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Characteristics of aluminium–scandium alloy thin sheets obtained by physical vapour deposition

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ABSTRACT

Thin sheets of an age-hardenable aluminium–scandium alloy were deposited by direct current magnetron sputtering. As targets an aluminium–scandium pre-alloy with a scandium content of 2.0 mass% (size 88 × 500 mm) was applied. The substrates to be coated consisted of thin steel sheets which after deposition were dissolved in an oxidizing medium. In this way, free-standing sheets of less than 30 μm thickness of the aluminium–scandium alloy were received. Two deposition temperatures, 37 and 160 °C, were applied. The as-received sheets showed a typical columnar structure. Two post-treatments of the sheets were applied: a cold isostatic pressing and an artificial ageing for 1 h at temperatures between 200 and 400 °C. The strength of the sheets was measured by tensile tests. The employed specimens had a width of 10 mm and were gained from the sheets by cutting. During testing, load and strain were measured by a 1000 N load cell and a video extensometer, respectively. The as-deposited specimens show a tensile strength of 350 MPa. Artificial ageing at 300 °C increases the tensile strength to more than 400 MPa. It could be shown that during tensile tests cracks are initialized at coating defects.

1. Introduction

Recently, aluminium alloys doped with scandium have been noted a special interest. Added to aluminium, scandium acts as a grain refiner and recrystallisation inhibitor. The high hardness and high-strength of aluminium–scandium alloys are caused by precipitation of the intermetallic phase Al_3Sc, which is coherent with the aluminium matrix. The coherency mismatch of about 1.2% results in significant lattice stress, which hinders dislocation motion. The maximum strength can be achieved by an artificial ageing at about 300 °C, a high temperature if compared to the optimum ageing temperatures of high-strength aluminium–copper–magnesium or aluminium–zinc–magnesium–copper alloys [1,2].

Physical vapour deposition is a well-known technique not only in engineering but also in other industries, such as: electronics, solar, glass and optical [3]. A typical application is the deposition of metal nitrides on high-speed steel or cemented carbide tools to enhance wear resistance. Pure metal coatings like aluminium, nickel or chromium can also be deposited and currently serve as heat reflectors, adhesion enhancers or magnetic storage. The generation of free-standing sheets is a scarcely applied technique but has already been described for aluminium–magnesium alloys by magnetron sputtering [4,5].

Background of the work reported here is the fabrication of micro-components. These can be assembled by lithographic technologies, where production with high accuracy can be realized, but costs are comparably high and the number of realized materials is quite limited [6]. Thin sheets of light-weight and high-strength materials are needed for numerable applications. In many cases the thickness of these sheets must not exceed 30 μm. Pure aluminium like Al 99.5 is easily available in such thickness but its strength is insufficient. On the other hand, high-strength aluminium alloys cannot be rolled down to such a thickness. Thus, it was considered to apply the PVD method to the production of aluminium–scandium alloy sheets.

2. Experimental

The deposition experiments took place in a magnetron sputtering unit with d.c. power supply. Sheets of an aluminium–scandium pre-alloy with a scandium content of 2.0 mass% and a size of 88 × 488 × 8 mm were used as targets and 100 μm thick sheets of unalloyed steel were used as substrates. The latter were mounted on a temperature controlled copper block in front of the target. The distance between substrate and target was 50 mm. To start the sputtering process, the chamber was filled with argon up to a pressure of 0.6 Pa and a voltage of about 500 V was applied to the targets. Thus, a self-sustaining glow discharge is generated. The applied voltage was controlled to keep the target power constant at either 1 or 2 kW during deposition. The substrate temperature

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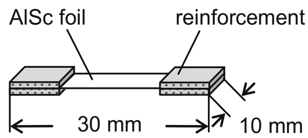

Fig. 1. Scheme of the tensile test specimen.

was kept constant either at 310 or at 433 K by heating or cooling the copper block during the whole deposition time. Temperature was controlled with a thermocouple mounted between the copper block and the back side of the substrate.

After deposition the Al–Sc coatings were separated from the steel substrate by dissolving the steel in a mixture of 15% sulphuric acid (H_2SO_4) and 30% hydrogen peroxide (H_2O_2). In this way, thin free-standing sheets of aluminium–scandium alloy were received.

In some experiments cold isostatic pressing (AAD-Hochdruck-technik, Bad Homburg) was applied as post-treatment in order to densify the deposited Al–Sc coatings. Pressing forces up to 400 MPa were available and were maintained for 1 h. To keep the hydraulic oil away from the Al–Sc sheets, the samples were hermetically sealed in polyethylene foil. A second post-treatment consisted of an artificial ageing at 300 °C for 1 h at atmosphere.

The morphology of the resulting layers was investigated by field emission scanning electron microscopy (FE-SEM) (S4004, Hitachi) and conventional scanning electron microscopy (SEM) (Camscan MV2300, EO Scan). The roughness was measured with atomic force microscopy (AFM) (Explorer, Veeco Instruments). The chemical composition of the coatings was analyzed by glow discharge optical emission spectroscopy (GDOES) (GDS 750A, Leco). The hardness of the resulting thin sheets was measured with a micro-hardness tester (Fisherscope H100C, Fisher) on the cross-section of the foil. Tensile tests were carried out on a electro-mechanical testing machine (E 1000, Instron).

Tensile specimens were gained from the sheets by cutting and had a width of 10 mm in the gauge region and. The sheets (size $30 \times 10\ mm^2$) had to be reinforced on both ends to enable mounting in the testing machine as shown in Fig. 1. During testing, load and strain were measured by a calibrated 1000 N load cell meeting the requirements of ISO 7500-1 and ASTM E4 and a video extensometer, respectively.

3. Results

3.1. Characterization of the aluminium–scandium micro sheets

All sheets obtained were found to have a columnar structure. In many cases adhesion between coating and substrate was poor and

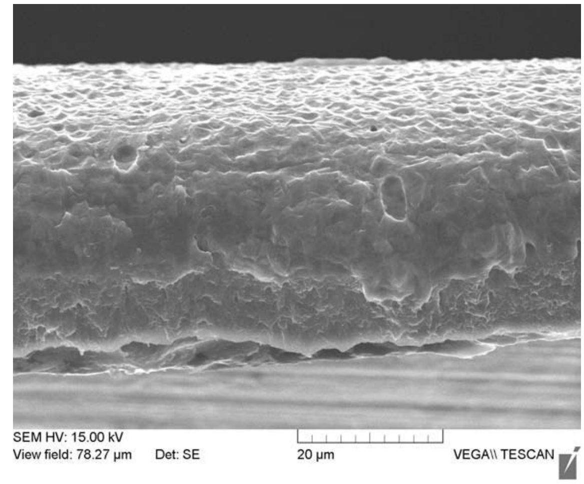

Fig. 2. SEM picture of a typical cross-section of an Al–Sc coating.

spontaneous delamination occurred. To get comparable results, all coatings were treated in an oxidizing media (H_2SO_4/H_2O_2). A typical structure in the cross-section of a separated coating is shown in Fig. 2. The visible section of the sheet was produced by cracking under liquid nitrogen (77 K). The outer surfaces of the separated coatings look different depending on their position during coating. According to their position to the substrate they will be further on called "free surface" or "contact surface". The contact surface is shown in Fig. 3a. It is relatively smooth compared to the free surface (depicted in Fig. 3b). On the latter the top ends of the columnar crystals are visible. The chemical composition, revealed by GDOES, resulted in a scandium content of 1.9 mass%.

3.2. Influence of post-treatments

The first post-treatment of the produced sheets consisted in a cold isostatic pressing at 400 MPa. Aim of this treatment was to reduce pores in the bulk of the sheets and furthermore to decrease the roughness of the free surface. The AFM tip scanned an area of $50 \times 50\ \mu m$ in both cases. The results of the contact surface and the free surface as-sputtered and after cold isostatic pressing are shown in Fig. 4. Due to the position of the contact surface during sputtering, its roughness is relatively low (Fig. 4a, $R_a = 25$ nm). The free surface, however, reveals a higher roughness (Fig. 4b, $R_a = 98$ nm) due to the columnar growth of the aluminium crystals. Cold isostatic pressing causes a slight decrease in roughness on

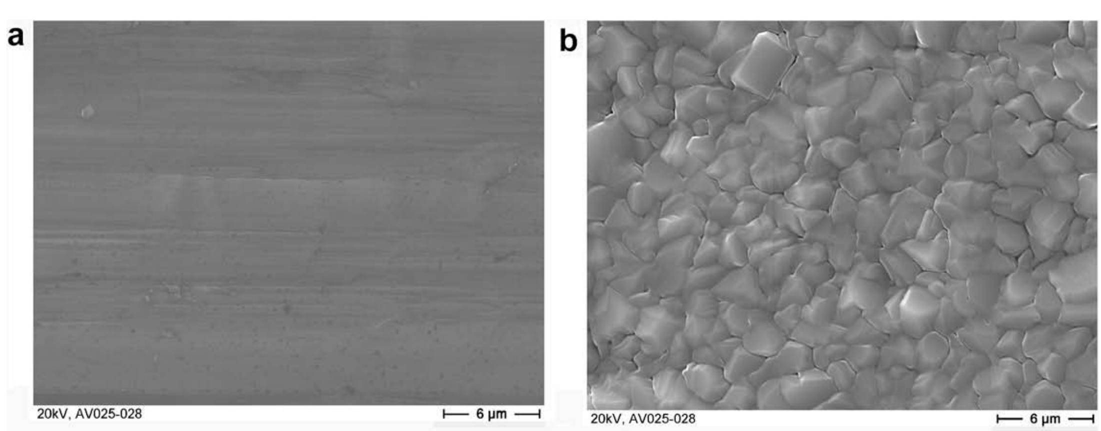

Fig. 3. Typical surfaces of a separated Al–Sc coating, (a) contact surface, during deposition in direct contact to the steel substrate and (b) free surface, during deposition opposite to the target.