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Microstructure and phase composition of microarc oxidation surface layers formed on aluminium and its alloys 2214-T6 and 7050-T74

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ABSTRACT

The influence of substrate on the microstructure and phase composition of surface layers synthesised by microarc oxidation (MAO) on aluminium and its alloys 2214-T6 and 7050-T74 is studied using scanning (SEM) and transmission electron microscopy (TEM) as well as cross-sectional X-ray diffraction. MAO layers are composed of three layers and are mainly made of gamma-Al₂O₃ and alpha-Al₂O₃ phases. The proportion of each phase depends on the substrate. The external porous layer is mainly composed of the gamma-Al₂O₃ phase. The internal dense layer can present two aspects according to the percentage of the alpha-Al₂O₃ phase. The so-called granular aspect indicates a high proportion of "dendrite" defect which results from discharge formation and implies a high percentage of the alpha-Al₂O₃ phase. In the latter, it is believed that a Zn alloying element can inhibit the growth of alpha-Al₂O₃. During the MAO process, discharges likely occur in the vicinity of the MAO layer/substrate interface, probably in the spherical porosities that result from oxygen generated in the thin layer localised at the interface.

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1. Introduction

Microarc oxidation (MAO) [1-4] also known as plasma electrolytic oxidation (PEO) [5,6] is a process of plasma-assisted electrochemical conversion of a metal surface, such as Al, Ti or Mg, or a metallic alloy to grow an oxide ceramic film [1-8]. The layers synthesized by the MAO process exhibit excellent wear and corrosion resistance, electrical and thermal properties.

It is usually admitted that three main steps govern the MAO layer synthesis [9,10]. In the first step, a number of separated discharge channels are formed in the oxide layer where its conductivity is the lowest because of defects. Due to the strong electric field, the anionic components are drawn into the channel. Concurrently, aluminium and alloying elements are melted out of the substrate, enter the channel and get oxidized. In the second step, Al is ejected from the channel into the MAO layer in contact with the electrolyte, thereby increasing the surface layer thickness in that location. In the last step, the discharge channel gets cooled and the reaction by-products are deposited onto its wall [9]. The above process repeats itself at discrete locations over the whole surface of the layer, leading to an overall increase in the MAO layer thickness [10]. It is now well established from micro-structural analysis that the MAO layer formed on Al and its alloys is composed of three layers: a surface layer, an internal layer with lower porosity and a thin layer, which exhibits a nano-scale polycrystalline microstructure, at the coating/substrate interface [11,12]. The higher cooling rate of molten alumina at the topmost surface favours the formation of gamma-Al₂O₃. On the over hand, alpha-Al₂O₃ is easily formed in the internal layer with a lower cooling rate [1,9,13].

Despite several investigations, the mechanism of the MAO layer growth remains unclear, particularly in terms of the local physical processes occurring during growth. The above description of the MAO layer formation requires fine characterizations because, to our knowledge, no micro-structural investigation has revealed crack defect caused by discharges formation in the coating. Moreover, the characterizations presented in the literature do not point out the relation between the substrate and the internal dense layer microstructure; on the other hand, the correlation between the dense layer microstructure and phase composition has not yet been undertaken.

Table 1.					
Chemical	composition	of 2214 T	6 and	7050 T	74 alloys.

Weight percent											
Alloy	Cu	Si	Mn	Mg	Fe	Zn	Ti	Cr			
2214-T6	3.9–5	0.5-1.2	0.4-1.2	0.2-0.8	0.3	0.25	0.15	0.1			
7050-74	2-2.6	0.12	0.1	1.9–2.6	0.15	5.7-6.7	0.06	0.04			

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Fig. 1. Schematic representation of cross-sectional X-ray micro-analysis at beamline ID 15 ESRF (Grenoble).

Therefore, it is clear that other characterizations are required in order to deeply understand the mechanism of MAO layer growth, particularly in terms of the local physical processes occurring during growth.

The present paper focuses on a fine micro-structural characterization and the phase composition of the MAO layer obtained on aluminium (purity 99.999) and its alloys 22A4-T6 and 7050-T74. Defects in the MAO surface resulting from discharges and gas formation are revealed and correlations are established between dense layer microstructure, phase composition and the nature of the substrate. First, we briefly present the conditions of MAO layer formation. Then, we explain the adopted methodology for sample preparation and present the equipments used. Finally, the obtained results are presented and discussed.



Fig. 2. SEM micrograph cross-sections and the corresponding X-ray spectra of MAO surface layers formed on (a) aluminium, (b) 2214-T6 and (c) 7050-T74 alloys.

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