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# Correlations between processing parameters and microstructure for YSZ films produced by plasma spray technique

Kristina Brinkiene*, Romualdas Kezelis

*Lithuanian Energy Institute, Breslaujos str. 3, LT-3035 Kaunas, Lithuania*

**Abstract**

Yttria-stabilized zirconia (YSZ) powders have been used to prepare plasma-sprayed zirconia films on various substrates employing non-equilibrium plasma spraying technology at atmospheric pressure. The properties of obtained YSZ films 20–60 μm in thickness are presented. This paper shows how the spray regime affects the microstructure of obtained YSZ films by using different shapes of plasma torch-Laval-like contour and cylinder. The influence of process parameters on formation and morphology of as-sprayed films has been investigated using the data of microstructural analysis. The results indicate plasma torch shape and started powder characteristics to be the most important parameters influencing the film microstructure.

*Keywords:* Films; Microstructure; Plasma spraying; $ZrO_2$

## 1. Introduction

At the present time low temperature plasma is widely used in fundamental research and industry. It also concerns thin ceramic films, deposited on various substrates.[1–3]

Plasma jet parameters, distance from the plasma outlet to film surface and the powder injection place mainly influence the film microstructure, which depends on whether the particle is partially or fully melted, its velocity and substrate temperature.[2,3] Unfitting spraying parameters produce a complex failure consisting of a wide variety of cracks, pores and other defects.[4,5] Such defects influence the conductivity and other properties of YSZ films.

The aim of this work was to study the relationship between plasma flow parameters together with initial powder properties and the process of formation of well adhered thin ceramic films deposited on the sheets employing non-equilibrium air plasma spraying technology at atmospheric pressure.

## 2. Experimental

When plasma technologies are used in industry or science investigations, it is important to have plasma torches with long operation time and stable outlet plasma jet parameters. For this aim in the Laboratory of Plasma Technologies of the Lithuanian Energy Institute a special test bench was built.[6,7] It consists of following main systems: electricity supplies, gas supply, cooling system and operation control and data monitoring system. Continual date monitoring of an operating plasma torch allows the test bench functioning. A schematic view of the experimental set-up is shown in Fig. 1.

The average outlet jet temperature and velocity were determined from heat balance. The capacity of the plasma torch, mass flow of gases, cooling water and its temperature were measured and gas enthalpy calculated. Gas temperature and velocity were determined numerically using gas property data for the ‘‘frozen’’ state of gases. Local jet temperature and velocity distributions were measured by means of a cooled calorimetric probe.[8,9]

The plasma torch was constructed so that powder injection could be provided internally or externally. Internal powder injection was arranged at different

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* Corresponding author.

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positions (Fig. 1). This allows feeding of high melting materials directly into the plasma core, expanding in this way spraying optimum conditions for different materials.

Parameters of the plasma torch ranged within the following limits: power supply (*P*)—35–40 kW, arc current (*I*) 120–200 A, voltage (*U*)—225–300 V, total gas flow rate (*G*)—4.9 g $s^{-1}$ (the main gas flow rate through the plasma torch—2.65 g $s^{-1}$, additional—2.25 g $s^{-1}$, hydrogen—0.12 g $s^{-1}$). Average plasma temperature in the powder injection place—3300–3700 °C, outlet plasma temperature—2750–3300 °C, the average velocity—650–1350 m $s^{-1}$, the working gas—air. Typical plasma spraying conditions are listed in Table 1.

Two shapes of anode exit mode were used for thin film deposition (Fig. 2). One of them was Laval-like exit contour (a). The powder was injected into the critical nozzle diameter (regime L). Laval-like exit contour results in a controlled expansion of the plasma core and leads to very high jet velocities. In this case the outlet plasma jet has extended hot core, delayed the detrimental influence of cold surrounding gas. The other was cylinder (b) 80 mm in length and 7 mm in diameter. In this case there was longer plasma–powder particle interaction time (regime C) and as result better particle-melting conditions.

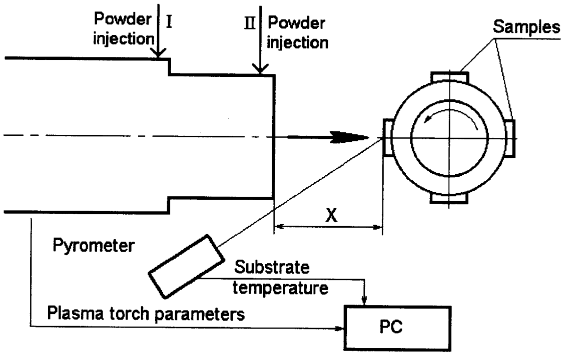


Fig. 1. Experimental set-up.

Table 1
Plasma spraying regimes for YSZ films deposition

| Regime<br>Anode exit shape | L<br>Laval contour | C<br>Cylinder |
|---|---|---|
| *P* (kW) | 45 | 47.5 |
| *G* (g $s^{-1}$) | 3.65 | 3.65 |
| *G*($H_2$) (g $s^{-1}$) | 0.1 | 0.1 |
| *T* (°C) | 3380 | 3180 |
| *x* (mm) | 20–100 | 20–100 |
| *v* (m $s^{-1}$) | 1120 | 980 |

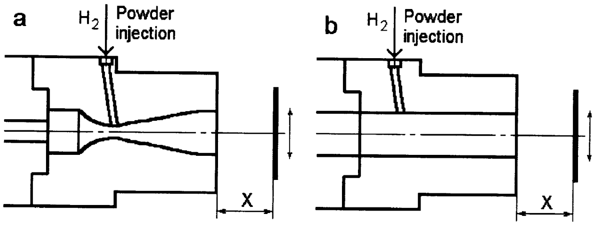


Fig. 2. Plasma torch anode exit shape: (a) Laval-like contour and (b) cylinder.

The substrates were mounted on a cylindrical fixture and rotated around their own axes. The plasma torch moving in horizontal direction creates the coating.

The objective of the present study is thus to investigate the effect of each spray regime on the structure of plasma-sprayed YSZ film. Major characterization techniques included scanning electron microscopy (SEM) and X-ray diffraction (XRD). The film thickness was evaluated by cross-sectional SEM observation. Specimens for cross-sectional microscopy were prepared by mounting the specimens in epoxy resin, followed by polishing through 0.05 μm alumina.

In this study the plasma spraying conditions for a yttria stabilized zirconia powder were optimized on the basis of the coating's structure and surface morphology. Among the parameters examined were the plasma jet parameters and started powder properties.

## 3. Materials

Commercial (Russian made) yttria stabilized zirconia powders (8 mol% $Y_2O_3$) were used in this work. The powders were sieved to obtain particles with sizes 50 μm (YSZ-1) and 63–100 μm (YSZ-2). After being dried, the powders were used to form plasma sprayed coating on various substrates.

Polished steal and nickel sheets were used as the substrate material. Prior to plasma spray, the substrate surface was hand-polished to 0.05 μm finishing. All substrates were cleaned with acetone and dried in air before they were used. To obtain a uniform coating, the substrates were placed on the fixture, which could rotate during plasma spray, 20–100 mm away from the exit of the torch, and heated by a plasma flame. The thickness of the steel and nickel substrates was 100 and 50 μm, respectively.

## 4. Results and discussion

As observed by scanning electron microscopy, the initial powder is in the form of agglomerates with wide size distribution (Fig. 3). The starting powders YSZ-l and YSZ-2 are irregularly shaped and differ only in size. Morphologies of plasma-sprayed powders YSZ-1 are shown in Fig. 3. YSZ-2 powder appears quite similar.

SEM observations of powders used have revealed a significant morphology differences between two spraying regimes. During plasma spraying by regime C, all size powders were completely melted and spheroidized. The spherical morphology promotes better flow ability of the powder during plasma spray deposition.[2] During the spraying regime L, the powders are heated at lower rate in this shape of plasma torch. Only some of small powders are partly melted; others have irregularly shaped surface morphology relating to higher jet velocity.

It was determined, that film properties strongly depend on started powder characteristics when powder is injected into Laval-like exit contour. The best results were obtained by spraying heat-treated (900 °C, 2 h) YSZ-2 powders (Fig. 4, regime L). Probably, the course grains impact the substrate with a higher momentum, and finally form a denser film.

From the data obtained with cylinder type plasma torch (regime C), it may be seen that homogeneous distribution of particles and pores is typical over the whole film thickness (Fig. 4). The better results were obtained by spraying powders with finer particle diameter (YSZ-1). The surface morphology of plasma sprayed YSZ film produced under regime C is more smooth and flat in comparison with film sprayed under operating regime L (Fig. 4).

For many of the plasma spraying conditions investigated in this work, it was observed that the density of

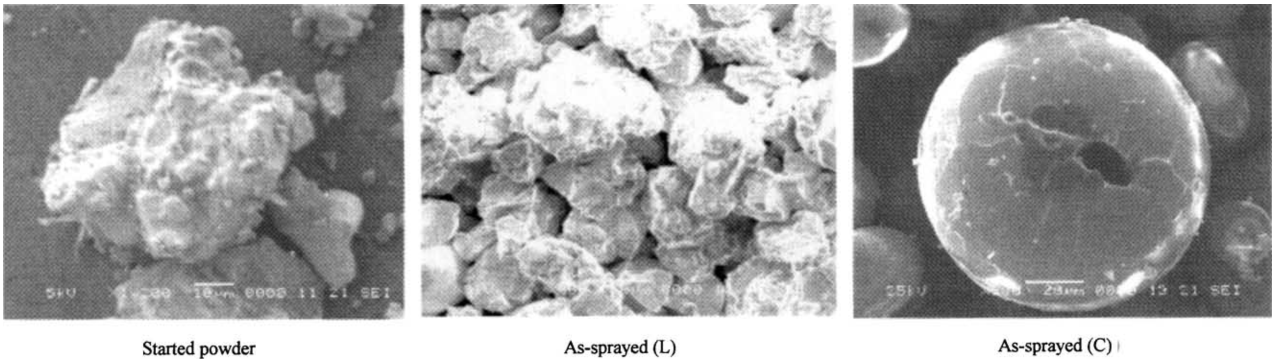

Started powder As-sprayed (L) As-sprayed (C)

Fig. 3. SEM micrographs of started powder and after passing through the plasma jet as a function of the processing regime.

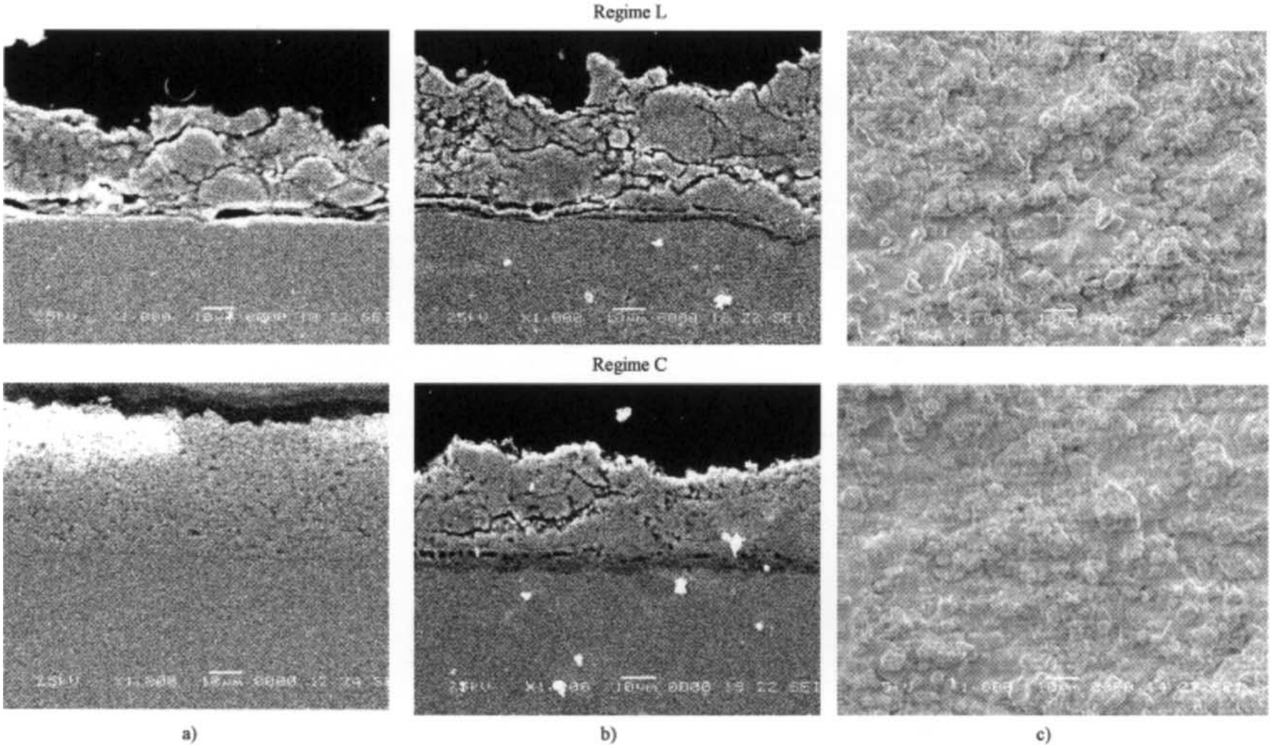
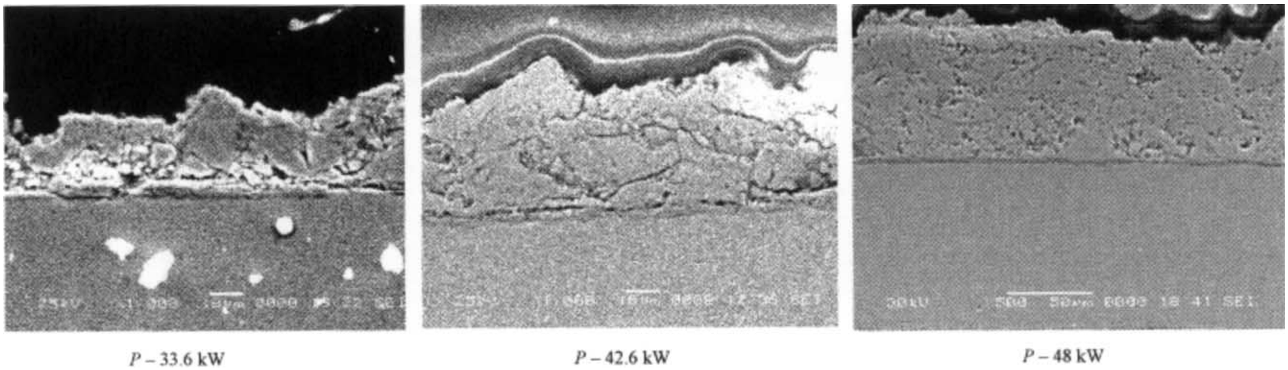


Fig. 4. Cross-sectional SEM micrographs (a, b) and surface morphologies (c) of sprayed YSZ films subjected to different spray regimes ($x$—50 mm): (a) YSZ-1, (b) YSZ-2.



*P* – 33.6 kW *P* – 42.6 kW *P* – 48 kW

Fig. 5. Influence of *P* on YSZ film properties. Regime C. *x*—70 mm. YSZ-1.

sprayed films varies greatly with the plasma spray conditions. An increase of density (3.7–5.21 g cm$^{-3}$) was observed by changing Laval-like contour to cylinder-shaped plasma torch. Increased plasma power (43–48 kW) produces a film with finer and reduced porosity and increased density from 4.97 to 5.21 g cm$^{-3}$ (Fig. 5). The reduced size of starting powder from 100 to 50 μm similarly affects the density of studied films.

The cross-sectional SEM micrographs showed that the thickness of all films was roughly between 10 and 60 μm subjected to the spray duration. The optimal spray duration is 60 s. The larger deviation in thickness is quite characteristic for plasma sprayed films deposited by regime L, using thermal treated powders due to the large surface roughness and worse degree of melting. The thickness of the sprayed film differs by 60 μm in thickest region up to 20 μm in thinnest region. It is determined, that the optimal spray distance for both regimes is 70 mm.

By the data of the XRD analysis (Fig. 6) no distinct peaks except cubic YSZ are found from XRD patterns of as-sprayed films.

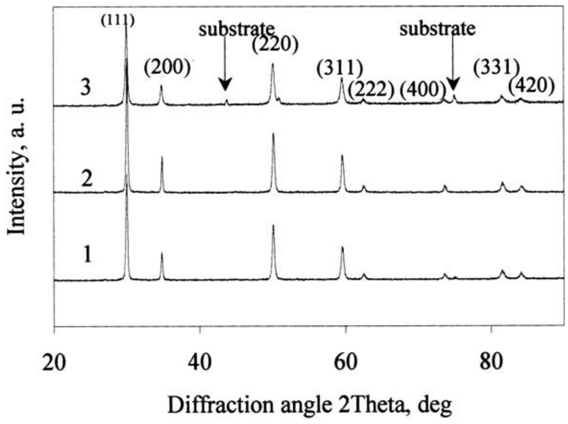


Fig. 6. The XRD patterns of YSZ: 1, initial powders (YSZ-1); 2 and 3, plasma-sprayed films obtained by regime C (2) and regime L (3).

## 5. Conclusions

In this study, the influence of plasma spray parameters on the microstructure of YSZ film was investigated with the aim of achieving better control of the process using two different regimes of plasma spray deposition technique.

Based on the results obtained, it can be deduced, that the variation of started powder properties and plasma parameters (especially plasma torch shape, plasma power and spray distance) has great influence on the structure of YSZ films. It was determined, that during the spraying process by regime L, the YSZ powders are heated at lower rate in this shape of plasma torch and this results in worse properties of sprayed film. The deposition of finer particle by regime C would produce film with relatively improved structure and increased density.

The thickness of film strongly depends on plasma spray parameters.

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