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Investigation of the Properties of Plasma Sprayed Titania

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Three different processing regimes with different processing temperature and their effect on coating microstructure and crystallinity have been investigated. The nano-sized coatings were deposited employing non-equilibrium plasma spraying technology at atmospheric pressure DC plasma torch. Microstructure of polished cross-section of the as-sprayed coatings was investigated by optical and scanning electron microscopy (SEM). Phase analysis and crystallite size of the plasma sprayed titania were identified by X-ray diffractometry (XRD). Coatings properties such as density and porosity were dependent on the spray conditions.

Keywords: coating, plasma spraying, microstructure, TiO_2.

1. INTRODUCTION

High temperature oxide ceramics, based on zirconia-, alumina-, and titania-oxide are widely used due to their superior properties, such as high heat resistance, high mechanical strength, and high ionic conductivity at high temperatures [1 – 3]. Zirconia-based ceramics fabricated using different processing techniques are successfully applied for solid oxide fuel cells components [4, 5]. Alumina, titania and zirconia coatings are used as thermal-barrier coatings, as bioinert bond coats and coatings for biomedical applications [6 – 8].

However, advanced electronics and up-to-date branches of industry need advanced methods for ceramic processing in these applications. The powder-based processing requires multi-step operations with high temperature sintering process to provide the final properties and dimensions of the final product. These steps are long-lasting and require a great deal of time to complete.

Ceramics can also be manufactured using non-powder based methods. Plasma spraying can be used as an economic alternative method to these processes. This process combines melting, rapid quenching and consolidation into a single step and can be used for any ceramic material that melts without decomposing [4 – 9]. Plasma spraying is gaining a great deal of interest in the high-temperature oxide ceramic field compared to other manufacturing methods. It concerns also thin ceramic films, deposited on various substrates.

A lot of processing variables including plasma gun configuration, process gases, flow rates, pressures and power, spray distance many others have influence on the properties of plasma sprayed coating [10 – 12]. The powder variables include particle size distribution, chemistry and morphology of the material used.

In this study the coatings structure and surface morphology were optimized on the basis of the plasma spraying conditions. Among the parameters examined were the plasma jet parameters and as-sprayed ceramic properties. The results reported here represent the structural data of plasma sprayed ceramic coatings from titania sprayed employing non-equilibrium plasma spraying technology at atmospheric pressure [13], suitable for various engineering applications such as thermal barrier coatings or biocoatings for health and medical systems.

2. EXPERIMENTAL

The coatings reported here were deposited on the polished titanium steel sheets employing non-equilibrium plasma spraying technology at atmospheric pressure DC plasma torch [9, 13]. The main operating parameters of the plasma torch: power supply (P) – 35 – 40 kW, arc current (I) – 120 – 200 A, voltage (U) – 225 – 300 V, total gas flow rate (G) – 4.9 gs^{-1} (the main gas flow rate through plasma torch – 2.65 gs^{-1}, additional gas flow – 2.25 gs^{-1}, hydrogen – 0.15 gs^{-1}), the average velocity (v) – 650 – 1350 m/s.

Powder injection was provided into a reactor, which was connected directly to the plasma torch anode. Outlet powder temperature was controlled by pyrometer. The plasma power used was in the range 30 to 36 kW. Table 1 summarizes the experimental conditions.

A commercial titania powder about 4 μm in size was used for the plasma spray deposition. After being dried the powders were used to form plasma sprayed coating on the substrates. To obtain a uniform coating, the substrates were placed on the fixture, which could rotate during plasma spraying, 20 – 100 mm away from the nozzle of the torch. Prior to plasma spray, the substrate surface was hand-polished to 0.05 μm finishing. All substrates were cleaned by acetone and dried in air before they were used. The substrate temperature was controlled by the optical pyrometer. The thickness of the titanium steel substrates was 1.2 mm.

Major characterization techniques included optical and scanning electron microscopy (SEM) and X-ray diffraction (XRD). The microstructure was characterized using a SEM (JSM 5600) and image analysis system, consisting of a microscope, personal computer and image software *NIH*

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Image 1.61. Phase composition of the powder as well as sprayed films were analyzed by X-ray diffractometer (DRON-6) with Cu-Kα radiation. The mean crystallites sizes were estimated from integral width of the diffraction profiles of rutile and anatase by XFIT program [14]. The density and porosity of plasma-sprayed films were evaluated from the data of SEM analysis. The film thickness was evaluated from the cross-sectional scanning electron microscopy photographs.

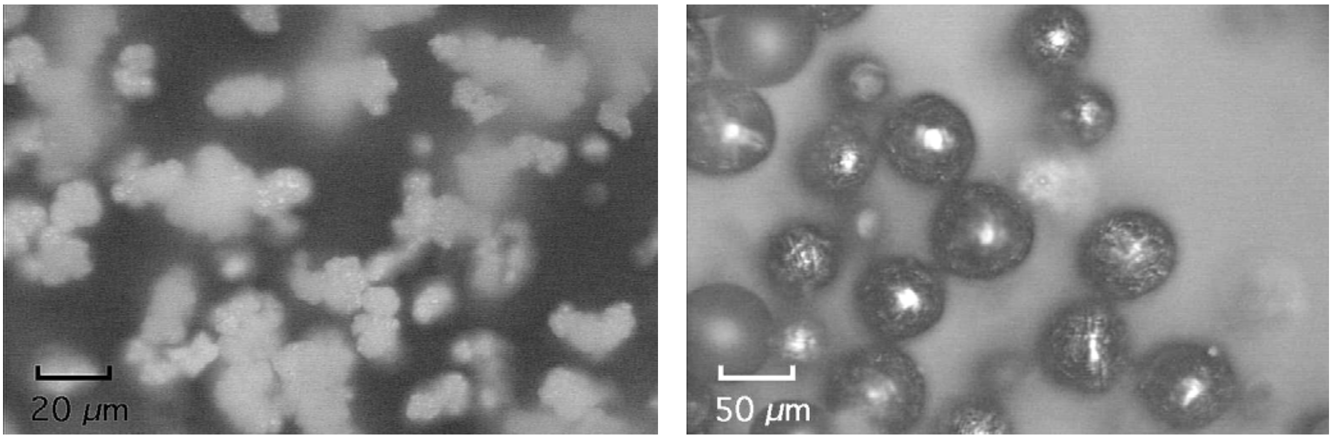
3. RESULTS AND DISCUSSION

To determine the effect of plasma spraying on the deposited ceramic properties, titania coatings were sprayed to produce samples for examination. The operating parameters for plasma deposition are presented in Table 1.

To understand the basic deposition process, the melting state of started powder was examined after passing the plasma jet. Morphologies of the initial as well as plasma-sprayed powders are shown in Fig. 1. The initial titania powder is in the form of agglomerates having average size 20 – 50 µm with slight powder size distribution having average particle size about 4 µm.

Table 1. Plasma spraying regimes for the ceramic films deposition

Spray regime	1	2	3
P, kW	30.8	35.8	36.1
G, g/s^{-1}	5.03	5	5.03
V, m/s	818	963	982
T, °C	2264	2546	2627
Spray distance, mm	70	70	70
Spray duration, s	20	20	10



a b

Fig. 1. Optical micrographs of TiO_2 powders: initial (a) and plasma-sprayed by regime 2 (b)

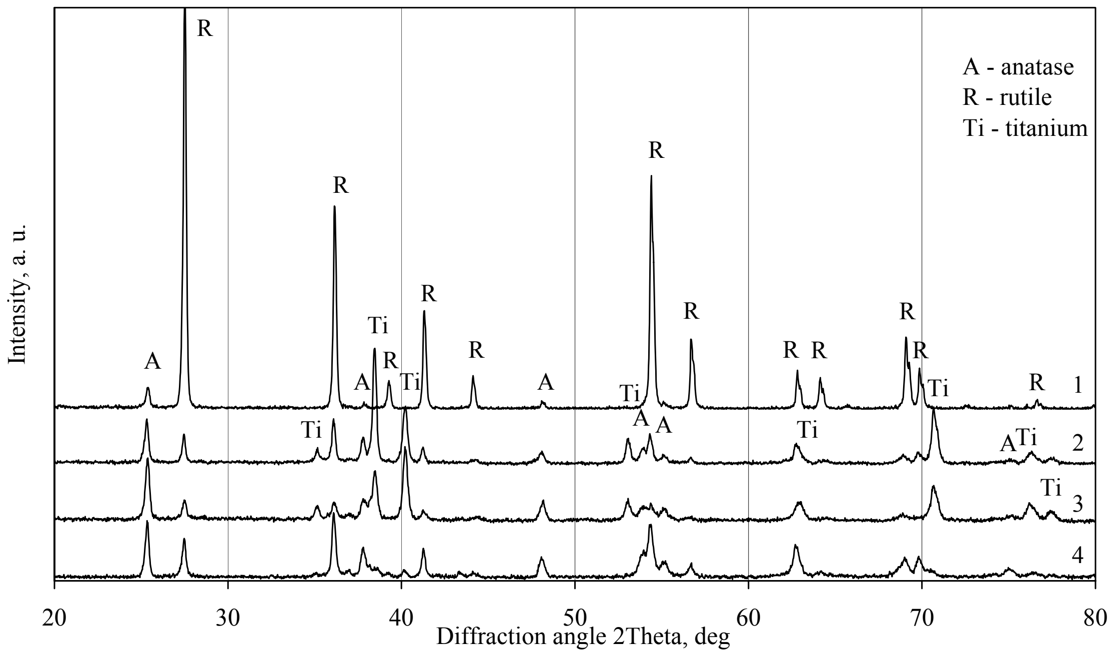


Fig. 2. The XRD patterns of titania: 1 – started powder and 2 – 4 – plasma sprayed titania: 2 – regime 2, 3 – regime 1, 4 – regime 3

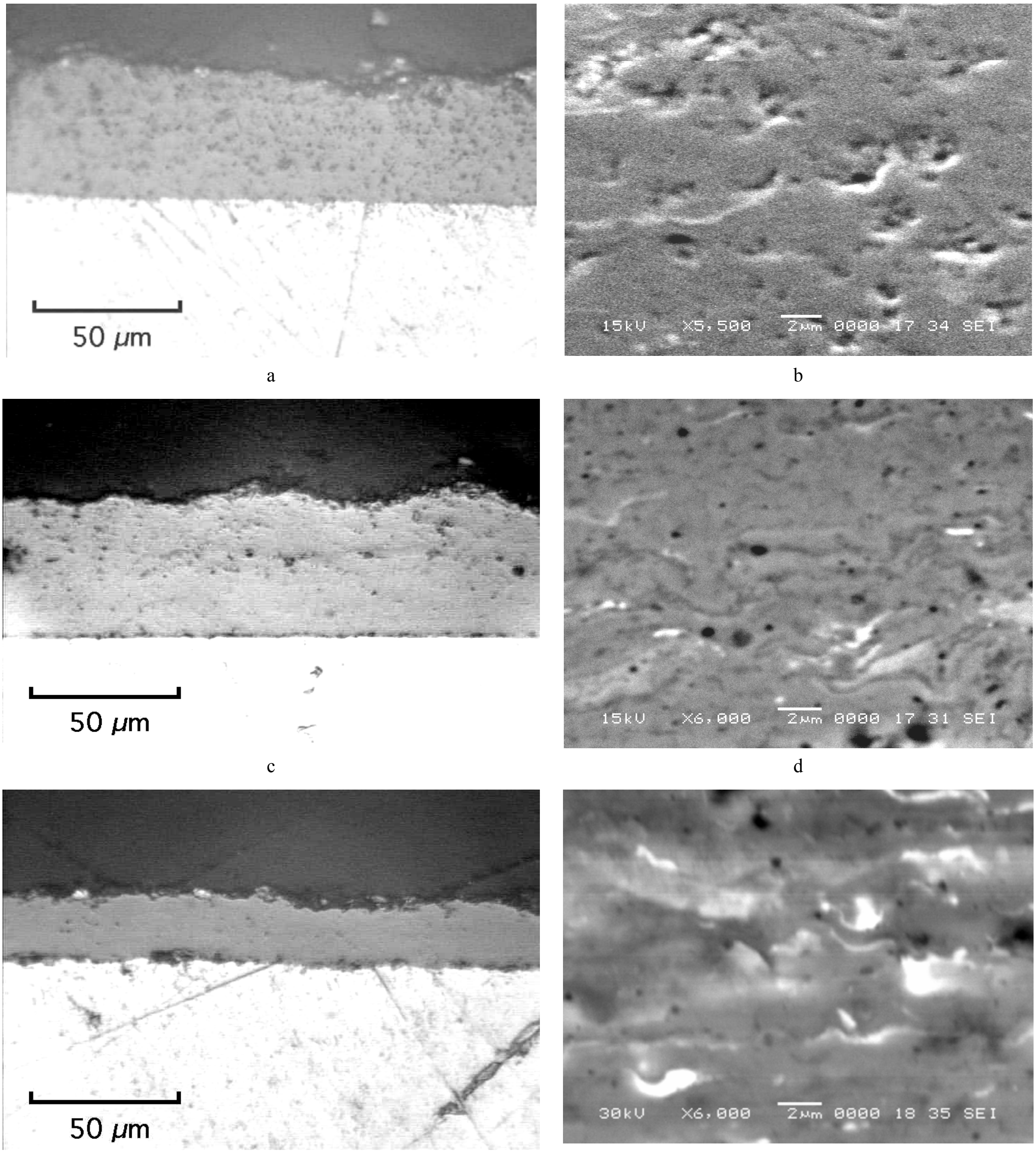


Fig. 3. Optical views (*a, c, e*) and SEM micrographs (*b, d, g*) showing the cross section surface morphology of plasma sprayed titania deposited by regime 1 (*a, b*), regime 2 (*c, d*) and regime 3 (*e, g*). Spray duration 20 s (*a* and *c*) and 10 s (*e*)

From the data obtained, it was determined, that during plasma spraying all titania powder was completely melted and spheroidized. Ball-shaped surface morphology is typical for all as-sprayed powder (Fig. 1, *b*). The average size of the powder after passing through the plasma jet is about 50 μm. The XRD patterns of started powder and deposited titania as a function of deposition regime are given in Fig. 2. The phase composition of started powder is the mixture of rutile (R) and anatase (A) (Fig. 2, 1).

The rutile is a dominating phase in the powder. The analysis of peak positions in the XRD patterns of plasma-sprayed titania indicated the presence of anatase (A) and rutile (R) mixture too. There is no new phase, except the titanium peaks (Ti), attributable to the substrate.

The peak intensities present some difference according to the deposition conditions. As the process temperature was increased (regime 3), the intensity of the main peaks, corresponding to (110), (101) and (111) of rutile and (101) peak of anatase is increased, indicating an increased degree of crystallographic texture in the plasma deposited titania.

Figure 3 shows cross-sectional optical views and SEM micrographs of the polished surfaces of as-sprayed titania films. In comparison the samples sprayed by different regimes, the morphology of deposited coatings substantially differs, as shown in Fig. 3, *a* – *g*. The structure of the titania deposited at lower temperature (regime 1) is characterized by porous nature (Fig. 3, *a* and *b*). From the SEM analysis, small ball-shaped and elongated pores (approximately < 1 μm diameter) predominate in the structure. The distribution of pores is quite homogeneous. Less porosity is specific for the coatings deposited at higher temperature (regime 2 and 3).

Titania coatings deposited at higher temperature are qualified by finer and reduced porosity and increased density (Fig. 3, *c* – *g*). A quantity of randomly distributed small pores of different sizes < 1 μm observed in the coating micrographs is marginal.

According to the XRD data, the deposited titania is nano crystalline. The crystallite sizes of the started powder and plasma sprayed titania coatings deposited by regimes 1 – 3 are presented in Figure 4. The crystallite size of rutile is considerably less for plasma sprayed titania in comparison with the precursor powder. The crystallite size of anatase is slightly bigger for plasma sprayed titania.

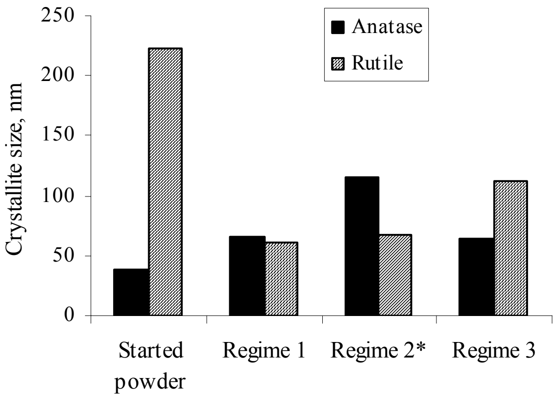


Fig. 4. The crystallite size of the started powder and plasma sprayed titania as a function of deposition regime. * Deposition temperature 2392 °C

The coatings adhesion and their thickness were evaluated from scanning electron microscopy data. According to the cross-sectional morphology features, the coating thickness depends on the spray duration and is about 50 μm, when the deposition time is 20 s and it is about 20 μm, in the case of spray duration 10 s. All the plasma sprayed titania coatings are well adhered to the substrate.

4. CONCLUSIONS

In this study, a plasma process for preparation of titania coatings is described. Use of the non-equilibrium plasma spraying technology at atmospheric pressure makes it possible to obtain well-adhered titania coatings with different porosity. The present study shows the data of phase analysis and the structural changes of the plasma sprayed titania deposited on the titanium steel sheets. According to the data of the XRD analysis two crystalline phases are dominated in the titania coatings – rutile and anatase. Plasma sprayed titania is nanocrystalline.

The process regime significantly affects the structure of titania coatings. The coatings deposited at higher temperature are qualified by finer and reduced porosity and increased density.

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