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Ultrasonic Technique for Measuring Porosity of Plasma-Sprayed Alumina Coatings

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Porosity is an important factor in plasma-sprayed coatings, especially ceramic coatings. Excessive porosity can adversely affect the performance of the coated component in various ways. An ultrasonic nondestructive measurement technique has been developed to measure porosity in plasma-sprayed alumina coatings. The technique is generic and can be extended to other ceramic coating systems. To test the technique, freestanding alumina coatings with varying levels of porosity were fabricated via plasma spray. Samples with varying porosity, obtained through innovative fabrication techniques, were used to generate a calibration curve. The ultrasonic velocity in the low-frequency range was found to be dependent on the density of freestanding coatings (measured via Archimedian techniques). This dependence is the basis of the development of a technique to measure the density of coatings.

Keywords alumina, nondestructive testing, porosity, ultrasonic velocity

1. Introduction

Thermal spray ceramic coatings provide specific functional characteristics, such as resistance to corrosion, heat, and wear, thereby protecting industrial components from extreme temperatures and/or harsh environments and extending their useful service life. One of the important factors in thermal spray coatings is porosity, as it can significantly affect service behavior and fitness for use.

State-of-the-art reports on thermal spray technology have identified an urgent need for the development of better techniques to measure porosity (Ref 1-3). In this context, nondestructive porosity measurement techniques offer considerable advantages.

Excessive porosity can affect the performance of a component in various ways. First, in applications where metallic and ceramic coatings are used to provide corrosion protection from harmful species, excessive interconnected porosity offers a channel for the diffusion of such species, thereby increasing the vulnerability of the base material to corrosion. Second, porosity has a significant influence on mechanical properties such as bond strength and fatigue behavior. Third, porosity is critical to the integrity, and thus the life, of the coating (Ref 4-6). Porosity also affects the thermal conductivity of the coating. Pores in the top coat act as air gaps, reducing conductivity. The pore shape and orientation also affect conductivity (Ref 7). These are important factors in the design and manufacture of thermal barrier coatings, where a degree of controlled porosity is desired.

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Consequently, measurement and characterization of various aspects of porosity—such as pore fraction and pore size, shape, and orientation—are important from both process development and service inspection standpoints. Current practices for characterizing porosity in coatings employ destructive metallographic techniques. These techniques are inherently time consuming, often unreliable in the case of ceramic coatings, and cannot be used on service parts. The development of a nondestructive technique to measure porosity promises benefits, including in-service inspection of variations in density to indicate potential premature failure.

Nondestructive techniques such as x-ray and ultrasonics have been demonstrated on bulk unsintered (green) and sintered ceramic materials. For example, computed tomography (CT) has been applied to measure the density and porosity distribution in green and sintered bulk SiC (Ref 8). This method uses the x-ray attenuation coefficients to reconstruct images of an internal cross section of a sample (Ref 9). The degree of porosity, as well as its size, shape, and orientation, significantly influence elastic properties such as Young's modulus, shear modulus, and Poisson's ratio. These properties and their directional variation were measured and characterized nondestructively using ultrasonic wave velocity techniques (Ref 10-14). However, these methods have not been applied to thermal spray coatings.

This paper reports on the development of a nondestructive technique based on ultrasound to measure the porosity of thermal spray coatings.

2. Ultrasonic Characterization of Density

Ultrasonic techniques have been demonstrated previously for the characterization of porosity in porous materials such as ceramics and powder metal alloys (Ref 11-14). However, these techniques have not been applied to thermal spray coatings. A technique has been developed for the quantitative measurement of porosity in thermal spray ceramic coatings. This technique is nondestructive and involves the measurement of the ultrasonic wave velocity in the coating. The wave velocity can be calculated from an ultrasonic time-of-flight measurement (using a commercially available ultrasonic system) and a thickness measurement (using a commercially available electromagnetic thickness sensor).

The porosity of the coating under test is determined using a previously established calibration curve that relates ultrasonic velocity to density of the coating. This calibration curve is generated by performing measurements of ultrasonic wave velocity and density (using Archimedian immersion techniques) on freestanding coating samples.

2.1 *Advantages and Benefits*

- The new technique is nondestructive.
- Elaborate sample preparation procedures needed for destructive metallographic characterization are avoided.
- The technique provides better accuracy and repeatability than existing destructive metallography techniques used for coating density measurement, which can have errors as high as 25% (Ref 10).
- The technique is rapid, easy to use, and capable of facile automation.

Other benefits include adaptability to routine in-service inspection of coated components as well as 100% postprocess inspection of manufactured parts. This technique could potentially be used as a quality control measure of the spraying process.

2.2 *Measurement Procedure*

Figure 1 shows a schematic diagram of the application of the technique. The ultrasonic wave velocity in thermal spray ceramic coatings is uniquely dependent on the porosity in the coating. The ultrasonic wave velocity can be calculated from a measurement of the time of flight of an ultrasonic pulse in the coating and the thickness of the coating. Time of flight can be measured using commercially available contact transducers and ultrasonic systems or modified hand-held ultrasonic thickness gages. The thickness of the coating can be measured using commercially available electromagnetic coating thickness measurement systems.

Relationships in the form of theoretical models or calibration curves can be established between ultrasonic velocity and coating porosity (hence, density). As the density of the coating increases, the ultrasonic velocity increases. These calibration curves can be generated by measuring ultrasonic wave velocity and density (using Archimedian techniques) on freestanding coatings. Once a relationship between ultrasonic velocity and density has been established on a freestanding coating, measurements of wave velocity in the applied coating can be used to infer its porosity.

Table 1 Data for generating the calibration curve

Longitudinal velocity, m/s	Density, g/cm^3
6494	3.428
6652	3.424
6293	3.409
5496	3.336
5678	3.323
4200	3.234
3160	3.170

3. Experimental Verification

Plasma-sprayed METCO 130 (Westbury, NY, USA) ($87Al_2O_3$-$13TiO_2$) was selected as the coating used for experimental verification of the method. Freestanding coatings were obtained by plasma spraying, in air, onto smooth steel substrates (AISI type 1020 low-carbon steel) using a Sulzer Plasma Technik A A3000S (Westbury, NY, USA) powder plasma spray system. To obtain samples with varying density, as typically encountered in industry, initially the plasma spray process parameters (e.g., gun current and standoff distance) were varied. This approach did not yield a sufficiently wide range of density.

Therefore, various mass percentages (5, 7, and 10%) of −325 mesh (<44 μm) copper metal powder were mixed with the ceramic powder and sprayed onto smooth (non-grit-blasted), thin steel substrates. The coatings were easily removed by bending the substrates to "popoff" the coating. Freestanding coatings thus obtained were immersed in dilute nitric acid to leach out the

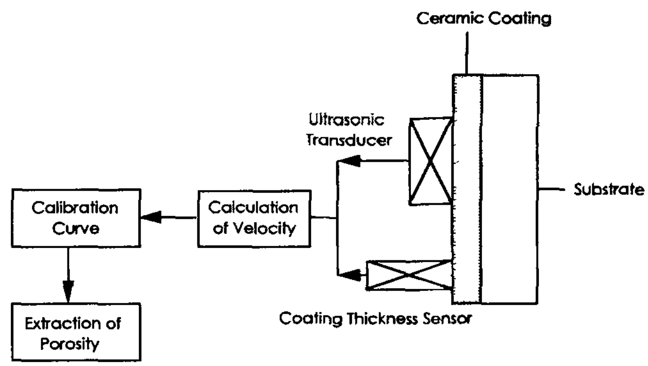

Fig. 1 Schematic of the porosity measurement system using the ultrasonic wave velocity measurement technique

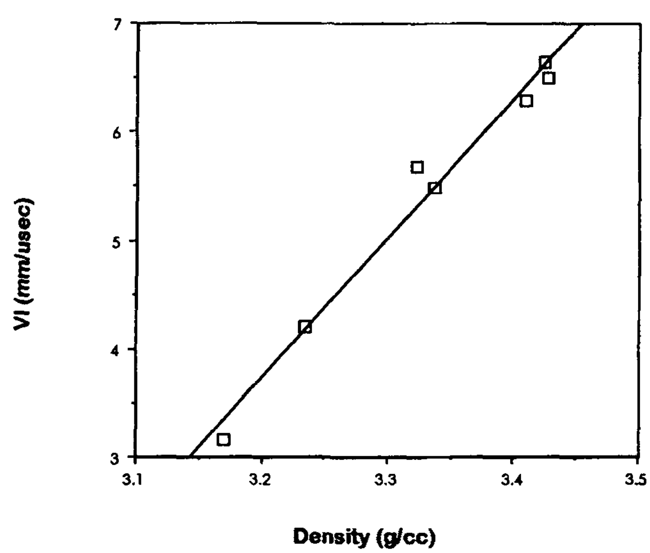

Fig. 2 Calibration curve relating velocity to density

copper and provide coating samples with high porosity. The samples used in this study had densities less than 94% of theoretical density.

Coating densities were measured using the Archimedian immersion procedure developed for porous materials (Ref 15). Longitudinal ultrasonic wave velocities were measured in these samples at 5 MHz via a through-transmission water immersion technique using a Panametrics 5052PR (Waltham, MA, USA) pulser receiver and a LeCroy 9400A (Chestnut Ridge, NY, USA) oscilloscope. Table 1 shows the measured densities and wave velocities. The ultrasonic velocity is a strong linear function of density.

A calibration curve can then be drawn by plotting the data in Table 1 and fitting a line through the data points (Fig. 2). The calibration curve relating longitudinal velocity, V_l, to density, ρ, is:

$$V_l = -37.549 + 12.897\rho \qquad \text{(Eq 1)}$$

with a correlation coefficient of $R_2 = 0.979$. Coating density measurements can now be made by measuring the wave velocity in the coating under test and then referring to the calibration curve to estimate the density.

For example, in a METCO 130 test coating, the wave velocity was measured to be 6165 m/s. From Eq 1, the coating is estimated to have a density of 3.390 g/cm^3. The density of the coating stripped from the steel substrate, measured using the Archimedian procedure, was 3.409 g/cm^3. This was quite close to the nondestructively estimated value. The error for determined density using this technique in this particular sample was 0.56%.

This procedure and the associated ultrasonic techniques can also be used on other porous ceramic coatings. Once a calibration curve has been developed for a material, routine measurements can be performed.

4. Conclusions

Ultrasonic techniques have been demonstrated for characterizing the density of plasma-sprayed alumina-titania coatings. Ultrasonic wave velocities measured on freestanding coatings are dependent on the density of the standard coatings measured by Archimedian immersion. This dependence can be used to measure the density of other, similar coatings. The errors in using this technique for the coatings tested are less than 1%. This technique could be extended to other ceramic coating systems through the development of similar calibration curves that relate density to ultrasonic velocity.

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