High-Temperature Oxidation of Al–Mg Alloys

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Surface-analysis techniques (x-ray photoelectron spectroscopy and Auger electron spectroscopy) and electron microscopy (SEM and TEM) have been used to study the mechanism of steady-state high-temperature oxidation of Al–Mg alloys. Two high-purity alloys containing 0.4 and 2.0 wt.% Mg were heat-treated in dry air at 550°C up to 90 hr. It was found that the oxide layer was composed of MgO and spinel (MgAl₂O₄), the major constituent being MgO. The molar concentration of MgO decreased with increasing depth, while that of spinel increased. The rate-controlling mechanism for the growth of the oxide layer in the Al–0.4Mg alloy was the solid-state diffusion of Mg in the MgO-spinel constituents. For alloys of higher-magnesium content, the growth of the oxide layer was controlled by solid-state diffusion of Mg through the adherent protective oxide areas and by the transport of Mg vapor across voids formed between the alloy substrate and the oxide layer.

KEY WORDS: Al–Mg alloys; oxidation; high-temperature mechanisms.

INTRODUCTION

When Al–Mg alloys are heat-treated in air at high temperatures, magnesium is depleted from the bulk with the formation of nonuniform and relatively thick, dark oxide layers on the surface. These oxide layers may cause undesirable effects during the fabrication and performance¹⁻⁴ of the alloys.

Surface-analysis techniques have been used in the study of the initial or transient stages⁵⁻¹⁰ of high-temperature oxidation of Al–Mg alloys. The aim of the present work was to use surface analysis and electron microscopy to study the mechanism of steady-state high-temperature oxidation of these alloys.

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EXPERIMENTAL

Two high-purity Al-Mg alloys were used in this investigation, containing 0.4 and 2.0 wt.% Mg. The alloys were cold-rolled to sheets of 2 mm thick. Before heat treatment, specimens of dimensions 2 x 5 cm were degreased in acetone and methanol. The samples were then oxidized in dry air at 550°C for 18 and 90 hr.

A JOEL-type scanning-electron microscope (SEM) was used to study the surface morphology of the oxidized specimens. For transmission electron microscopy (TEM), the oxide layers were stripped chemically using a solution of iodine in absolute methanol (5 gm iodine/100 ml methanol) and were then washed in methanol.

X-ray photoelectron spectroscopy (XPS) and Auger electron spectroscopy (AES) in conjunction with argon-ion sputtering were used to study elemental composition and chemical binding (oxide or metal state) of the oxidized surfaces. These investigations were performed in a combined AES/XPS/SIMS Leybold-Heraeus LHS 10 instrument. The ultimate pressure of the instrument was 10^-9 mbar.

For XPS the specimens were excited by MgKα x-rays (1253.6 eV). The Auger electron spectra were obtained with an electron beam (I = 260 nA) scanned over an area of 0.3 x 0.3 mm². The data were recorded in the E N(E) mode. For AES depth profiling, an area of 0.05 cm² around the area of emitted Auger electrons (∼0.001 cm²) was sputter-etched by 3 KeV Ar⁺ ions (angle of incidence: 60° to normal). The total sputter-ion dose density for each depth profile was about 2 x 10¹⁸ Ar⁺/cm². The depth scale for sputtering was calculated by assuming a sputter yield Y of about 1 (emitted atom/primary ion) for 3 KeV Ar⁺ ions and a particle density of 10¹⁵ atoms per 0.4 nm of the oxidized specimen surface.

In depth profiling, the KLL Auger lines of O, Mg, and Al were investigated. The Auger lines were integrated numerically by a computer after smoothing and linear background subtraction. Sensitivity factors for Al KLL and O KLL were used to relate the measured integrated Auger peak intensities with the relative atomic concentrations of Al and O in the oxide layer. The sensitivity factor for Mg KLL in MgO was determined by comparing Auger spectra with XPS data.

RESULTS AND DISCUSSION

Figure 1 shows a complete XPS spectrum for the surface of the Al-0.4Mg alloy, after oxidation for 90 hr at 550°C. The main constituents were O, C, and Mg, but not Al. It has been assumed that carbon appearing in the analysis is a normal laboratory contamination. The surface of the alloy