

# A facile preparation of ceramic coatings on Ti alloys for thermal protection systems

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## ABSTRACT

In order to reduce the effects of solar energy on the instruments and the apparatus in the outer space, two different kinds of ceramic coatings were prepared on Ti6Al4V alloys by plasma electrolytic oxidation (PEO) in zirconate electrolytes. The phase composition, microstructure, thickness, and roughness of coatings were examined by XRD, SEM, EDS, thickness measurement gauge, and roughness measuring instrument, respectively. The infrared emissivity and solar absorbance of the ceramic coatings were studied with the UV–vis–NIR spectrophotometer and infrared reflectometer. The results show that ZrO<sub>2</sub> coatings are dense and deep gray–green, and mainly composed of *m*-ZrO<sub>2</sub>, *t*-ZrO<sub>2</sub>. In addition, a small amount of TiO<sub>2</sub>, ZrTiO<sub>4</sub>, and the amorphous compound containing P exists in the coating. The intrinsic solar absorbance and emissivity of ZrO<sub>2</sub> coatings are 0.92 and 0.76, respectively. The KZr<sub>2</sub>(PO<sub>4</sub>)<sub>3</sub> coating is rough, thick, and white with the main crystalline of KZr<sub>2</sub>(PO<sub>4</sub>)<sub>3</sub>. The intrinsic solar absorbance and emissivity of the KZr<sub>2</sub>(PO<sub>4</sub>)<sub>3</sub> coating are 0.62 and 0.91, respectively. For both coatings, the absorbance decreases while the emissivity increases due to an increase in the real surface area of the coatings by increasing thickness and roughness of the coatings as the PEO time progresses. The porous structure of the KZr<sub>2</sub>(PO<sub>4</sub>)<sub>3</sub> is proved to be ideal for high emissivity and low absorbance materials with the lowest solar absorbance (0.239), the highest infrared emissivity (0.99), and a corresponding balance temperature of the coating of 276 K.

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## 1. Introduction

The conversion and utilization of solar energy is an important issue under the condition of the current shortage of energy [1–3]. The disposal of solar energy, *i.e.* reducing the harm caused by the sun, is also worthy of attention. When the spacecraft is operating in the orbits, the large thermal gradient between the sunlit and shadowed sides is detrimental to the performance of the precise instruments and apparatus and their components, such as circuit boards, microchips, and solar cell [4–6]. Fortunately, such problems by the sun radiation can be eliminated through a thermal protection system to a certain degree. Among various thermal protection systems, using temperature control coating as a method of passive thermal control works through adjusting the solar absorbance ( $\alpha_s$ ), the fraction of incident solar energy that a surface absorbs, and the emissivity ( $\epsilon$ ), which is the fraction of heat that

the surface radiates compared to that of a blackbody at the same temperature.

In recent years, various methods of temperature control coatings have developed, such as painting and brushing [7], plasma spray [8], sol–gel [9,10], anodizing [11], chemical vapor deposition [12], magnetron sputtering [13,14], and ion implanting [15]. Consequently, various temperature control coatings have been successfully prepared with different ratios of absorbance and emissivity ( $\alpha_s/\epsilon$ ). However, these methods are usually of high cost or of weak adhesion between the coating and the substrate. Therefore, it is of great significance to develop a novel preparation method for temperature control coating to reduce the effects of the solar energy on the satellites and space vehicles in the aerospace field.

Thus, we proposed the well-developed plasma electrolytic oxidation (PEO) technique for the preparation of the temperature control coating. The oxide ceramic coatings prepared by this technique have excellent adhesion strength with the substrate and decent stability under various corrosive environments. The composition and structure of the ceramic coatings can easily be adjusted by the doping of the electrolyte and the changing of

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process parameters [16–20]. In this study, we successfully prepared two kinds of temperature control ceramic coatings on Ti alloys by plasma electrolytic oxidation in  $K_2ZrF_6$  and  $NaH_2PO_4$  electrolyte solution. The composition, structure, and temperature control property of coatings were also investigated, and their inherent relations were discussed.

## 2. Experimental details

### 2.1. Preparation of ceramic coatings

Plate samples of Ti6Al4V with dimension (40 mm × 40 mm × 0.8 mm) were polished by abrasive papers and washed in distilled water. A homemade, pulsed electrical source of 10 kW was used for plasma electrolytic oxidation of samples in a water-cooled electrochemical bath made of stainless steel, which was served as the counter electrode. During the PEO treatment, the sample was the positive electrode, and the temperature was controlled to be below 30 °C using a cooling water flow. The electrolyte solution contained 6 g/L  $K_2ZrF_6$  and 1 ml/L  $H_3PO_4$  (85%). The coatings were prepared at the current density of 10 A/dm<sup>2</sup>, the duty ratio of 45%, and the working frequency of 50 Hz for different times. After the treatment, the coatings were flushed with distilled water and dried in air.

### 2.2. Coating characterizations

The surface morphologies and elemental compositions of the coatings were examined by a JSM-6480 scanning electron microscopy (SEM, Japan's Hitachi LTD) equipped with energy dispersive spectroscopy (EDS, JED-2200, Japan). The phase composition of coatings was examined by TTR-III type X-ray diffraction (XRD, D/max-rB, Ricoh, Japan). The coating thickness was measured with an eddy current coating thickness measurement gauge (TT260, Time Group Inc., China). The thickness was the average of 10 measurements made at different locations of the sample. The coating roughness was examined with surface roughness measuring instrument (TR200, Time Group Inc., China).

### 2.3. Evaluation of temperature control property and calculation of balanced temperature

The emissivity of the coatings was measured by a TEMP 2000, a portable infrared emissivity spectrometer in the range 250–2500 nm at room temperature. The Perkin–Elmer Lambda 950 UV–vis–NIR spectrophotometer was used to measure the absorbance of PEO coatings.

The balanced absolute temperature ( $T$ ) in K can be described by Eq. (1) [21]

$$T = \sqrt[4]{\frac{S\alpha_s A_p}{\sigma \epsilon A_R}} \quad (1)$$

where  $S$  is the solar constant (0.135 w/cm<sup>2</sup>),  $\alpha_s$  is the solar absorbance of the exterior surface,  $A_p$  is the projected surface area in cm<sup>2</sup> of the spacecraft perpendicular to the solar rays,  $\sigma$  is the Stefan–Boltzmann constant,  $\epsilon$  is the emissivity at temperature ( $T$ ), and  $A_R$  is the total “effective” area in cm<sup>2</sup> for heat radiation. The smaller the value of  $\alpha_s$  and the larger the value of  $\epsilon$ , the lower the surface balance temperature.

## 3. Results and discussion

### 3.1. Cell voltage–time curves

Fig. 1 shows the typical voltage–time curve of the PEO process [19,20], which can be divided into three simple stages. Within the initial minutes, the voltage rapidly ascended until the spark voltage of the oxide film was reached; this is referred to as Stage a. Then, the voltage increased slowly as the spark discharged onto the metal electrode surface, which is the main period for coating growth. According to the slope of the voltage increase, it can be divided into Stage b and Stage c. It is clear that the increasing speed of the voltage at Stage b is less than that of Stage c, which may be related to the different growth characteristic at different stage.

### 3.2. Structure and composition of the coatings

Fig. 2 shows the thickness and roughness of the ceramic coating. The coating thickness increases slowly within the initial 10 min, then presents a quick increasing trend, which means that the coating growth speed at the second stage and the third stage is distinctively different, which corresponds to the voltage–time curve in Fig. 1. In addition, the coating roughness increases with

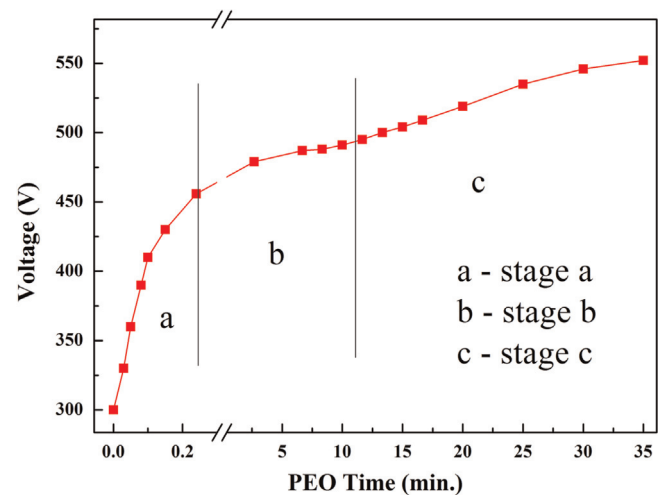


Fig. 1. The voltage–time curve of the PEO process (current density: 10 A/dm<sup>2</sup>, duty ratio: 45% and frequency: 50 Hz).

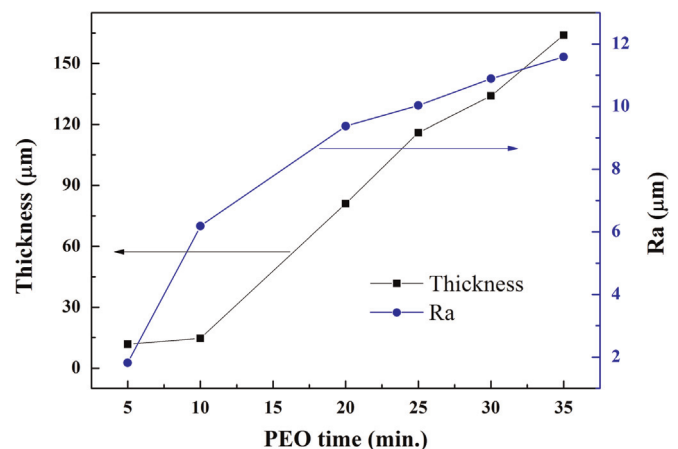


Fig. 2. Thickness and roughness of the ceramic coatings prepared for different time.

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