work has been needed to understand the relationship between the age hardening and the structure of the alloy (2,3,4). The cold hardening has been attributed to the growth of GP zones which are present immediately after quenching. The warm hardening is believed to be caused by the growth of platelets of the metastable γ' phase until coherency with the matrix is lost and the material starts to soften.

The size of zones immediately after quenching is the higher, the lower the cooling rate becomes (2). Therefore a difference between the properties of SQ and LQ (liquid quenched) specimens was to be expected. The advantage of studying the behaviour of specimens in the course of isochronal annealing is twofold: first, measurements can be carried out throughout the whole temperature range on one single specimen, and secondly, the first information on the difference in behaviour of differently quenched specimens can be obtained within comparatively short time. The annealing time applied in our experiments was one hour.

Experimental procedure

The quenching from the liquid state was performed by the splat two-piston technique. The Vickers microhardness was measured on electropolished surfaces of the SQ

specimens and on the untreated surfaces of the LQ specimens. A 30 ponds load was applied to specimens exceeding 20 μm in thickness, and a load of 10 ponds to thinner specimens. In order to obtain the average value of microhardness, twenty indentations were made.

The zone sizes were measured by the small-angle X-ray scattering technique. The scattering was registered by means of photographic films. Because of the small size of the splat cooled specimens a Kessig camera with a pinhole collimated beam was applied. Because of the low intensity of scattering of some samples, a small specimen-to-film distance (10 cm) was chosen. Scattering angles of less than $40'$ were inaccessible to measurements. Filtered cobalt radiation was applied to ensure a better resolution of the camera. The characteristic diffraction rings which decrease in the process of ageing were not resolved after annealing at higher temperatures. The zone sizes were measured by applying the Guinier approximation to the outer part of the diffraction rings or to the tail of the scattering curves where the tail alone was accessible for interpretation. Due to the unfavourable experimental conditions the values of the zone sizes are only approximative.

Experimental results

Fig. 1 (a) shows the isochronal curves of microhardness of four SQ specimens. The curves indicate the cold and warm hardening stages and are similar to those reported by Köster (1). Within the explored temperature range maximum hardness is reached in all specimens during ageing at 250°C .

Three types of isochronal microhardness curves of the LQ specimens can be distinguished. In Fig. 1 (b) both the cold and warm hardening can be discerned; in Fig. 1 (c) the cold hardening is hardly perceptible while the curves presented in Fig. 1 (d) show a continuous increase in hardness. As can be seen, there is no correlation between the thickness of specimen and type of microhardness curve. The common feature of all LQ specimens is that maximum hardness is reached at a lower temperature than in the case of the SQ specimens.

Fig. 2 (a) shows the increase in zone radii in one SQ specimen during isochronal annealing. After annealing at 250°C X-ray diagrams showed only the streaks scattered by the platelets of the γ' phase.

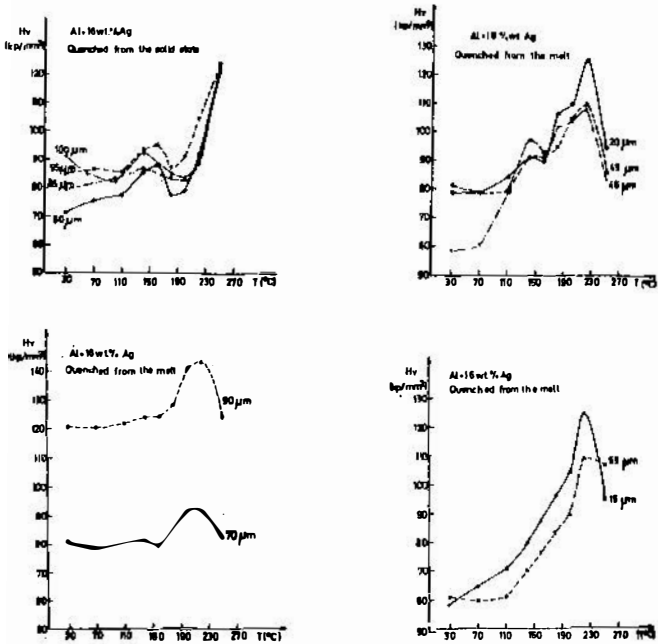


FIG. 1

Microhardness of SQ and LQ specimens in the course of isochronal annealing for one hour

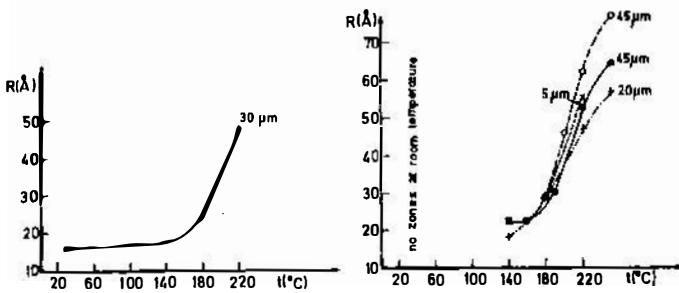


FIG. 2

Radii of GP zones in the course of isochronal annealing for one hour. (a) Specimen quenched from the solid state; (b) liquid quenched specimens of different thicknesses.

Fig. 2 (b) shows the zone radii in the splat cooled specimens of different thickness. At room temperature no diffraction rings could be detected by means of our technique. After annealing at 60°C and 100°C , and after prolonged exposure times, very weak rings appeared which were much larger than in the case of the SQ specimen. The zone sizes at these temperatures are not indicated in Fig. 2 (b) as the scattered intensity was too low to allow quantitative interpretation of the scattering curves. From 140°C on, the trend of curves in both the SQ and LQ specimens is similar. (The upper curve in Fig. 2 (b) was obtained by the use of a Kratky camera which allows the detection of scattering at much lower angles). Because of very small grains no individual streaks of β' platelets were observed at high temperatures. All the strong scattering was interpreted as due to the zones.

Discussion of the results

No zones could be detected in splat cooled specimens at room temperature. The same result was obtained by A. Daniel (5) with an Al-20 wt.% Ag specimen. Obviously, the time period during which the vacancies are mobile is not long enough to allow the transport of a sufficient number of silver atoms to zones of a measurable size.

No GP zones could be detected even after ageing for three months at room temperature. This indicates that the number of the quenched-in excess vacancies is small. On the basis of recent studies of defects in conventionally quenched Al-Zn alloys (6) and liquid quenched pure aluminium (7) one possible explanation is that during the quenching most of the excess vacancies condense into dislocation loops which act as traps for the remaining vacancies. The large area of grain boundaries, due to small grain size, may also contribute considerably to vacancy annihilation.

The growth of zones above room temperature is slow since only a small number of thermally created vacancies takes part in the diffusion of silver atoms. In trying to estimate the zone size of the specimen $20\ \mu\text{m}$ thick at 60°C and 100°C we supposed that the product of the diameter of the diffraction ring and the radius of zones remains constant at temperatures up to 140°C . The form of the

miscibility gap justifies this assumption but only when the metastable equilibrium is attained within one hour of ageing, which may not be the case with ultra rapidly quenched specimens. The zone sizes as obtained from the diameter of diffraction rings were 13.5 \AA at 100°C , and 8.8 \AA at 60°C . Extrapolating the radius-temperature curve up to room temperature, a radius of about 4 \AA was obtained. This is quite a reasonable result. However, more sensitive X-ray techniques should be used to check the presence of such small zones.

The growth of δ' platelets cannot be observed in small-angle X-ray diagrams of polycrystalline samples as readily as in single crystals. A faster growth of δ' platelets up to critical size is indicated by the fact that the maximum hardness is reached at lower temperature than in the case of SQ specimens. The early nucleation and rapid growth of δ' platelets may be related to the presence of many stacking faulted regions which have been observed by Daniel (5) in splat cooled aluminium-silver alloys.

All three types of microhardness curves could be explained in a similar way. However, this qualitative explanation should be verified by further and more detailed experiments, using different techniques. Although the same supersaturation of an aluminium-silver alloy can be achieved by quenching from the solid state, further investigation of liquid quenched specimens would be of interest because it would offer the opportunity of studying the kinetics of alloy decomposition from the early stages.

Acknowledgements

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References

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DISCUSSION :

- J. Lendvai : Have you quenched samples from the solid state to a lower temperature than room temperature ?
- K. Kranjc : No.