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A Method of Measuring Non-Linear Elastic Properties of Thermal Barrier Coatings

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Abstract

Accurate characterization of the elastic properties of thermal barrier coatings (TBC's) is important for failure prediction. Thermally sprayed coatings often exhibit anisotropic and non-linear elastic properties due to the coating microstructure that results from the thermal spray process. A method was developed for determining the elastic behavior of TBC's on substrates by measuring the in-plane modulus as a function of residual coating stress. The in-plane modulus was determined by resonant frequency measurement, and the residual stress was measured from the substrate curvature. The residual stress was varied both by increasing the temperature of the TBC and substrate and by applying compressive plastic strain to the metal substrate. The stress-strain behavior of the TBC was derived from the data for modulus versus residual stress, and significant non-linear elastic behavior was observed.

THERMAL BARRIER COATINGS (TBC's) are used on metal surfaces in the hot sections of gas turbines to decrease the metal temperatures and extend life (1,2). One way to allow higher gas-side temperatures, which increase turbine efficiency, is to guarantee the reliability of the TBC. Accurate elastic properties measurements of thermal barrier coatings are required for life prediction. The thermal strain in the TBC is determined in part by the in-plane elastic modulus, and the elastic properties are required for modeling the stress states of TBC's on engine hardware. It is well known that the elastic properties of thermally sprayed ceramic coatings, such as TBC's, differ from the bulk material properties (3). Thermally sprayed coatings typically have pores and microcracks which reduce the elastic modulus of the coating from the bulk value (4). In addition, anisotropy has been observed in the elastic properties of thermally sprayed metal (5) and ceramic (6) coatings, which had different moduli in the plane of the coating than in the through-thickness direction.

However, up to now little attention has been given to the non-linear elastic behavior of thermally sprayed TBC's. In a study by Wesling, et al. (7), stress-strain curves for air plasma sprayed (APS) 8 wt% Y_2O_3-ZrO_2 (YSZ) were measured in compression for free-standing hollow cylinders with wall thicknesses of 1.5 mm. The stress-strain curve was non-linear and exhibited a factor of 3 increase in the modulus over the strain range from 0 and -0.008. Special preparation of the free-standing samples was required, and the use of self-aligning hydraulic grips limited the experiment to loading in compression only. In this paper a new method is described for the determination of the non-linear elastic properties of TBC's on metal substrates, in which the modulus was measured as a function of the coating residual stress. The residual stress was varied by changing the temperature of the TBC-substrate system and by deforming the substrate plastically. The non-linear stress-strain behavior of the TBC was derived from the data for modulus versus residual stress.

Experimental

The TBC-substrate system used in this study was APS 8 wt% Y_2O_3-ZrO_2 topcoat on a 0.25 mm thick APS NiCrAlY bondcoat on a 3.2 mm thick Inconel 718 substrate. All specimens were fabricated by GE. The specimens had topcoats with thicknesses of either 1.4 mm or 0.5 mm, as specified below. Residual stress and elastic modulus were measured on samples that were rectangular bars approximately 25.4 mm long by 6.4 mm wide by 3.95 mm thick. The residual stress in the TBC, σ, was measured from the difference in the curvatures of the composite beam measured before and after removal of the TBC (8). The stress was deduced by the equations relating beam curvature to the stress in laminated beams, which are included in mechanics texts such as Timoshenko and Goodier (9). There is a stress gradient through the thickness of the coating, and reported here is the average stress in the TBC layer. TBC removal was done by lapping off the TBC using metal-bonded diamond abrasives. This procedure can also be used to determine the stress in sub-layers of the TBC by removing only part of the entire TBC layer.

The curvature was determined in two ways. For measurements at room temperature, curvature was determined by scanning the back-face of the substrate with a Tencor profilometer and fitting the shape to a section of a circle. The curvature was measured before and after removal of the TBC. Care was taken to assure that the trace was always recorded in the same location on the substrate back-face. For measurements at elevated temperatures, the sample was placed in a furnace in a three point loading configuration (10). A Mo push-rod contacted the sample at mid-length and was used to monitor the deflection of the sample relative to the fixture. Curvature was determined from the deflection assuming the sample deformed elastically in the shape of a section of a right circular cylinder.

The TBC elastic modulus was measured from the resonant flexural frequency of the composite (substrate, bondcoat and TBC) beam. The resonant flexural frequencies were measured by suspending the rectangular beam specimens from support wires at the node positions for the fundamental flexural resonance (11). The specimens were excited by impacting the center of the beam with a small zirconia sphere in a manner similar to that described in the ASTM standard C-1259-94 entitled Elastic Modulus by Impulse Excitation of Vibration. The resulting resonant vibrations were sensed with a Model 4165 Bruel & Kjaer condenser microphone located near the midpoint of the specimen. The resonant frequencies of the specimens were determined by feeding the microphone output to a digital storage oscilloscope with fast fourier transformation capabilities. The resonant frequency for the fundamental mode of flexural vibration, specimen mass and specimen dimensions were used to calculate the in-plane Young's modulus of the TBC. Resonant frequencies for higher vibration modes were measured at the same time and were used to give confidence in the identification of the fundamental frequency. Resonant frequency was measured for samples both at room temperature and in a modified tube furnace, as shown in Figure 1a. For measurements at elevated temperatures a vertical waveguide of fused-silica tubing with one end in close proximity to the bottom mid-span of the specimen was used to transmit the vibrations out of the furnace to the microphone.

In previous measurements of the resonant flexural frequency of composite beams (12,13), specimens were oriented with the node lines parallel to the plane of the composite layers, and the amplitude of flexural vibration was normal to the plane of the coating. Removal of the coating caused a shift in the position of the neutral axis of the beam which had to be recalculated for the resonant frequency determination. In the method used here, the specimen was oriented as shown in Figure 1b so that the direction of the node lines was normal to the layers and the amplitude of vibration was parallel to the plane of the coating. Coating removal, then, did not change the position of the neutral plane. The technique gave the advantage of simplifying the mathematical analysis. The modulus of the coating can be determined in a straight-forward manner

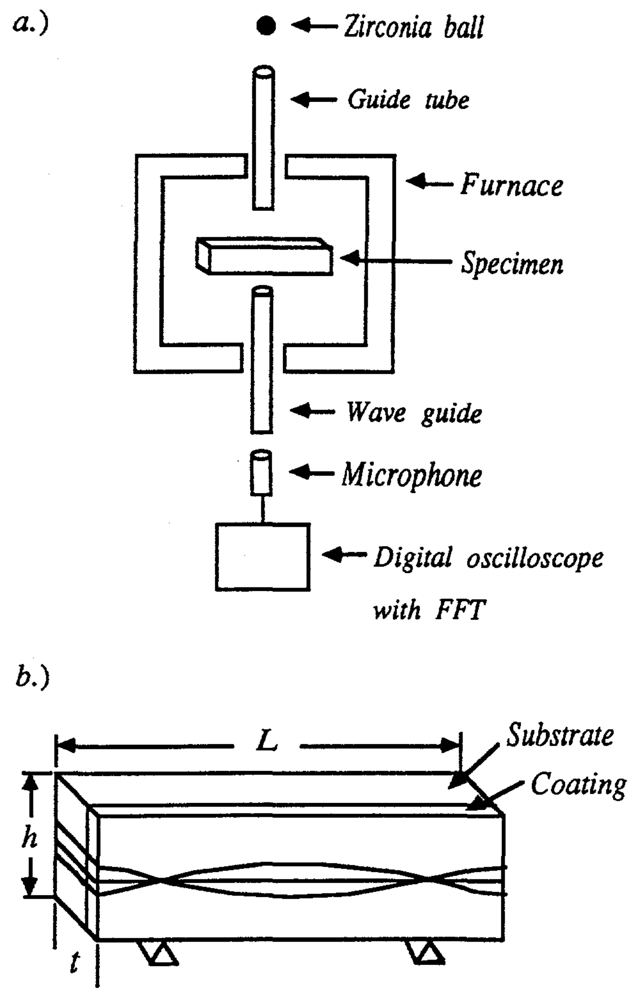

Figure 1 - a.) Schematic diagram of the apparatus for measuring resonant flexural frequency at elevated temperatures. b.) Schematic diagram of flexural bending in the specimen during the resonant frequency measurement. The amplitude of the vibration is in the plane of the coating. Specimen dimensions are indicated in the figure.

from the restriction that the stiffness of the composite beam is equal to the sum of the stiffnesses of the components (13):

$$E_{c+s} I_{c+s} = E_s I_s + E_c I_c \qquad (1)$$

where E is Young's modulus, I is the moment of inertia and the subscripts $c+s$, c, and s denote that the quantity is for the composite beam with both the coating and the substrate, for the coating only and for the substrate only, respectively. Young's modulus for a homogeneous rectangular beam is related to the resonant frequency by (11):

$$E = 0.94642 \frac{TL^3}{h^3} \frac{mf^2}{t} \qquad (2)$$

where L, h, and t are the length, height, and thickness of a homogenous specimen with dimensions as indicated in Figure 1b, T is a dimensionless shape factor (dependent on the specimen length, height, and Poisson's ratio), m is the mass and f is the resonant frequency of the fundamental mode of vibration. Combining the two equations leads to the following expression for the modulus of the coating, E_c:

$$E_c = E_s \left[\frac{m_{c+s}}{m_s} \left(\frac{f_{c+s}}{f_s} \right)^2 - 1 \right] \frac{t_s}{t_{c+s} - t_s} \qquad (3)$$

E_s is determined from the resonant frequency of the bar after the coating is removed, and E_c is deduced by the change in resonant frequency and sample dimensions between the composite beam with and without the coating.

TBC modulus was measured as a function of residual stress, and the stress-strain curve was determined from the data. The residual stress was varied in two ways. First, measurements of TBC modulus and stress at elevated temperatures gave modulus measurements for residual stresses that were less compressive than for the specimen at room temperature. Second, the residual stress was made more compressive by plastically deforming the substrate in compression before measuring the modulus and stress.

Results

The non-linear elastic behavior of TBC's was determined first from measurements of TBC modulus and stress at elevated temperatures. Since the intent of the paper is to describe a method for measuring elastic behavior of TBC's on substrates and to demonstrate that the elastic behavior exhibited significant non-linearity, normalized plots of stress and modulus are sufficient to illustrate the method and non-linear effect. TBC residual stress and modulus, normalized to the room temperature values for one of the coatings, are given as a function of temperature in Figure 2 and Figure 3. Three TBC beam samples with a TBC thickness of 1.4 mm were prepared from the same coated substrate. The three samples were treated identically except for the maximum temperatures of the thermal treatments, which were room temperature, 400°C, and 700°C. Curvature and resonant frequency were measured for the TBC's in their as-deposited state at room temperature, and then curvature and frequency measurements were made at various temperature intervals for the samples taken to elevated temperatures. The TBC was removed from the substrate, and curvature and frequency measurements were made on the substrate itself for the stress and modulus determinations. The specimen dimensions used in the calculations were corrected for thermal expansion.

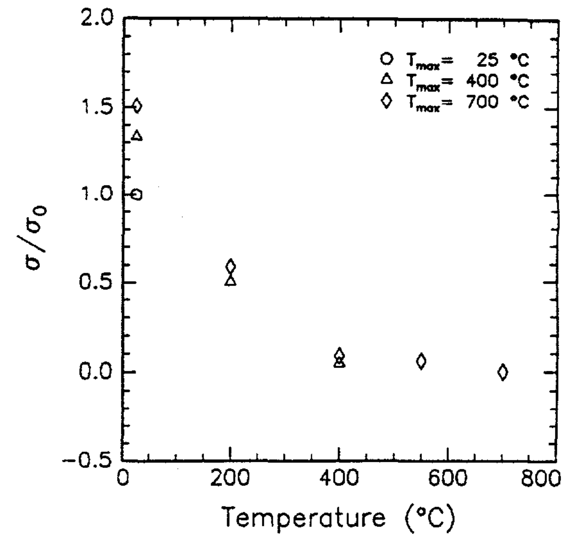

Fig. 2. Plot of the residual stress versus measurement temperature for three samples taken to different maximum temperatures. The stress is normalized by the residual stress of the sample kept at room temperature.

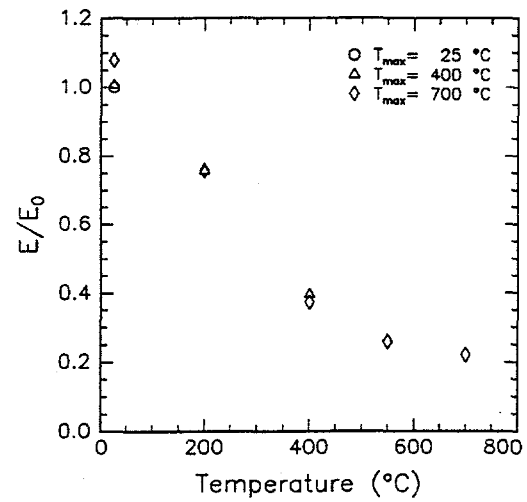

Fig. 3. Plot of the modulus versus measurement temperature for three samples taken to different maximum temperatures. The modulus is normalized by the modulus of the sample kept at room temperature.

The maximum temperature, 700 °C, was high enough to produce a stress-free state in the coating at the highest temperature, but it was kept below 900 °C because a previous study (10) showed that APS 8 wt% Y_2O_3-ZrO_2 underwent irreversible shrinkage at temperatures above 900 °C, most likely due to conversion of metastable strained-cubic phase material to tetragonal phase. In the present study, a change in the room temperature residual stress was observed for the specimens after their first heat treatment to either 400 °C or 700 °C. However, subsequent exposure to those temperatures did not cause further

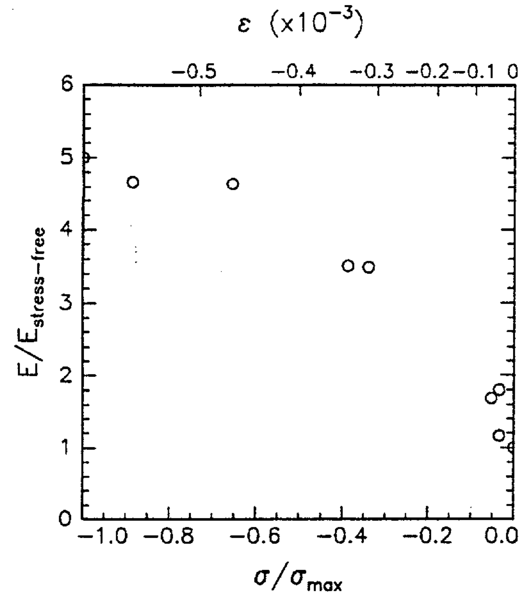

Fig. 4. Plot of modulus versus residual stress showing non-linear elastic behavior. The modulus is normalized by the modulus of the stress-free coating and the stress is normalized by the maximum stress measured in the experiment. The strain was calculated by the same procedure as for Figure 8.

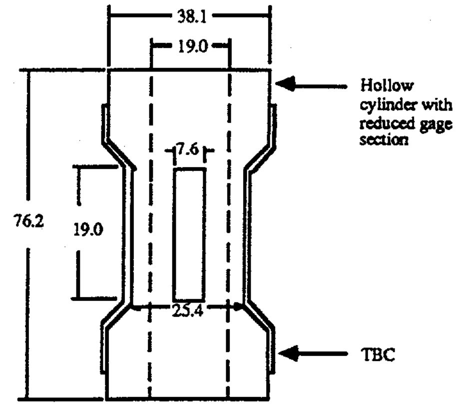

Fig. 5. Schematic drawing of the sample used to apply compressive plastic strain to the substrates of the TBC's. Putting the TBC free edges on the wide part of the specimen allowed the attainment of large plastic strains without TBC failure. Dimensions are in mm.

changes in the room temperature properties, and there was good agreement in the elevated temperature properties for the two specimens that were heated to different maximum temperatures. The effect of temperature was to reduce the compressive residual stress, which caused the change in TBC modulus. The change in modulus due to temperature alone was negligible for these samples.

In Figure 4, the modulus, normalized to the stress-free modulus, is plotted versus the stress, normalized to the maximum compressive stress. For reference, the strain, calculated using a procedure described later, is also plotted. The non-linear elastic behavior is evident from the plot. For the strain range between 0 and -0.0006, the modulus increased by a factor of 5. Of course, the modulus would have been constant with stress if the coating had exhibited linear elastic behavior.

The compressive strain range over which the elastic behavior was determined was extended by plastically deforming the substrate in compression before measuring the modulus and stress. A special test specimen, shown in Figure 5, was used for the plastic deformation. The specimen was a hollow IN718 cylinder with a reduced diameter gage section, and four flat faces were milled into the gage section along its length. The 0.5 mm thick TBC covered the gage section and extended onto the wider ends of the specimen, where the TBC free edges experienced much less strain than the coating in the gage section. That allowed the application of a large plastic substrate strain before TBC delamination. Strain gages were attached to the inner surface of the hole on the inside of the specimen. The specimen was loaded in uniaxial compression until yielding occurred in the gage section, and samples were prepared with a series of different plastic strain levels. Specimens in the shape of rectangular bars were cut, using abrasive water-jet cutting, from the regions of the gage section that contained the flat faces. The curved back-side of the substrate was polished flat to yield a rectangular parallelpiped similar to that used in the experiment described above. The stress state in each specimen was deduced from the difference in substrate curvature before and after removal of the TBC layer, and the modulus was determined at room temperature from the change in resonant flexural frequency upon removal of the TBC.

Figure 6 is a summary of the stress and modulus, normalized to their values for zero plastic substrate strain, versus applied plastic strain. The non-linear elastic behavior is evident in the fact the modulus changed by

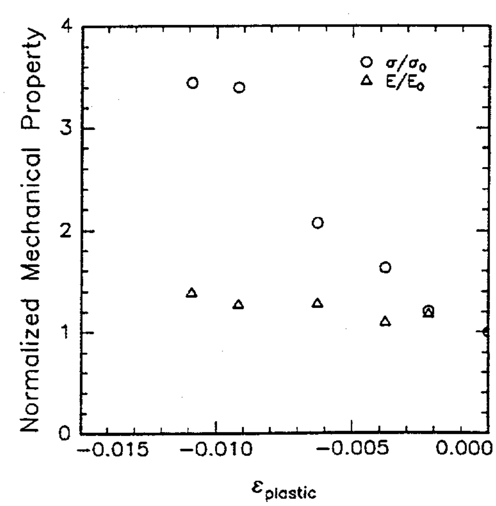

Fig. 6. Plots of stress and modulus versus applied plastic strain. The stress and modulus are normalized by the values for the specimen with zero applied plastic strain.

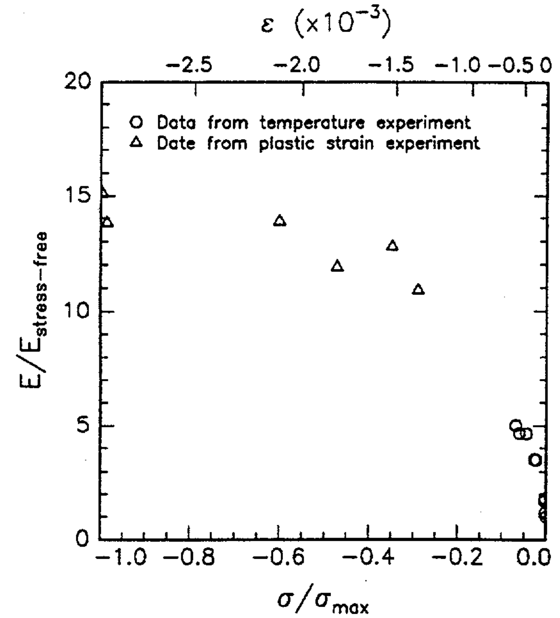

Fig. 7. Plot of modulus versus residual stress for the data from the experiments in which residual stress was varied using temperature (circles) and using plastic strain (triangles). The modulus is normalized by the stress-free modulus and the stress by the maximum stress measured in the experiments. The strain was calculated by the same procedure as for Figure 8.

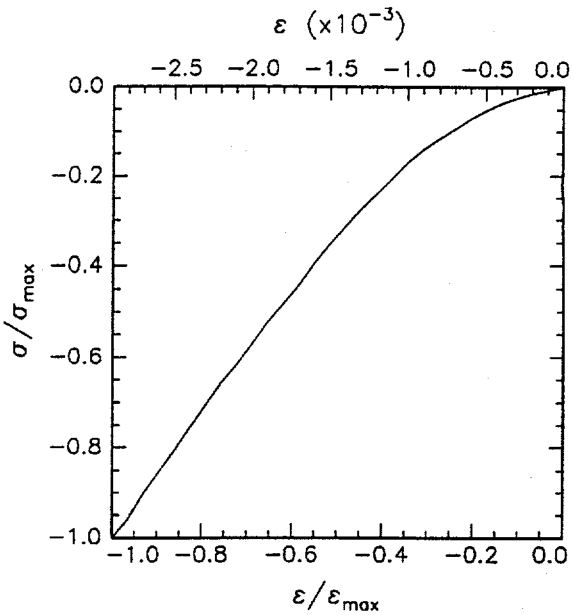

Fig. 8. Plot of stress versus strain curve deduced from the measurements of Young's modulus at different residual stress levels. The stress and strain are normalized by the maximum stress and strain measured in the experiments.

about 40% over the range of applied strain. Figure 6 also shows that the residual stress became more compressive with applied strain, as expected. The compressive residual stress and the modulus of the as-deposited specimens were different from those for the specimens used in the first experiment because the coatings were deposited at different times. However, since the coating microstructures were similar, the data from the two experiments are plotted together in Figure 7. The modulus, normalized by the stress-free modulus is plotted versus the residual stress, normalized by the maximum stress tested. It can be seen that with increasing stress, there is a decrease in the change in modulus and the modulus begins to approach a constant value, indicative of linear elastic behavior.

A procedure was developed for deriving the stress-strain relationship from the measurement of Young's modulus at a number of residual stress levels. The procedure consisted of first fitting the measured Young's modulus versus the stress points, $E(\sigma)$, with an arbitrary function, such as a polynomial. Young's modulus measured by the resonant frequency technique is the slope of the stress-strain curve at the stress of the coating (the tangent modulus) because the flexural bending imposes a small strain variation on top of the coating's original stress state. That leads to the following differential equation:

$$\frac{d\sigma}{d\varepsilon} = E(\sigma) \qquad (4)$$

Integration of the differential equation, as given in Eq. 5, was performed numerically to derive the stress-strain curve.

$$\int \frac{d\sigma}{E(\sigma)} = \int d\varepsilon \qquad (5)$$

The integration resulted in an arbitrary constant which was determined by assuming that the stress-strain curve went through the point (0,0). As an example of the result of the procedure, Figure 8 gives a plot of the stress-strain curve with the stress and strain normalized to their maximum values. Also given is the absolute strain level. The non-linear elastic behavior is evident in the shape of the curve.

Conclusions

A method was demonstrated for determining the stress-strain curve of TBC's on substrates from measurements of Young's modulus at different TBC residual stress levels. Temperature and applied plastic strain were used to vary the residual stress levels. Young's modulus was determined from a change in resonant frequency and the residual stress from a change in substrate curvature before versus after coating removal. Over the strain range between 0 and -0.0029, significant non-linear elastic behavior was observed. The modulus changed by a factor of 15 over that range. Previous measurements (7) on free standing APS YSZ showed a factor of 3 increase over the strain range between 0 and -0.008. The non-linear elastic behavior is expected to result from details of the coating microstructure produced by air plasma spraying.

Acknowledgments

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