Oxidation Behaviour of Microcrystalline AlSiX Alloys Studied by AES Technique

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Rapid solidification from the melt allows rapid extraction of heat during the transition from the liquid state to solid material and thus permits large deviations from equilibrium which, in turn, offers a range of desirable properties: high homogeneity, very fine grains and intermetallic phases. Subsequent compacting of microcrystalline (MC) ribbons from AlSi alloys at elevated temperatures makes it possible to use this material for number of practical purposes. The oxidation of the surface, taking place during heating, prevents adequate interribon bonding and thus has a deleterious effect on the mechanical properties of the final product.

The oxidation kinetic data (Fig. 1) for the studied MC ribbons from AlSi11 (alloy A), AlSi11Mg0.2 (alloy B), AlSi11Mg0.2Sb0.11 (alloy C) and AlSi11Mg0.2Sr0.025 (alloy D) show that the presence of additives intensifies the oxidation. The most pronounced effect is observed with (Mg+Sr) addition but in this case very high densities were measured after compacting of the ribbons. As far as a strong correlation between the oxide growth velocity and the protective properties of the oxide layer is not observed, additional methods for studying the oxidation behaviour are needed. Therefore, thermogravimetry and Auger Electron Spectroscopy (AES) combined with Argon ion sputtering were used to study the oxide growth and the oxide constitution after annealing at 535°C for a number of AlSi microcrystalline alloys.

Comparison between the depth profiling results taken from a chosen region and AES spectra gathered from different microspots of the MC alloys studied, showed a general tendency for initial oxide film formation during the solidification. In Fig. 2a a depth profile for an as-cast alloy is presented. As can be seen, aluminium based oxide film with minor magnesium content is formed on the surface. This oxide scale is negligible in thickness when is placed on the silicon phase and sufficiently thicker on the aluminium matrix. In this early stage of oxidation neither strontium nor antimony were found in the layer.

When the samples were annealed at 535°C, for different times, a thicker oxide layer was formed due to the thermally activated diffusion of

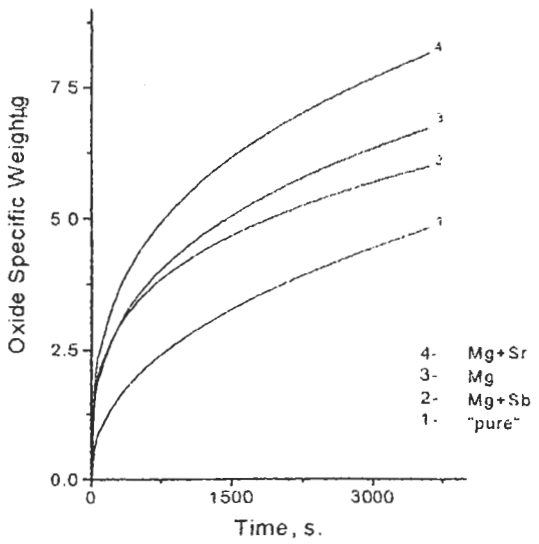

Fig.1 Oxide specific weight as a function of the annealing time for pure and doped microcrysalline AlSi11 alloys

the elements. The analysis of the depth profiles of annealed MC alloys (Fig. 2b) shows that the magnesium and the oxygen become prevailing components of the upper part of the layer. The relative content of magnesium increases with the annealing time. The oxygen signal was found to keep nearly the same level. The measured aluminium content after annealing of the ribbons is much smaller than the one of magnesium. It can be seen, that the decrease of magnesium leads to an increase of aluminium content. For prolonged periods of heating (120 min.) no aluminium can be detected in the upper part of the oxide layer (Fig. 2b).

When the MC alloys are doped additionally with Sr or Sb substantial changes are observed in the oxide layer formation. For short periods of annealing (up to 2 min.) the results do not differ sufficiently from these for the as-cast alloy B. With increasing of the annealing time (up to 45 min.) strontium (antimony) together with magnesium takes part in the oxide growth. Strontium, which has greater affinity to the oxygen participates quite actively in the oxidation and favours the formation of defects. From the upper part of the oxide layer of alloy D high strontium signal is registered due to predominant strontium presence in the burst blisters' scales. These blisters are formed on all samples of alloy D after prolonged periods of annealing. They strongly disturb the continuity of the surface. Unhomogeneity both in morphology and composition of the oxide is extended with the annealing time, causing this way, brittleness and falling-outs of the particles of the oxide. The protective properties of the oxide film decreases in strontium doped ribbons, but the existence of peeled off parts from the surface facilitates better compacting into ingots with good density and mechanical properties.

For prolonged periods of heating (up to 120 min.) the oxide layer of antimony doped ribbons is getting thicker but remains undisturbed. Antimony is strongly involved in the formation of the homogeneous oxide film, but it does not reach the upper part layer where magnesium oxide is prevailing. Addition of antimony in Mg doped MC ribbons improves significantly the protective properties of the oxide film.

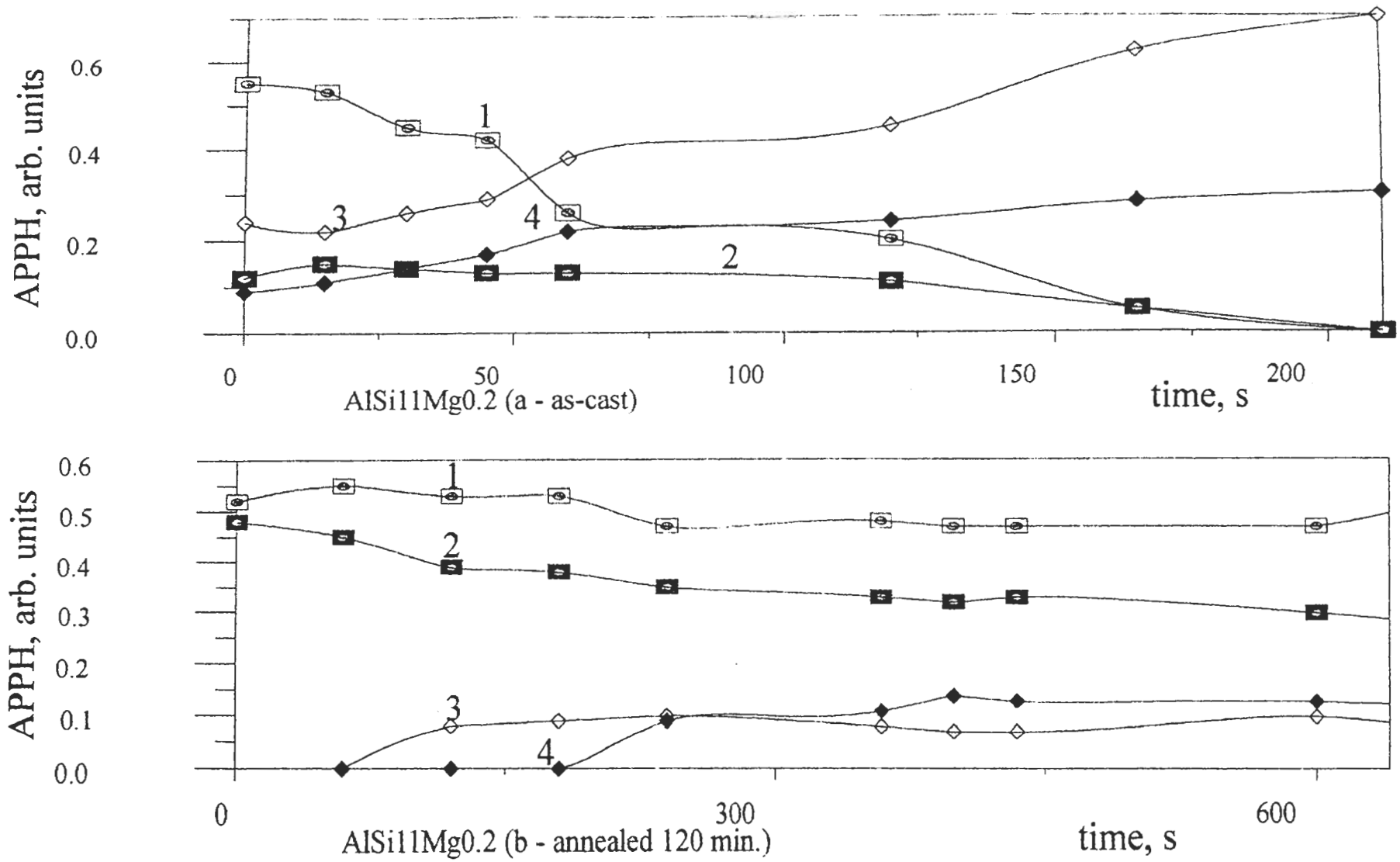

Fig. 2 Auger peak-to-peak height (APPH) of O-1, Mg-2, Al-3, Si-4 as a function of the etching time.