Evaluation of Various Adhesive Options for Tensile Attachment Strength Testing per ASTM F1147

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INTRODUCTION: Porous and nonporous coatings and structures applied to the fixation surfaces of devices undergo tensile and shear stresses in vivo. Coatings can be adhered to a substrate through a variety of methods including sintering, mechanical fixation, diffusion bonding, etc., or porous structures can be printed via additive manufacturing. It is important to ensure that these porous structures have adequate mechanical strength within the porous structure as well as between the porous structure and the substrate. Tensile attachment strength (TAS), shear strength, and shear fatigue are three types of tests performed on porous structures to evaluate mechanical integrity. Typically, these tests are performed on cylindrical coupons representative of the device material on which the porous structure has been applied. The primary adhesive mentioned in ASTM F1147-05R17E01 which has been used to perform these tests historically, FM 1000 polyamide-epoxy unsupported adhesive film, has been discontinued recently. The purpose of this study was to evaluate three alternative adhesives as a replacement for FM 1000.

METHODS: Three different epoxy adhesives were evaluated: Loctite EA 9658 AERO, FM 309-1u, and 3M Scotch-Weld Epoxy 2214 NMF. The 2214 adhesive is mentioned as an alternative in the appendix of ASTM F1147. Adhesive-only strength was measured to ensure the effectiveness of the adhesives and cure parameters. TAS testing was performed to evaluate adhesive performance for a porous plasma sprayed titanium (PPS-Ti) coating provided by an outside supplier, specifically for testing alternate adhesives. Each coated coupon was adhered between a pair of tensile stubs to create a test assembly. Adhesive cure parameters are in Table 1. After all assemblies were cured, each was tested in a manner consistent with ASTM F1147. The peak load was divided by the nominal cross-sectional area of the coupon to calculate the TAS of the coating for each coupon tested. Coupons were also examined for penetration of the adhesive to the substrate.

RESULTS: A plot of tensile attachment strengths for adhesive-only samples and coated coupons is in Figure 1 with the means ± standard deviations labeled. The adhesive did not appear to penetrate to the substrate for any of the cure parameters or adhesives used (Figure 2). The primary failure for coupons tested with EA 9658 occurred between the coating and the substrate. Coupons tested with FM 309 failed primarily within the coating. Coupons tested with 2214 primarily failed between the coating and the substrate; however, two coupons failed on the uncoated surface (backside) of the coupon.

DISCUSSION: All adhesive-only samples exceeded the minimum tensile strength of 34.5 MPa required by ASTM F1147. All coated coupons had valid results as defined in ASTM F1147 with no signs of adhesive penetration to the substrates. All adhesives also appeared to penetrate sufficiently into the coating to ensure adequate grip on the coating during testing. Comparisons were limited in sample size; however, average TAS of coated coupons tested with any of the three adhesives was similar (one way ANOVA, p>0.05). Some of the EA 9658 flowed down the assemblies, which required removal after the samples had cooled. Thus, it was beneficial to cure the EA 9658 with the coating oriented facing down so less of the adhesive covered the coating/substrate interface. The 2214 resulted in similar attachment strengths with lower variability. However, the film adhesives such as EA 9658 and FM 309 were easier to handle when setting up assemblies for curing. The FM 309 resulted in lower average TAS for coated coupons, but it did not flow around the fixture like the EA 9658.

SIGNIFICANCE: The ability to perform reliable strength testing on porous and nonporous coatings and structures was disrupted with the discontinuation of the FM 1000 adhesive. Three adhesive alternatives were tested and proven sufficient to meet test method requirements outlined in ASTM F1147.

Table 1: Cure parameters used for each adhesive.

Adhesive	Time (h)	Temp (°F)	Pressure (psi)	Amount of Adhesive	Coating Orientation
EA 9658	2	350	20	2 films	up
FM 309	2	350	20	2 films	up
3M 2214	2	250	0.7	0.05"	down

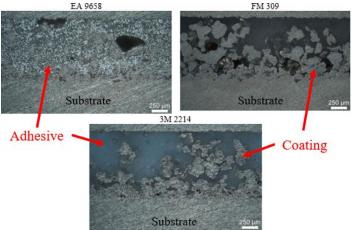


Figure 2: Cross-sectional images of adhesive penetration for each adhesive

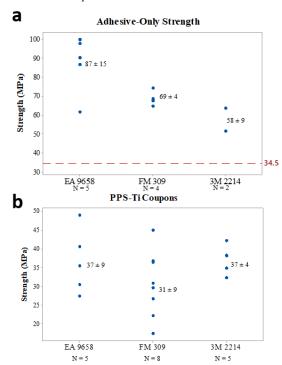


Figure 2: Attachment strength value plots for (a) adhesive-only samples and (b) coated coupons.