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Cracking in a Flame-Sprayed Epoxy

Kurtis R. Ford, Sandia National Laboratories, PO Box 5800, MS 0346, Albuquerque, New Mexico 87185-0346, USA

Mark E. Stavig, Sandia National Laboratories, PO Box 5800, MS 0958, Albuquerque, New Mexico 87185-0958, USA

Robert S. Chambers, Sandia National Laboratories, PO Box 5800, MS 0346, Albuquerque, New Mexico 87185-0346, USA

Abstract Summary

The objective of this work is to understand the cracking of aluminum flame spray on an epoxy thermoset. In the experiments presented here, epoxy cylinders are uniformly coated with flame spray. The cylinders are put into a state of tensile stress by taking them to elevated temperatures and similarly put into a state of compression by taking them down to cold temperatures. Surface cracks on the outside of the cylinders are photographed and compared. The cylinders are cross-sectioned at room temperature to study how the aluminum surface cracks propagate into the epoxy. It is shown that thicker aluminum generates observable surface cracks at a lower temperature than a thinner coating does. The surface cracks cannot be seen at room temperature. However, some of the coating cracks propagate into the substrate and can be seen at room temperature when the cylinder is cross-sectioned. The substrate cracks tend to be deeper with a larger coating thickness. Similarly, cracks are deeper when the substrate with a given thickness is taken to higher temperature. Supplementary examples that contain the addition of a hard inclusion between the aluminum and epoxy substrate at elevated temperatures are discussed as well as delamination of the aluminum film at cold temperature.

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(Extended-Abstract – not to exceed 8 pages)

INTRODUCTION

Thermal spray is a deposition process that injects molten particles into a jet of gas. The jet pushes the droplets onto a substrate where they rapidly solidify upon contact to create layers of cohesively bonded splats [1]. Thermal spray coatings are used in many applications [1] with various materials including epoxy [3],[2]. Typically, this technology is used to apply metallic coatings as protection against corrosion [4] and wear [5]. Applying metal coatings to an epoxy substrate with this technology has been less common because the high temperature gradients and abrasive blasting in preparation for the flame spray tend to damage the epoxy [6]. Nevertheless, the application of thermal sprays has been extended to flame spray coatings on epoxy parts. Coating an epoxy substrate with a layer of metal has allowed epoxy parts to be used in applications that require thermal shielding or electrical conductivity [6],[7]. Methods for reducing damage to the epoxy substrate have been reported [6], but to our knowledge, post-process damage imparted by the flame spray on the epoxy substrate during an operation environment has not been reported. Substrate damage in an aluminum coated epoxy after temperature cycling is considered in this study.

We first noticed damage imparted by flame spray on an epoxy substrate in electrical experiments involving flame-sprayed epoxy cylinders. In all of these tests, the cylinders were temperature-cycled and electrically tested. In some cases, cracking in the flame spray was noted. In one round of tests, epoxy cylinders coated in aluminum flame spray were temperature-cycled 60 times from room temperature down to -55 °C with a 5-hour dwell at the temperature extremes to saturate the cylinder. After the series of tests had finished, cracking in the flame spray was visible with the naked eye. See Figure 1. An adhesion test was done on the resulting cracks to show that the region around the cracks had delaminated from the epoxy substrate. The adhesion test involved placing a piece of tape on a non-cracked region to show that it would not peal of the epoxy. The same type of tape was then stuck to the cracks in Figure 1 to show that the flame spray peals off around the cracks.

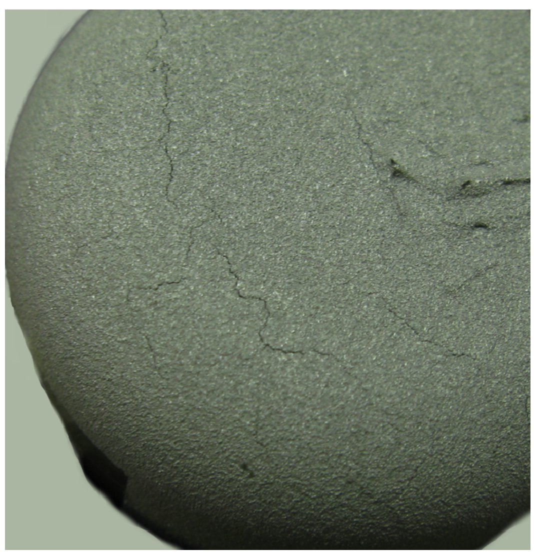

Figure 1: A photo of the cracks in the flame spray of the epoxy cylinder that was taken to -55^{o} C 60 times.

In another test, similar epoxy cylinders were cycled three times between +71 °C and -55 °C with 5 hour dwells at each temperature extreme. No cracking in the flame spray was noticed.

However, in a follow up test a copper strip was adhered to the epoxy cylinder underneath the flame spray and taken through the same temperature cycle. After the experiment, large cracks in the flame spray and epoxy were noticed in front of the copper strip. See Figure 2. The cracks in front of the copper strip could not be reproduced in tests that did not include flame spray. Experimentalists wanted to know the conditions under which flame spray cracks generated and if the cracks could propagate into the epoxy. If so, flame spray cracks could be responsible for the cracking observed in front of the copper strip.

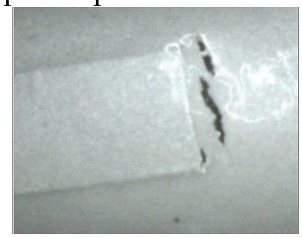

Figure 2: (a) A photo of cracks in front of the copper strip that penetrate into the epoxy substrate, and (b) a photo of the cracks in the flame spray of the epoxy cylinder that was taken to -55^{o} C 60 times.

In an effort to answer the questions above, epoxy cylinders were flame-sprayed and temperature-cycled to determine under which conditions the flame spray could crack and if flame spray cracks could propagate into the epoxy.

TEST METHOD/OVERVIEW

Epoxy cylinders were flame-sprayed and temperature-cycled to isolate conditions that could lead to cracking. Each cylinder was built according to the drawing in Figure 3. The edges of the cylinders that were covered in flame spray were given a 3.2 mm radius to prevent the stress singularity of a sharp corner. A portion of the cylinder was left without flame spray to minimize handling of the coating. The flame spray was applied to the epoxy cylinders at room temperature. describes the flame spray process that resulted in an approximately 0.1 mm coating of metal.

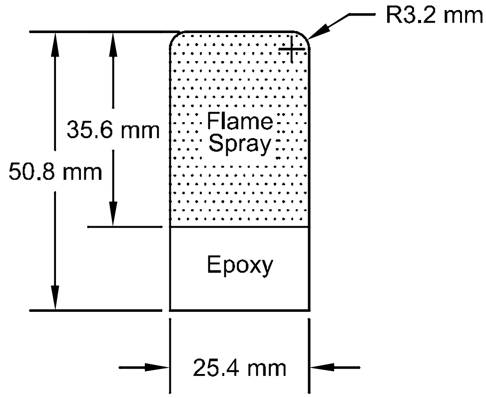

Figure 3: A drawing of the flame-sprayed epoxy cylinders used in the experiments.

The epoxy cylinders are composed of adduct of digylcidyl ether of bisphenol A (Epon 826) filled with Hycar 1300x18 carboxyl terminated butadiene acrylonitrile rubber (CTBN) and cured with diethanolamine (DEA)--for conciseness, this material is refered to as 826-CTBN (DEA). In one of the experiments the coefficient of thermal expansion of this material is lowered by replacing the CTBN with D32 GMB (826-GMB (DEA)). Both epoxies are cured by warming them to 50 °C for 20 hours, ramping them to 70 °C, and holding for 6 hours before bringing them back down to room temperature to be flame sprayed. In preparation for the flame spray process, the epoxy cylinders are sandblasted for better adhesion. Cracks due to sandblasting have been reported [6]; therefore, a single 826-CTBN (DEA) cylinder was sandblasted with the others and temperature-cycled but not flame-sprayed to allow for crack inspection.

TEST 1 -- CRACKING EPOXY AT HIGH TEMPERATURE

The first experiment was done to identify a temperature at which tensile cracking occurs in a flame-sprayed 826-CTBN (DEA) cylinder and verify that the cracking propagates into the epoxy. After the flame spray process, the cylinders were heated at a rate of approximately 2 °C/min and visually inspected through a window in the heating chamber for cracks. The first inspection occurred at the post-gelation cure temperature of 70 °C, which corresponds to the glass transition temperature of the epoxy. Additional inspections were made in 20 °C increments. Faint cracking was observed at 90 °C. At 110 °C , the cracking in the flame spray had progressed to such a point that it could be captured with a camera. The cylinder was further heated to 120 °C and soaked at for 4 hours to reach thermal equilibrium. After the high temperature soak the cylinder was cooled back down to room temperature. After the temperature cycling, cracks could not be seen with the naked eye on the surface of the flame spray (Figure 4). The cylinder was cross-sectioned and examined under an optical microscope; cohesive cracks were found in the epoxy that were approximately 1 mm long. Each of the cohesive cracks in the epoxy corresponded to a surface crack in the flame spray, but every surface crack did not correspond to cohesive cracking. No cracks were detected in the control cylinder that was sand blasted but not flame sprayed when subjected to the same temperature environments.

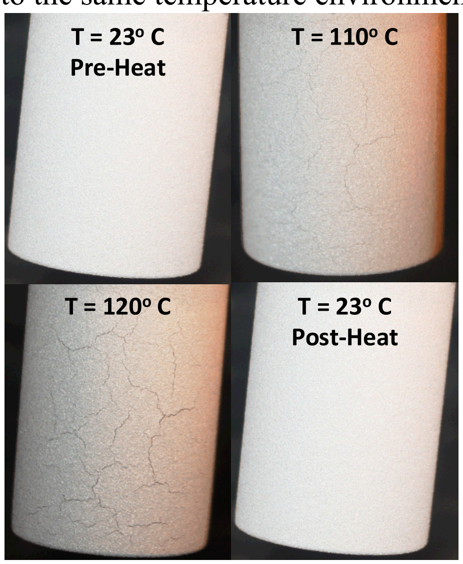

Figure 4: Photographs of the same flame-sprayed 826-CTBN (DEA) epoxy cylinder at different temperatures to show how cracking varied.

The above process was repeated with an epoxy filled with GMB rather than CTBN. The GMB filler lowered the coefficient of expansion and changed the macroscopic material properties [8]. At 120 °C cracking occurred in the 826-CTBN (DEA) as shown previously, but no cracking was observed in the 826-GMB (DEA) sample. The implication is that the strain created by the 826-GMB (DEA) was not sufficient to impart the stresses needed to reach the critical energy release rate for the flame spray coating.

TEST 2 -- FURTHER CHARACTERIZATIONS OF EPOXY CRACKS AT HIGH TEMPERATURES

The 826-CTBN (DEA) experiments were repeated with three cylinders each having a different thickness of flame spray: half nominal, nominal, and twice nominal (~0.05 mm, ~0.1 mm, ~0.2 mm, respectively). The different thicknesses were created with the same process as before except the lateral speed of the flame spray gun was adjusted to achieve the desired thicknesses. Two sets of temperature cycles were performed. First, a cylinder with each of the three thicknesses was heated at 2 °C/min until 100 °C, and then they were cross-sectioned and examined at room temperature with an optical microscope. A second set of three specimens identical to the former were taken to 155 °C and likewise inspected. In both sets, cracking was observed in the nominal and twice nominal cylinders but not the half-nominal or uncoated control cylinders.

At temperature, the cracks in the flame spray appeared largest in the cylinders with the thickest films. The cylinders taken to 155 °C had the largest cracks at temperature. Cracks in the specimens taken to 100 °C could only be seen at temperature through the oven window in the twice nominal case.

Each of the cylinders described above were cross-sectioned and illuminated to show cracks that would otherwise be invisible to the naked eye at room temperature. The illuminated cross-sections for the 100 °C samples in Figure 5 and the 155 °C samples in Figure 6 look similar. The thick samples have less cracks in the flame spray, although the cracks open wider when the cylinders are taken to temperature. The crack pattern observed in the thicker specimens is less connected than that of the nominal case. The cylinder with the nominal coating taken to 155 °C has more channel cracks than the equivalent specimen that was only cycled to 100 °C . The twice nominal coating has nearly the same number of flame spray cracks at 100 °C as it does at 155 °C. The half nominal cylinder has a low enough splat density of the flame spray that the light shines through.

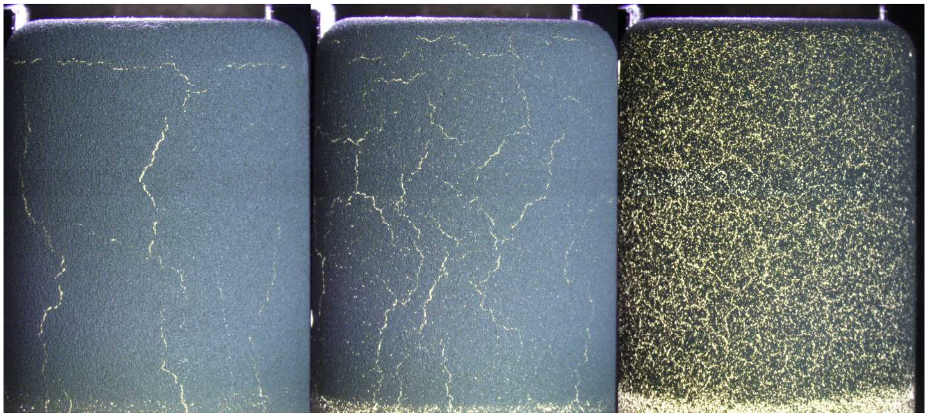

Figure 5: Optical image of the cracks in the twice nominal (left), nominal (middle), and half nominal (right) with cracks at room temperature that resulted from heating to 100 °C . The cracks are seen by shining a light through the epoxy substrate; they are not visible at room temperature otherwise.

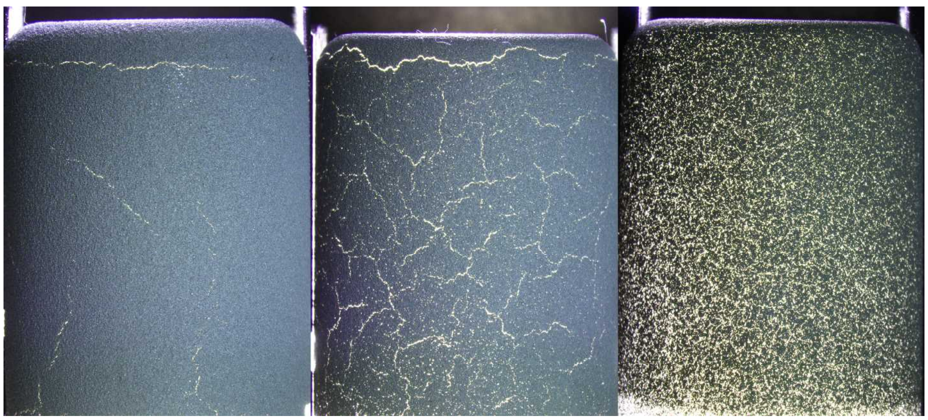

Figure 6: Optical image of the cracks in the twice nominal (left), nominal (middle), and half nominal (right) with cracks at room temperature that resulted from heating to 155 °C . The cracks are seen by shining a light through the epoxy substrate; they are not visible at room temperature otherwise.

The epoxy side of the cross-sectioned cylinders was examined for cohesive substrate cracks with an optical microscope. The entire perimeter of each cylinder was inspected and charted in Figure 7 and Figure 8. The cylinders that were taken to 100 °C had only 1 cohesive crack in the nominal case and 3 cracks in the twice nominal case. In contrast, the cylinders that were taken to 155 °C had 8 substrate cracks on the twice nominal case and 5 cracks on the nominal case. There were not cracks on the half nominal in any set. Although the cracks in Figure 7 and Figure 8 are not drawn exactly to scale, their relative size and direction are representations that can be used for comparison. The longest observed crack (Figure 9) occurred in the twice nominal case taken to 155 °C .

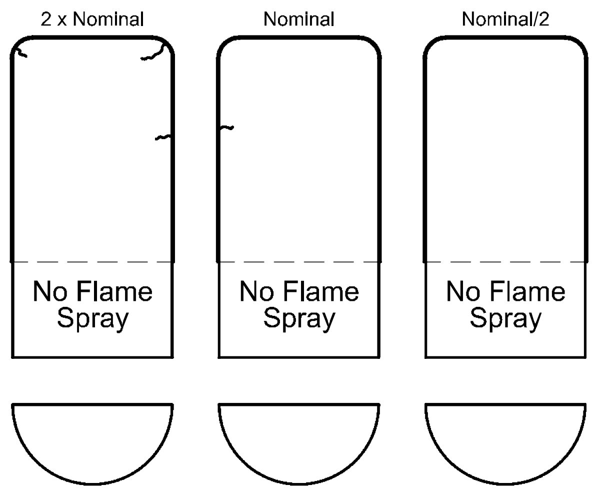

Figure 7: A chart showing the locations of epoxy cracks as observed with a microscope on the cross-sectioned surface after being heated to 100 °C .

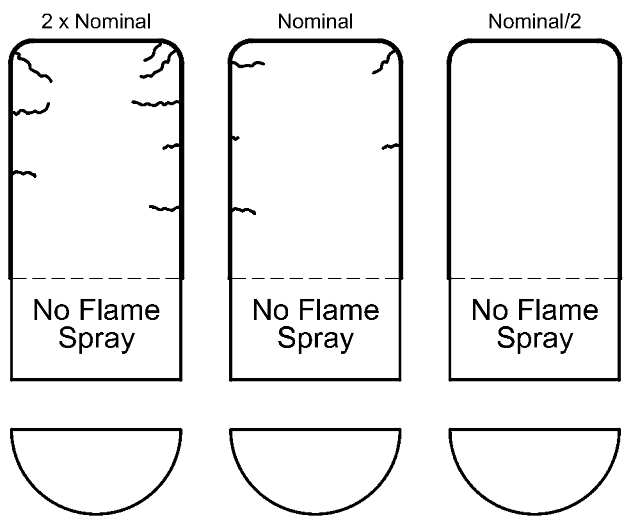

Figure 8: A chart showing the locations of epoxy cracks as observed with a microscope on the cross-sectioned surface after being heated to 155 °C .

Figure 9: Optical microscope image of a cohesive crack near the top right fillet of thetwice nominal cylinder taken to 155° C.

The cohesive cracks in the twice nominal case tend to be longer in each set. The extent of substrate damage is greater when the cylinders are taken to higher temperatures. In the nominal cases, many flame spray channel cracks exist without substrate damage. It is possible that cracks begin in the flame spray and propagate into the substrate. This type of mechanism would explain the crack in front of the copper strip that could not be reproduced without flame spray.

TEST 3 – THIN FILM BEHAVIOR AT LOW TEMPERATURE

A set of three 826-CTBN (DEA) cylinders half nominal, nominal, and twice nominal flame spray thickness were next cooled to -80 °C at 2 °C /min to create delamination similar to those observed in the electrical experiments. Room-temperature cycles to cold result in a compressive state that could cause buckled delamination in the thin film [9]; however, that was not observed in this round of experiments. After being cooled to -80 °C and soaked for 5 hours no cracks of any kind were observed through the window of the temperature chamber. After warming back to room temperature and inspecting with the naked no flame spray surface cracks due to delamination were found. It may be that the delamination observed in the electrical experiment initiated at an existing channel crack [10],[11]. Future work will attempt to delaminate the flame spray after channel cracks have been introduced.

CONCLUSIONS

Flame spray coated epoxy cylinders tend to crack at elevated temperature. Channel cracking in the flame spray with and without substrate damage have all been observed. Experiments have shown that substrate damage in 826-CTBN (DEA) exists at 100 °C and is exacerbated as the temperatures increase to 155 °C. Furthermore, the severity of the substrate damage increases with thicker films of aluminum. No cracking was observed in sandblasted 826-CTBN (DEA) cylinders without a thin film of metal, even with temperature cycling. Likewise, no cracks were observed in 826-GMB (DEA) coated in flame spray and heated to 120 °C. Channel cracking that occurs at high temperatures with a 0.1 mm film cannot be seen at room temperature without illuminating the epoxy substrate behind the film. It has been shown that cracks in an aluminum flame spray coating have the potential to damage an epoxy substrate.

ACKNOWLEDGEMENTS

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