ANL/ET/CP--91637   CONF-970111--7

USE OF INDENTATION TECHNIQUE TO MEASURE ELASTIC MODULUS OF PLASMA-SPRAYED ZIRCONIA THERMAL BARRIER COATING*

J. P. Singh, M. Sutaria, and M. Ferber[#]

Energy Technology Division
Argonne National Laboratory
Argonne, IL 60439

[#]Oak Ridge National Laboratory
Oak Ridge, TN 37831

RECEIVED
MAR 2 5 1997
OSTI

January 1997

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

MASTER

DISCLAIMER

Portions of this document may be illegible
in electronic image products. Images are
produced from the best available original
document.

USE OF INDENTATION TECHNIQUE TO MEASURE ELASTIC MODULUS OF PLASMA-SPRAYED ZIRCONIA THERMAL BARRIER COATING

J. P. Singh, M. Sutaria, and M. Ferber[#]
Energy Technology Division, Argonne National Laboratory, Argonne, IL 60439
[#]Oak Ridge National Laboratory, Oak Ridge, TN 37831

ABSTRACT

Elastic modulus of an yttria partially stabilized zirconia (YSZ) thermal barrier coating (TBC) was evaluated with a Knoop indentation technique. The measured elastic modulus values for the coating ranged from 68.4 ± 22.6 GPa at an indentation load of 50 g to 35.7 ± 9.8 at an indentation load of 300 g. At higher loads, the elastic modulus values did not change significantly. This steady-state value of 35.7 GPa for ZrO_2 TBC agreed well with literature values obtained by the Hertzian indentation method. Furthermore, the measured elastic modulus for the TBC is lower than that reported for bulk ZrO_2 (≈190 GPa). This difference is believed to be due to the presence of a significant amount of porosity and microcracks in the TBCs.

INTRODUCTION

Thermal barrier coatings (TBCs) have shown great potential for increasing turbine inlet-gas temperature by reducing heat transfer from hot gas to air-cooled blades. Turbine superalloys melt in the range of 1230-1315°C [1]. Because the combustion gas environment is at ≈1370°C, the superalloys are air-cooled to protect them from the oxidation, creep, melting, and thermal fatigue that reduces efficiency. Efficiency is traditionally improved by improving the design of the cooling system. An alternative approach is the use of TBCs. A typical TBC system consists of an insulating outer ceramic layer and an inner metallic "bond coat" layer between the ceramic layer and the metallic substrate. TBCs provide improved performance and

increased efficiency by lowering the metal temperature and reducing the cooling air requirements [2-4].

However, premature failure of the ceramic coating layer is a concern for the reliability and long service life of gas turbine components [5-7]. Such failure of ceramic coatings may be related to factors such as residual stresses developed in the ceramic layer during cooling because of the expansion mismatch between the ceramic and the metallic layers [5-9]; cyclic inelastic strain in the coating due to thermal cycling during service [10-11]; as well as oxidation, plastic deformation and creep of the bond layer, and phase transformation and sintering of the top layer.

Many of these causes (especially those that are stress-related) are greatly influenced by the elastic modulus of the coating. Therefore, the elastic modulus becomes a critical parameter to be evaluated for improved understanding and prediction of TBC failure.

Conventional techniques used for bulk ceramics have been used to measure elastic moduli of ZrO_2 coatings with limited success. These techniques consist primarily of loading coated specimens in uniaxial tension or flexural mode and then estimating the elastic modulus from load-deflection plots. Inconsistent results observed in many cases have been attributed to the errors introduced in measurements because of the small thickness of coating samples. To this end, measurement of elastic modulus by an indentation technique offers great promise [12-14].

In a recent study [12], Hertzian indentation testing has been used to generate contact damage in plasma sprayed ceramic coatings and subsequently the measurements were made to evaluate elastic modulus of the coatings. In addition, Knoop indentation testing [13,14] has been used to measure elastic modulus of bulk ceramics. This method is based on measurement of elastic recovery of surface impression of the indentation diagonals (a and b) made by the Knoop indenter, as shown in Fig. 1. The elastic recovery is independent of load and is correlated with hardness (H) to elastic modulus (E) ratio as follows:

$$\frac{b'}{a'} = \frac{b}{a} - \alpha\frac{H}{E} , \qquad (1)$$

where a' and b' are the dimensions of the Knoop diagonals after elastic recovery, and α is a constant. The value of α was determined as 0.45 by curve-fitting the

experimental data to Eq. 1. In the equation, values of a, b, a', and b' can be obtained from Knoop indentations, and H can be measured by using Vickers indentation [15,16] to estimate E. As indicated by Marshall et. al. [13], the relative error in the estimation of H/E is ≤10% for most brittle materials.

The purpose of this study is to demonstrate the applicability of Knoop indentation testing for the measurements of elastic modulus of ceramic coatings and compare the results with those obtained by other techniques (especially Hertzian indentation testing).

EXPERIMENTAL PROCEDURES

Material

The TBC sample consisted of substrate bars machined by electro-discharge machining (EDM) from 1.0 in. round stock of Waspaloy, supplied by Teledyne Allvac/Vasco. The alloy composition (in wt.%) was Ni-58.6, Cr-19.3, Co-12.5, Mo-3.9, Ti-2.93, Al-1.4, Fe-1.1, Zr-0.07, Cb(Nb)-0.06, Si-0.06, Mn-0.04, Ta-0.03, Cu-0.02, S-0.0003, C-0.006, P-0.005. One face of the substrate bar was grit-blasted (24 grit Al_2O_3 at 70 psi), after which the specimen was cleaned ultrasonically in methanol. The substrate was first coated with 5 mil of NiCrAlY and subsequently with ≈10 mils of yttria partially stabilized zirconia (YSZ) by air plasma spraying. The YSZ powder contained 7-9 wt% Y_2O_3. A typical photomicrograph of the polished cross-section of the TBC is shown in Fig. 2; significant porosity in the zirconia top coat (180 μm) is clearly evident.
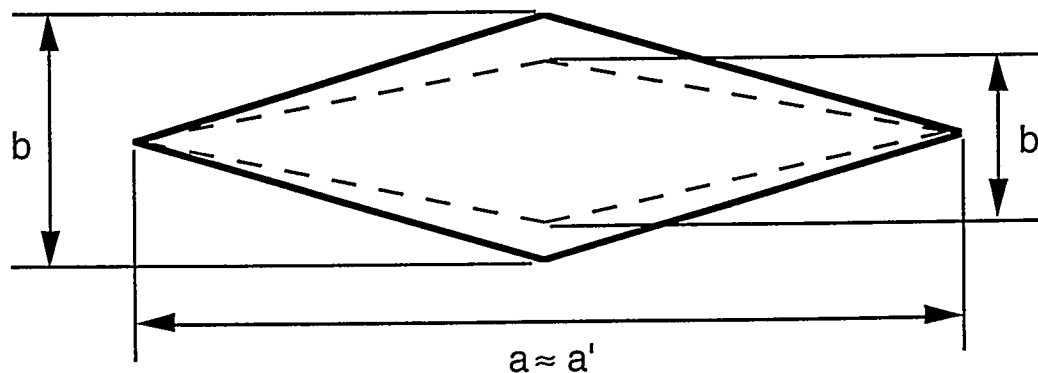

Fig. 1. Schematic showing elastic recovery of a Knoop indentation.
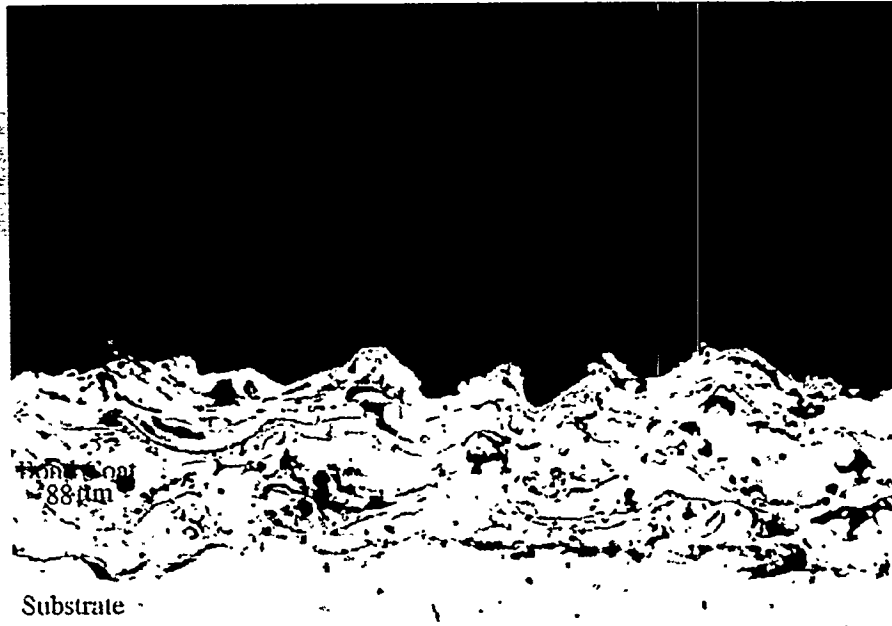
Fig. 2. Photomicrograph of typical polished cross section of TBC.

Indentation Testing

First, the sample was fine-polished (0.5 μm) and then its hardness (H) was measured at various positions using a Vickers indenter. The hardness was calculated by Equation 2, as shown below:

$$H = 1854.4 \frac{F}{d^2} , \qquad (2)$$

where F is the indentation load (g) and d is the indentation diagonal (mm). Indentations were made at 50, 100, 300, and 500 g loads, and the loading time was kept constant at 15 s for all indentations. Thirty to forty indentations were made for each condition to obtain a statistically reliable value.

Subsequently, the surface was fine-polished and Knoop indentations were made at the same locations with identical loads and times as mentioned above. The Knoop indentation diagonals (as shown in Fig. 3) were carefully measured with a high-magnification optical microscope. Elastic recovery (b'/a' - b/a) was evaluated with a known value of a/b (7.11) for the Knoop indenter geometry, and elastic moduli were calculated by Eq. 1.
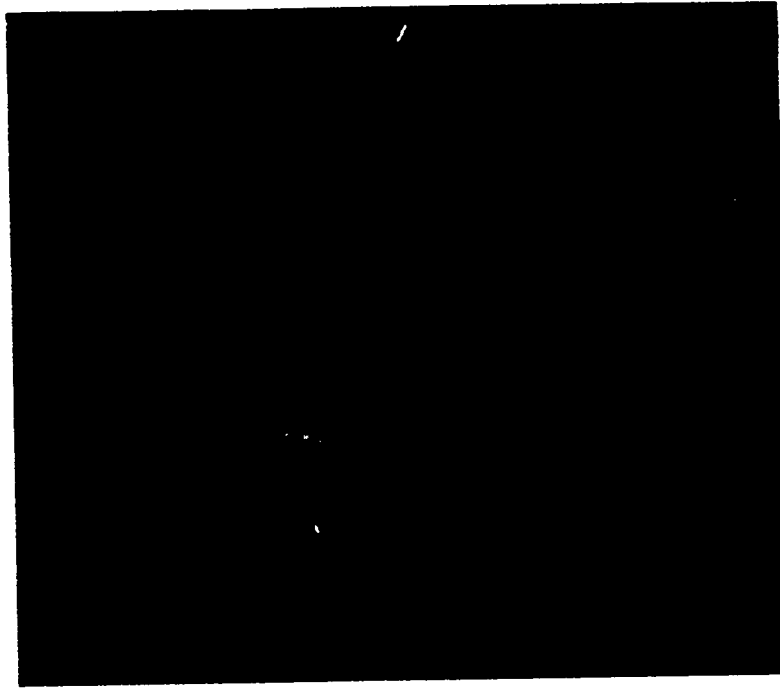
Fig. 3. Photomicrograph of typical Knoop indentation.

RESULTS AND DISCUSSION

Figure 4 shows the variation of hardness with indentation load. Hardness decreases from 6.1 GPa at an indentation load of 50 g to 3.3 GPa at an indentation load of 300 g. At a higher load of 500 g, hardness appears to approach a steady state intrinsic value of 1.8 GPa. Similar dependence of hardness was also observed by Hirao and Tomozawa [17] for vitreous silica and by Swain and Bradt [18] for yttrium and aluminum garnet. Several models have been proposed to explain this dependence. Recently, Li and Bradt [19] explained this dependence in terms of Newtonian-like proportional specimen resistance (PSR). According to this model, the effective indentation test load is equal to the applied indentation load minus PSR, where PSR increases with indentation load and size. The analysis also suggests a critical load below which hardness is load-dependent. Above the critical load, the hardness is independent of indentation load and relates to intrinsic hardness value.
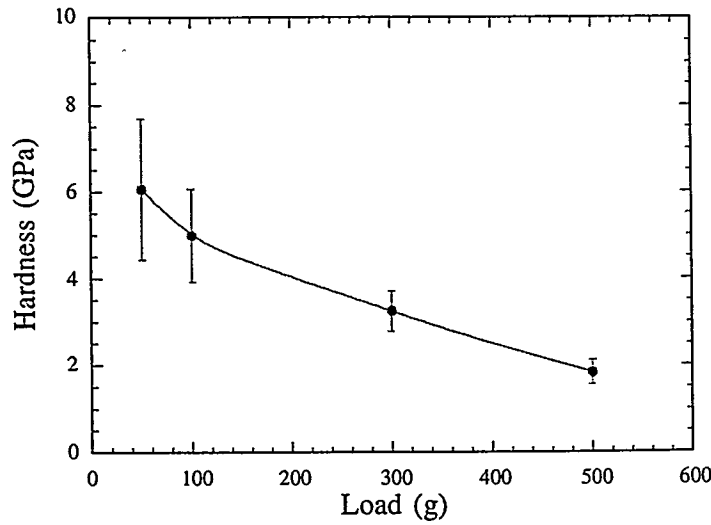
Fig. 4. Variation of hardness with indentation load.

Figure 5 shows the dependence of elastic modulus on indentation load; the elastic modulus decreases from 68.4 ± 22.6 GPa at an indentation load of 50 g to 35.4 ± 13.4 GPa at an indentation load of 500 g. As expected, at lower loads the indentation encompasses only a relatively small region of the TBC. The indentation size is on the order of a single grain or a lamellae of ZrO_2 coating. This minimizes the extent of defects (such as pores, grain/lamellae boundaries, microcracking, etc.) encompassed by the indentation, which results in observed higher elastic modulus values. On the other hand, at higher loads, the indentation size is large and encompasses many of the above-mentioned microstructural defects, as shown in Fig. 6. This results in the observed decrease in elastic modulus. After a critical load ($\approx$300 g in the present case), the extent of defects encountered per unit area or per unit volume by the indentation becomes relatively constant. Therefore, the resulting elastic modulus value becomes insensitive to the indentation load after the critical load is reached and this value can be used for materials selection and component design.

Table I compares the elastic modulus value obtained in the present study by Knoop indentation with that obtained by the Hertzian indentation method [12]. The elastic modulus value of 35 GPa obtained by Knoop indentation agrees very well with the value of 30 GPa obtained by Hertzian indentation. Also, the hardness value (1.8 GPa) obtained by Vickers indentation method in the present study is the same as that obtained by Pajares et al. [12].
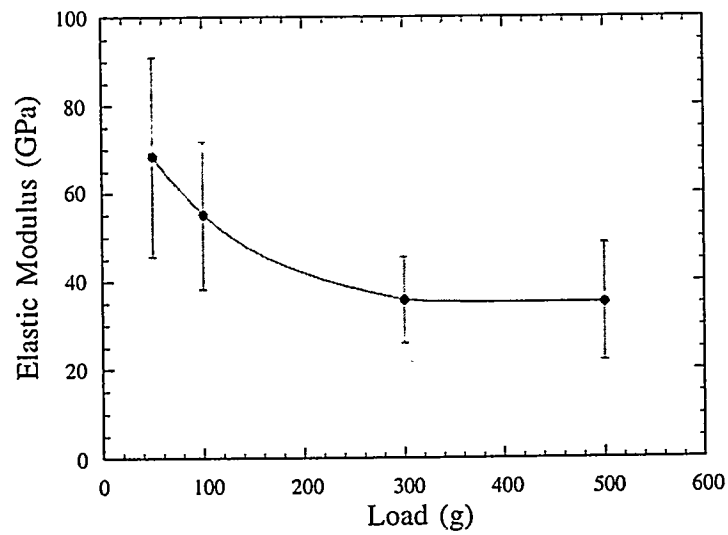
Fig. 5. Dependence of elastic modulus on indentation load.

Fig. 6. Typical photomicrograph of ZrO_2 coating showing microstructural defects such as microcracks and porosity.

Table I.   Comparison of elastic moduli resulting from
Knoop and Hertzian indentation methods

Testing Method	Elastic Modulus (GPa)
Knoop indentation	35 ± 13
Hertzian indentation [12]	30 ± 6

It is also to be noted that the measured elastic modulus values ($\approx$40-65 GPa) for the ZrO_2 TBC are much lower than those reported for fully dense bulk ZrO_2 ($\approx$190 GPa). This difference is due to the presence of significant porosity and microcrack [20], as shown in Fig. 6.

CONCLUSIONS

The hardness and elastic modulus of ZrO_2 thermal barrier coating was measured by the Vickers and Knoop indentation techniques. Hardness and elastic modulus values decreased with increasing indentation load and approached a steady state value of 1.8 and 35 GPa, respectively, at an indentation load of 500 g. These values agree very well with those measured by the Hertzian indentation method, demonstrating the applicability of Knoop and Vickers indentations for reliably measuring the elastic modului of thermal barrier coatings.

ACKNOWLEDGMENTS

REFERENCES

1.   S. M. Meier and D. K. Gupta, "The Evolution of Thermal Barrier Coatings in Gas Turbine Applications," J. Engineering for Gas Turbine and Power, Trans. ASME, 111, 301-305, 1989.

2. D. L. Ruckle, "Plasma Sprayed Ceramic Coatings for Turbine Vane Platforms," Thin Solid Films, 73, 455, 1980.

3. R. A. Miller, S. R. Levine, and S. Stecura, "Thermal Barrier Coatings for Aircraft Gas Turbines," AIAA Paper No. 80-0302, 1980.

4. D. S. Duvall and D. L. Ruckle, "Ceramic Thermal Barrier Coatings for Turbine Engine Components," ASME Paper No. 82-GT-332, 1982.

5. R. A. Miller and C. E. Lowell, "Failure Mechanisms of Thermal Barrier Coatings Exposed to Elevated Temperature," Thin Solid Films, 99, 265, 1982.

6. S. J. Grisaffe and S. R. Levine, *Proceedings of the First DOE/EPRI Conference on Advanced Materials for Alternate Fuel Capable Directly Fired Heat Engines,* Castine, ME, 1979.

7. D. L. Ruckle, "Evaluation of Plasma Sprayed Ceramic Coatings for Turbine Engine Components," Thin Solid Films, 64, 327, 1979.

8. P. F. Becher, R. W. Rice, C. C. Wu, and R. L. Jones, "Factors in the Degradation of Ceramic Coatings for Turbine Alloys," Thin Solid Films, 53, 225, 1978.

9. S. Rangaswamy and H. Herman, "Thermal Expansion Study of Plasma-Sprayed Oxide Coatings," Thin Solid Films, 73, 43, 1980.

10. J. T. DeMasi-Marcin, K. D. Sheffler, and S. Bose, "Mechanisms of Degradation and Failure in a Plasma-Deposited Thermal Barrier Coating," ASME Journal of Engineering for Gas Turbines and Power, 112, 521-526, 1990.

11. "Thermal Barrier Coating Life Prediction Model Development-Final Report, No. NASA CR-180807," Performed by General Electric Co., Cincinnati, OH; Sponsored by National Aeronautics and Space Administration, Lewis Research Center, Cleveland, OH, Nov. 1988.

12. A. Pajares, L. Wei, B. R. Lawn, N. P. Padture, and C. C. Berndt, "Mechanical Characterization of Plasma Sprayed Ceramic Coatings on Metal Substrates by Contact Testing," Materials Science and Engineering A, 208,158-165, 1996.

13. D. B. Marshall, T. Noma, and A. G. Evans, "A Simple Method for Determining Elastic-Modulus-to-Hardness Ratios using Knoop Indentation Measurements," Comm. Am. Ceram. Soc., 65 (10)C-175-C-176, 1982.

14. B. R. Lawn and V. R. Howes, "Elastic Recovery at Hardness Indentations," J. Mater. Sc., 16 (10) 2745-52, 1981.

15. A. G. Evans and E. A. Charles, "Fracture Toughness Determinations by Indentation," J. Am. Ceram. Soc., 59(7-8)371-72, 1976.

16. G. R. Anstis, P. Chantikul, B. R. Lawn, and D. B. Marshall, "A Critical Evaluation of Indentation Techniques for Measuring Fracture Toughness: I," J. Am. Ceram. Soc., 64(9)533-38, 1981.

17. K. Hirao and M. Tomozawa, "Microhardness of SiO_2 Glass in Various Environments." J. Am. Ceram. Soc., 70(7)497-502, 1987.

18. M. V. Swain and R. C. Bradt, "Nano and Micro Hardnesses of Single Crystal Yttrium Aluminum Garnet (YAG) on the {111} Plane," Plastic Deformation of Ceramics, eds. R. C. Bradt, C. A. Brookes, and J. L. Routbort, Plenum Press, New York, 1995.

19. H. Li and R. C. Bradt, "The Indentation Load/Size Effect and the Measurement of the Hardness of Vitreous Silica," J. Non-Cryst. Solids, 146, 197-212, 1992.

20. D. P. H. Hasselman and J. P. Singh, "Analysis of Thermal Stress Resistance of Microcracked Brittle Ceramics," Ceram. Bull., 58(9), 856-60, 1979.

DISCLAIMER