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# Splat Formation and Nanohardness Investigation of Atmospheric Plasma Sprayed Alumina

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## ABSTRACT

Atmospheric plasma spraying (APS) is a well established process for preparing ceramic coatings. In this process, powder particles injected into the plasma jet are heated and accelerated simultaneously in the plasma stream and are deposited in molten or semi-molten droplets conditions onto a prepared substrate. During droplet impingement, these molten droplets rapidly solidify to form single splats. The coating is built up by piling up the layers of splats. Among plasma-sprayed ceramic and cermet coatings, alumina ($Al_2O_3$) is the most widely established coating material because of their dielectric and wear resistance properties.

Much work has been done to characterize the complex structures which form plasma sprayed ceramic deposits. The majority of alumina related research has been carried out on deposits close to the interface with the substrate, and macro-features of the microstructure, e.g. splat shape, distribution alignment, and inter-splat interfaces. To enhance the quality and performance of APS coatings, a degree of understanding of coating structure and mechanical properties at the microscale and the nanoscale is required. However, this information is generally not available. Mechanical properties of ceramic coatings are usually difficult to measure, due to their comparatively small size in thickness and high brittleness.

Depth-sensing nanoindentation is a very useful tool for investigating mechanical properties of plasma sprayed ceramic coatings [1]. The nanoindentation results of brittle materials can be made with higher scatter and reduced reproducibility, which is caused by the stochastic indentation response. After a detailed literature search, the author found that although a lot of effects have been made to study the plasma sprayed coatings using nanoindentation technique [2,3], studies on the nanoscale mechanical properties of alumina splats are very rare in the open literature. Therefore, the object of the present investigation is to carry out a detailed investigation on as-sprayed alumina splats using nanoindentation. Two distinct forms of alumina splats, the disc shaped and the distorted splashing shaped splats were prepared on smooth silicon wafers at different spray distances from the plasma gun. Also thick APS alumina coatings layer were prepared on a grit-blasted mild steel substrate to study their nanohardness and elastic modulus.

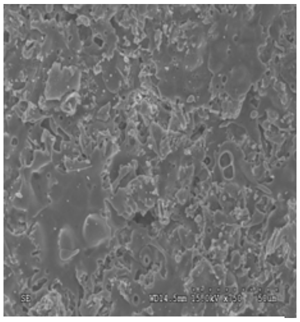

Fig.1 SEM micrograph of APS alumina coating

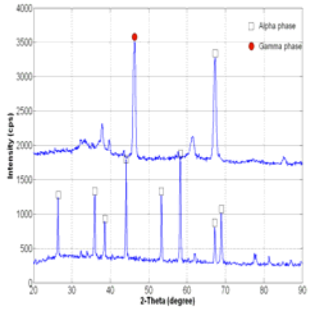

Fig.2 XRD patterns of alumina starting powders and coating

For the alumina coating (Fig.1) on mild steel sample, indentation tests were performed at different locations on the sample surface separated by a spacing of at least 50 μm. The APS alumina coating exhibits bimodal distribution of nanohardness values. The average nanohardness value of one apex is 10.8 GPa, and the second one is 15.5 GPa. As shown in Fig. 2 (top), the APS alumina coating mainly consists of α $Al_2O_3$ and γ $Al_2O_3$ phases. The starting powder in an APS process is usually α $Al_2O_3$. Previous studies [4,5] reported that the transformation of α-alumina to γ-alumina could take place only when the powder particle is completely molten. It is well-known that the alpha phase is harder than gamma phase. Thus, the bimodal character in hardness testing on the APS alumina coating is due to the presence of two different alumina phases, α and γ, in the top coating. The average Young's modulus calculated from the nanoindentation tests also has bimodal character, one is 195.6 GPa, and the other one is 253.2 GPa.

For the disc shaped splat sample, a total of three kinds of indentations were made on a single splat. Two indentations (A and B) were made in the center region of the splat and the last indentation (C) was made on the rim of the splat, shown in Fig. 3. For the splashing shaped splat sample, only one indentation test was performed on each suitable location due to the small size of the splashing alumina fingers. The residual impression of the indents was examined and imaged using the scan mode of the nano-indenter, Fig. 4.

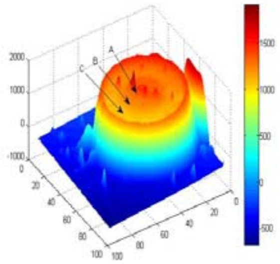

Fig.3 Surface scan of a disc shaped $Al_2O_3$ splat

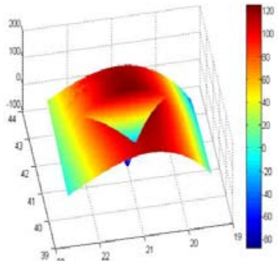

Fig.4 Residual shape after nanoindentation

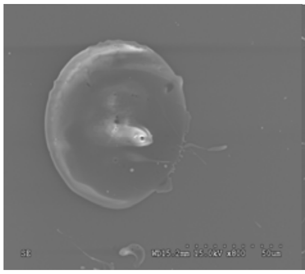

Fig. 5 SEM micrograph of a disc shaped splat

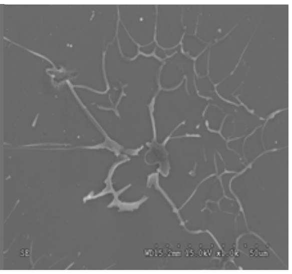

Fig.6 SEM micrograph of splashed shaped splats

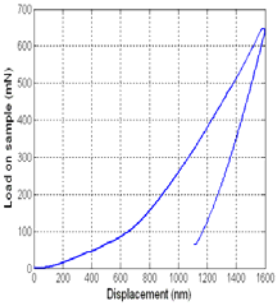

Fig.7 Typical naoindentation load-displacement curve

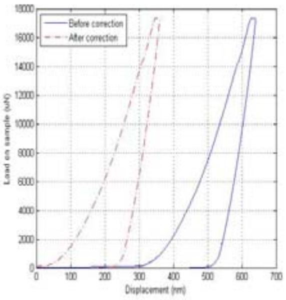

Fig.8 Load-displacement curve with zero point shift

From the nanoindentation testing, the averaged *H* at the rim of the disc shaped alumina splat is about 8.0 GPa while the averaged hardness for the locations near the splat center is about 9.4 GPa. The small difference could be caused by the different phase compositions due to the different cooling rates during the solidification stage; and it could also be caused by different geometry confinements, where the physical restriction for deformation near the rim area is much less than in the center region of the splat.

During the nanohardness test process, there are some uncertainties associated with the measurements. Although most of the force-displacement curves obtained on single disc shape splats are smooth, as shown in Fig. 7, a small number of tests show a sliding behavior, Fig. 8. The uncertainty caused by the zero point assignation is noticed on both disk shaped and splashing shaped splats. Figure 3 shows the peaks on a disk shaped splat may have a height in the order of several hundred nm. For the uneven surface, when the indenter met one of the tiny peaks on the splat, the system would generate a false "contact" signal and thus a wrong measurement in displacement.

Especially for the nanoindentation tests performed on splashing shaped splats, the zero point shifts phenomenon is more obvious. As shown in Fig. 8, after "contact", up to about 300 nm in displacement, the force reading is close to zero. The authors feel it is reasonable to shift the displacement signal to overcome this wrong "contact" reading. The corrected hardness after zero point reassignment for a splashing shaped splat is found to be around 5.5 GP.

## ACKNOWLEDGEMENTS

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