

## **Micro-Macro Behavior Near the Crack Tip In a Composite Materials Based on Anodic Hard Ceramics**

Maksim Kireitseu

Department of Mechanics and Tribology

Institute of Machine Reliability (INDMASH), National Academy of Sciences of Belarus

Lesnoe 19 - 62, Minsk 223052, Belarus e-mail: [indmash@rambler.ru](mailto:indmash@rambler.ru)

An important engineering problem in structural design is evaluating structural integrity and reliability. Depending on the structural design, material type, service loading, and environmental condition, the cause and degree of strength degradation due to the different aging mechanisms differs. One of the common causes of strength degradation is the result of crack development in the structure. In this study, centrally cracked specimens were used to study micro-macro damage and deformation processes near the crack tip in a composite materials based on anodic hard ceramics. The specimens were made of alumina-based hard oxide ceramic coating. It is expected to extend fatigue life of the alumina layer by hardening with chrome carbide deposited on the coating. Experimental result reveals that, porosity, hardness, heterogeneity of the microstructure, interface and substrate, the initial distribution of flaws within the system, and the magnitude and sign of the residual stress in the layer play a key role for local damage and strain distribution near the crack tip. In addition, the damage fields near the crack tip were investigated. The strain fields and stress maps were discussed.

For more information, contact:

Dr. Maksim V. Kireitseu

Department of Mechanics and Tribology

Institute of Machine Reliability (INDMASH)

Lesnoe 19 - 62, Minsk 223052,

Belarus

Email: [indmash@rambler.ru](mailto:indmash@rambler.ru)

## Introduction

A ductile substrate like aluminium or its alloys coated with a brittle thin layer is one of common coating-substrate structures encountered in engineering applications. Fracture behaviour of the thin alumina-based composite coating and the coating adhesion are among the major considerations in evaluating the integrity and quality of such coating-substrate systems. A reliable and consistent measurement of these properties is also critical in improving the thin alumina-based composite processing technologies.

An important engineering problem in structural design is evaluating structural integrity and reliability. Depending on the structural design, material type, service loading, and environmental condition, the cause and degree of strength degradation due to the different mechanisms differs. One of the common causes of strength degradation is the result of crack development in the structure.

As the size of a mechanical structure used in smart systems (such as accurate microsystems, bearing units etc.) becomes susceptible to defects, the effect of such defects on structural strength has a vital importance. Fracture toughness is one of the most important mechanical properties, especially for designing and operating systems and constructions. Fatigue-induced cracking of materials becomes a serious problem when long-term reliable operation is required in a system.

## Objective

An objective of the work is to investigate micro-macro damage and deformation processes near the crack tip in composite materials based on hard alumina ceramics formed by micro arc oxidizing. The strain fields, damage fields and failure maps were discussed.

## Experimental technique

The electrostatically actuated test device was used to evaluate microfracture properties through a microcrack diagnosis of materials forming the systems for improvement of

micromechanical reliability. The test device consists of comb drives for loading and a suspending beam for testing. The sharp notch is introduced to the suspending beam in the test device.

Alumina layer is fabricated by micro arc oxidizing process. Figures 1 shows the SEM photograph of the pre-cracked alumina layer 200  $\mu\text{m}$  in thickness. CrC layer was produced by pyrolytic deposition from vapor phase of metalorganic solution. The CrC layer fills the pores and surface cracks that it is expected will improve fatigue of the alumina layer and near crack tip behavior.



Figure 1. SEM micrograph of alumina layer with cracks.

## Stress Contours & Failure Maps

It is possible to combine the results of the researches and generate stress contours and failure maps to illustrate which mechanisms are expected to operate under particular conditions.

Figure 2 shows crack path initiated in the layer under localized stresses that distributes through the coating under applied load. The picture was generated from SEM images by software approximation using finite element analysis in detail see upcoming works.



Figure 2. Crack propagation under localized loading of initial crack tip.

In fig.2 dark regions show internal voids, pores and defects with initiated cracks propagating in the layer under loading of the initial crack tip shown as darker segment in the top of the fig. 2. In fact, cracks propagate across the alumina layer concentrating in the porous and defect zones. The rest light region of the layer exhibits relatively dense and pore free layer.



Fig 3. SEM micrograph of crack path at a grain boundary site of alumina layer.

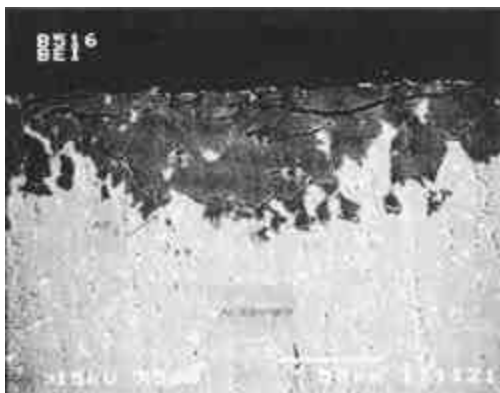


Fig. 4. SEM micrograph of crack along side the surface of alumina layer.



Fig 5. SEM micrograph of alumina layer showing the distribution of precipitates and cracks across the layer (perpendicular to the surface).

Recent study of Lawn<sup>1</sup> revealed that there are two fundamentally different fracture/damage

mechanisms in ceramic composites that are usually determined by their microstructures. First class of overviewed ceramics consists of very brittle materials, such as single crystals, and fine-grained ceramics. In this ceramics Hertzian indentation produces macroscopic cone cracks when the indentation load exceeds a certain critical value for a given material<sup>2-6</sup>. This type of cone cracking results in severe strength degradation<sup>7,8</sup>.

In contrast to the last case, in some of new composite ceramic coatings with advanced strength behavior, such as glass-ceramics and alumina based composites the cone cracking is suppressed, because of crack deflection along the composite sub layers, its micro granular submerged particles and hard microstructural elements. Along with some cone and ring cracks a damage zone is formed within the region of high shear-compression concentration just under the indenter<sup>8-11</sup> that has well shown at fig.6. In some cases both the highly destructive macroscopic cone cracking and the damage dispersion occur in the material. However, the growth and linkup of the microcracks under a high load or repeated loading conditions could lead to severe surface damage and ultimate material peeling off from the surface<sup>12,13</sup>.

One potential approach for avoiding such a lack is to use a multilayer composite structure, in which layers strengthens one another. In view of oxide ceramic layer another one should fill the pores, smooth sharp edges of alumina crystals and health other microstructural defects.

The view to this approach comes from the characteristic of the spatial distribution of Hertzian shear stresses in the alumina layer<sup>14</sup>. Figure 6 gives the contours of shear stresses ( $\sigma_v$ ) along the plane perpendicular to the loading axis (the r-z plane), and fig. 2 shows the contours of the maximum shear stresses ( $\sigma_{vmax}$ ). The stresses are presented in units of mean pressure. Comparing figs. 6 and 7 shows that (a) the highest value of  $\sigma_{vmax}$  is 50% of the mean pressure, whereas that of tensile stresses  $\sigma_{tz}$  is only 35% of the mean pressure; (b) throughout

the field,  $\sigma_{vmax} > \sigma_{rz}$ ; (c) for a given stress value, the area within the  $\sigma_{vmax}$  contour is larger than that within the  $\sigma_r$  contour.

Therefore, if the microstructural weaknesses, such as pores and defects, can be strengthened along the r-z plane, the failure and cracking of coating, and its dispersion and failure can be reduced to a certain degree.

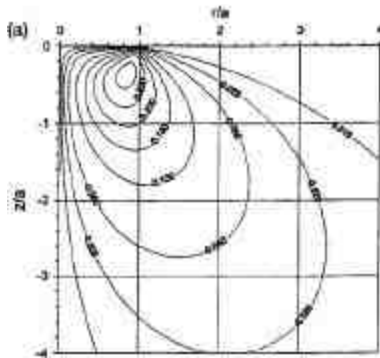


Fig. 6. Contours of shear stresses along the plane perpendicular to the loading axis.

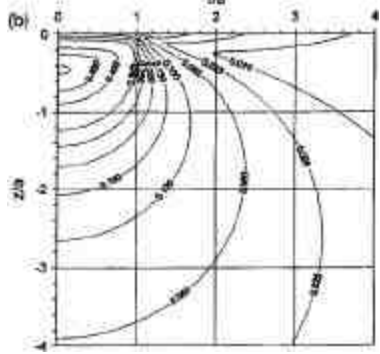


Fig. 7. Contours of maximum shear stresses.

Two failure stresses fields are shown in Fig. 6, 7 for the coating systems. The conditions for two possible modes of failure may result from a split in the alumina layer. Delamination along the interface is one possibility. Alternatively, the split may extend as a crack into the substrate. The conditions for delamination have been previously, while the split can propagate into the substrate<sup>15</sup>. Based on the tests it is expected that for the composite system it is if

$$K_I \cdot \sigma / \sqrt{t} < 1.2-1.95 \quad (1)$$

Once the crack has entered the substrate, it is possible for it to turn and propagate parallel to the interface as shown in fig. 4 and it is expected to be expressed as:

$$K_I \cdot \sigma / \sqrt{t} < 0.3-0.58 \quad (2)$$

Figure 6, 7 compares the conditions that will determine whether a crack initially lying along the interface will continue in that plane, or whether it will kink into the substrate. The condition for this latter event to occur is<sup>16,17</sup>

$$K_I \cdot \sigma / \sqrt{t} < 0.7-0.9 \quad (3)$$

In both of these stress field maps, the contours through the origin show the effect of varying the stress in the alumina layer while keeping the material properties constant. Two very important results should be noted. The first is that failure does occur along the maximal stress concentration zone and along weakest path; however, the mechanics of the problem will dictate different driving forces for crack propagation at different locations. This difference may result in the fracture condition being met along a trajectory with a high fracture resistance before it is met along one with a lower resistance.

A crack will propagate more readily in the substrate than along the interface. In other words, the fracture toughness of the substrate can be twice that of the interface before delamination becomes an issue in the failure of thin layers. A second observation to be made from these plots is that conditions may be such that more than one mode of failure is energetically favorable. Under these conditions, kinetic effects become important and failure will be dominated by the mechanism that has the faster kinetics of crack growth.

It is found that when fracture occurs in the alumina-based layer or substrate it will do so under pure mode-I conditions, and it is therefore appropriate to use the mode-I fracture toughness,  $K_a$  for alumina layer or  $K_s$  for

substrate, for these materials. Failure mechanism that involves propagation of a crack along the interface will introduce the problem of mixed-mode fracture. Ideally, the fracture resistance of the interface should be measured under the mixed-mode conditions that are appropriate; however, this is not always possible and some choice of failure criterion has to be made. For the purposes of this discussion, the choice that  $K_I = K_{II}$ , (the mode-I fracture toughness of the interface) has been made.

### Failure mechanisms

A crack, whether it develops in the alumina layer, interface or substrate, is driven by the relaxation of residual stresses and the consequent decrease in elastic energy stored in the system. In a homogeneous material, a crack will follow a trajectory in which it is subject only to an opening mode of deformation at its crack tip.<sup>18,19</sup> This is known as mode-I fracture.

The first mechanism to be considered in detail involves the extension of a crack along the interface between a layer and substrate. General results for the asymptotic values of the stress-intensity factors that are pertinent when the crack length  $a$  is much longer than the layer thickness ( $t$ ) have recently been obtained by Suo and Hutchinson.<sup>20</sup> The mixed-mode nature of the delamination process, the asymptotic values are approached as the crack length increases depends on the type of flaw that initiates delamination. If the crack grows in from the edge of the sample, then the stress-intensity factors increase with crack length, having values when  $a/t < 1$ .<sup>21</sup>

$$\begin{aligned} K_I \cdot \sqrt{t} &= 0.36 \cdot (a/t)^{1/2} \\ K_{II} \cdot \sqrt{t} &= 0.63 \cdot (a/t)^{1/2} \end{aligned} \quad (4)$$

The existence of a steady-state solution in these two cases is a direct result of the assumption that the problem is a two-dimensional one and that there is no variation in the geometry across the width of the samples.

Figure 3, 4 show a crack initiated from either an external in Fig. 3 or an internal Fig. 4 boundary of the layer. The lack of a steady-state regime in which stress intensity factor is independent of the crack length is very evident from these plots. Similarly, if a flaw initiates from a finite split in the layer, delamination is limited to the region around the split and scales with its length.<sup>22,23</sup>

### Alumina-based composite coating splitting.

A tensile stress in a brittle alumina-based composite coating may be relieved by the formation of a periodic array of parallel cracks growing across the alumina layer (Fig. 1, 5). The value of  $K_I$  which acts to split the alumina-based layer depends on the spacing between the cracks.<sup>24</sup>

Once splitting does occur, the analysis suggests that the crack spacing should be no larger than about eight times the alumina thickness; cracks further apart than this have a negligible interaction with one another.

Although these characteristics have been observed in at least one study,<sup>25</sup> most observations suggest that the crack spacing is much larger than the range predicted in view of defect concentration, and stress distributions respectively in Fig. 2, 6, and 7. There are two possible reasons for this discrepancy. The first is that this figure shows only the thermodynamic minimum spacing and that to obtain such spacing would require a suitable density of initial flaws. In practice, it is expected that the cracking pattern may be dominated by the statistical distribution of defects.<sup>26</sup> The other reason to be mentioned is that the crack spacing is determined by the critical shear stress at the interface<sup>27,28</sup> when the alumina layer is deposited on a ductile substrate, or if alumina layer cracking is associated with delamination at the interface. It can be shown that under these conditions<sup>27</sup>, the crack spacing is not dependent on the fracture toughness of the alumina-based layer and that, in contrast to the elastic analysis,  $\bar{\epsilon}$  increases with ( $t$ ):



$$\tilde{\epsilon} = 2\delta_0 \cdot t/\hat{\sigma} \quad (5)$$

In this equation  $\hat{\sigma}$  is the critical shear stress at which slip occurs and it may be controlled either by the yield stress of the substrate or by friction at the delaminated interface. It is revealed that although the spacing is independent of  $K_a$  for alumina layer in this shear-lag analysis, the critical stress at which splitting can occur still depends on it.

Finally, of interest is note that when there is a biaxial stress in the alumina-based layer, the periodic array of parallel cracks would be replaced by a pattern commonly known as "mud racking". The alumina layer fractures into a series of localized clusters similar to that seen in areas of drying mud. Although the details of the mechanics are slightly different, the general trends outlined in this section are expected to be similar.

#### *Substrate cracking.*

The third location at which a crack may propagate to relieve the strain energy stored by alumina layer under tension is within the substrate. There is a steady-state regime in which the stress-intensity factors are independent of the crack length. In this regime,  $K_I$ , and  $K_{II}$  depend on the elastic properties of the alumina layer and substrate and on the depth of the crack beneath the interface. Of particular note is the existence of a depth at which  $K_{II} = 0$ .

The analysis of the area confirms that this is the depth of the trajectory along which a crack will propagate. Experiments demonstrate that this trajectory has a remarkable stability which can be explained because any perturbation of the crack from this path results in a shear stress that acts to drive it back again.

Two effects of changing the modules of the layer and substrate can be observed in the alumina-based composite. The first is that as relation in Young modules of alumina-based layer and substrate decreases, the stable trajectory approaches the interface and the

resulting spall is therefore thinner. The other concerns the magnitude of the driving force, which appears to increase with the compliance of the layer. However, it should be remembered that if the stress is a result of some residual strain, such as might arise from a lattice mismatch or from a difference in thermal expansion,  $\delta_0$  scales with the modulus of the layer, and the dependence of  $K_I$  on the layer compliance will be reversed.

Suo and Hutchinson<sup>29</sup> have presented general results that allow the effect of any stress state, elastic constants and substrate thickness to be calculated. One of the important results confirmed as it was in the work<sup>30</sup> is the sensitivity of the crack path to the thickness of the Al substrate.

#### *Buckling.*

The mechanisms by which a compressive stress in a coating may be relieved are much more limited. From an energetic argument, it is conceivable that delamination along the interface could occur in a similar fashion to that described previously.

The compressive stress required to buckle a portion of the coating over an initial strip of delaminated interface is given by<sup>17, 26</sup>

$$\sigma_c = -\delta^2 E \cdot (t/a^2)/12 \quad (6)$$

It is expected in the considered case that stress increases as the crack begins to propagate along the interface and, consequently, a rich diversity in behavior is possible. Delamination may be an unstable process. It may be stable, in which case an increase in stress intensity factor requires an increase in the magnitude of stresses. An intermediate range of behavior is also possible in which an initial instability is followed by stable crack growth. The details of the delamination process are generally controlled by the initial size of the defect at the interface and by the value of the interfacial toughness.

It has recently been shown that when delamination occurs by this mechanism it does so under mixed-mode conditions.<sup>27</sup> These characteristics can have a substantial influence on the fracture behavior in alumina-based composites.

Prospectively, it is expected that there is a maximum defect length for which no further growth by this mechanism of combined buckling and delamination is possible. Once the defect has reached this length, further failure can occur only by plastic deformation or by cracking of the alumina-based layer in buckled region. The existence of this maximum does not depend on the precise details of the mixed-mode failure criterion; it only depends on a criterion in which the fracture resistance increases rapidly as the ratio  $K_I/K_{II}$   $\rightarrow \infty$  as it was shown.<sup>31</sup>

A biaxial stress state appears to produce an even richer behavior. An initial analysis suggests that buckling and delamination should occur by the formation of a circular blister that is found to be by Hutchinson<sup>32</sup>. In fact, failure under these conditions seems to be often associated with nonaxisymmetric and sinusoidal patterns of buckling.<sup>31,33</sup>

## Conclusion

A number of mechanisms by which alumina-based composite coatings may crack or delaminate have been reviewed in this paper. The failure of alumina-based coating is controlled by a number of parameters including the mechanical properties of the layer (porosity, defects, voids, and grain structure), interface and substrate, the initial distribution of flaws within the system, and the magnitude and sign of the residual stress in the layer. The presence of a tensile stress can cause delamination along the interface, or cracking in the layer or substrate. Failure induced by a compressive stress involves a complex interaction between buckling of the layer and delamination along the interface.

Cracks that propagate in the alumina-based composite or substrate do so under the influence

of stress intensity factor, and the fracture resistance is controlled by the mode-I fracture toughness of the appropriate material. Conversely, delamination generally occurs under mixed-mode conditions in which both shear and normal stresses play a role in the fracture process. Beyond the generalization that they increase the fracture resistance, the effect of shear stresses on the failure of an interface is not completely understood. If a mixed-mode fracture criterion is assumed, it is possible to compare different failure mechanisms and determine which ones will operate under particular conditions. It is important to appreciate that failure does not necessarily occur along the weakest path. There may be conditions in which the mechanics dictate that the driving force be greater along a trajectory with a larger fracture resistance, and this may dominate the failure process.

It is expected that fracture resistance and fatigue life of the alumina-based composite coatings can be increased by healing pores, structural defects and internal voids by smart particles. In this view upcoming work will highlight in detail the new alumina-based composites hardened by particles of chrome carbide and ultra dispersed diamonds.

## References

1. B. R. Lawn, N. P. Padture, H. Cai, and F. Guiberteau, "Making Ceramics Ductile. Science, 263, pp. 1114-1116 (1994)
2. B. R. Lawn, Fracture of Brittle Solids. (Cambridge University Press. Cambridge, U.K., 1993).
3. J. P. Tillett. Fracture of Glass by Spherical Indenters. Proc. Ph's. Soc. London. Vol. 8, 869, pp. 981-992, (1956)
4. Suhotski P. & Kireytshev Maxim. Tribomechanic properties of Al-Al<sub>2</sub>O<sub>3</sub> and Al-Al<sub>2</sub>O<sub>3</sub>-CrC composite coatings based on the anodic oxide ceramics. Proceedings of the SEM Annual Conference. June 4-6, OR, USA pp. 14-15 (2001)
5. B. R. Lawn, "Hertzian Fracture in Single Crystals with the Diamond Structure. J. Appl. Phys.. 39, (1968)

6. M. T. Laugier. "Hertzian Fracture of Sintered Alumina. J. Mater. Sciences. 19, (1984)
7. L. An, H.-C. Ha, and H. M. Chan. "High-Strength of Alumina/Calcium Hexaluminate Layer Composites" J. Am. Ceram. Soc., 81 [12] (1998).
8. H. Cai, M. Kalceff, & B. R. Lawn. Deformation and Fracture of Mica-Containing Glass Ceramics in Hertzian Contacts. Mater. Res., 9 [3] (1994)
9. S.F. Guiberteau, N. P. Padlure. and B. R. Lawn, "Effect of Grain Size on Hertzian Contact in Alumina. J. Am. Ceram. Soc., 77 [7] (1994)
10. L. An, "Fracture and Contact Damage Behavior of Some Alumina-Based Ceramic Composites" Ph.D. Dissertation. Lehigh Univ. Bethlehem, PA. (1996).
11. S. Cho. H. Moon, B. J. Hockey, and S. M. Hsu. "The Transition from Mild to Severe Wear in Alumina During Sliding." Acta Metal. 40, (1992)
12. N. P. Padture and B. R. Lawn, "Contact Fatigue of a Silicon Carbide with a Heterogeneous Grain Structure. J. Am. Ceram. Soc.. 78 [6] (1995)
13. M. T. Huber, Zur Theorie Der Berührung Fester Elastischer Körper. Ann. Phys, Leipzig, 43 [61] 1904)
14. B. R. Lawn & T. R. Wilshire, Fracture of Brittle Solids. Cambridge University, Cambridge, (1975).
15. H. Tada, P. C. Paris, and G. R. Irwin, The Stress Analysis of Cracks. Handbook (Del Research Corporation, St. Louis, 1973).
16. M. D. Thouless, H. C. Cao, and P. A. Malaga, J. Mater. Sci. 24, 1406 (1989).
17. M. Y. He and J. W. Hutchinson, J. Appl. Mech. 56, 270 (1989).
18. F. Erdogan and G. C. Sin, J. Basic Eng. 85, 519 (1963).
19. M. D. Thouless, A. G. Evans, M. F. Ashby, and J. W. Hutchinson, Acta Metall. 35, 1333 (1987).
20. Z. Suo and J. W. Hutchinson, Int. J. Fract. 43, 1 (1990).
21. M. D. Thouless, A. G. Evans, M. F. Ashby, and J. W. Hutchinson, Acta Metall. 35, 1333 (1987).
22. M. D. Thouless, Acta Metall. Mater. 39, 1135 (1990).
23. H. M. Jensen, J. W. Hutchinson, and K. S. Kim, Int. J. Solids Struct. 26, 1099 (1990).
24. M. D. Thouless, J. Am. Ceram. Soc. 73, 2144 (1990).
25. E. M. Olsson, et al. Appl. Phys. Lett. 58, 1682 (1991).
26. G. Gille, in Current Topics in Materials Science, edited by E. Kaldis (North-Holland, Amsterdam, 1985).
27. M. S. Hu and A. G. Evans, Acta Metall. 37, 917 (1989).
28. D. C. Agrawal and R. Raj, Acta Metall. 37, 1265 (1989).
29. Z. Suo and J. W. Hutchinson, Int. J. Solids Struct. 25, 1337 (1989).
30. Y. H. Chiao and D. R. Clarke, Acta Metall. 38, 251 (1990).
31. J. W. Hutchinson, M. D. Thouless, and E. G. Liniger Int. J. Solids Struct. 22, 565 (1994).
32. J. W. Hutchinson and Z. Suo in Advances in Applied Mechanics, ed. by J. W. Hutchinson (1993).
33. G. Evans and J. W. Hutchinson. Acta Metall. 37, 909 (1989).