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Microstructure, temperature estimation and thermal shock resistance of PEO ceramic coatings on aluminum

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ABSTRACT

Plasma electrolytic oxidation (PEO) is a novel surface technique for producing ceramic coatings on valve metals and their alloys. In this paper, microstructure, temperature estimation and thermal shock resistance of the ceramic coatings formed on pure aluminum by heteropolar pulsed current ceramic synthesizing system were investigated. Results show that the coating roughness becomes greater and the plasma discharge channel populations in the ceramic coatings decrease while the pores enlarge with PEO treatment time. In addition, the surface morphology of the ceramic coating indicates that melting and solidifying process happen alternately in the process. The results also show that the coatings have a good thermal shock resistance. Moreover, the temperature and the temperature rise rate of the plasma discharge channels were investigated, and a formula for calculating the temperature and the temperature rise rate of the plasma discharge channels at any time during the PEO process was deduced.

1. Introduction

Aluminum and its alloys have been used in industry for many years. However, their comparatively low resistance against wear and corrosion has limited their working life and wider use. One way to overcome these problems is the application of suitable coatings on their surfaces. The protection of aluminum alloys from wear and corrosion by synthesizing ceramic coatings is currently of great interest. Plasma electrolytic oxidation (PEO) is a novel technique for depositing ceramic coatings on aluminum alloys in an aqueous electrolyte. The coating synthesized by PEO process has excellent wear and corrosion resistance, and adhesion to the substrate (Yerokhin et al., 1999; Gnednekov et al., 2001; Gu et al., 2006). The technique is similar to the conventional anodic oxidation in process form, but PEO process is more complicated, combining electrochemical oxidation with a high voltage plasma spark discharge, micro-melting and micro-solidifying of oxides in an environmentally friendly electrolyte. There is no need to keep very low electrolyte temperature in the process. The plasma discharge process stimulates the growth of the surface coatings, and makes them thicker and harder than that produced by conventional anodic oxidation. The PEO coatings are composed of aluminum oxides and other

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compounds with some electrolyte components (Rudnev et al., 2001, 1999). The coating is being exploited for various applications, including those for which wear resistance, corrosion resistance and thermal protection of work-pieces are required (Tian et al., 2002; Nie et al., 2002; Yerokhin et al., 2000; Butyagin et al., 1748; Gnedenkov, 2000). However, there is still much work to be done before the coatings can be efficiently and widely used. Especially, relatively little is known about the thermal shock characteristics of PEO coatings, in spite of extensive study of the deposition process (Yerokhin et al., 2002; Sundararajan and Krishna, 2003) and of coating microstructures (Han et al., 2003; Wenbin Xue et al., 2000). High thermal shock resistance of the coatings is very important to make the coatings have an effective thermal protection function to the metallic components serving at high temperature condition because of some obvious differences in thermo-physical properties between the coatings and their substrates, such as in thermal expansion coefficient. Curran and Clyne provided some valuable basic data for studies of the thermal shock property of the coatings on aluminum alloys in their recent investigations (Curran and Clyne, 2005a,b). The thermo-physical property data of PEO coatings is helpful for understanding the thermal shock resistance of the coatings.

In the present work, an oxide ceramic coating was produced on industrially pure aluminum using the PEO technique. Thermal shock resistance of the coatings was investigated in this paper. Furthermore, in order to estimate the thermal property related temperature rise rate of the discharge channels and their average temperature at any time during the PEO process, a model was set up and a formula was deduced by means of analyzing the surface microstructure changes of the coating.

2. Experimental procedure

Test pieces (30 mm × 3 mm × 0.5 mm) of industrially pure aluminum were used as substrates for PEO coating formation and growth. The PEO coatings were fabricated using a 50 kW hetero-polar PEO ceramic synthesizing system. One output of the power supply was connected to the electrolyte bath and the other to a test piece immersed into the electrolyte. A constant current density (10 A/dm^2) at the coating surface was maintained by regulating the voltage between the two electrodes. More details about the equipment can be found elsewhere (Weichao Gu et al., 2006). An aqueous electrolyte was used, mainly containing $(NaPO_3)_6$ and NaOH, and other additives such as $NaAlO_2$. The PEO processing time was within 45 s and 35 min. The temperature of the electrolyte was maintained within 30 ± 2 °C by a circulation water cooling-controlling system. The surfaces of the aluminum test pieces were cleaned of mechanical contamination, rinsed with acetone and alcohol, and then laundered with distilled water. The test piece and the stainless steel bath immersed in the electrolyte served as anode and cathode, respectively, during the PEO process of the present work. After the process, the test pieces were laundered with distilled water again to be ready for further investigations.

The surface morphology of the coatings was examined with a scanning electron microscope (SEM JSM5800). The surface roughness of the coatings was investigated with an XP-2TM surface smoothness meter. The population and dimension of the pores of the coating surfaces was estimated by a quantitative image analyzing apparatus.

The thermal shock resistance of the coatings was investigated through a cyclic heating–cooling test. Two kinds of experiments were carried out with each PEO coated sample. Firstly, the sample was heated up to a high temperature of 450 °C and had a dwell time of 5 min in air, then a dwell time of 3 min in room temperature air. This is called an air-cooling thermal shock test. In the second experiment, the sample was heated up to a high temperature of 450 °C and had a dwell time of 5 min in air, then a dwell time of 1 min in the room temperature water. This is called a water-cooling thermal shock test. Before and after the thermal cycling test, the surface characteristics of the specimens were inspected visually using SEM.

3. Results and discussion

3.1. *Roughness and surface morphology of the PEO coatings*

Under the experimental parameters described above, some observations of the surface characteristics can be made:

(a) All of the coatings have a uniform white color appearance.
(b) Roughness of the coatings does not seem to have much relation to the original surface roughness of the substrates. When a PEO coating is formed on a smoother substrate surface, the sample may become rougher, or otherwise.
(c) The surface roughness of the coatings mainly depends on the current density and treatment time, that is, the higher the current density and/or the time is, the higher the roughness of the coatings will be. This effect is shown in Fig. 1.
(d) The peaks and valleys of the roughness signals in the roughness curves become wider and wider with the PEO treatment time, which is related to the pore size and population in the coatings as shown in Fig. 2.

Fig. 2 illustrates the surface morphology of the ceramic coatings for different treatment time. It was shown in Fig. 2(a) that after a PEO process of 45 s, scrub traces on the substrate surface still exist. Many small pores with discharge melting feature can be seen on the surface at larger magnification, Fig. 2(b). The pores serve as the discharge channels. Most of the molten oxide in the channels was expelled out of the channels and solidified onto the coating surface around the channels, which creates a lot of craters as can be seen in Fig. 2(b) and (c). It also can be seen in Fig. 2 that the pore size apparently increases and the pore population decreases with the treatment time.

The statistical results of the channel diameter and number versus the PEO time at the current density of 10 A/dm^2 are shown in Fig. 3. The diameter increase of the pores conforms to a linear relation with a slope of 0.22 μm/min. The decrease of the pore population with treatment time is very fast at first,