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Photoacoustic microscopy of indentation lateral cracks

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Abstract: Understanding the fracture process in advanced ceramics is of obvious use for improving their strength, toughness and ultimately performance. The lateral cracking process is related to wear in particular. Photoacoustic microscopy is used here to view lateral cracks produced in a controlled manner by Vicker's indentations in hot pressed silicon nitride (NC 132, Norton Co.). Eventually, a figure of merit related to bearing performance may emerge.

1. INTRODUCTION

Because of their combination of hardness, wear tolerance, corrosion resistance, high temperature capability and low density, ceramics are being applied in valve train applications. Two examples are the new Detroit Diesel Series 60 engines with ceramic cam followers and the Advanced Ceramic Technology Insertion Program for the 8V71T/LHR Self Propelled Howitzer. As exemplified in a Chrysler effort however, problems remain. It was noted there [1] that there were no material properties that correlated with bearing performance. Hence, there is a need for a measure of the bearing performance potential of a material to guide development. Again, R. N. Katz [2] observed that ceramic bearing failures were like those of M50 steel bearings, i.e., subsurface failure followed by lateral cracking and subsequent spallation. To generate controlled lateral and vertical cracking, diamond indentors can be used. Indentation testing, which is widespread in the ceramics field because of its ease of implementation, provides hardness values, but also provides fracture toughness as it was noted [3] that the length of radial cracks from the corners of Vickers indents correlated with other measures of fracture toughness. Indentation testing for fracture toughness has proven remarkably robust despite misconceptions of the actual crack system present and the many different equations used for numerical determination [4].

By analogy with the observation on the relation of the surface trace of the radial cracks and fracture toughness, ranking of candidate materials based on the lateral cracks could lead to a figure of merit related to bearing performance. The problem has been that the lateral crack system in opaque ceramics for industrial use has been inaccessible.

Figure 1 schematically shows a Vickers indentation. The surface radial cracks from the indent are shown. Though a vertical "half-penny" crack is shown in the cross section associated with the surface crack of the plan view, surface radial cracks do not guarantee half-penny cracks in general [5]. The lateral cracks are usually thought to emanate from the bottom of a plastic zone underneath the indentation as shown. Thermal wave imaging of indentations has been accomplished before [6,7,8]. This paper delves more deeply into the lateral crack system so revealed.

Interpretation of the images is based on previous theoretical and experimental models of the response at vertical cracks, slanted cracks and exposed and buried edges [9-12]. Cracks here are not completely planar, will have roughness on the crack faces and may lie in a "crack process zone" of deformed material and microcracks. In addition, indentation generates intersecting crack systems.

Fig. 1. Schematic of a Vickers indentation.

2. EXPERIMENTAL

Laser generation with gas cell detection is used [13]. For this application, gas cell detection has an appropriate resolution, has a simpler interpretation since it responds only to the temperature without convolutions of elastic properties and emissivity (the crack structure is complicated enough already), and is insensitive to perfectly vertical cracks. By imaging at a range of modulation frequencies, the thermal diffusion length is varied revealing depth structure.

Indents were made with loads of 2, 5, 10, 15, 20, 25 and 30 Kg in hot pressed silicon nitride (NC132, Norton Co.). These are higher loads than normally used for hardness testing, but are necessary to generate the radial cracks for fracture toughness measurement. Silicon nitride is a leading bearing candidate.

The microstructure is composed of beta silicon nitride grains, typically 1-2 μm in diameter and 10-15 μm in length, predominantly perpendicular to the hot pressing direction [14]. They lie in the plane of the images shown. Radial cracks will tend to cross these grains while laterals will propagate parallel to them.

3. RESULTS

Image sets of scanned optical, magnitude and phase at frequencies from 175 Hz to 10 KHz are shown in figure 2 of two 20 Kg indents. The focal spot was 8.1 μm and the step size 4.0 μm for this set.

If there were no laser power variations, the images in the optical column would be identical. The surface deformation arising from the indentations can be seen since the heating beam was slightly off center giving rise to shadowing. The faint circular feature to the right of center is a water mark left from cleaning of the sample. This serves as a landmark and an example of a purely surface feature. The modulation frequencies are shown on the left. On the right hand edge is a reference bar indicating that, in this picture, higher numbers correspond to whiter pixels.

The most striking feature is the rosette pattern of the right indent in the phase at 2071 Hz. A corresponding faint black fringe exists in the 7600 Hz magnitude. A black fringe is what would be expected in the phase and magnitude for the particular lock-in amplifier used. This pattern is clearly larger than the surface deformation. From one dimensional theory [15], which is generally valid in three dimensions [10,16], the edge of the black ring in the indent phase image would be about 90 μm deep compared to the indent apex at about 66 μm . The "sharpness" of the black edge argues for the origin of its thermal contrast in cracking rather than in residual stress fields.

Fig. 2. Two 20 Kg indents in NC132 imaged from 175 Hz to 10 KHz. The area scanned is 1.53 X 0.508 mm. Scanned optical and gas cell magnitude and phase are shown.

The series of photoacoustic images show the black ring shrinking towards the indent as the modulation frequency rises. This demonstrates that the lateral crack system begins at a shallower depth near the indent and generally dives down and flattens out at its extremities. The non-symmetry of the black fringe demonstrates that we are not confusing an edge effect for lateral cracks. The edges of the indent have high symmetry and are outlined by a white box having a width of about one thermal diffusion length at the image's modulation frequency as would be expected [9]. The depth of the lateral crack system shown at 10 KHz is 40 μm or less. The gas cell response below the left indent in figure 2, bound sharply on the left by the radial crack at 7 o'clock, is interpreted as a shallow lateral crack [5].

A material inhomogeneity is particularly observable on the bottom, left of center, in the magnitude image at 175 Hz, where its effect is larger than that of the indents. It is clearly sub-surface. The presence of such unexpected thermally revealed structure demonstrates that much remains to be learned in these materials. The images in general have a "thermal microstructure" exhibiting a periodicity of about 2.6 μm as measured in other image sets, using a 3.4 μm spot size. This can be compared with the microstructure mentioned above.

Images of indents at 2 Kg had indistinct lateral crack systems with a greatest apparent extent of the effect of the indentation on the thermal wave response at 3528 Hz. The greatest apparent extent was at 2751 Hz for 10 Kg indents and 1 KHz for 30 Kg.

4. DISCUSSION

Previous mirage experiments found that mirage indicated radial crack length was at least 90 μm longer than the optically observed radial crack length [8]. However, compressive residual stresses in prepared surfaces of Si_3N_4 down to about 20 μm are known [4] and could easily account for some surface closure of radial cracks. Mirage and gas cell measurement of indents as the surface is polished down would be of interest.

The lateral crack system in NC 132 begins at the indent sloping downward and flattening out at its extremities. The lateral crack system is deeper for more heavily loaded indents and the lateral cracks are generally bound by the radial cracks. This is generally expected.

The lateral crack system that is observed does not appear to originate below a deformation zone below the apex of the indentation as generally assumed. Shallow lateral cracks mentioned by Cook and Pharr [5] as "a new and unexplored crack system" are common. They have obvious relevance to wear and erosion. Interaction of the radial and lateral cracks, which are usually considered separately, is apparent as in the bright cracks next to radial cracks.

The observed scale of the "thermal microstructure" and the indefinite lateral crack systems observed at loads of 2 Kg and below, indicates that models seeking to explain lateral crack development in NC 132 must allow for microstructural influence and micro-residual stress to considerably larger scale than has been assumed so far. Observations have been made that fracture toughness from "large" radial cracks has not correlated with "small cracks" responsible for material abrasion and wear performance [17]. The lateral cracks experience a different stress field than the radials which arise at the sharp corners of the indent, and so the crack path can be more tortuous and dependent on material microstructure as the crack seeks easier paths.

Questions remain. Near the indent thermal contrast may arise from deformed material and/or microcracking. High circumferential tensile stress (~ 700 MPa is required to form cracks) is present, but the stress is highly compressive radially. Depth estimates using the magnitude are half or less of those from using the phase as was done above.

In the long range, it is hoped that exploration of the lateral crack system of ceramic alloys will lead to a figure of merit for materials to be used for bearings.

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