

School of Materials Engineering

Effect of Spray Angle on the Microstructure and Mechanical Properties of Suspension Plasma Sprayed Dense Vertically Cracked Yttria-Stabilized Zirconia Thermal Barrier Coatings

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Thermal barrier coatings applied to complex surfaces through suspension plasma spraying changing the angle of incidence between the plasma spray and substrate surface. The hardness and microstructural properties of coatings were characterized through Vickers hardness testing and SEM imaging and analysis. The results demonstrated a dense vertically cracked microstructure in coatings sprayed at 90, 75, and 60 degrees, with a slight increase in porosity and decrease in hardness at 60 degrees. Coatings sprayed at 45 degrees demonstrate a combination of dense vertically cracked and columnar microstructure that resulted in a ~5-7% increase in porosity and ~230-310 MPa decrease in hardness.

Project Background

Motivation: Yttria-stabilized zirconia thermal barrier coatings (TBCs) are used to protect superalloy blades within gas turbine engines from the extremely high temperatures required for a gas turbine to run efficiently. A popular application process of TBCs is suspension plasma spraying (SPS) due to the ability to spray micron and sub-micron ceramic powders. Spraying fine particles by SPS at 90-degrees to a substrate surface and a short stand-off distance can result in coatings with a controlled dense vertically cracked (DVC) microstructure. DVC coatings are desirable because of their high hardness, and ability to withstand the strains induced by dimensional changes of the superalloy during heating and cooling cycles. However, DVC YSZ coatings sprayed by SPS onto parts with complex geometries, like gas turbine engine blades shown in Fig. 1, demonstrate worse mechanical properties in areas where the angle of incidence between plasma spray and surface varies from 90 degrees, like at curved surfaces.



Fig. 1 Examples of coated (three blades on the left) and uncoated (three blades on right) gas turbine engine blades.

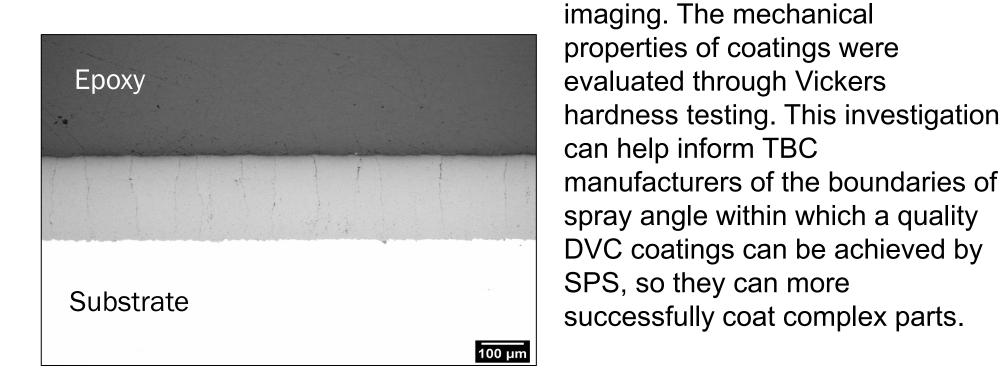


Fig. 2 Optical image of dense vertically cracked SPS YSZ coating sprayed at 90 degrees.

Results & Discussion

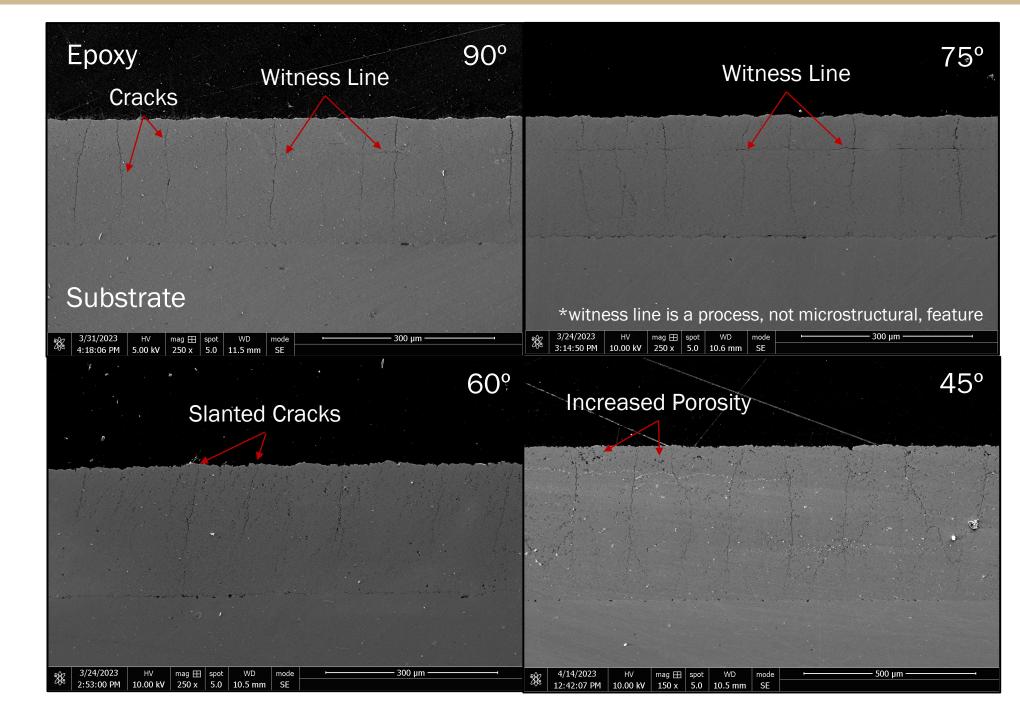


Fig. 6 SEM images of 90, 75, 60, and 45-degree spray angle coating cross-sections. The cross-sectional SEM images shown in Fig. 6 demonstrate the dense vertically cracked microstructures observed in all respective samples sprayed at 90, 75, and 60-degrees. Observationally, the 60-degree spray

angle coatings show a slight slant in the vertical cracks, decrease in crack density, and increase in porosity. The 45-degree spray angle coating also

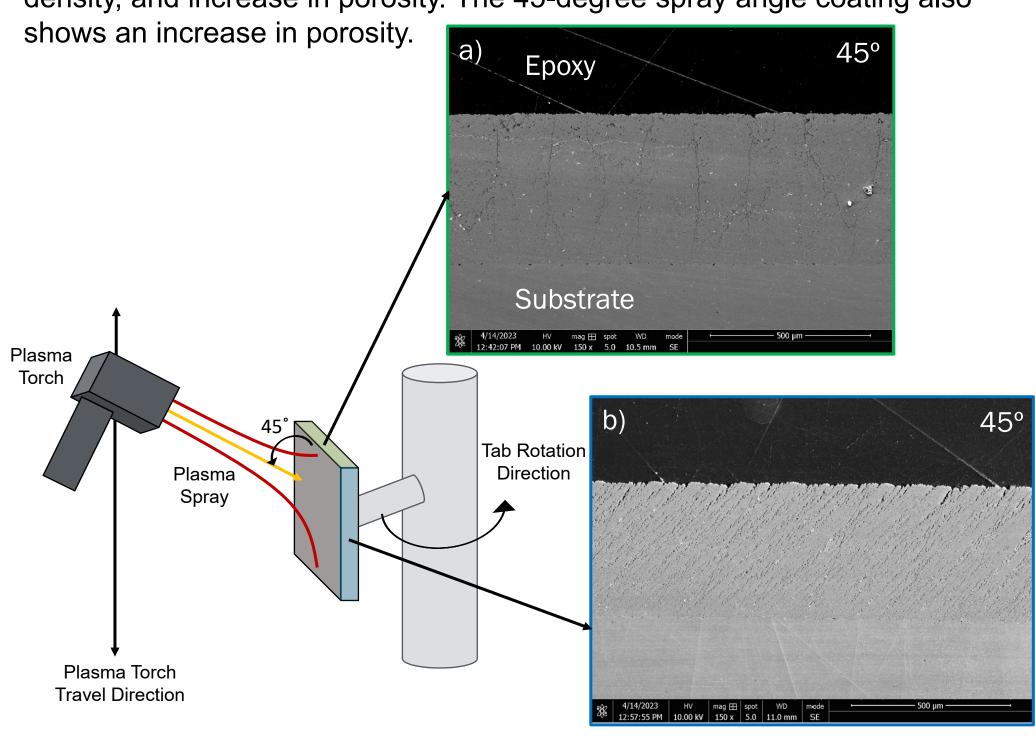


Fig. 7 Diagram of a sample sprayed at 45-degrees and the SEM images of taken of a 45degree sample mounted on a cross-section (a) perpendicular to and (b) parallel to the plasma torch travel direction.

However, while the microstructure observed for the 90, 75, and 60-degrees was consistently DVC in all samples and locations, the observed microstructure in the 45-degree spray angle samples was dependent on the cross-sectional plane imaged. Samples mounted and imaged on a cross-sectional plane perpendicular to the axis of plasma torch travel demonstrate a DVC microstructure, shown in Fig. 7(a). Samples mounted and imaged on a plane parallel to this axis reveal a highly porous microstructure with a combination of DVC and columnar features, shown in Fig. 7(b). In this microstructure both cracks and dense areas separated by porous regions are present. The cracks and porous regions are slanted towards the spray direction.

The combination of a DVC and columnar microstructure suggests the samples sprayed at 45 degrees experienced different spray deposition behavior than the other spray angles. Possibly, at a 45-degree tilt, fewer melted droplets impacted the substrate surface with enough momentum to exit the plasma plume and adhere directly to the surface. Instead, plasma drag forces cause these droplets to travel parallel to the surface, where they impacted and grew at surface asperities. This deposition behavior could result in columns of dense and porous regions, slanted in the spray direction, within a dense vertically cracked coating.

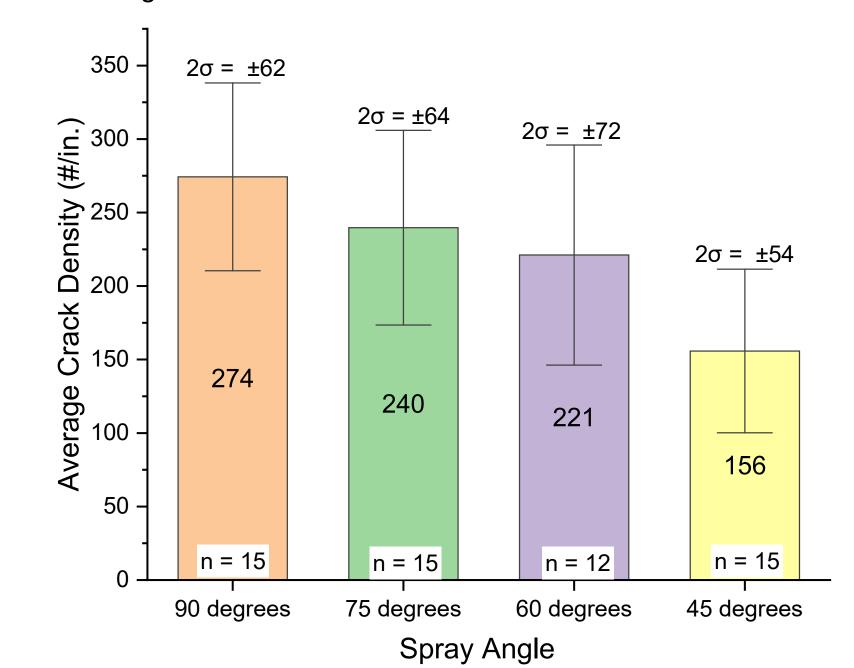


Fig. 8 Plot of average crack density versus spray angle with error bars representing two standard deviations.

Fig. 8 shows the continuous decrease in average crack density with spray angle, the largest drop being at 45-degrees. Potentially, as the angle of incidence becomes shallow enough to introduce a high amount of porosity, as seen in the 45-degree samples, the porous regions prevent the growth of cracks during coating cooling. A lower crack density may limit the strain tolerance of coatings during thermal expansion of the substrate.



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Results & Discussion

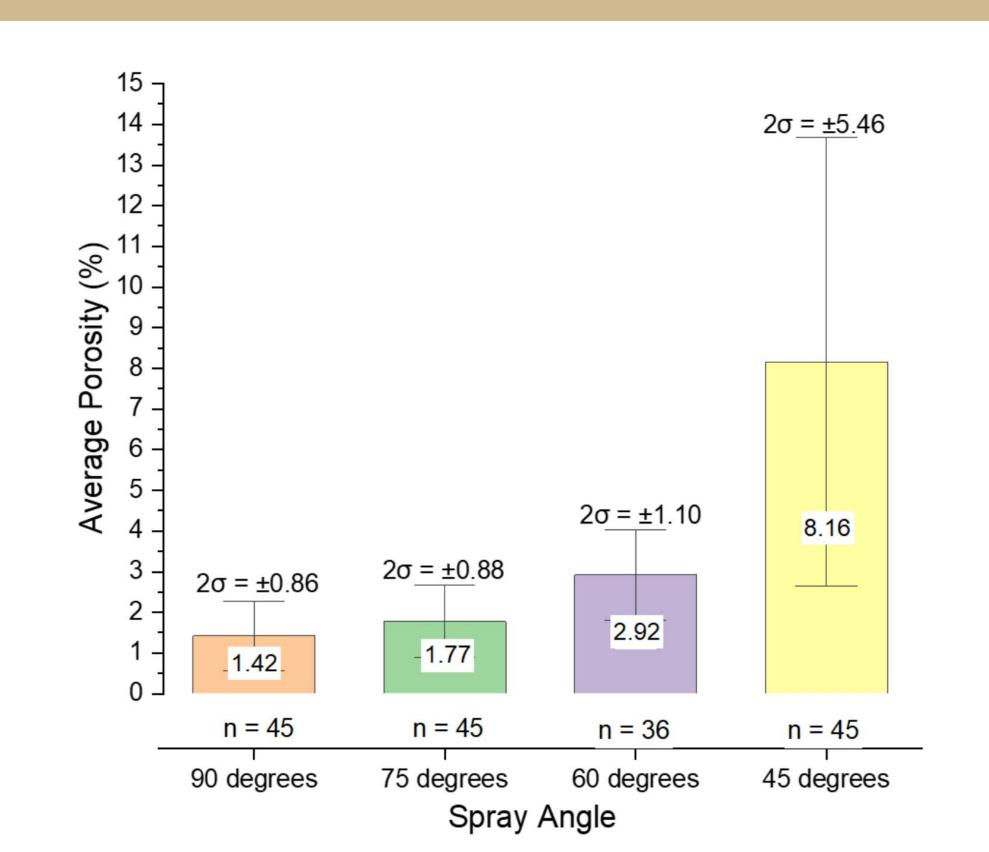


Fig. 9 Plot of average porosity versus spray angle with error bars representing two standard deviations

The average porosity measured from the cross-sectional SEM images is similar between samples sprayed at 90 and 75 degrees. The average porosity increases for the 60-degree spray angle samples by roughly 1.2 to 1.5%. The average porosity increases more for the 45-degree spray angle samples, by almost 5%, due to the development of a combination of columnar and DVC microstructures. Increases in porosity are concerning for mechanical properties such as hardness and coating adhesion strength. The porosity standard deviation for the 45-degree spray angle samples is also much larger than the others, suggesting more variation in porosity and microstructure within the 45-degree spray angle samples.

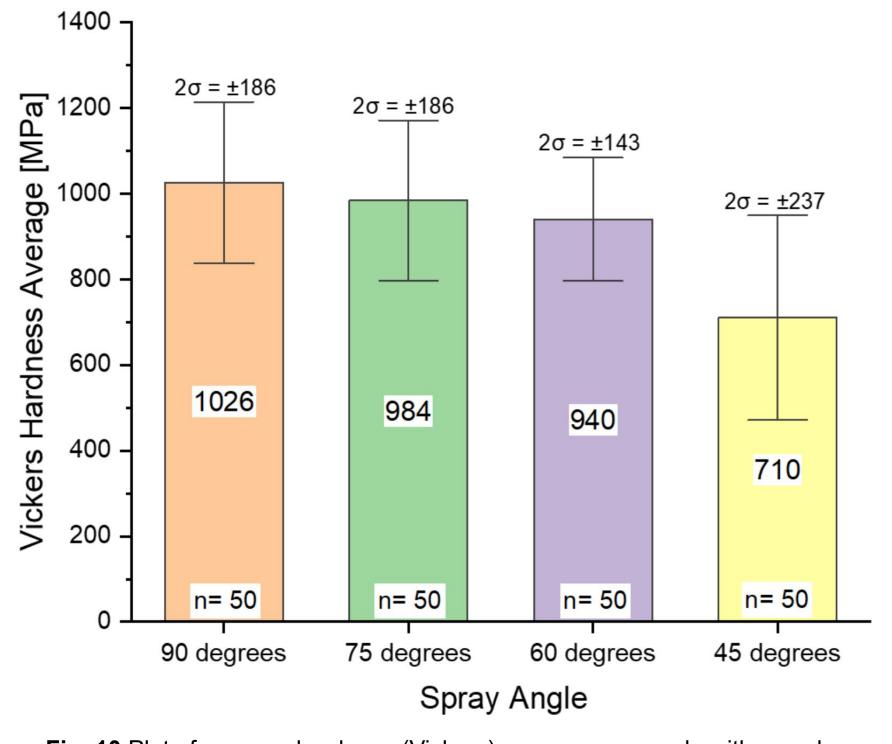
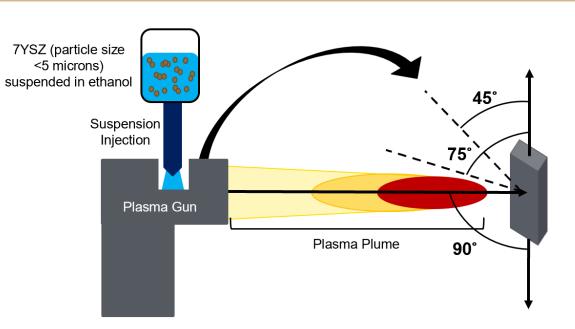


Fig. 10 Plot of average hardness (Vickers) versus spray angle with error bars representing two standard deviations

Fig. 10 demonstrates a slight decrease in average Vickers hardness from 1026 to 984 to 940 MPa exhibited by coatings sprayed at 90, 75, and 60-degree spray angles, respectively, in keeping with the porosities presented in Fig. 9. However, the 45-degree samples exhibited an average hardness of 710 MPa. This decrease in hardness is resultant of the large porosity increase in the DVC-columnar microstructure formed when the incident angle was decreased to 45 degrees. The 45-degree sample data set also demonstrated a wider range in hardness, as shown by the larger standard deviation.

Experimental Methods

Sample Preparation: Test specimens were made by Praxair with a suspension plasma spray process. Particles of 7YSZ with diameters less than 5 microns were suspended in a colloidal suspension of ethanol. The solution is fed through a tube into a plasma stream of a specified velocity and power. The plasma stream vaporizes the ethanol,



Project Goal: This project aims to

incidence between plasma spray

and substrate surface, referred to

microstructure and mechanical

properties of DVC YSZ coatings

sprayed by SPS. To do so, nickel

at the typical 90-degree angle to

degrees at incident angles of 75,

microstructures of coatings were

evaluated through optical and SEM

60, and 45-degrees. The

superalloy coupons were sprayed

the plasma spray and tilted off 90-

investigate how the angle of

as spray angle, effects the

Fig. 3 Diagram of SPS process used to coat one-inch samples at different spray angles.

melts the powder into droplets and carries them to the substrate. Vertical passes were sprayed at a consistent stand-off distance onto 1"x1" tabs of a nickel superalloy at angles of 90, 75, 60, and 45 degrees, as seen in Fig. 3, to create the coatings. Eight coupons for each angle were sprayed simultaneously while rotating as shown in Fig. 7. Five coupons from each angle were mounted on their cross-section in epoxy and polished on a LECO GPX200 up to a 0.05 colloidal silica, as shown in Fig. 4. Three samples from each angle were left unmounted for surface imaging.

Optical and SEM Imaging: Sample cross-sections and surfaces were optically imaged with an Olympus BX41M microscope at 10x and 20x. Samples were also imaged with a Quanta 650 FEG SEM at various magnifications after carbon coating with a SPI Sputter Coater.

Microstructural Analysis: Microstructural properties including coating thickness, crack length, and crack density were measured on the optical images of sample crosssections. Crack bricking length and porosity were measured on the SEM images. The porosity was calculated by

adjusting the grayscale threshold on areas between cracks, as seen in Fig. 5, and using the particle analysis function in ImageJ. The mud cracking area was measured on the optical images of the unmounted sample using the ImageJ software.

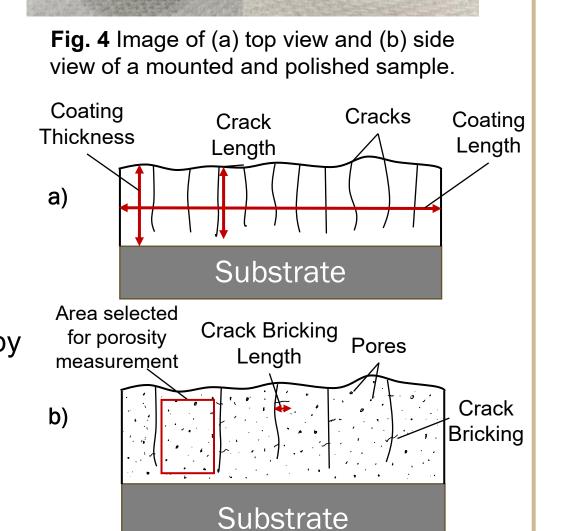


Fig. 5 Diagram of microstructural surfaces. All measurements were taken measurements taken from (a) optical images and (b) SEM images.

Vickers Hardness Testing: Vickers hardness measurements were taken on a Wilson Tukon 1202 Hardness testing with a 300-gram weight (2.942N) and a 10 second dwell time. Each sample was tested 10 times near the center of the sample. A total of 5 samples were tested for each spray angle.

Conclusion

The results of this investigation demonstrate that spray angle impacts the microstructure and hardness of SPS DVC YSZ coatings. Coatings sprayed at angles of 90, 75, and 60-degrees produced DVC microstructures across 1 square inch surfaces with acceptable crack lengths and densities. Coatings sprayed at 60 degrees showed a slight slant in vertical cracks and increase in porosity, resulting in a small decrease in average hardness. The most obvious microstructural differences were observed in the samples sprayed at 45 degrees. SEM images of cross-sectional planes parallel and perpendicular to the torch travel direction show a mix between a DVC and columnar microstructure in which slanted columns of dense and porous regions are present within dense areas with vertical cracks. The mixed microstructure likely resulted from deposition of droplets both directly upon surface impact and at surface asperities while travelling parallel to the surface still within the plasma plume. The DVC and columnar microstructure combination resulted in an increase in porosity of roughly 5 to 7% and a decrease in average hardness of roughly 230 to 310 MPa in coatings sprayed at 45 degrees.

Future investigations should be done on the range between 60 and 45degree spray angles, along with investigations into how the microstructural differences affect coating adhesion strength and thermal life cycle properties.