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OPTICAL COHERENCE TOMOGRAPHY FOR INSPECTION OF HIGHLY SCATTERING CERAMIC MEDIA: GLASS POWDERS AND PLASMA-SPRAYED COATINGS

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ABSTRACT. Optical coherence tomography (OCT) is used to collect images of spherical glass powders with mean diameter ranging from 10 to 150 microns. Appropriate peak distance measurements on image interferograms provide a good approximation of particle size distribution. The relationship between the light penetration depth inside powders and the particle diameter is also discussed based on Mie theory. Thereafter, OCT images of plasma-sprayed ceramic coatings are presented as well as an approach for establishing light penetration depth inside zirconia coatings.

Keywords: optical coherence tomography, powder sizing, ceramic coating

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INTRODUCTION

Optical coherence tomography (OCT) is a nondestructive technology that has been predominantly developed for cross-sectional imaging of internal microstructure in living biological systems [1]. OCT combines the principles of low coherence interferometry and optical heterodyne detection to achieve both a high sensitivity to weakly backscattered light and a high axial resolution. The actual performance of OCT systems varies widely according to selected design and data acquisition hardware, but a sensitivity of -90 dB and an axial resolution up to 2 μm can generally be obtained [2]. Such performance ensures a promising future to OCT technology for the characterization of several translucent materials. In fact, this technique has already proved its capabilities to image polymer matrix composites and help to predict their permeability and mechanical properties [3], to detect and characterize subsurface defects in Si_3N_4 ceramic balls [4] and to measure the hole pattern in microstructured optical fibers [5].

In the present work, two additional applications of OCT are explored. First, optical coherence tomography is applied to study the size distribution of five different glass (silica) powders with mean diameter ranging from 10 to 150 μm . Appropriate inter-peak distance measurements on powder interferograms are used to retrieve the distribution, and the results are then compared to the actual glass powder distribution obtained by laser

diffraction. The light penetration depth is also determined from these interferograms and its relationship to powder distribution is discussed based on Mie scattering theory.

In a second application, OCT is evaluated as a promising technique for microstructure characterization of plasma-sprayed ceramic coatings. Plasma-sprayed ceramic coatings are widely used as thermal barrier and protective coatings against wear and corrosion in the automotive and aeronautical industries. In these applications, the quality of ceramic coatings is critical since they should resist to extreme conditions of utilization. However, plasma spraying is a stochastic process through which feedstock ceramic powder is melted and accelerated before impinging the substrate to rapidly solidify and form splats. Therefore, the resulting microstructure of coatings is characterized by the existence of interlamellar pores, globular pores and splat interfaces which significantly influence coatings thermal conductivity and mechanical properties [6]. It thus becomes imperative to detect the presence of such porosity, ideally with a nondestructive evaluation (NDE) technique, in order to avoid catastrophic failure of the material and optimize its performance. In this regard, OCT presents an axial resolution compatible with splats optical thickness as well as a good sensitivity to ceramic/air interfaces. In particular, OCT is expected to be sensitive to interlamellar contact.

In this work, OCT interferograms of yttria stabilized zirconia (YSZ) plasma-sprayed coatings are collected and analyzed individually to successfully gather information related to light penetration depth inside YSZ coatings. This interferogram analysis also allows the evaluation of the refractive index of YSZ non-transformable tetragonal phase.

PRINCIPLES OF OCT

As shown in Figure 1, an OCT system is actually based on a Michelson interferometer. The light emitted by a broadband source (superluminescent diode – SLD) is divided at a beam splitter into two distinct optical paths that are respectively oriented towards the reference mirror and the sample. When the light returns, the measurement is performed by evaluating the group delay into the sample arm as compared to the reference arm. When this delay is shorter than the light coherence length, the backscattered field from the sample combines with the reference field to produce an interference signal called interferogram. The resulting interferogram is then collected by the photodetector and submitted to further electronic processing. By recording this interference signal as the reference mirror is synchronously translated, the axial profile of the sample backscattering properties can be obtained [7]. The axial resolution in this profile depends on the coherence length of the light source and a high resolution can be achieved independently of the beam focusing conditions, thus of the sample arm optics. In fact, for a light source with a Gaussian spectral distribution, it can be shown [7] that

FIGURE 1. Schematic representation of an OCT system.

FIGURE 2. SEM micrographs and size distributions (as obtained using a commercial laser diffraction system) of three different glass powders.

the axial resolution Δz of the OCT system is inversely proportional to the bandwidth $\Delta\lambda$ of the power spectrum centered at a wavelength λ :

$$\Delta z = \left(\frac{2 \ln 2}{\pi} \right) \left(\frac{\lambda^2}{\Delta\lambda} \right). \quad (1)$$

MEASUREMENTS IN GLASS POWDERS

Experimental

Spherical glass powders of five different size distributions were obtained by induction plasma spheroidization [8]. Glass powder shape and distribution were respectively characterized by scanning electron microscopy and by laser diffraction (Beckman Coulter LS 13 320 Particle Size Analyzer). Results are shown in Figure 2 for three of the powders.

The glass powders were poured and compacted into small cups to form dense glass particle stacks and were thereafter characterized using an OCT system. A representation of the sample arm is shown in Figure 3a. The optical source coupled into the interferometer was a Covega SLED-236 which emits over a 70.2 nm wide bandwidth centered at a wavelength of 1320.3 nm. The maximum emitted power was 17 mW. In the sample arm, the 4-mm wide collimated beam was focused onto the powder surface using a lens with a focal length of 25 mm. For each powder, 500 A-scans (OCT interferogram envelopes) were acquired to form a single B-scan and the procedure was repeated 50

FIGURE 3. Schematic representation of the OCT sample arm used to characterize a) glass powders and b) plasma-sprayed ceramic coating.

FIGURE 4. Typical OCT B-scans for three different glass powders. The logarithmic amplitude is represented on a gray scale, black being the absence of signal and white standing for larger signal.

times. The step size between two successive A-scans was 5 μm , and the separation between two consecutive B-scans was 20 μm .

Results and discussion

For spherical glass powders, the objective was to identify relevant interferogram parameters related to powder diameter and thus, to retrieve powder size distribution from available images. Figure 4 shows typical OCT B-scans obtained for the three powders presented in Figure 2. As the powder diameter increases, the B-scan image takes a granular look and the expected spherical shape can be observed in the first particle layer of powder 5. Moreover, light penetration depth seems longer for larger powder diameters. Thus, two approaches are investigated to retrieve powder size from the B-scans: a processing method for first particle layers (from which a diameter distribution for surface particles will result) and another method using all particle layers in a powder section.

The first approach is based on the hypothesis that the maximum signal peaks will be obtained for the imaging conditions shown in Figure 5. The sample beam impinges right on the top particle surface, gets through its diameter and exits at the bottom surface. Both air/glass interfaces along the optical path result in an abrupt refractive index variation which generates a reflected signal towards the interferometer. Therefore, by evaluating the distance between the two corresponding peaks on the interferogram, a measure of the glass particle optical diameter ($\phi_{\text{optical}} = n_{\text{glass}} \phi_{\text{geometric}}$) is obtained. This procedure of inter-peak distance measurement is repeated for all appropriate A-scans selected by the application of an amplitude threshold. The resulting distribution of geometric diameters are plotted in Figure 6 and compared to the distribution obtained by laser diffraction.

The OCT powder size distributions agree well with those obtained by laser diffraction, except for the small powder. This observation can be explained by the fact that the axial resolution limit of the OCT system, as calculated by Equation 1, is 11 μm . Consequently, glass particles having a geometric diameter smaller than 7.3 μm will not be identified by the system, thus explaining the observed OCT distribution right shift for the small powder. Nevertheless, the correlation between both techniques is quite excellent and

FIGURE 5. Relationship between inter-peak distance measurement and glass particle diameter.

FIGURE 6. Comparison of powder size distributions as obtained by optical coherence tomography (OCT) and laser diffraction (LS).

validates the hypothesis of applying a threshold criterion to select appropriate A-scans and retrieve first layer particle distribution.

The second approach to retrieve powder diameter relies on the dependence of light intensity attenuation (or light penetration) with powder diameter. According to Mie scattering theory, the extinction cross-section (C_{ext}) per unit particle volume (v) decreases with increasing particle radius (r) larger than $1 \mu\text{m}$ [9], as shown in Figure 7a. Thus, starting with the Beer-Lambert law of light attenuation through a slab of particles, the light penetration depth z_p at $I/I_0 = 1/e$ can be defined as:

$$I = I_0 e^{(-\alpha_{ext} z)} \Rightarrow z_p = \frac{1}{\alpha_{ext}} = \frac{1}{x \frac{C_{ext}}{v}} \propto r, \quad r > 1 \mu\text{m} \quad (2)$$

where I_0 and I are respectively the initial light intensity and the light intensity after a distance z into the slab, α_{ext} is the attenuation coefficient and x is the volume fraction of spheres into the considered slab. The proportionality to radius r is obtained by replacing the calculated extinction cross-section per unit sphere volume with its asymptotic limit shown in Figure 7a. Therefore, the light penetration depth inside the studied powder should increase linearly with powder diameter. If this relationship is verified and properly calibrated, mean powder diameter could be retrieved from light penetration depth.

FIGURE 7. a) Extinction cross-section per unit volume particle as calculated using Mie scattering theory. b) Determination of the penetration depth from A-scan attenuation curve fitting.

FIGURE 8. a) A-scan penetration depth as a function of position along the B-scan. An 8-point moving average filter was applied before plotting. b) Relationship between the average penetration depth and the mean powder diameter.

In an attempt to verify the above relationship, a light penetration depth measurement was performed on each A-scan. According to Equation 2, the plot of logarithmic light intensity as a function of the optical distance results in a straight line of slope $-\alpha_{ext}$, from which the light penetration depth (z_p) can be calculated. Figure 7b shows an interferogram where local maxima were selected for the linear regression used to calculate light penetration depth. No peaks were identified for optical depths larger than 1250 μm since it corresponds to a region where multiple scattering is not negligible. Then, light penetration depths were plotted as a function of position along a B-scan (Figure 8a) and variations were found to be relatively small; a representative average penetration depth can therefore be evaluated. As expected, Figure 8b shows a linear relationship between the average light penetration depth and the mean powder diameter.

Also, as a simple statistical measure of light penetration depth inside powders, an average of A-scan amplitude center position in depth (by analogy to a mass center) was calculated for each powder. The average values obtained (results not shown here) were found to be representative of light penetration and also showed a linear relationship with mean powder diameter. This statistical method (which uses a faster algorithm and which is more robust than the slope identification) should be further explored.

Thus, the second approach, which considers the whole powder volume, can also be used to retrieve mean powder diameter. However, even though the concept has been verified, further modeling and validation with very narrow distribution of glass powders is needed. Moreover, the calibration step would have to be repeated for given material under study, the scattering cross-section per unit particle volume being affected by material refractive index.

MEASUREMENTS IN PLASMA-SPRAYED CERAMIC COATINGS

Experimental

The HCST Amperit 825.1 yttria stabilized zirconia (YSZ) powder was sprayed on a steel substrate using a Sulzer Metco F4-MB plasma gun. Argon and hydrogen were used as the plasma gas mixture and the injected plasma torch power was held to 35.2 kW. The resulting porosity, as determined by SEM micrograph analysis, was found to be 10%. Thereafter, the sprayed zirconia coating was detached from its steel substrate and polished on both sides to form a wedge. It was then placed over a silicon wafer (Figure 3b) which provides two parallel reference surfaces for subsequent OCT imaging. Silicon was chosen for its high refractive index and its very low attenuation at the selected wavelength. The OCT system parameters were slightly changed as compared to those for powder imaging. The collimated beam was 2.4-mm wide and only one B-scan (formed by 1000 A-scans, separated by a 10 μm step size) was collected.

FIGURE 9. a) OCT B-scan of an YSZ coating placed over a silicon wafer. b) Selected A-scans, one from the Si reference zone and one from the YSZ zone, to determine the light penetration depth and the refractive index of YSZ non-transformable tetragonal phase.

Results and discussion

OCT imaging of the YSZ coating wedge placed onto the silicon wafer was performed. The resulting B-scan, shown in Figure 9a, was used to identify two representative A-scans in order to evaluate the light penetration depth inside the coating. The first one was selected from a zone where the sample beam impinges only on the reference Si wafer, and the second one was extracted at the location where the sample beam gets through both the YSZ coating and the Si wafer (e.g. at the Si back surface detection limit). This selection of the latter A-scan ensures that light penetrates the coating to a depth of at least its thickness at this point, thus evaluating a conservative light penetration depth. The corresponding A-scans are shown in Figure 9b. Dashed lines are plotted to appreciate the peak position shifts in optical depth at the coating top surface and at the Si wafer bottom surface. Therefore, the geometric coating thickness (through which light passes) is given by the simple profilometry difference $z_1 - z_3$ and the back surface shift (involving the coating refractive index and thickness) is given by $z_4 - z_2$. Obviously, there should be an equivalent back shift for the Si wafer top surface peak position when light gets through the coating, but this peak is undetectable. It cannot be clearly distinguished among other multiple reflection peaks.

Knowing that the coating contains about 10% porosity, the index of refraction n_{YSZ} of YSZ non-transformable tetragonal phase (which refers to a stable form of the tetragonal phase resulting from plasma spraying [10]) can be extracted from Equation 3:

$$[(0.9n_{YSZ} + 0.1n_{air}) - 1]e = d \quad (3)$$

where n_{air} is the air refractive index, $e = z_1 - z_3$ is the coating thickness and $d = z_4 - z_2$ is the Si wafer back surface shift. Thus, having a coating thickness $e \approx 100 \mu\text{m}$ and a back surface shift $d \approx 48 \mu\text{m}$, the index of refraction of YSZ non-transformable tetragonal phase is evaluated as $n_{YSZ} \approx 1.53$. To the authors' knowledge, this is the first evaluation of the refractive index of YSZ non-transformable tetragonal phase. In comparison to the reported refractive index of about 2.1 for YSZ cubic phase [11], the value found seems quite small. However, the phase transition from cubic to tetragonal of pure evaporated zirconia (which comes with a few percent increase in volume) has been found to influence significantly the refractive index, bringing it to a value close to 1.7 [12]. The same behavior apparently affects plasma-sprayed yttria stabilized zirconia.

CONCLUSION

Optical coherence tomography has been successfully used to retrieve information related to glass powder diameter. OCT B-scans were processed both for first particle layers and for larger volume penetration. It was found that the distribution of inter-peak distances on first particle layers, which is a measure of the particle diameter, shows an excellent agreement with the real size distribution obtained by laser diffraction. On the other hand, it was shown that the light penetration depth linearly depends on powder diameter, but further modeling and calibration are needed to apply the inverse relationship, that is, to retrieve powder diameter from light penetration depth.

An OCT B-scan of plasma-sprayed YSZ coating wedge placed onto a silicon wafer was exploited to determine an approximate light penetration depth inside coating and to evaluate its refractive index. From the observed peak shifts, it was found that light penetrates to a depth of at least 100 μm inside the coating and that the refractive index of YSZ non-transformable tetragonal phase was close to 1.53.

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