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Fracture Toughness of Thermal Spray Ceramic Coatings Determined by the Indentation Technique

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The indentation technique for determining material toughness is applied to spinel and yttria-stabilized zirconia plasma-sprayed coatings in this investigation. Fracture toughness of the coatings ranged from 1.9 to 3.4 MPa$\sqrt{m}$ for spinel and 2.0 to 3.3 MPa$\sqrt{m}$ for yttria-stabilized zirconia. These results are in good agreement with those obtained by other experimenters for bulk materials.

1. Introduction

THE determination of fracture toughness of a ceramic material has assumed greater importance as these materials are required to meet increasing operational stresses. Mechanical testing is therefore necessary to predict the service behavior of these ceramics. Testing does not guarantee specific component performance, but it is critical in both assessing new materials and in ranking materials for specific application.

Being inherently brittle, ceramics do not deform in a forgiving manner, as do metals and cermets. Rather, failure occurs catastrophically. However, through the application of fracture mechanics, it is possible to determine a fracture toughness parameter that permits calculation of failure stress in the presence of a crack.[1] This parameter, denoted as fracture toughness, is a measurable property, and the measurements when made by conventional ASTM techniques are generally acknowledged to yield reliable results if properly performed. However, the application of this technique is difficult. Specimen preparation is tedious and expensive for ceramic coatings. The difficulties are much greater, and the required specimen thickness and geometry are very difficult to achieve. Aspects of this topic have been discussed at length in other review articles.[2-7]

Because of these problems with the standard ASTM techniques, there has been an increasing interest in the application of indentation fracture mechanics as a simple technique for determining the fracture toughness values of brittle materials.[8-10] Using a combined fracture mechanics and dimensional analysis technique, Evans and Charles[11] characterized the surface cracks generated by Vickers indentation of brittle materials. Their analysis was successful on materials that had fracture toughness values that spanned a large range. This analysis[12] included hardness, Young's modulus, and crack length as parameters in the calculation of fracture toughness values.

This test method can be used[12,13] for measuring fracture toughness of brittle materials, and valid data can be obtained from ceramic specimens. The technique uses a diamond indenter to initiate and propagate a crack on a polished ceramic surface. The length of the observed cracks that extend from the four corners of a Vickers microhardness indentation are mathematically related to the force exerted on the indenter and the material fracture toughness. In addition to its simplicity, this technique has several attractions as a tool for characterizing the mechanical response of brittle materials. The geometry and the size of the crack patterns can be controlled accurately, and the location of the contact site is predetermined.

The main objective of the present investigation is to adapt this technique to measure the fracture toughness of several different plasma-sprayed ceramic coatings such as spinel (i.e., MgO-ZrO_2) and yttria-stabilized zirconia and compare the results with those of bulk ceramics generated by standard ASTM techniques.

Key words: ceramics, cracking in ceramics, fracture toughness, indentation technique, spinel, yttria-stabilized zirconia

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2. Experimental Procedure

The materials chosen for this investigation were provided by Fisher-Barton, Inc., Watertown, Wisconsin. These materials consist of spinel and an yttria-stabilized zirconia coating. Coatings were applied by plasma spray using optimized spray parameters. Initial planar grinding was performed with the MINIMET using a 30-µm diamond suspension on a METALAP 10 platen. Samples were initially polished using a 6-µm diamond suspension on a TEXMET polishing cloth. Final polishing was conducted on a vibratory polisher using a TEXMET cloth and MASTERPOLISH™ 2.[14] Specimens were indented using a Rockwell superficial hardness machine equipped with a Vickers diamond pyramid indenter.

Optical microscopy was used to measure the indentation parameters of crack length, *c*, and impression diagonal, *a*. An estimated accuracy of ±3 ocular divisions was used in obtaining the crack length measurements and the accuracy of the impression diagonal was estimated to be ±2 ocular divisions. This corresponds to an accuracy of ±0.001 mm at 100× magnification. The crack lengths were measured immediately after releasing the load, and the fracture toughness was calculated from the following equation:[13]

$$K_{Ic} = 0.016\,(E/H)^{1/2}\,(P/C^{3/2}) \quad [1]$$

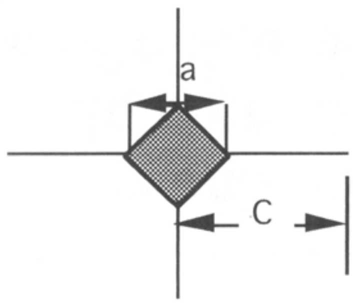


Fig. 1 Schematic of Vickers indentation setup showing characteristic dimensions of crack length (c) and hardness impression (a). This is also known as the median crack length.

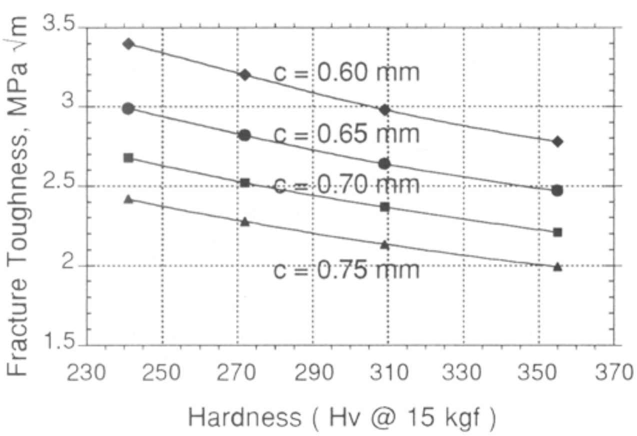


Fig. 2 Fracture toughness at 30-kgf load as a function of hardness calculated from median crack (Eq 1) in spinel.

Table 1 Cracking load determination

Material	Load, kgf, for $c > 2a$
Spinel	30
Yttria-stabilized zirconia	60

where K_{Ic} is the fracture toughness, MPa$\sqrt{m}$; P is the indenter load, kgf; E is the Young's modulus, GPa; H is the hardness, GPa; and C is the crack length (mm), as shown in Fig. 1.

3. Results and Discussion

The indentation loads necessary to produce cracks suitable for both materials are shown in Table 1.

These loads were based on the criteria $c > 2a$ without chipping.[15] As shown in Table 1, no crack extensions were observed for loads below 30 kgf in the case of the plasma-sprayed spinel coating and below 60 kgf for the yttria-stabilized zirconia coating. However, at loads of 30 kgf and 60 kgf, cracks were obtained that were sufficiently long to satisfy the $c/a > 1.8$ criterion of the Evans and Charles method.[11] At five randomly chosen points on each prepared specimen, an indentation was

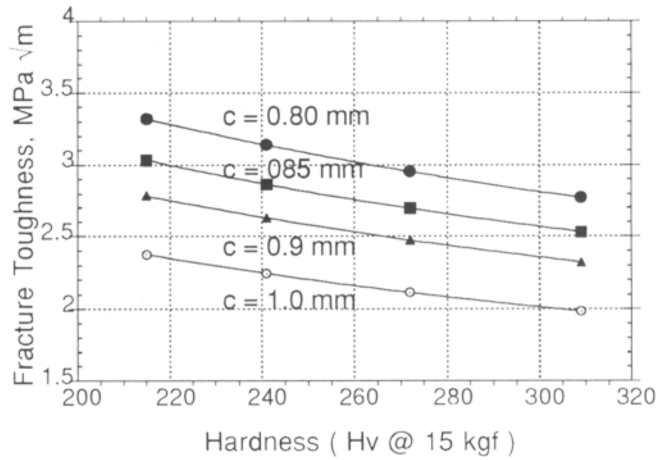


Fig. 3 Fracture toughness at 60-kgf load as a function of hardness calculated from crack (Eq 1) in yttria-stabilized zirconia.

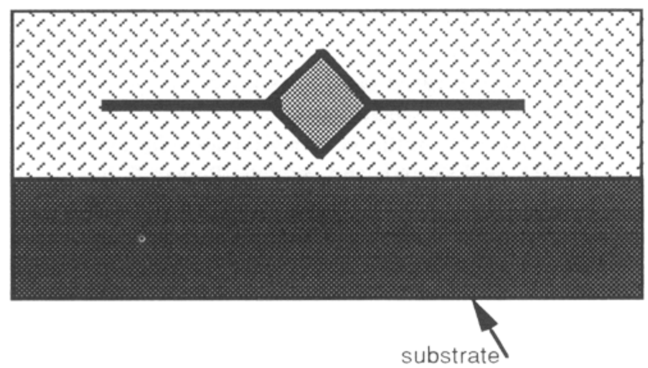
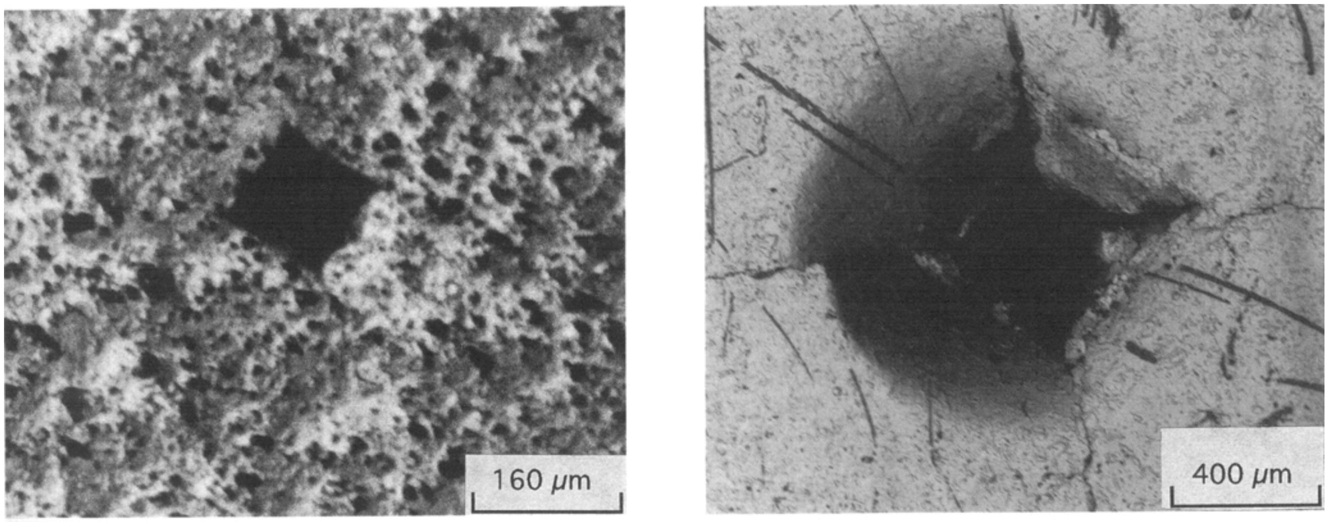


Fig. 4 Schematic representation of sharp indentation induced crack patterns in plasma-sprayed coating.[16]

made using the same load at each point. This load was kept constant for each coating type. The crack length was measured for each indentation, and these crack lengths were then averaged arithmetically. There were two specimens of each coating type, and the average crack length of the individual specimens was combined to produce an average crack length for each coating type. The hardness value for each material was measured using the same method as that used to obtain the crack length. Having obtained the crack length and the hardness and using an estimated elastic modulus of 145 GPa for spinel[16] and 284 GPa for yttria-stabilized zirconia,[17] the fracture toughness of each coating material was calculated.

The procedure averaged out variations in crack length and hardness and produced a single value for fracture toughness. Another approach would be to produce an indent at low load to measure hardness and then increase the load at the same indentation to produce a crack. This would yield the precise K_{Ic} at that point. Unfortunately, with the present equipment, this is not possible. Therefore, to determine the range of K_{Ic} values for each coating type, the following procedure was used, and the results of the procedure are shown in Fig. 2 and 3. First, on a single specimen of each coating type, four indents were made under



(a) (b)

Fig. 5 Vickers indent in spinel plasma-spray coating **(a)**. Microcracking propagation from the Vickers indentation in spinel plasma-spray coating **(b)**.

Table 2 Comparison of fracture toughness values

Material	K_{Ic}, MPa$\sqrt{m}$	Ref
Zirconia (metastable phase)	6-9	16
Zirconia (stable phase)	1.1	21
Spinel	1.2-1.9	22
Yttria-stabilized zirconia (mixed phase)	2.0-3.3	Present study
Spinel	1.9-3.4	Present study

15-kgf load, and the hardness of each was recorded. Figures 2 and 3 show the range of fracture toughness values that may be calculated for crack lengths from 0.60 to 1.00 mm. Second, on the same specimen, four indents were made under the specified load for each coating type, and the crack lengths were measured and recorded, from which another estimate of the fracture toughness value could be calculated (Fig. 2 and 3). Then, from each combination of hardness and crack length, a K_{Ic} value was calculated. From these combinations, the range of K_{Ic} values for each coating was obtained, as shown in Fig. 2 and 3.

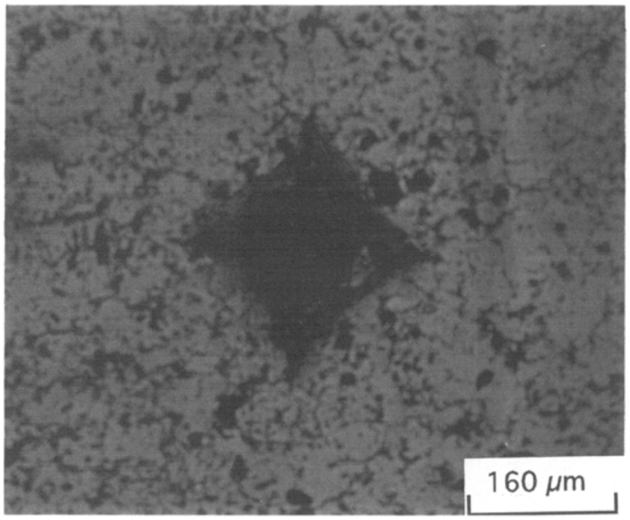
A description of the crack patterns resulting from Vickers indentation in the plasma-sprayed coatings is given in Ref 18. These crack patterns are produced by indenting a cross section of the coating. The crack pattern is identified as crack type I (Fig. 4), where the dominant cracks are parallel to the substrate and emanate from or near one or two of the impression diagonal corners. This pattern can be viewed as (1) resulting from the indent position in a dense portion of coating where complete transmission of indentation stresses to the surrounding material would be expected to produce a Boussinesq stress field,[19,20] with the resultant surface traces, or (2) resulting from stress field interactions with large coating pores or defects located near one or both of the impression diagonals.

In the present study of plasma-sprayed coatings, indents were made perpendicular rather than parallel to the substrate, which produced cracks emanating from four corners of the indent instead of two. These results (cracks from each corner) are consistent with those found in the literature on fracture toughness measurement of bulk ceramics. The presence of porosity and the fact that plasma-sprayed coatings do not have homogeneous microstructures produce variations in the fracture toughness values obtained by the indentation method. The fact that the values obtained from use of the indentation method on plasma-sprayed materials had a smaller variance than values obtained from the same materials in the bulk form does not imply that there is no relationship between K_{Ic} and microstructure, but instead demonstrates the superiority of the indentation method compared to the conventional techniques.

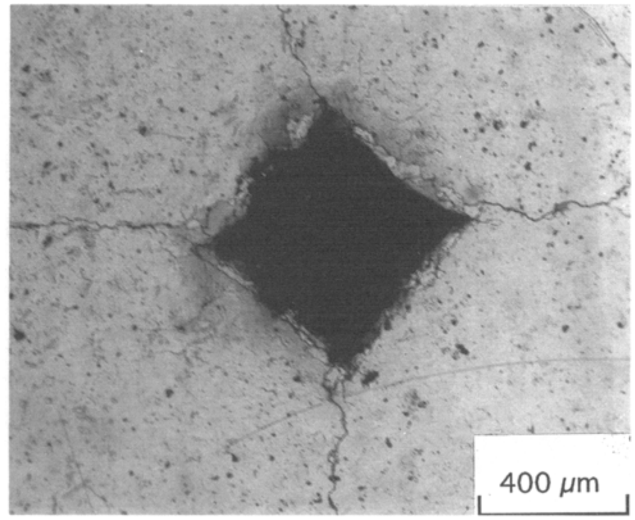
The fracture toughness, as generated by the indentation method, ranged from 1.9 to 3.4 MPa$\sqrt{m}$ for the spinel coating and from 2.0 to 3.3 MPa$\sqrt{m}$ for yttria-stabilized zirconia coating (Table 2). Figures 5 and 6 show the indents for a spinel coating and an yttria-stabilized zirconia coating before and after crack initiation.

4. Conclusion

Fracture toughness values for plasma-sprayed spinel and yttria-stabilized zirconia coatings were determined using the indentation method. As shown in Table 2, good agreement was found between the indentation method and conventional techniques for spinel and mixed phase yttria-stabilized zirconia. The effect of microstructure should be taken into consideration, because the plasma-sprayed coatings have different levels of porosity and microstructure than that of bulk materials. Despite possible differences in microstructure, the present results agree reasonably well with experimental data on bulk ceramics.



(a)



(b)

Fig. 6 Vickers indent in yttria-stabilized zirconia plasma-spray coating (**a**). Microcracking propagation from the Vickers indentation in yttria-stabilized zirconia plasma-spray coating (**b**).

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