Al-12.5 wt% Si ribbons prepared by melt spinning

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Aluminium-silicon alloy with composition 12.5 wt% Si was rapidly quenched from the melt at a cooling rate of about $10^6$ Ks$^{-1}$ using the melt-spinning technique. The resulting ribbons were investigated by resistivity measurements and X-ray diffraction. The resistivity of the 20 μm thick ribbons was 26.6 μΩcm. This is 4.4 times its bulk resistivity value. The activation energy calculated from isothermal ageing was 1.14 ± 0.1 eV. The limit of primary solid solubility is extended almost to the eutectic composition and the large supersaturation is relieved by thermal annealing.

1. Introduction

Since the splat-quenching experiments of Duwez et al. [1] there has been considerable interest in the formation of metastable phases through rapid solidification. From studies on rapid solidification of some Al-metal alloys, supersaturated solid solutions, metastable crystalline phases and even amorphous phases have been reported [2–6]. Rapidly solidified Al-Si alloys of various compositions and thickness have been prepared by splat cooling [7–9] and by melt spinning [9–12].

These studies centred mainly on the variation of lattice parameter with the amount of silicon assumed to be present in solid solution. Some microstructure studies were also made.

In the present work, long uniform ribbons of Al-Si eutectic alloy were prepared by melt spinning. The structure of the as-quenched and annealed ribbons was studied by X-ray diffraction. Also, isothermal resistivity measurements at ageing temperatures from 100 to 187 °C have been made.

2. Experimental procedure

Aluminium-silicon alloys with composition 12.5 wt% Si were prepared from 99.999 wt% pure Al and 99.99 wt% Si, and the alloy was cast into a copper mould to obtain rods 2.5 cm long and 0.4 cm in diameter. Specimens in the form of ribbons were prepared by a melt-spinning apparatus [13]. In this technique a stream of the molten alloy with a temperature of 750 °C was ejected by pressurized argon from a 0.35 mm diameter orifice on to a copper wheel rotating at 2950 r.p.m. Continuous uniform ribbons of thickness 20 μm were obtained. A cooling rate of about $10^6$ K s$^{-1}$ was estimated from the time of stay of the ribbon on the wheel.

Resistance changes at room temperature were followed by using a double Thomson bridge. The accuracy was 0.05% with reproducibility better than 1%. Annealing was done by placing the foils in a low heat-capacity Al container in the furnace for the specified time and then quenching in alcohol before the measurement. Each data point corresponds to an average of measurements on five specimens. X-ray examination of the ribbons was conducted using filtered CoKα radiation. Short lengths of the ribbon to be X-rayed were stuck on a glass slide parallel and close to each other using Vaseline.

3. Results

3.1. Electrical resistivity

The resistivity depended on the thickness of the ribbons produced. Ribbons having smaller thickness had higher resistivity values. Ageing of melt-spun ribbons at any temperature resulted in a decrease in the electrical resistivity. The decrease occurred for all thicknesses. The rate of decrease of the electrical resistance ratio $R(t)/R(0)$ with ageing time for Al-12.5 wt% Si varied with ribbon thickness, being smaller for thicker ribbons.

Detailed ageing studies were done on ribbons with the least thickness obtained which possess the highest cooling rate. The state of these ribbons is expected to differ appreciably from the equilibrium state. So, ageing was done on the 20 μm thick ribbons.

The variation of electrical resistance ratio $R(t)/R(0)$ with ageing time at ageing temperatures of 95, 128, 142, 154, 162, 173 and 187 °C for melt-spun Al-12.5 wt% Si, 20 μm thick ribbons is shown in Fig. 1. A rapid decrease in $R(t)/R(0)$ is followed by a slower one for ageing temperature above 128 °C. At a lower temperature such as 128 °C, the decrease is slow all
over the curve as shown in Fig. 1. For ageing temperatures lower than 128 °C, the resistivity remains unchanged for a certain period of time. A representative plot at 95 °C is shown in Fig. 1 indicating an incubation period of 3 h. This incubation period increases with decreasing ageing temperature.

The measured resistivity of as-cast rods having 0.4 cm diameter and 2.5 cm length, and having the same composition as the ribbons, was 6.1 μΩ cm compared with 26.6 μΩ cm for the 20 μm thick as-spun ribbon.

3.1.1. Activation energy

From the isothermal annealing data (Fig. 1) the activation energy \( E \) was calculated. The time \( t \) taken to reach a given value of a property \( P \) is given by

\[
\ln t = \frac{E}{kT} + \text{constant}
\]

where \( k \) is the Boltzmann constant and \( T \) the absolute temperature.

A plot of the logarithm of the time to reach different values of \( R(t)/R(0) = 0.8, 0.7, 0.6 \) and 0.5 versus the reciprocal of the ageing temperatures is shown in Fig. 2. From the slope of the curves activation energies of 1.24, 1.11, 1.1 and 1.1 eV were obtained, corresponding to value of \( R(t)/R(0) \) of 0.8, 0.7, 0.6 and 0.5, respectively, with an average of 1.14 ± 0.1 eV.

3.2. X-ray diffraction

The X-ray diffraction patterns of the as-quenched Al–12.5 wt % Si ribbons (20 μm thick) for 20 from 20 to 80° show the first three reflections from Al, but no Si lines were observed.

However, the X-ray diffraction pattern of ribbons annealed for 5 h at 200 °C show the presence of the first three Si lines which were absent in the as-prepared ribbons. Table I shows the intensities of observed reflections compared with the reported values [14].

From Table I it can be seen that in the as-quenched ribbons the relative intensities of Al lines do not conform with the published values, indicating some degree of preferred orientation. This can happen because, on quenching the melt, the direction of heat gradient and the way the ribbon separates from the melt affect the orientation of the Al crystals to a significant degree. The degree of preferred orientation decreases slightly after annealing. On the other hand, the relative intensities of the Si lines match the published values fairly well. This can be attributed to the fact that Si was present as a solid solution in Al in the as-quenched ribbons. When the ribbons are subjected to heat, it may be that Si starts precipitating randomly. So, the newly formed crystallites do not have any reason to show preferred orientation.

Although Si lines were absent in the as-prepared ribbons, an estimate of about 2 wt % Si could be lost, without being observed, in the background in the X-ray diffraction pattern. So an amount of about 10 wt % Si present as solid solution is estimated.