

Polymer-Filled Metal Foams for Contamination Resistant Aircraft Leading Edges

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Introduction

Adhesion of contaminants has been identified as a ubiquitous issue for aeronautic exterior surfaces.¹⁻⁴ Organic coatings have been demonstrated to enable reduction in insect residue adhesion as well as a reduction in ice adhesion strength.⁵⁻⁷ These approaches however, have yet to demonstrate sufficient durability to enable application to commercial aircraft wing leading edges. High durability materials have been developed in parallel to these efforts;⁸ contaminant adhesion issues are not addressed. This work describes a first attempt to identify a solution to the seemingly competing issues of contaminant adhesion and durability.

A co-continuous network of organic and metallic domains may provide greater durability, while still enabling the changes in surface-contaminant interactions that have been realized. To that end, unique properties have been achieved through fabrication of composite metal foam materials.⁹⁻¹¹ Likewise, extensive research has been dedicated toward development of polymeric coatings for mitigation of insect residue adhesion.¹² This work describes initial results from infusion of a low surface free energy epoxy resin into a composite metal foam substrate for the purpose of generating a durable contaminant adhesion-resistant material.

Experimental

The composite metal foam utilized in this work consisted of a combination of hollow steel spheres (approximately 1 mm diameter) and steel powder (325 mesh size, approximately 44 μm diameter). These materials were sintered in a vacuum press under mild conditions to minimize consolidation and prevent collapse of hollow spheres. The generated composite metal foam was determined to have a density of approximately 2.7 g/cm³ (Figure 1).

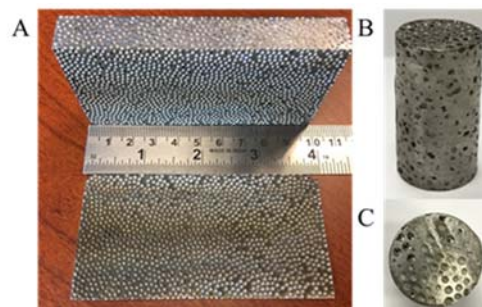


Figure 1. Composite metal foam rectangular prism (A) and cylinder (B and C) samples. The length and diameter of the cylinder shown is 2.54 cm and 1.8 cm, respectively.

A series of epoxy resins were generated to determine which formulation exhibited the most favorable properties prior to infusion into the metal foam specimens. The epoxy matrix consisted of DER 331 (Dow, epoxy equivalent weight, EW_e , of 187.5 g/mol) and Ethacure 100 (Albemarle, amine equivalent weight, MW_n , of 44.6 g/mol). This formulation was modified with aminopropyl-terminated polydimethylsiloxane oligomers (Gelest, DMS series, MW_n from 62.3 to 8950 g/mol), which imparted low surface energy properties to the resin.

Resins were generated for each DMS oligomer with loading levels ranging from 1-15 wt%. The resin components were combined using a Thinky® mixer (ARE 310) and transferred to a silicone mold, lightly coated with silicone grease. This was placed in a vacuum oven at 70 °C and 1 torr to remove any additional trapped gas. The sample was cured at 100 °C for 2 hrs and 175 °C for 4 hrs under ambient pressure. Once cured, the epoxy specimens were polished using a Buehler lapping instrument to a mirror finish and evaluated using water contact angle goniometry, pin-on-disc tribometry, and insect impact studies. Details of this work will be provided in a future publication; however, the target properties were an increase in water contact angle values and a decrease in wear rate and retained insect residues. Based on these results, it was determined that the resin containing the longest DMS oligomer (DMS-A32) at the highest loading level (15 wt%) exhibited the most promising properties. Therefore, this formulation was selected for composite metal foam infusion and further analysis.

Composite metal foam specimens were cut to dimension using wire electric discharge machining (EDM) prior

to being infused using a vacuum-assisted thermal infusion process (Figure 2). This was very similar to the cure conditions for the epoxy specimens with the composite metal foam submerged in the epoxy resin. Under vacuum conditions (2-5 torr), the entrapped air was removed from the metal foam. The vacuum was then vented to drive resin into the empty pores. The cylindrical samples were machined to the final working diameter (1.5 cm) on a lathe.

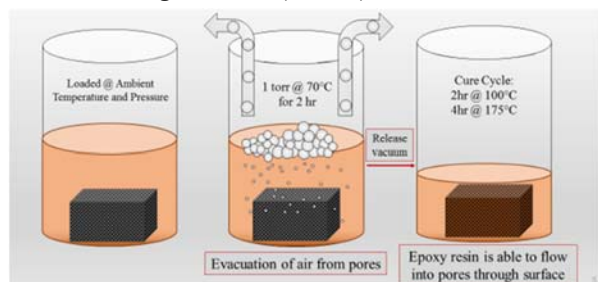


Figure 2. Thermal vacuum-assisted resin infusion into a composite metal foam specimen.

Two specimen geometries were fabricated, rectangular and cylindrical specimens. The resin-infused composite metal foam (RiCoM) rectangular specimens were characterized using the same methods as the epoxy resins described previously. Additionally, durability was assessed using a pen-delivered grit-blasting instrument (Hunter, microjet 200) followed by repeated assessment of water contact angle goniometry and insect impact studies.¹² The cylindrical specimen was prepared for determination of impact ice adhesion using a custom-built ice adhesion rotary test stand.¹³

Results and Discussion

For brevity, only a portion of the results from this work is presented here. The remaining details will be described in a future publication. Analysis of the entire series of epoxy coupons indicated that resins containing the largest aminopropyl-terminated siloxane at the highest loading level (15 wt%) resulted in the greatest increase in water contact angle value, lowest level of insect residue contamination, and lowest wear rate. Therefore, this resin was selected for infusion into composite metal foam samples.

The degree of infusion was determined using cross-sectional microscopy. Images were collected on the polished surfaces of a RiCoM-rectangular sample and contributions from the resin, metal, and void spaces were determined using threshold analysis in ImageJ (public domain, image processing software, Figure 3). Based on this analysis, it was determined that approximately 40% of the image area consisted of metal, 53% of the area was epoxy, and 7% was comprised of unfilled space. This resulted in a degree of infusion of ~88% at 70 °C and 1 torr.

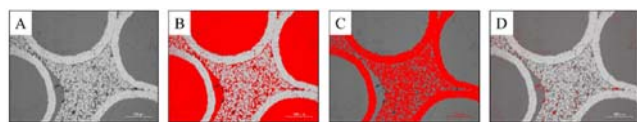


Figure 3. Threshold analysis of an (A) epoxy-infused composite metal foam micrograph indicating relative amounts of (B) resin, (C) metal, and (D) unfilled void space.

Durability of these materials was assessed by grit blasting the polished RiCoM-rectangle surface at 100 psi and held approximately 2.5 cm from the surface for 120 s. Grit blasting was performed through a small (1.6 mm) window and a wear rate was determined by measuring the loss of material determined by step height measurements performed using a contact profilometer (Table 1). These results were compared to other surfaces of relevance including the epoxy resin (A32 15 wt%), a stainless steel surface (304), and an aluminum surface (6061-T6). As can be seen, the wear rate for the RiCoM specimen was significantly lower than the epoxy-only specimen indicating that this would be likely to exhibit a greater operational lifetime than an epoxy coating alone.

Table 1. Grit blasting wear rate determination.

Material	Depth (μm)	Wear Rate ($\mu\text{m/s}$)
A32 15 wt%	325	2.7
RiCoM-rectangle	104	0.9
Stainless steel	27	0.2
Aluminum	67	0.6

The change in surface properties before and after grit blasting was also important to consider. A change in water contact angle could indicate a difference in response of the surface with respect to contaminant adhesion. Therefore, water contact angle values were measured on each of these surfaces before and after grit blasting (Figure 4). The neat epoxy was not considered for this analysis as the grit material was observed to partially embed in the surface. As can be seen, the reduction in water contact angle value for the RiCoM-rectangle specimen was negligible, while stainless steel exhibited a dramatic reduction.

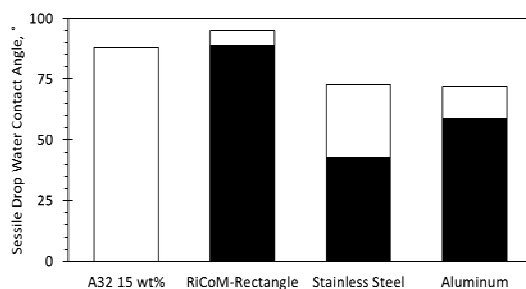


Figure 4. Sessile drop water contact angle values before (white) and after (black) sand blasting.

Similar to the contact angle measurements, insect impact experiments were performed before and after grit blasting to ascertain how erosion changed the response of each surface to contaminant exposure (Figure 5). For all surfaces, grit-blasting resulted in a reduction in insect residue areal coverage. This can be related to the increased surface roughness on the grit-blasted portion of the surface which has been correlated to reduced stability of expanding residues leading to reduced retention of residues.¹⁴

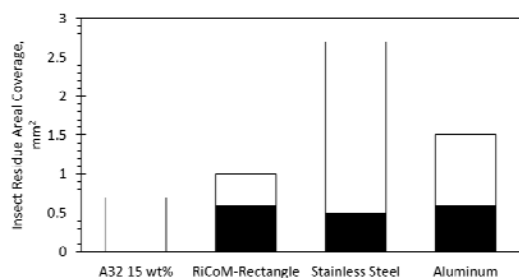


Figure 5. Insect residue areal coverage values before (white) and after (black) sand blasting.

The RiCoM-cylinder performance regarding accreted ice adhesion under flight conditions (impact ice) was also assessed. A roughened specimen (control surface in Figure 6A) and a test specimen were mounted on a refrigerated centrifuge rotor and spun at approximately 5500 rpm while exposed to super-cooled water droplets ($\sim 20 \mu\text{m}$ diameter). Ice was accreted on both specimens with the test ending when the ice was shed from the test specimen.

Each sample was evaluated at -12°C and performed in triplicate. An ice adhesion shear strength (IASS) was calculated based on the mass differential between the control and test specimens and the ice detachment area from the test specimen (**Conclusion**

Initial results indicate that resin-infused, composite metal foam materials may provide greater durability and an extended operational lifetime, relative to an organic coating, for leading edge applications. Further work to improve resin adhesion and ascertain an operational wear rate will advance this technology.

Table 2). Although the standard deviation is significant for these tests, the values determined for the stainless steel and epoxy-coated aluminum samples were determined to be statistically significant, student t-test p-value of 0.06%. Thus, the epoxy resin itself provides a nominal decrease in IASS. However, the RiCoM-cylinder sample exhibited a higher IASS value, relative to the stainless steel specimen. This was likely partly due to an unanticipated increase in macroscopic surface roughness imparted on the cylinder as a result of the machining process. Epoxy resin appeared to be removed from filled macropores as the specimen was machined (Figure 6C).

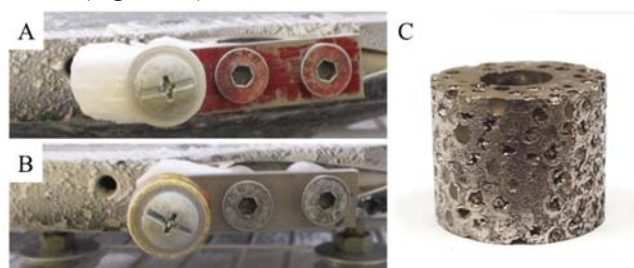


Figure 6. Accreted ice on a control (sanded stainless steel) surface (A). The ice shed from the epoxy-coated stainless-steel surface (B). Surface of the machined RiCoM-cylinder sample used for ice adhesion testing (C).

Conclusion

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Table 2. Ice adhesion shear strength values, -12°C testing.

Surface	IASS, kPa
Stainless steel	460±50
Epoxy-coated aluminum	390±100
RiCoM-cylinder	590±50

Acknowledgements

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