



Enhancement of scattering and reflectance properties of plasma-sprayed alumina coatings by controlling the porosity

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ARTICLE INFO

Available online 23 May 2012

Keywords:

Alumina
Optical properties
Atmospheric plasma spraying
Suspension plasma spraying
Porosity
USAXS

ABSTRACT

The plasma-spraying process generates materials with typical, porous and complex, microstructures. Inspired by dielectric multilayer mirrors (DMMs), thermal sprayed media may be used in the field of optics, particularly for making scattering and reflecting coatings suitable for a large range of wavelengths. In fact, pores inside plasma sprayed matrix create numerous optical index discontinuities, similarly to the gaps created in DMMs, in order to obtain high reflectivity.

The porosity of coatings microstructure can be customized by selection of plasma sprayed process parameters. This study aimed to optimize scattering and reflectance properties in porous alumina by the control of spray parameters resulting in the optimized porosity. A self-supporting bi-layer with a diffuse reflectance over 90% over a large band of wavelengths was obtained.

The first layer (micro-structured), which is thick enough to support the free standing, was prepared by atmospheric plasma spraying (APS). The second layer (nanostructured) was manufactured by suspension plasma spraying (SPS) over the first layer in order to enhance the reflectance at short wavelengths.

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1. Introduction

The increasing use of reflecting optics in ultraviolet (UV), visible, and infrared (IR) devices has stimulated a great interest in research on coatings for mirrors [1]. A way to improve reflectivity is to manufacture multilayer materials with different optical indexes between successive layers [2]. This gap of optical index is also observed in heterogeneous materials, such as porous plasma sprayed coatings, between the matrix and the pore [3]. Therefore, with sufficient control of the main features of the porosity (value and main pore size), these coatings could be another way to provide reflecting coatings [4].

Thanks to their high damage resistance, even in extreme conditions, plasma sprayed ceramics should be suitable for manufacturing of optical devices for high power laser. The French high power laser project (Laser Mégajoule) plans to initiate a fusion reaction by laser confinement [5]. A key point of this project is to characterize the interaction between the laser and the plasma during the experiments. As the energy flux is locally highly concentrated, an idea is to use a precisely calibrated panel made of ceramic material which will scatter the radiation and consequently redirect only a part of it towards the detection cameras. The spectral range of operation is from 300 nm to 800 nm.

Plasma-sprayed materials have typically heterogeneous, usually porous, microstructure. The porosity produces optical index gaps between the air within the pores and the matrix resulting from this volume scattering depends on the shape and the size of the heterogeneities [6]. In order to enhance the Mie scattering, the microstructure has to be suitable to the incident radiation and the pores size has to satisfy the following relationship:

$$x = \frac{\pi \times \text{Re}(n^*) \times d}{\lambda_0} \approx 1 \quad (1)$$

where x is the size parameter, n^* the complex optical index of alumina, d the average pore diameter and λ_0 the wavelength of the incident radiation in vacuum.

This equation shows that when the wavelength decreases, the pores sizes have to decrease too to conserve the size parameter. This relationship also allows us to estimate the approximate optimal pore sizes as function of wavelength to observe Mie diffusion as calculated in Table 1. With an incident radiation at $\lambda = 300$ nm, the efficient pore sizes to provide Mie diffusion is about between 25 and 100 nm; at $\lambda = 800$ nm, the pore sizes has to be between 100 and 300 nm.

In this study, a self supporting bi-layer was manufactured by plasma spraying. Alumina was chosen for suitable optical properties, particularly its low absorption coefficient in the considered wavelengths range [7,8]. The first layer and its pores size distribution are specifically adapted for

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Table 1

Values of calculated diameters d of pores according Mie parameter and incident wavelength.

	$x = 0.5$	$x = 1$	$x = 2$
$\lambda = 300$ nm	26 nm	53 nm	106 nm
$\lambda = 800$ nm	70 nm	140 nm	282 nm

the higher wavelengths in the working range. The second nano-structured layer is manufactured by suspension plasma spraying over the first one in order to enhance optical properties at short wavelengths. This layer has much smaller pore size distribution to enhance Mie scattering at these short wavelengths.

2. Experimental procedure

2.1. Coating preparation procedures

As explained above, two techniques were used to produce the bi-layer system: atmospheric plasma spraying and suspension plasma spraying processes. Aluminum substrates (25 cm^2 in surface and 2 mm thick) with an average roughness (R_a) of about $4\text{ }\mu\text{m}$ and with a thermal expansion coefficient ($20 \times 10^{-6}\text{ K}^{-1}$) were chosen. The substrates were coated with fused alumina powder ($-22 + 5\text{ }\mu\text{m}$, H.C. Starck, Germany) for obtaining the microstructured layer. Next, the nanopowder (100 nm, Goodfellow, France) was mixed with distilled water, ultrasonically and mechanically stirred to break up the agglomerates. The concentration of the solid in the suspension was 10% in weight. No dispersant was added to the suspension since a sedimentation test showed that the sedimentation time was far higher than the spraying time. The suspension was injected into the plasma jet by using a mechanical feeder consisting of a pressurized reservoir in which the suspension is stored and forced through a precision nozzle ($250\text{ }\mu\text{m}$ in internal diameter) to provide the nano-structured part of the bi-layer system [9].

The two layers were manufactured with the same F4-VB plasma torch (Sulzer-Metco AG, Wohlen, Switzerland) equipped with 6 mm internal diameter nozzle. The operating parameters are summarized in Table 2 [10,11].

At the end of the process, the coatings were submerged in liquid nitrogen in order to create a thermal shock to separate the coatings from the substrates.

2.2. Phase and microstructure analysis

The phase analyses were carried out by X ray diffraction (XRD) using a Siemens D501 diffractometer with the $\text{Cu-K}\alpha$ radiation ($\lambda = 1.5406\text{ }\text{\AA}$) at an acquisition rate of $0.005^\circ\text{ s}^{-1}$.

The coating microstructures were observed by optical microscopy and with the scanning electron microscope (SEM) XL30 (Koninklijke Philips Electronics N.V., Amsterdam, The Netherlands) on polished and fractured cross sections.

Table 2

Plasma sprayed experimental setup for the microstructured layer and the nanostructured layer.

	APS	SPS
Ar flow rate (SLPM)	12	40
He flow rate (SLPM)	45	0
H_2 flow rate (SLPM)	3	10
Current (A)	500	600
Voltage (V)	53	68
Spray distance (mm)	100	40
Gun traverse speed (m s^{-1})	1.5	–
Feeding rate (g min^{-1})	20	–
Pressure injection (bar)	–	3

The open porosities were determined by the hydrostatic pressure method.

Pores size distributions of microstructured coatings and of bi-layer coatings were measured by mercury intrusion porosimetry (Micromeritics Autopore III 9410). The measurable pore sizes ranged between a few nanometers and $100\text{ }\mu\text{m}$ by applying pressure from 0.04 up to 400 MPa and assumed mercury wetting angle of 130° .

Pores microstructures of the coatings were also characterized by ultra-small angle X-ray scattering (USAXS) [12] to obtain quantitative pore size distributions for pores smaller than about $2\text{ }\mu\text{m}$ of the two layers following methods described in Ref. [13–15]. The experiment was conducted at the ChemMatCARS beamline 15-ID [16] at the Advanced Photon Source, Argonne National Laboratory. The instrument was using X-ray energy of 16.9 keV to optimize the sample transmission and minimize effects of multiple scattering. Beam size was about 2 mm (width) $\times$ 0.8 mm (height). Samples thickness was about 0.1 mm , giving total scattering volume of sample characterized around 0.16 mm^3 . These samples were sprayed by APS and SPS separately, with the same parameters used to obtain the bi-layer system. The coatings were separated from the aluminum substrates by thermal shock. Data were analyzed using Irena data analysis package [17].

2.3. Optical properties

The optical properties reflectance properties of the coatings were measured from 300 nm to 800 nm with the help of a UV–visible Varian Cary 5000 spectrometer (Varian Inc., Gloucester, MA, USA). The measurements were made on coatings removed from their substrates. As plasma sprayed coatings are highly scattering media, an integrating sphere was mounted to collect the reflected hemispherical flux. This sphere is made of a highly-reflecting inner coating (Spectralon®) so that the reflected light undergoes multiple reflections within the sphere and eventually reaches a detector mounted on the sphere. Spectralon® was used as a standard with 100% of reflectance.

3. Results and discussions

3.1. Phases and microstructure analyses

The XRD analyses (Fig. 1) indicates that all alumina plasma sprayed coatings crystallize in a mixture of α and η phases. The presence of the η phase is due to the high thermal flux within the plasma, which causes the decomposition of alumina [18].

The observation by optical microscopy of the bi-layer material (Fig. 2) shows a good adherence between the two layers. The micro-structured layer, which is manufactured by APS, has a typical porous microstructure

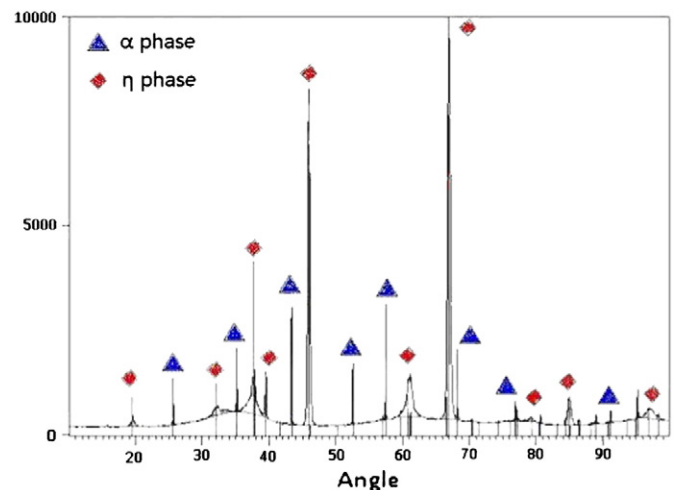


Fig. 1. XRD pattern of plasma sprayed alumina coatings.

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