

Thermal Spraying of Nylon-11 and Nylon-11/Silica Coatings: Modeling and Characterization of Coating Microstructure

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High Velocity Oxy-Fuel (HVOF) thermal spraying is a powder coating technique that has been successfully used produce polymer and composite coatings based on empirical optimization of spraying parameters. In this project both mathematical models and experimental observations were used to improve understanding of the thermal spray coating formation process, including microstructure-properties-processing relationships, during thermal spray deposition of pure nylon-11 and nylon-11/silica coatings. During thermal spray deposition, jets of high temperature and high velocity gases are used to melt and accelerate materials injected into the jet and propel them towards the surface to be coated. Upon impact at the surface, multiple hot particles deform, cool and consolidate to form a coating. Fully coupled mathematical models have been developed to predict nylon-11 particle acceleration and heating in the High Velocity Oxy-Fuel (HVOF) jet and a 3-D model of particle splatting and heat transfer upon impact with a substrate. The predicted shapes of deformed particles exhibited good qualitative agreement with experimentally observed splat shapes including a characteristic “fried-egg” shape with a large nearly-hemispherical core in the center of a thin disk. This shape was formed by polymer particles having a low temperature, high viscosity core and a high temperature,

low viscosity surface. Composite feedstock powders were produced by dry ball-milling Nylon-11 together with 10 vol. % overall ceramic phase loadings with multiple scales of reinforcements. Dry ball-milling polymer and ceramic particles together resulted in a shell-core powder morphology with ceramic-rich shells around polymer-rich cores. The morphology and microstructures of the feedstock powders and sprayed coatings were characterized by optical and SEM-EDS microscopy. The microstructure of the HVOF sprayed composite coatings has a cellular lamellar structure with ceramic reinforcements agglomerated at splat boundaries. Multi-scale ceramic reinforcements reduced scratch depths by as much as 40 to 50% relative to pure polymer coatings, and by up to 20-30% compared to single-scale reinforcements.

Thermal spraying has been used for almost 100 years to deposit coatings from metal, ceramic and cermet powders that extend component life and protect infrastructure against the destructive influences of heat, corrosion and wear¹. More recently, high velocity oxy-fuel (HVOF) combustion spraying has been used to deposit ceramic-reinforced polymer composite coatings for improved scratch, wear and corrosion resistance²⁻⁴.

HVOF combustion spray process has been demonstrated as a viable approach for producing nano-scale and multi-scale reinforced semi-crystalline polymer composite coatings by controlling both the particle dwell time and substrate temperature. HVOF sprayed polymer matrix composites incorporating nominal 10 Vol. % of multiple size ceramic reinforcements (multi-scale) ranging from 7 nm to 15 μm were studied to bridge the nano and conventional micron size scale regimes⁵.

Composite feedstock powders were produced by ball milling Nylon-11 powders with silica powders of varying particle sizes (7 nm – 15 μm). Dry ball-milling polymer and ceramic particles together resulted in a core-shell powder morphology with ceramic-rich shells surrounding polymer-rich cores.

The microstructure of the HVOF sprayed composite coatings was a cellular lamellar structure with ceramic reinforcements agglomerated at splat boundaries. EDS analysis confirmed the concentration of ceramic reinforcements at splat boundaries in sprayed coatings as shown in 1 and 2. The effect of particle size on dispersion and distribution, and the influence of substrate temperature on coating adhesion, were investigated. Microstructural characterization was used to analyze the dispersion and distribution of the ceramic reinforcements within the polymer matrix. Changes in reinforcement content, as determined by TGA were correlated to nano/multi scale coating microstructures, reinforcement loadings and processing parameter variations.

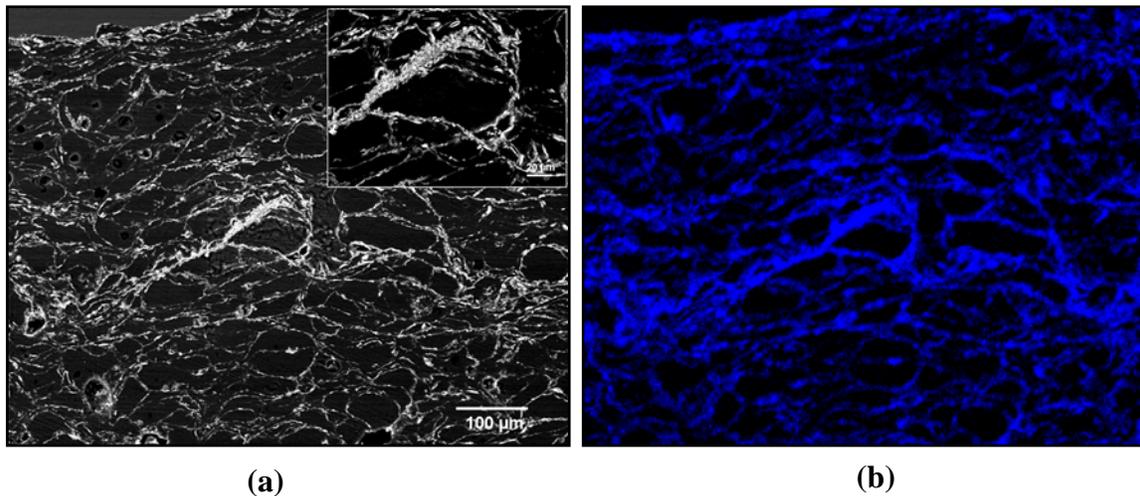


Figure 1: SEM-BSE (a) and SEM-EDS (b) image of a cross-section of an HVOF sprayed multi-scale reinforced coating showing the two phases present. The inset is a high magnification image that corresponds to Figure 2a for EDS analysis.

Thermo gravimetric analysis (TGA) was used to confirm the ~10 Vol. % loading of ceramic reinforcement within the composite feedstock powders. The amount of ceramic reinforcement incorporated within the sprayed coatings was measured by TGA to be ~5 Vol. %, which indicated a ~50% loss of reinforcement during spraying.

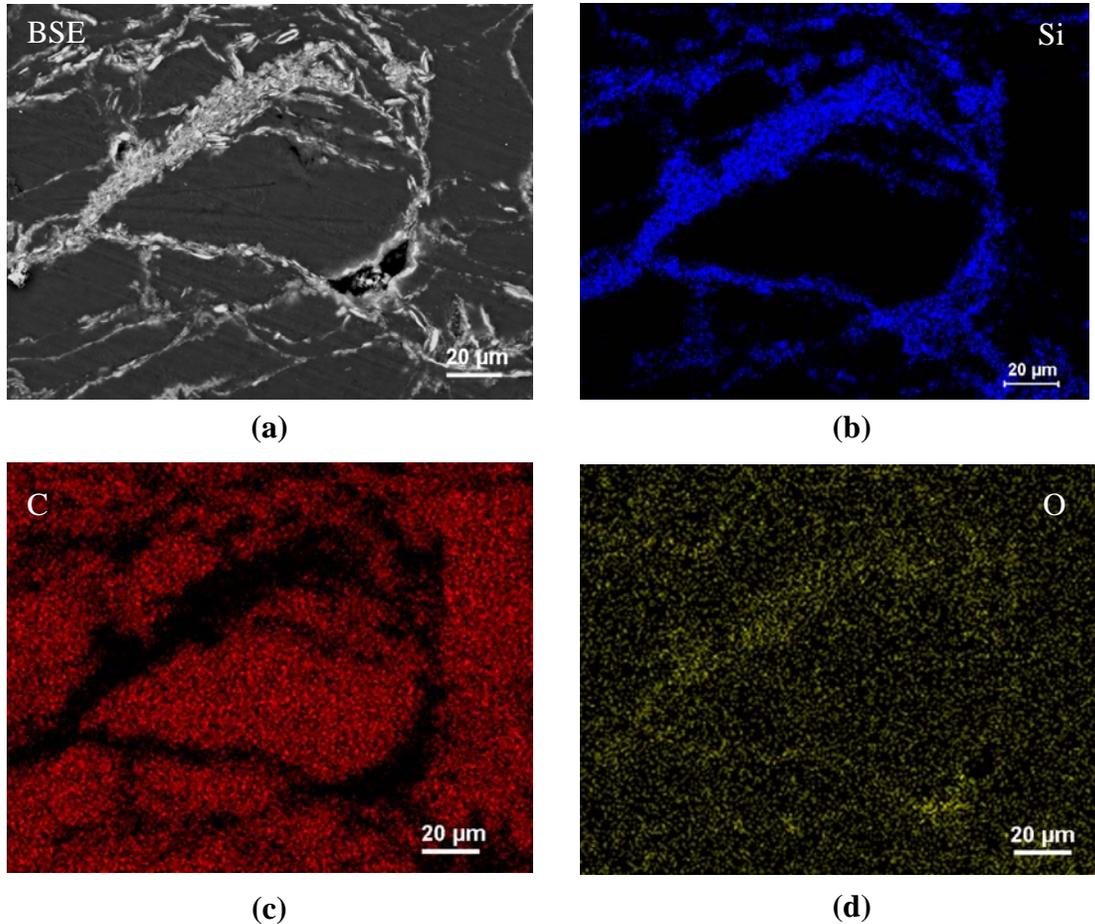


Figure 2: Chemical microanalysis of an HVOF sprayed multi-scale reinforced coating confirming the distribution of the ceramic phase within the polymer matrix by EDS dot maps (a) SEM-BSE shows the elemental contrast between nylon and silica, (b) silicon (blue), (c) carbon (red), and (d) oxygen (yellow) EDS dot maps confirming the spatial distribution of elements.

The HVOF sprayed coatings were characterized for mechanical properties such as scratch resistance. Multi-scale composite coatings exhibited improved scratch resistance

over HVOF sprayed pure Nylon-11 and single scale (nano-scale) reinforced composite coatings. Multi-scale ceramic reinforcements reduced scratch depths by as much as 50% relative to pure polymer coatings and by up to 28% compared to single-scale reinforcements, as shown in the Figure 3a and b. Improvement in the mechanical properties was likely due to mechanical reinforcement/load transfer provided by the ceramic particles due their better dispersion and distribution within the polymer matrix.

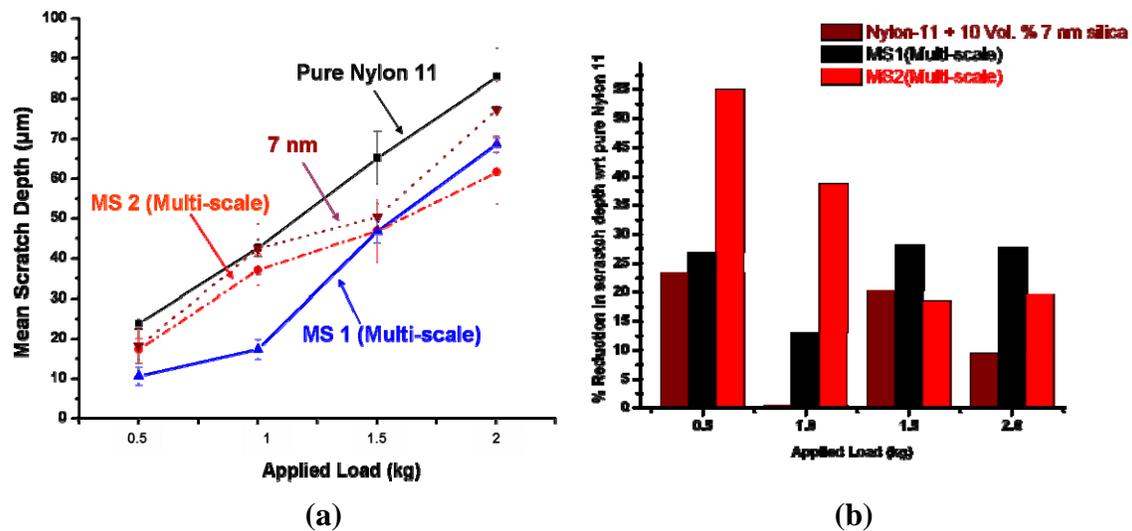


Figure 3: Scratch test performance of pure Nylon-11 and composite coatings: (a) Coating scratch depth vs. load for pure Nylon-11, 7 nm and multi-scale coatings and (b) percentage reduction in scratch depth relative to a pure Nylon-11 coating.

References

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