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Nondestructive Measurement of Porosity in Thermal Barrier Coatings

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Porosity is an integral part of thermal barrier coatings (TBC) and is required to provide thermal insulation and to accommodate operational thermal stresses. The effective use of TBC in hot-section components of aircraft engines requires nondestructive testing (NDT) methods to detect porosity variations and measure thickness changes to reduce the risk of damage to the coating due to such variations. The eddy current method has been used to measure the thickness of a plasma-sprayed TBC coating and either ultrasonic or capacitance techniques have been applied to assess porosity content based on thickness values obtained using the eddy current tests. The porosity values estimated by the NDT methods have been confirmed by destructive testing, which included metallography and vacuum volumetric measurement using nitrogen absorption.

Keywords capacitance measurement, eddy current testing, nondestructive testing (NDT), porosity, thermal barrier coating (TBC), thickness, ultrasonic testing

1. Introduction

Thermal barrier coatings (TBCs) are used to protect the hot-section components of aircraft engines from wear, erosion and high-temperature degradation. In such applications, TBCs provide thermal insulation of the metallic components against thermal transients (hot spots) and improve the parts longevity. Typically, the use of a 250 μm thick ceramic layer on turbine blades can reduce the metal average temperature by up to 80 °C and the hot-spot temperature by 170 °C or more.[1,2] In addition, the application of TBC can improve engine performance or thrust by either increasing the combustion temperature or reducing the cooling airflow,[3] both resulting in fuel economy.[4]

TBCs are thin ceramic layers of low thermal conductivity such as zirconia deposited on the metallic substrate using either plasma spraying or electron beam physical vapor deposition processes. An intermediate layer of a bond coat is normally applied prior to deposition of zirconia to minimize the thermal expansion mismatch between the metallic substrate and the ceramic coating. The bond coat, which is a modified aluminide alloy such as MCrAlY (where M is Co, Fe, Ni, or a mixed combination),[5,6] also serves to improve the bonding of the ceramic to metallic substrate and to protect the substrate from oxidation. The thickness of the bond coat is typically 75-150 μm and the ceramic zirconia is in the 80-500 μm range.

Partially stabilized zirconia (PSZ) is most commonly used in modern gas turbine engines[7] due to its high durability and low thermal conductivity compared with the other ceramics.[8] A stabilizer (e.g., yttria) is required to avoid the phase transformation of the zirconia at elevated temperatures.[9-12] Controlled pores and microcracks are deliberately introduced in the coating to reduce the through-thickness heat transfer and achieve the desirable thermal insulation properties. Porosity and microcracks are essential elements in the functionality of the TBC influencing the thermal conductivity as well as other performance parameters such as fracture behavior and resistance to erosion. However, the right amounts of pores or microcracks and their uniform distribution are essential for optimal performance of a TBC system. The TBC lifetime is limited by mechanical and thermal stresses generated in the coating due to the mismatch of thermal expansion coefficients of the ceramic coating and the metallic substrate as well as the oxidation of the bond line.[2,3] An improper amount or uneven distribution of porosity can adversely affect the durability of the coating, particularly at areas where coating is thinner. Therefore, there is a need for nondestructive testing (NDT) techniques to measure the coating thickness for uniformity and to assess the porosity content and uniformity after manufacture. For this paper, the eddy current method was used for thickness measurement, and ultrasonic or capacitance techniques were used to assess porosity content in a plasma-sprayed TBC coating.

2. Specimens

The TBC specimens were made by Standard Aero Limited of Winnipeg, Manitoba using commercial 8% yttria stabilized zirconia ceramic powder (Saint-Gobain 204, −100/+400 mesh, Worcester, MA). The powder was directly deposited onto In-

Table 1 TBC Thickness Values and Porosity Range of TBC Specimens Investigated

	Thickness Values, μm		Porosity Range, %	
Specimen No.	Micrometer	Eddy Current	Manufacturer	NRC
I5-1	358	355	<3	0.4-3.3
I5-2	272	279	3-6	3.0-4.2
I5-3	264	254	5-10	4.4-9.7
I5-4	510	530	5-10	5.7-9.3

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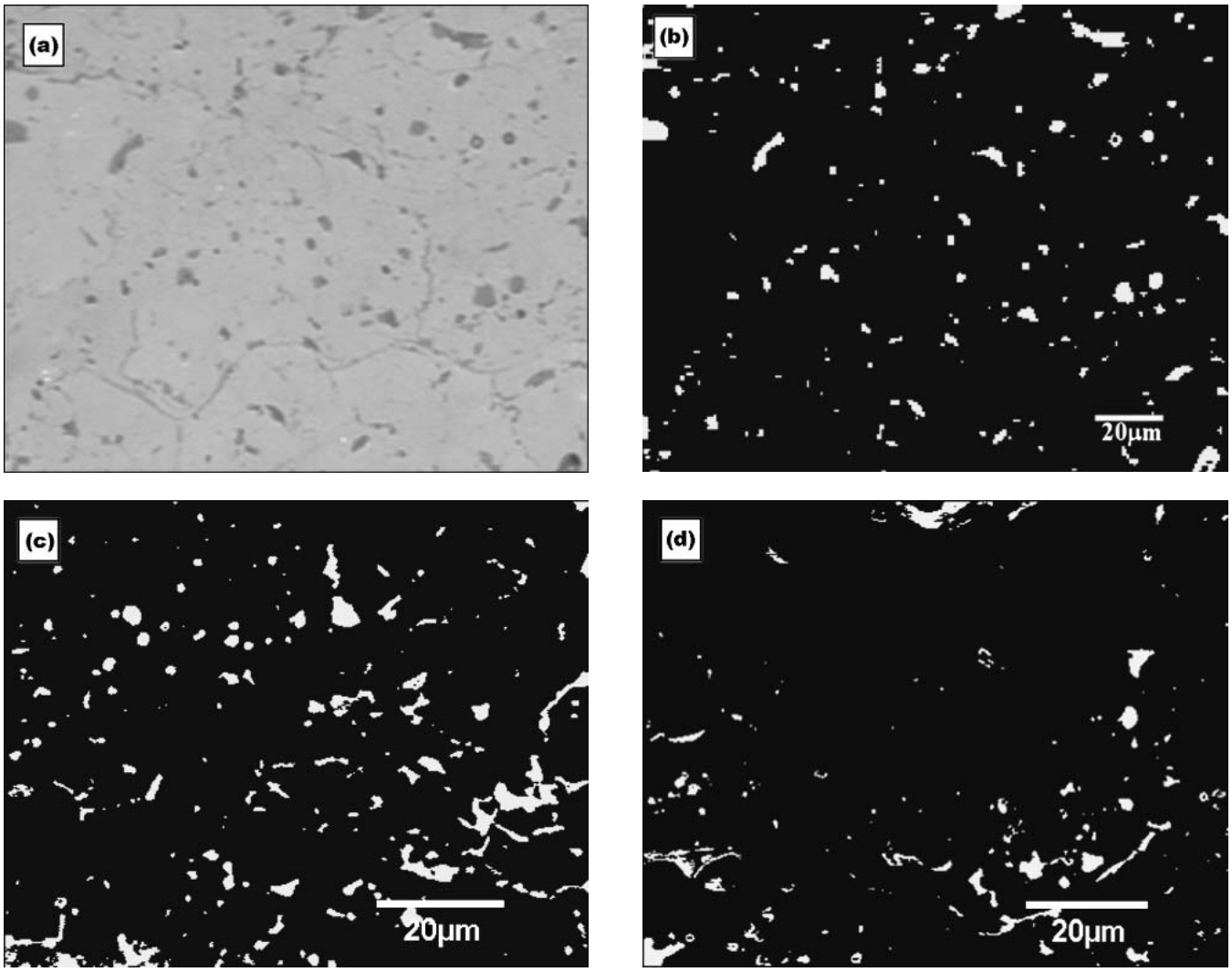

Fig. 1 **(a)** Optical micrograph of specimens I5-4 and **(b)** the equivalent back and white image used for porosity measurement; images of the same specimen corresponding to **(c)** maximum porosity and **(d)** minimum porosity contents

conel 600 substrate using Sulzer Metco's 9 MB plasma spray system (Westbury, NY). No bond-coat was applied prior to coating, but the surface of the substrate was grit blasted. The process parameters, in particular the spray distance, were changed to achieve different porosity levels, without the introduction of micro-cracks. The coating porosity range for the specimens was measured by the manufacturer using optical microscopy and is provided in Table 1. For this purpose, coated samples were carefully sectioned, mounted, and polished using conventional semi-automatic procedures to avoid introduction of defects by separation of surface grains. The measured porosity contents are not single fixed values but cover a range. This is inherent to plasma-sprayed ceramic coatings due to a combination of factors, including powder particle size and shape variation, and softening and distortion of particles during flight and on impact. Also, the pores have varying shapes, with the vast majority being non-spherical. These will have a bearing on the results obtained using NDT techniques. The manufacturer porosity range was verified independently at National Research Council Canada (NRC) by carefully polishing the specimen cross-section and taking optical micrographs from five different locations of the polished coatings (Fig. 1a). Using a standard image processing software, the grayscale optical micrographs were first converted into black and white images (Fig. 1b), and then the ratio of the total white areas (pores) to the total surface area of the same picture was calculated. This ratio corresponds to the average porosity content of this particular location of the specimen. The minimum and the maximum values of the average porosity of the five locations provide the porosity range for the specimen. Figure 1(c) and 1(d) correspond to areas where porosity is minimum or maximum, respectively. Table 1 also provides the porosity range measured by NRC, indicating that the porosity values provided by the manufacturer and those measured at the NRC are similar.

Accurate measurement of the coating thickness was carried out using a conventional eddy current technique.[13] Given that the ceramic topcoat is nonconductive, the measurement of its thickness by eddy current is a lift-off (i.e., a probe-to-specimen spacing) measurement. Thus, the quantification of the ceramic

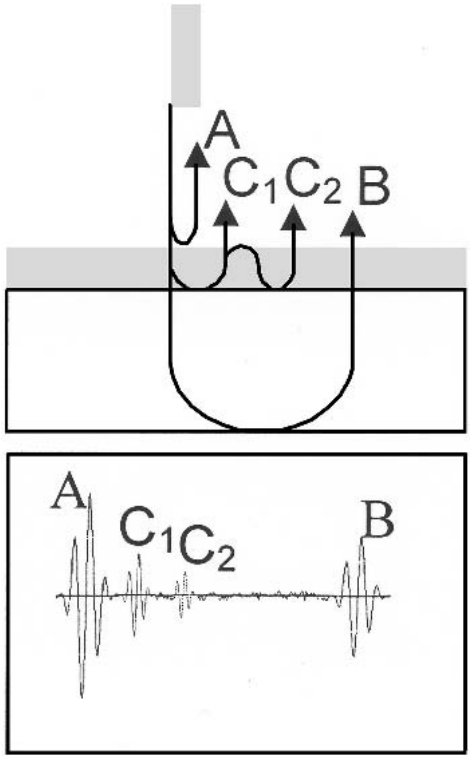

Fig. 2 Schematic diagram of ultrasonic echoes in a coated flat specimen

Table 2 Ultrasonic Longitudinal Wave Velocity and Electrical Permittivity Values Measured for TBC Specimens

Specimen No.	Velocity Immersion Probe, m/s	Velocity Contact Probe, m/s	Relative Permittivity
I5-1	4300	4250	29
I5-2	3920	3870	23.9
I5-3	3820	3820	20.0
I5-4	3410	3650	14.4

coating thickness is achieved by comparing the lift-off signal of the coated side to that of the substrate measured on the bare metal side of the same specimen. The thickness of the specimens was also confirmed by ball-end micrometer measurements and is provided in Table 1. In the latter case, the coating thickness was obtained by subtracting the substrate thickness (0.888 mm) from the overall specimen thickness, assuming that the thickness of the substrate was uniform. The accuracy of thickness values is important since these values are used for determining the ultrasonic velocity and effective permittivity of the coating that in turn are used to estimate the coating porosity.

3. Ultrasonic Testing

Ultrasonic testing is widely used for flaw detection and, to a lesser degree, for material characterization or quality control. The ultrasonic velocity (V) is generally dependent on the elastic constant (E) and the density (ρ) of the material. The general expression is

$$V \propto \sqrt{\frac{E}{\rho}}.$$

For a given material, since there is a direct relationship between density and porosity content, the ultrasonic velocity may be used to estimate porosity if elastic constant (E) does not change significantly with porosity or its changes can be measured and compensated for. It has been shown that the ultrasonic velocity in porous ceramics and plasma-sprayed coatings is linearly dependent on their porosity level.[14,15] Using this as a basis, the ultrasonic longitudinal velocity in the coating can be determined by dividing the coating thickness, that is provided by eddy current measurement, by the time-of-flight (TOF) of the ultrasonic waves in the coating.

In the pulse-echo method used here and illustrated in Fig. 2, the ultrasonic time-of-flight in the coating is half the time between the echo reflected off the coating top surface (A) and the first echo from the coating-substrate interface (C1). The time-domain resolution of these echoes is dependent on the coating thickness and the ultrasonic frequency or wavelength (velocity-frequency ratio). An increase of the frequency provides a better resolution of the echoes; however it also increases the attenuation that results in a decrease of the signal amplitude. Due to the high attenuation of TBC, it is not easy to use frequencies higher than 10 MHz, and therefore this frequency was used. At 10 MHz frequency, the wavelength of the longitudinal waves in the coating is significantly larger than the coating thickness and in practice the surface echo and the interface echoes overlap, making TOF measurements impossible. However, at this frequency, the substrate thickness-to-wavelength ratio is about 1.5; that allows the resolution of echoes reflected off the coating top surface (A) and the substrate bottom surface (B). Thus, the TOF in the coating was established by subtracting TOF in the substrate from TOF in the whole specimen. The advantage of this method is that it is applicable even to specimens with variable substrate thickness since the substrate equally affects the two TOF readings. Finally, the longitudinal wave velocity in the coating was calculated from the TOF measurements using thickness values provided by the eddy current technique (Table 2).

The TOF measurements were carried out using a commercial ultrasonic instrument (Panametrics 5601A/TT, Waltham, MA), a digital oscilloscope with a 500 MHz sampling frequency, and commercial immersion or contact transducers with a center frequency of 10 MHz. The velocity values provided are the average of up to ten TOF measurements on different locations on each specimen. The results for the immersion technique are plotted in Fig. 3 against the porosity range providing a trend-line between the velocity and porosity for the coatings investigated. The trend line can then be used to estimate the porosity content of other parts with the same coatings by measuring their thickness and velocity using NDT methods.

4. Capacitance Measurement

The capacitance technique involves the measurement of the dielectric properties of the coating and is based on the fact that a decrease in the effective permittivity of the ceramic coating is directly proportional to an increase in its porosity.[16] Experimentally, it is possible to determine the capacitance of the ce-

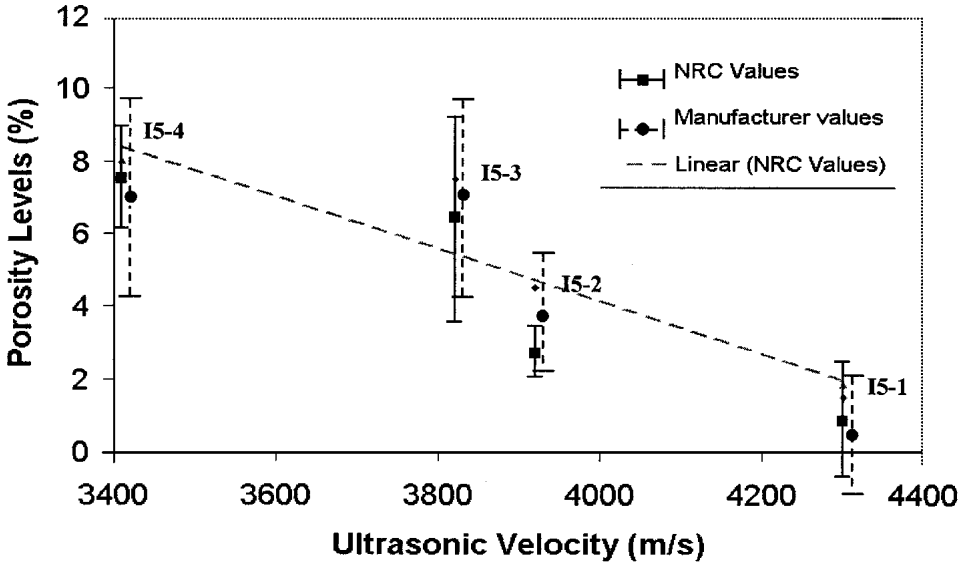

Fig. 3 Ultrasonic velocity of TBC measured with an immersion probe versus porosity values measured using optical micrographs

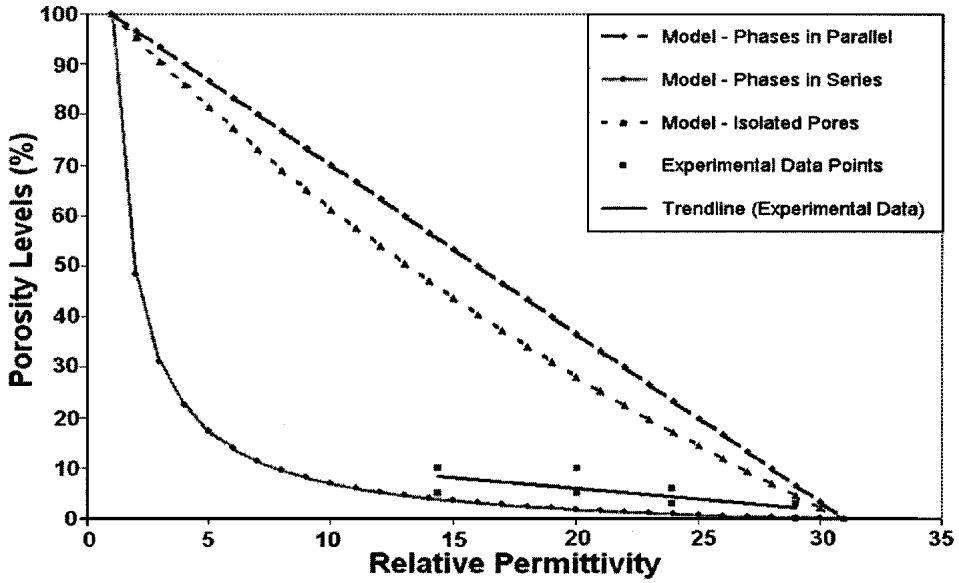

Fig. 4 Calculated porosity versus dielectric constant for different phase geometries

ramic coating using a commercial impedance analyzer (e.g., Hewlett-Packard Impedance Analyzer HP4192A, Palo Alto, CA) equipped with a probe that uses pressure electrodes. The effective permittivity can be determined by knowing the distance between the electrodes (d), the measured capacitance of the coating (C), and the coating area (A) interrogated by the probe. This area is determined by calibrating the instrument with respect to a reference specimen of known permittivity. The expression is

$$\varepsilon = \frac{C\,d}{A}.$$

The effective permittivity of the TBC specimens relative to the vacuum permittivity (relative permittivity) measured with this technique is presented in Fig. 4 and Table 2. This figure also provides three other curves developed from Gerhardt's models[16] that suggest a porous coating is a composite of two phases, namely the coating material and the porosity. The two phases can be in parallel, in series, or isolated from each other. Based on this model, the plasma-sprayed coatings consist of isolated and interconnected pores and microcracks that are arranged primarily in series and separated by melted, partially melted or unmelted powder splats. The experimental results of Fig. 4 essentially support this model.

5. Discussion

The ultrasonic velocities measured with immersion and contact probes were somewhat different, however in three out of

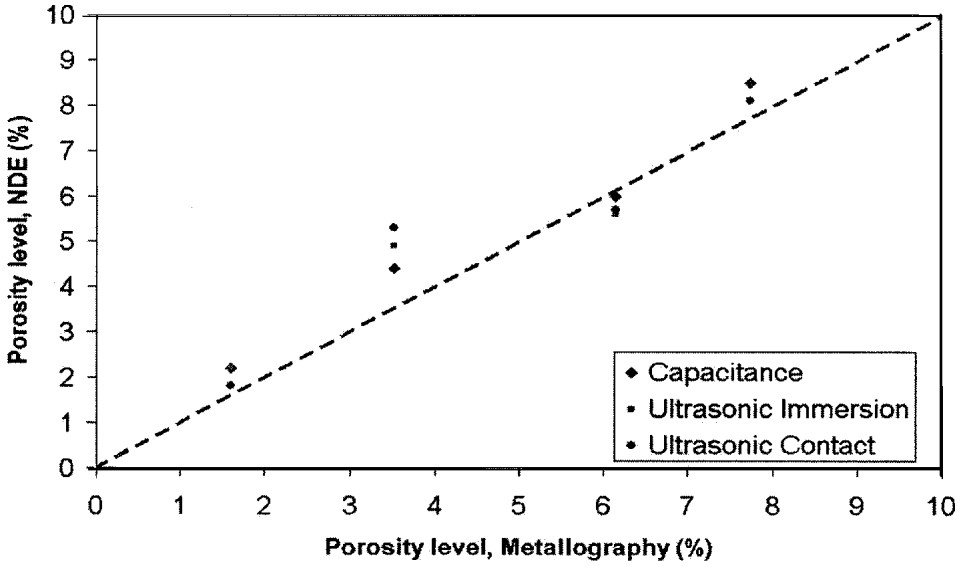

Fig. 5 Porosity in the TBC specimens investigated using ultrasonic and capacitance methods as compared with the average of porosity values measured using optical micrographs

Table 3 Porosity Values Determined by Different Methods

Specimen No.	Ultrasonic Immersion	Ultrasonic Contact	Dielectric Capacitance	Metallography
I5-1	2.2%	1.8%	2.2%	1.6%
I5-2	4.9%	5.3%	4.4%	3.5%
I5-3	5.6%	5.7%	6.0%	6.1%
I5-4	8.5%	8.1%	8.5%	7.7%

four specimens, the difference was less than 50 m/s or <1%. In one specimen (I5-4), the ultrasonic signal-to-noise was relatively low due to its higher porosity content and difficulty in measuring TOF accurately. Nevertheless, in the porosity range of the investigated specimens, a linear relationship was observed between the porosity content and the ultrasonic velocity, as seen in Fig. 3.

The average porosity measurements taken from the three independent NDT methods are similar, but the NDT porosity values are slightly higher than those measured using metallography (Fig. 5 and Table 3). To verify these data, the porosity of sample I5-1 was also established by comparing its density to that of a fully sintered zirconia of the same composition. The density of the free-standing coating was determined from its weight, measured using an accurate electronic scale, and its volume determined by vacuum volumetric measurements using nitrogen absorption.[17] The comparison of the measured density (5988 kg/m^3) and that of the fully sintered zirconia with 8%-yttria (6100 kg/m^3[18]) provides a porosity of about 1.8%. This value is in the range of porosity data obtained for specimen I5-1 by NDT methods (1.8-2.2%) and by metallography (1.6%) indicating the validity of both NDT and metallographic measurements.

For the TBC specimens used here, both the ultrasonic velocity and the permittivity decrease with increasing the coating porosity. In the case of ultrasonic velocity, the decrease can be associated with changes in both density and elastic constants that together affect ultrasonic velocities. In the case of capacitance, an increase in porosity is comparable to the introduction of more cavities in a dielectric.

The correlation between the ultrasonic velocity and the relative permittivity suggests that porosity affects these two physical parameters in the same manner, and either parameter can be independently used to estimate the porosity. In situ measurements of ultrasonic longitudinal velocity can be carried out using readily available instruments and contact probes; however a fluid is always needed to provide coupling between the probe and the specimen. This method may be time-consuming and not quite applicable to thick TBC coatings that have a high ultrasonic absorption. The capacitance method is simple, inexpensive and more robust; however, it is still a laboratory technique and commercial probes are not readily available. Work will continue to further develop this approach and the associated instrumentation or probes for in situ applications in manufacturing environments.

6. Conclusions

The potential of NDT techniques for thickness and porosity assessments in thermal barrier coatings was studied. The conventional eddy current method can be used to determine the thickness of TBC coatings, and if the thickness is known, ultrasonic techniques can then be used to obtain an estimate of the porosity. Capacitance measurements can also be used to determine the porosity if suitable probes are available. Both techniques can be applied in situ to check the uniformity of the porosity content in TBC coatings after process; however, the capacitance method is simpler to use and more practical. It may be possible to combine either of these methods with eddy current in a single instrument for simultaneous assessment of the uniformity of TBC thickness and porosity.

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