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SCRATCH TESTING AND ACOUSTIC EMISSION OF NANOSTRUCTURED PARTIALLY STABILIZED ZIRCONIA COATINGS

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ABSTRACT

The crack propagation and fracture surface of nanostructured and conventional partially stabilized zirconia (PSZ) coatings were evaluated via scratch test, acoustic emission (AE), Vickers indentation at high loads, and scanning electron microscopy (SEM). Under the same experimental scratch test conditions, the nanostructured coatings exhibit lower AE activity than that of the conventional ones. This effect is attributed to the localized zones of plasticity exhibited by non-molten nanostructured particles in the coating microstructure.

KEYWORDS

Nanostructure, thermal spray, partially stabilized zirconia, scratch test, crack propagation.

INTRODUCTION

The mechanical performance of thermal spray coatings may improve with the use of nanostructured materials. This class of materials is characterized by microstructure sizes of 2 to 100 nm [1]. Different views are considered concerning the performance of nanostructured metals and ceramics. In metals, nanostructured materials increase their yield strength according to the Hall Petch relationship [2]. It is suggested that the benefits of these materials lie in the increased ductility and yield strength. This ductility has been speculated to be due to the increase in the grain boundary area and, thus, an increase in the grain boundary sliding [1]. Strain energy is absorbed and the potential for cracking is reduced because the material can deform.

Recently Lima et al. [3, 4] have observed that plasma sprayed nanostructured partially stabilized zirconia (PSZ) coatings demonstrate a bimodal distribution with respect to the coating mechanical properties. This bimodal distribution originates from the two-phase composite like structure of the coatings; i.e., the molten and non-molten nanostructured PSZ particles contained in the coating microstructure. Nanostructured PSZ feedstock particles are formed by a successive agglomeration of individual nanosized particles. These nanosized particles (~100 nm diameter) are agglomerated into clusters (1 – 5 μm diameter), which are then post agglomerated into spherical microscopic particles (10 – 160 μm diameter) [3, 4]. These spherical microscopic particles are porous and the clusters are poorly bound among each other. As a consequence, mechanical properties, such as microhardness, will exhibit values due to the weak binding of the clusters. The bimodal distribution will be given by (i) the molten particles (dense phase) and (ii) the non-molten particles (soft phase) encountered in the coating microstructure [3, 4].

The presence of non-molten particles in the coating microstructure was one of the objectives of this work. If the nanostructured feedstock particles were totally molten, then any important property that the nanostructured PSZ may demonstrate would be destroyed during plasma spraying. The present paper is a continuation of previous work [3, 4]. Therefore, it is hypothesized that the presence of non-molten nanostructured PSZ particles in the coating microstructure creates zones or sources of localized plasticity. This localized plasticity would be caused by cluster yielding and rearrangement upon the application of a load. Due to this characteristic, nanostructured coatings may present a higher resistance to crack propagation and enhanced toughness when compared to conventional coatings.

A key tool that can be employed to study the fracture of TBCs is acoustic emission (AE). The physics of acoustic emission can be characterized as a stress or pressure wave that travels through a dense medium because of dynamic processes occurring in the material [5] The dynamic process under consideration in this paper is the production of cracks initiated within the thermal spray coating produced by the scratch indenter during the scratch test. Once AE is emitted from a crack, the pressure wave propagates through the material and is then received by the transducer and recorded for post-processing and detailed analysis.

In this study, nanostructured PSZ was plasma sprayed with parameters that produced a varying degree of nanostructured phase within the coating. Scratch testing and acoustic emission were performed on the coating top surface to investigate the fracture properties of the material. Conventional PSZ was also plasma sprayed to compare the crack propagation behavior of nanostructured and conventional coatings.

EXPERIMENTAL PROCEDURE

Sample Preparation

The nanostructured PSZ feedstock (ZrO_2-7wt.%Y_2O_3) Nanox S4007 (Inframat Corp., North Haven, CT, USA) was sprayed using a Metco 3MB plasma torch (Sulzer-Metco Inc., Westbury, NY, USA) with the following configuration: GH nozzle and powder port #2. The conventional PSZ feedstock (ZrO_2-8wt. %Y_2O_3) Metco 204 NS (hot oven spherical particle – "HOSP") was sprayed using the identical plasma torch, configuration and spray parameters employed for spraying the nanostructured material. Low carbon steel substrates were used for all coatings.

Acoustic Emission

Acoustic emission was recorded during the scratch testing of conventional and nanostructured coatings. A 5 mm diameter ultrasound transducer (Physical Acoustics Corp., Princeton Junction, NJ, USA) was placed on the back of the steel substrate beneath the scratched area. The placement of the transducer was selected to increase the reception of the acoustic signal. The system was calibrated prior to scratching the sample to affirm that background noises did not contribute to the data set. A 40-dB pre-amplifier (Model 2/4/6-AST, Physical Acoustics Corp, Princeton Junction, NJ, USA) was used to process the signal prior to input by the data acquisition board in the personal computer. The system threshold was fixed at 35 dB to filter the background noise associated with nearby mechanical and electrical equipment. Data was collected using a conventional software program (Mistras 2001, Physical Acoustics Corp., Princeton Junction, NJ, USA).

Scratch Testing

Scratch testing was performed using an in-house built machine. The scratch tip was a Vickers diamond indenter. The direction of the indenter was oriented with the corner of the diamond at the leading edge. The scratch speed and distance was controlled using a personal computer and stepper motor controller card. The scratching speed was set as 0.03mm/sec. This speed was chosen to permit a significant amount of AE and force data to be gathered during a test. The length of the scratch was set to 2 mm to ensure that a large number of particles/splats would be traversed and provide sufficient data for statistical analysis of the AE. A normal load of 1000 g was applied for each sample. The total time of each test was 60 seconds. All samples were polished with 0.01 μm alumina suspension prior to scratch testing.

RESULTS AND DISCUSSION

Coating Microstructure

Two types of nanostructured coatings were chosen [4]. Each coating has a different amount of non-molten nanostructured particles embedded in its microstructure, 38% and 23% in area; respectively. It is expected that the two distinct microstructures produce different responses to the mechanical stresses generated during the scratch tests. When comparing a coating sprayed with an agglomerated nanostructured feedstock (Fig. 1a) to a coating sprayed with a conventional feedstock (Fig. 1b); the nanostructured coatings (Fig. 1a) exhibit two distinctive regions or phases. A dense region consisted of fully molten particles, equivalent to the overall conventional coating microstructure (Fig. 1b), and another region consisted of low density areas represented by non-molten agglomerated nanostructured particles. The fully molten particles retain the non-molten particles in the coating microstructure [3, 4, 6].

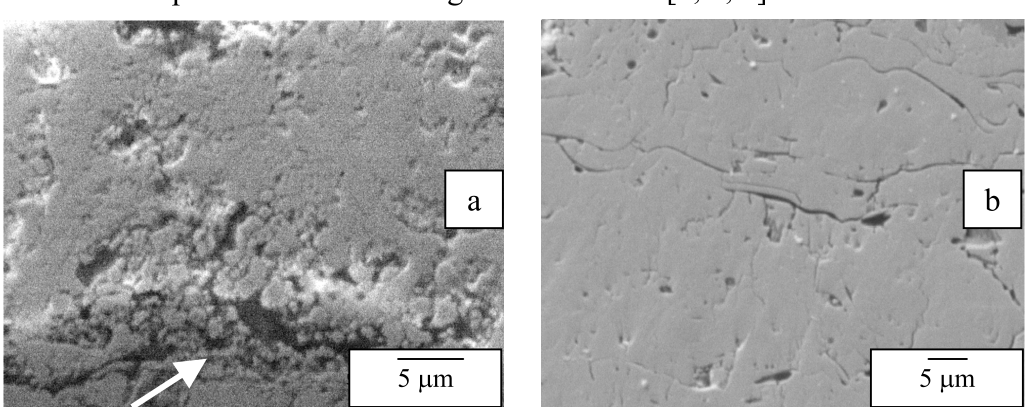

Figure 1: (a) Typical microstructure of a nanostructured coating. The white arrow indicates a region where non-molten agglomerated nanostructured particles are observed. (b) Typical microstructure of a conventional coating. The majority of the particles are fully molten.

The non-molten region of the nanostructured coatings, due to its low density zone should provide cluster mobility, rearrangement and yielding. Such mechanical behavior is the mechanism by which localized plasticity is speculated to operate, as previously discussed. The ability to deform by cluster yielding provides a mechanism through which strain energy is dissipated and crack propagation is resisted [3, 4]. Due to the reduction in the strain energy, any AE energy emitted during deformation is low and, thus, a low number of AE signals may be detected. Furthermore, a coating that exhibits a predominant dense phase (fully melted particles) in its microstructure, may result in release of energy via transgranular cracking. This might account for the greater count of AE events in the coatings than coatings where particles were fully melted.

Scratch Scars and Crack Activity

The collection of the AE emitted from the samples was used to characterize the degree of energy absorbed by the coating system during the scratch process. The AE information that is found to be characteristic of the cracking event is the maximum amplitude of the impulse related to the crack energy release and the number of events occurring during the scratch process [7]. Based on these characteristics, the maximum amplitude and number of events (cracks) for nanostructured and conventional coatings will be analyzed.

Figure 2 shows the number of AE events, representative of the number of cracks, for the nanostructured and conventional coatings. It is evident that there is a difference in the number of cracks for the nanostructured and conventional coatings. The conventional coatings demonstrate on average of ~9 times more cracks than the nanostructured ones. Explanations for this behavior are; (i) that the AE in the nanostructured coatings would be reduced due to localized plastic deformation rather than cracking, (ii) the energy associated with major cracking mechanisms in conventional coatings would be considerably high because the crack path may exhibit not only intergranular but also a transgranular crack mode, and (iii) the energy associated with fracture and removal of the nanostructure material is low due to the presence of non-molten particles in the coating microstructure.

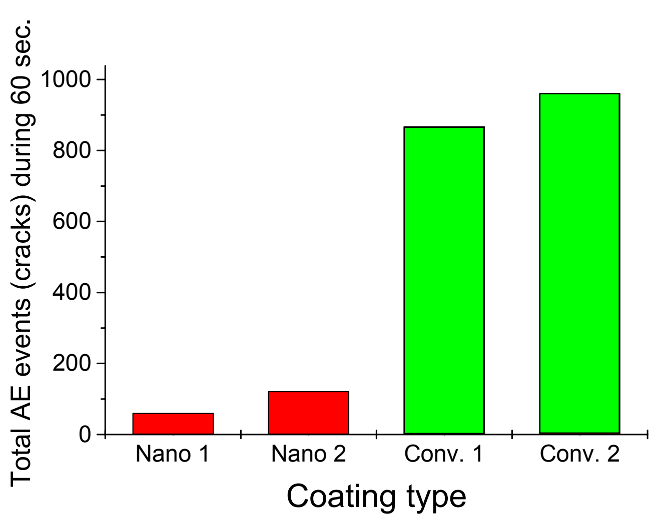

Figure 2: Total AE events (cracks) for each type of nanostructured and conventional coatings.

The SEM micrographs of the nanostructured and conventional coatings (Fig. 3) indicate that the scratch scar appears to be smooth or polished and suggests a cutting process with the diamond tip for the conventional material (Fig. 3c). These features have similar characteristics with those of Fig. 3b, which represents the sample with the lowest amount of nanostructured material. Further analysis of the conventional coating shows that there are cracks parallel to major scratch scar. Apparently the top surface of the conventional coating (Fig. 3b) was locally depressed as the indenter went through it. Since conventional coatings exhibit predominantly fully molten particles, their ability to comply to an applied stress is limited and the regions immediately surrounding the indenter stylus collapse in a catastrophic mode during the scratch. This phenomenon could explain the high AE activity observed for the conventional coatings.

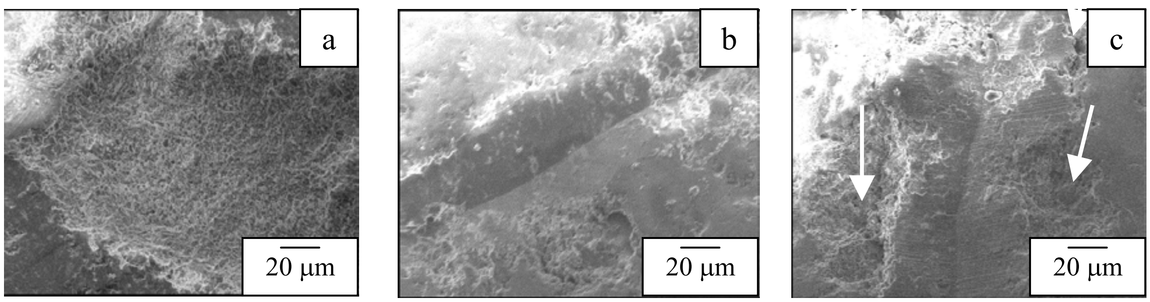

Figure 3: SEM pictures of the scar scratch made on nanostructured and conventional coatings. (a) Coating containing 38% of non-molten nanoparticles. (b) Coating containing 23% of non-molten nanoparticles. (C) Conventional coating. The white arrows represent parallel cracks following the scratch scar.

This effect was not observed for nanostructured coatings because they demonstrate localized plasticity.

Crack Propagation via Indentation

In the present work, the indentation tests (Vickers – 20 kgf load) were used to investigate further the fracture response of the materials under load. The conventional coating exhibits a higher degree of cracking, Fig. 4a. For the nanostructured coating (Fig. 4b), just one distinctive crack is observed from the corner of the indenter.

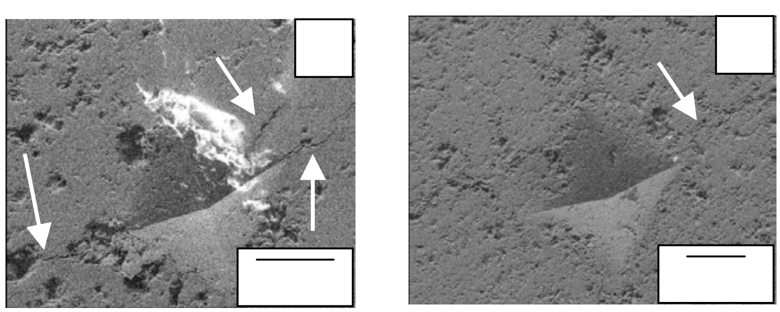

Figure 4: Vickers indentation on (a) conventional coating and (b) nanostructured coating. The white arrows are pointing towards cracks.

CONCLUSIONS

The nanostructured PSZ coatings resist crack propagation more than conventional PSZ coatings. It is thought that the non-molten agglomerated nanostructured particles embedded in the coating microstructure yield under stress, creating localized plastic deformation zones in the coating structure. The conventional coatings, due to a lack of plasticity, tend to release strain energy via crack propagation.

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