Fracture of hard alumina-based ceramic composite coatings on a metal substrate

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A ductile substrate like aluminum with a brittle thin layer coating is one of common coating-substrate structures encountered in engineering applications. Fracture behavior of the thin layer 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 layer processing technologies. Based on our recent study of anodic alumina coatings on aluminum alloys, a new methodology has been developed to better measure the fracture property of the thin alumina coating and the interfacial strength of alumina-metal interface. The essential aspects of the methodology include (a) in-situ tensile testing of plastically deforming coated substrate under optical microscope, scanning electron microscope, acoustic emission and (b) a nonlinear finite element analysis of the coating composite system. In this talk, we will detail the theoretical and experimental basis of this new methodology and the results of its application to the anodic alumina-aluminum metal and other brittle layer-ductile systems.

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Introduction

Alumina ceramic coatings formed by micro arc oxidizing process are widely used in industry today. Important use of alumina layers on aluminum is as protective coatings. There is considerable interest in characterizing alumina layers and surfaces and in understanding the mechanical properties of alumina layers under stress.[1-3]

In the paper have been investigated the emission of different types of particles from alumina during uniaxial tensile deformation. In general, the observed AE is closely associated with the production and growth of cracks in the alumina coating although the mechanism is not at all clear. In this paper, new experimental data relating AE and cracking of the alumina layer is discussed, leading to a better understanding of this phenomenon. An important new addition to our measurements is the detection of acoustic emission (AE) from the samples during their deformation. It was revealed that the AE observed actually accompanies the propagation of a crack in the alumina layer.

The objective of the paper is to investigate fatigue of alumina layer formed on the aluminum-steel base as well as to highlight an approach to nonlinear finite element analysis of the composite coating system.

Experimental technique

The base material of the plate sample was steel. Prior to thermal flame spraying of aluminium to be oxidized, surface of base metal was prepared by grinding with water jet contained SiC, Al2O3 particles with average size of 2 mm. In result, profile of the surface was arranged in the quincunx order with roughness of 400 µm. Apply thermally sprayed bond coating to suit the application and to facilitate good chemical and mechanical bond between coating and core. Grit blast and preheat surface. Air consumption was 0.4-0.45 m^3/min. Distance of spraying is 120-140 mm. Polymer granular powder was used to coat surface with polyamide layer of 420 µm in thickness and 6 GPa in Young's modulus. Some semi empirical calculations show that aluminum layer formed by thermal flame spraying do not overheat polymer layer as temperature of Al particles do not exceed 120^0Ñ.

To form aluminum layer was used both aluminum cord with diameter of 2 mm or aluminum powder with granules size of 60-100 µm and purity of 99.85%. Aluminum thickness was about 3 mm. Butane was a heating gas of the system to provide temperature about 1200^0Ñ for melting the aluminum powder. The parameters provide optimal set of mechanical properties of aluminum layer that are porosity of 1-3%, Young's modulus of 68-72 GPa, and fine structure.

Finally, aluminum layer was transformed into hard alumina aluminum with micro arc oxidizing process. Micro arc oxidizing have been done by special equipment at current frequency of 50 Hz, voltage of 420 V and current density of 10-12 À/dm^2. The coating was formed during 60 min.

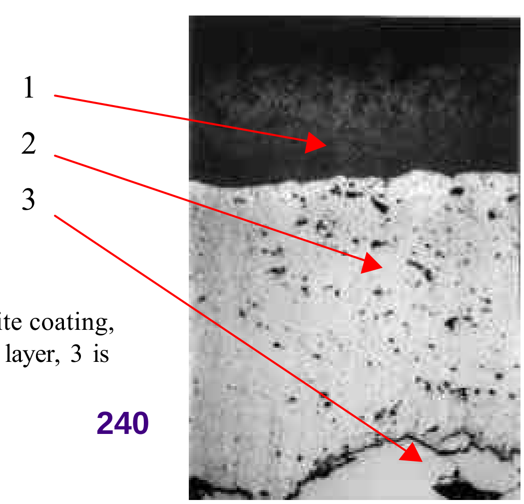

Figure 1. Microstructure of the composite coating, where 1 is alumina layer, 2 is aluminum layer, 3 is rough steel base.

An acoustic transducer was attached to the center of the specimen on the side opposite the alumina layer. The transducer was attached by a rubber band and a layer vacuum grease provided acoustic coupling to the specimen. A broadband preamplifier with 160 dB gain was used with appropriate filters to boost the signal-to-noise ratio. The signals associated with the AE during deformation were in the form of discontinuous bursts observed as ring down pulses. The frequency of ringing was 170 kHz and typically 500 ìs in duration. The ringing occurred at 2 MHz for approximately 100 ì s. In most of our experiments a pulse discriminator was set slightly above the noise level to detect the start of the acoustic signal and to produce an easily handled, single pulse in near coincidence with this event. Extreme care was taken to measure one AE pulse per burst and to insure that the discriminator output was as near as possible in coincidence with the beginning of the AE burst. The transducer estimates that the discriminator output pulse is 1.8 ± 0.5 ìs after the onset of the AE event, taking into account both the time required for the stress wave to reach the transducer crystal and amplifier rise times.

Results & Discussion

With the exception of a small peak which occurs in the elastic portion of the stress-strain curve (also observed with anodized samples), the AE bursts observed were negligible in number. The anodized samples, on the other hand, produced a total of 60 000 to 70 000 detectable AE bursts when layer is strained to under 50 N applied load. This led us to the hypothesis that the AE bursts are due to cracking of the alumina coating. Further supporting evidence was obtained by directly observing the initiation and incremental growth of cracks in the alumina in air under an optical microscope (x400) as shown in fig.3 while simultaneously listening to the demodulated AE bursts over a loudspeaker. A significant number of the two events appeared to coincide.

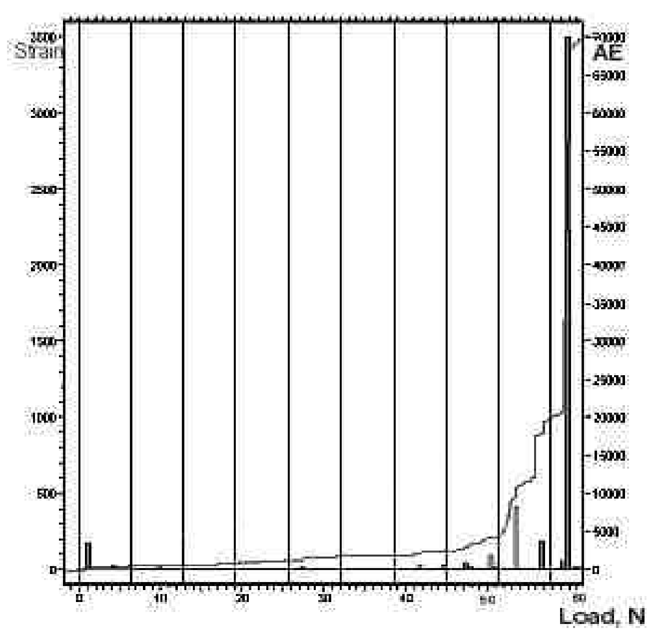

FIG. 2. Acoustic emission and strain vs. applied load.

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Optical microscopy (see in fig. 3) shows that these are the initial cracks formed in the alumina layer whereas the cracking occurring later is primarily crack extension. In the central portion of the emission curves the ratio is near unity and constant. Finally, the ratio climbs as the AE curve is seen to drop off faster than the EE curve. It is expected that this increase is due to chemi-emission.

In addition, the average length of both new cracks and the increments of crack growth were observed under the microscope to be about 0.1 mm. A measurement of total crack length in the oxidized area leads to an estimate of roughly 12 000 increments in crack growth during the experiment (10% strain). This is consistent with the total AE counts observed, again indicating that the alumina cracking is the source of the AE.

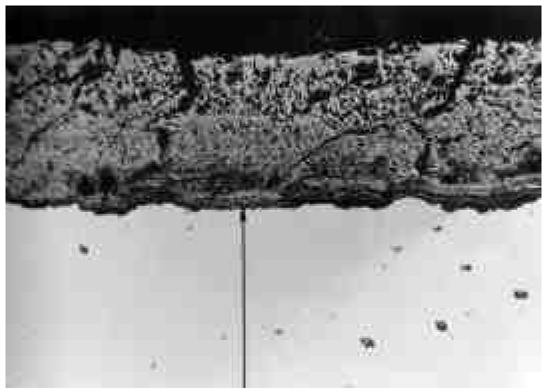

Fig. 3. Initial cracks formed in the alumina layer.

These differences are attributed to the changes in the different mechanical properties of the alumina produced while processing of sprayed Al.[3,6] For alumina coated samples, optical microscopy shows that the rate of crack growth as a function of strain, correlates very well with the AE characteristic curve. Again, this supports the hypothesis that the observed AE bursts result from alumina cracking.

The AE bursts which we detect for these samples are clearly associated with the cracking of the alumina coating and closely accompany actual propagation of the crack tip.

Within our uncertainty in the position applied load about 0, the peak of the AE occurs in coincidence. The decay from the peak exhibits a number of time constants and suggests that a number of mechanisms may be involved. Also, it has been found that the highest rate of AE occurs very near the onset of crack propagation. For brittle materials, cracks propagate at near the speed of sound,[8] implying that 0.1 mm long cracks grow in times on the order of 0.1 ìs.

The slower 5 ìs decay, which involves a substantial fraction of the emission, occurs after the crack tip has come to rest. The exponential decay indicates a mechanism which relaxes relatively slowly when compared with most electronic processes. A highly localized thermal excitation (i.e., thermionic emission) as suggested by Arnott and Ramsey[9] is not inconsistent with such a decay. Also, recent measurements on cracks propagated through glass and quartz by Weichert and Schonert[8] show that temperatures on the order of 3000-4000 K were reached. Such temperatures could produce thermionic emission from the alumina. Another mechanism which is not inconsistent with our results in the electrified fissure model[10] wherein crack propagation in the alumina produces charge separation across opposing crack faces. This charge separation produces strong electric fields which lead to field emission from the crack walls. The leakage of the charge to the substrate could account for the decay.

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Finally, figures 4 show short fragment of procedure to nonlinear finite element analysis of the composite coating by software. First right side picture shows an approximated view of the coating structure as it appears in optical microscope (fig.1).

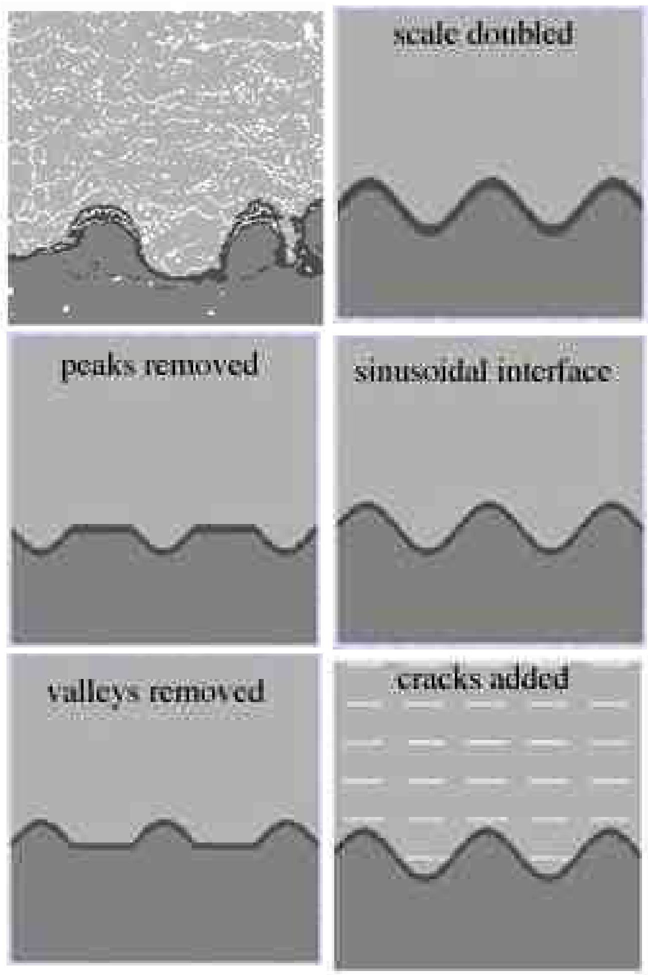

Fig. 4. Fragment of nonlinear finite element analysis

Then structure interface passed through the computing is illustrated as sinusoidal, peaks or valleys removed scale that depends on particular surface to be used. Finally, segmented picture can be shown with introduced cracked areas and types of cracks observed to be used in nonlinear finite element analysis in detail see upcoming works.[11]

Fracture of Alumina Hardened by CrC

Figure 5 shows Vickers indentations obtained on the surface of the coated and uncoated ceramic specimens. It is found that, whereas typical Palmquist cracks appear after the indentation of the uncoated specimen, there are not cracks near the corners of the Vickers indentation on the surface of the coated ceramic specimen.

On the other hand, the indentation on the surface of the specimen at the load 50 N has a rounded shape, unlike that obtained on the surface of the Al_2O_3 ceramics depended on the energy state of the

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near-surface layer of the ceramics. The appearance of the rounded shape of the indentations is evidently related to the redistribution of stresses at the surface when there is indentation due to the presence of the coating. This leads to minimization of the surface energy and formation of the indentation of the most equilibrium rounded shape. The retardation of the Palmquist cracks' initiation and propagation by the coating is determined to occur during the Vickers indentation by use of loads of up to 200 N (fig. 5).

This phenomenon can also take place as a result of the presence of residual compressive stresses in the coatings. Though coefficient of thermal expansion of the coating is not known since the coating is not single phase, the difference between the thermal expansion of the substrate and the coating can take place when taking into account the difference among coefficients of thermal expansion of Al_2O_3, chromium and chromium carbides.

There is no exfoliation of the coating after the indentation, which is evidence of its excellent adhesion to the ceramic substrate. The absence of the coating exfoliation after indentation at high loads is related to the considerable rate of the substrate-coating interaction during deposition. Microscopical studies of the indentations have revealed that the boundary line of the indentation at the surface of the uncoated.

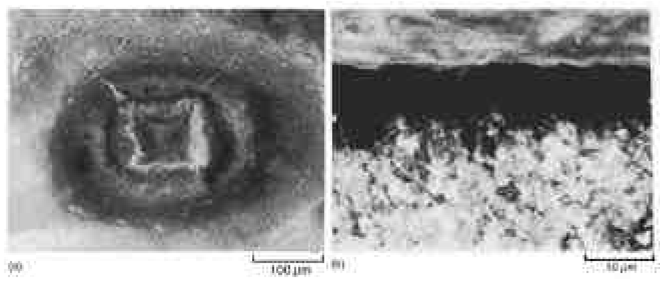

Fig. 5. Vickers indentations on the surface of specimen.

In general, ceramic specimen is characterized by numerous microfailures and microcracking in the structure. In the same time, the boundary line of the indentation track on the surface of the coated specimen views uniform and smooth. Of interest is the fact that the Vickers microhardness near the interface between alumina and CrC layer, which was found to be 20.0 GPa, is higher than that of the uncoated one (about 15.0 GPa)[11].

Numerous microcracks is found to be present across the boundary line of the Vickers indentations of the alumina specimen under relatively low applied load (2 N). Whereas various surface defects typically presented in alumina coating are healed by the CrC coating leading to retardation in the cracks' initiation and propagation under indentation. This results in improved toughness and hardness of the near-surface layer of the coated specimens.

Formation of the more uniform near-surface layer of the ceramic specimens and healing various surface defects lead to increase in transverse rupture strength (TRS) of the alumina layer. The TRS was determined to be 24 MPa for the uncoated specimens and 31 MPa for the coated ones. The difference in the TRS values determined in this study was around 15%.

To the contrary, the coated specimen comprises only a few main cracks in the near-surface layer, which propagate into the substrate by an angle close to 90° (they are visible in right Fig. 5) by the dark shaded near-surface zone). These main cracks are evidently formed at the relatively uniform and perfect surface of the coated ceramic specimen, where there are only a few surface defects initiating the crack propagation. The electrochemical studies mentioned above and the SEM investigations have revealed

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that the coating is fully dense and virtually defect-free. In the case of the uncoated specimen, the failure seems to start by "opening" up a great number of defects present at the specimen surface, which results in reduced resistance of the ceramics against initiation and propagation of these microdefects. This phenomenon can occur at a subcritical mode, when a crack grows at loads lower than a breaking load. In this case, there is not any preferential orientation of the microcracks with regard to the specimen surface since they can grow relatively easily toward various directions. Evidently, in the case of the coated ceramics, the failure occurs by the initiation and propagation of the main crack, which has the most favorable orientation with regard to the specimen surface from the viewpoint of the surface energy minimization, i.e., by an angle of 90° to the specimen surface.

Conclusion

It is expected that this new information will provide a basis for testing theoretical models of AE during and following crack propagation. The CrC coating strengths the alumina layer and enhances its microhardness.

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