Super-saturated solid solution of Al-5.7 at.% Ag alloy prepared by splat-quenching

The preparation of super-saturated solid solutions of Al–Ag alloys by conventional solid state quenching methods has been known to be difficult except for the cases of low Ag concentration (< 2.8 at. % Ag) [1-3]; the analysis of the integrated intensity of X-ray small-angle scattering indicated that the phase separation takes place during quenching and the electrical resistivity, when aged after quenching, decreases monotonically with time indicating that the early stages of ageing is already finished.

By the technique of rapid conduction cooling of liquid metals (splat-cooling), it is possible to create a condition of high cooling rate ($\sim 10^7$ °C sec^{-1}) [4] and resulting high solidification rate ($\sim 10^2$ cm sec^{-1}) [5]. With this method many instances of widely extended solid solubilities in binary alloys have been reported including several works on solute rich Al–Ag alloys [6-8]. Recently we reported the ^{57}Fe Mössbauer experiment of splat-cooled Al–Fe alloys [9, 10] and showed that the local order of solutes is still present in the solid solution whose solid solubility limit appears to be extended.

The purpose of this paper is to present the results of the studies on the splat-cooled structure of Al-5.7 at. % Ag alloy by X-ray small-angle scattering and electrical resistivity measurements and by observation with a transmission electron microscope. The splat-quenching was carried out following the method developed by Duwez and Willens [11]. X-ray small-angle scattering intensities were measured at room temperature using Ni filtered Cu$K\alpha$ radiation. The electrical resistivity was measured at liquid nitrogen temperature by the four-probe potentiometric method. The defect structures were observed using a transmission electron microscope with an accelerating voltage of 1000 kV (JEM 1000).

Fig. 1 shows the X-ray small-angle scattering curves; one is the curve for a specimen splat-cooled from 660°C onto a copper substrate at room temperature and the other is the curve for a specimen prepared by the solid state quenching; quenched from 550°C into ice water.

The comparison of these two curves shows that for the splat-cooled specimen there is no solute clustering which contributes to the small-angle scattering whereas for the solid state quenched specimen the existence of the solute rich zones (the so-called G.P. zones) is evident. The average zone radius estimated by the Guinier approximation was 14 Å.

Typical electron micrographs of a splat-cooled specimen are shown in Fig. 2a, b and c. These pictures were taken without application of any thinning treatment. A great number of prismatic dislocation loops having different shapes and sizes and defect free regions along the grain boundary are observed. This structure is interesting since the defects observed in solid state quenched alloys are either helical dislocations or imperfect dislocation loops with stacking faults [12-15]. The concentration of quenched-in vacancies in a splat-cooled specimen was estimated from the size and density of dislocation loops to be (0.7 to 1.0) $\times 10^{-3}$. This value may be compared with that reported for splat-cooled

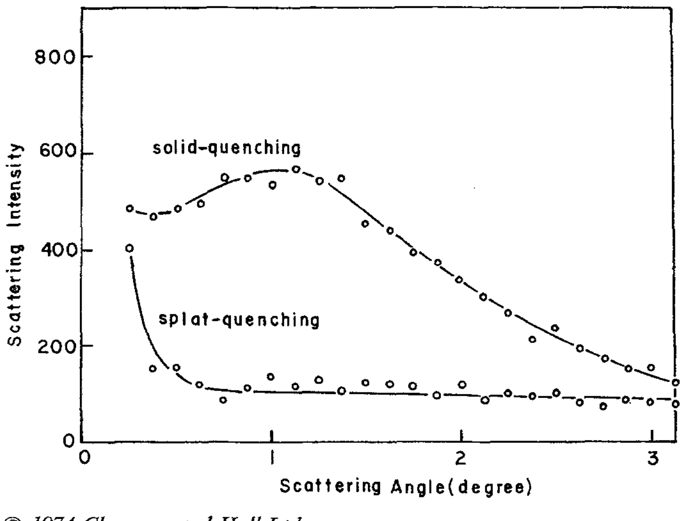
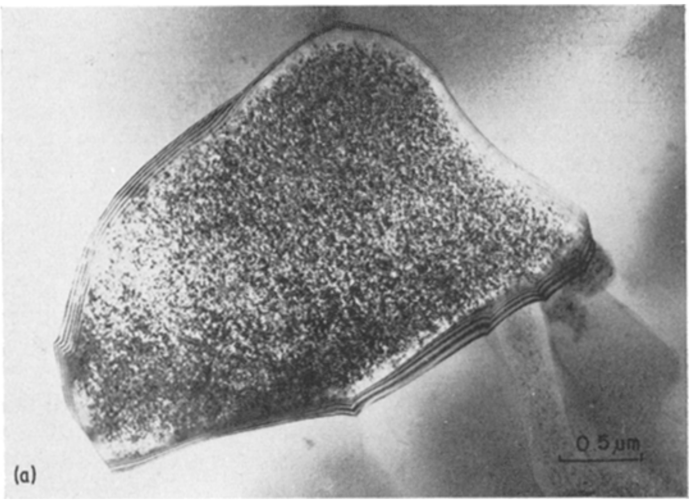


Figure 1 Comparison of X-ray small-angle scattering curves between splat quenching and solid quenching.



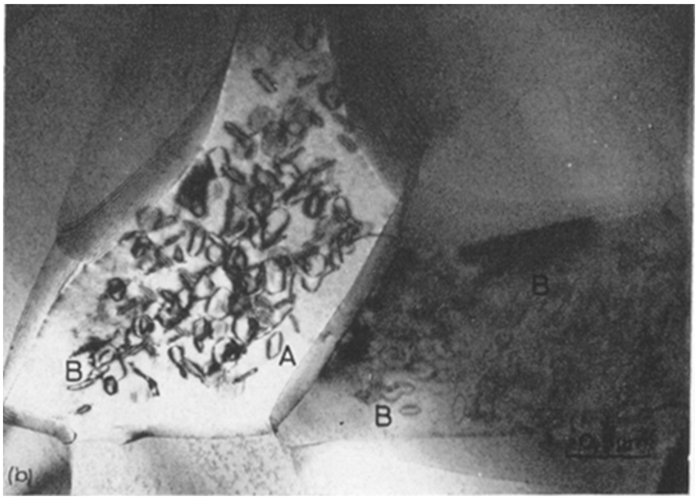
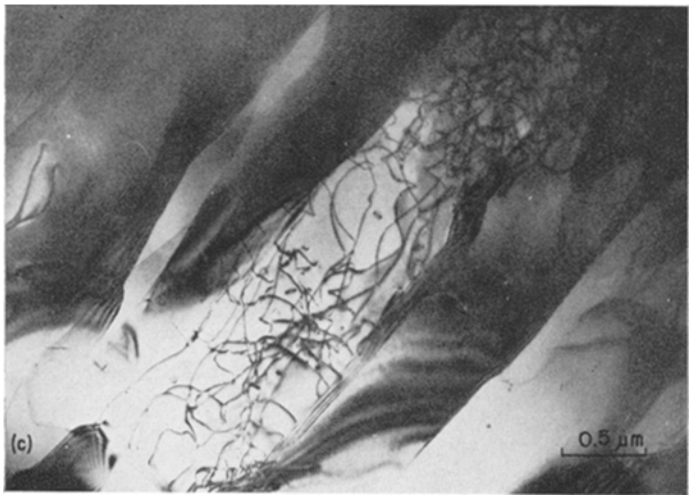


Figure 2 Defect structures in a splat-cooled Al–5.7 at.% Ag alloy. (a) A large number of black spot defects which may be either very small dislocation loops or vacancy clusters. (b) Various dislocation loops; regular hexagons, parallelograms (A) and large loops with irregular shapes (B). Loop density, 2.3×10^{14} cm^{-3}; loop diameter (average), 1800 Å; estimated concentration of quenched-in defects, 0.7×10^{-3}. (c) Tangled dislocations.



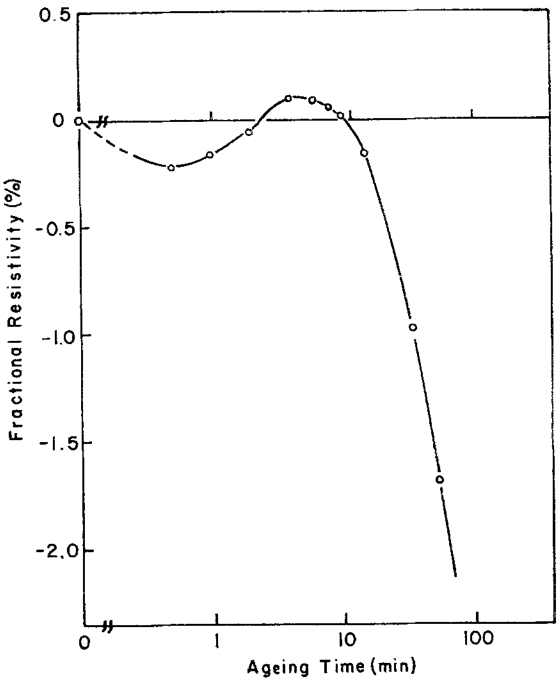


Figure 3 Change of electrical resistivity of splat-cooled Al–5.7 at. % Ag alloy during the isothermal ageing at 82°C.

pure aluminium, (1.0 to 1.3) × 10^{-3} [16] and 0.2 × 10^{-3} [17].

In Fig. 3, the resistivity change is shown of a splat-cooled specimen during the isothermal ageing at 82°C. The resistivity first decreases by a small amount and then after a small increase, decreases again rapidly. This resistivity change is beyond the experimental error (0.04 %) and a similar tendency was observed at 71 and 92°C ageings. The first decrease of resistivity is attributable to the recovery of quenched-in defects and the final rapid decrease is explained as due to the zone growth. After the ageing for 104 min, the X-ray small-angle scattering was measured for the same specimen. The Guinier approximation was almost satisfied and the average zone radius was about 8 Å.

These experimental results indicate that there are no solute clustered regions in splat-cooled specimens, or, if clusters are present, their average size is less than 8 Å; a size not determinable by the small-angle X-ray scattering technique.

It is also noted that neither a metastable nor another second phase was detected by the X-ray Debye-Scherrer method. It is therefore concluded that the splat-cooled structure of Al-5.7 at. % Ag alloy is a super-saturated solid solution with high density of lattice defects formed by quenched-in vacancies.

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