FABRICATION OF TITANIUM BONDED JOINT SPECIMENS FOR HIGH TEMPERATURE TESTING

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ABSTRACT

Four sets of adhesively bonded, titanium lap-shear coupon specimens were fabricated for ultimate strength testing according to the ASTM D1002 and D3165 standards. Important features of the fabrication methods, processing details, and lap-shear test results are presented for specimens fabricated using a modified bismaleimide adhesive, EA 9673, on titanium. Surface treatment of the titanium was performed using surface abrasion followed by one of two separate chemical etching processes. Although cure cycle requirements are different among most adhesives, a single surface preparation method was sought as the preferred method for conditioning the titanium specimens prior to bonding and curing. A fabrication process using a combination of low-pressure grit-blasting of the titanium surface followed by anodization with a sodium hydroxide solution applied to the D1002 specimen geometry provided the highest lap-shear strengths in the study. Additionally, difficulties documented during the fabrication process of the D3165 specimens along with features of the D3165 geometry were identified as factors that contributed to lower lap-shear strength results for the D3165 specimens as compared to the results for the D1002 specimens.

KEY WORDS: Adhesives/Adhesive Bonding, Joining/Joints/Bonding, Metals – Titanium

1. INTRODUCTION

Incorporating minimum-weight aeroshell designs and advanced materials that can withstand the severe heating loads associated with a variety of baseline entry conditions are important objectives that were developed to achieve program goals under the In-Space Propulsion (ISP) program at the National Aeronautics and Space Administration (NASA). The fabrication and testing of adhesively bonded joint specimens is a key component of this research as aeroshell

designs representing state-of-the-art technology are typically fabricated from aluminum or, on occasion, conventional carbon fiber composite materials that are bonded to a relatively heavy layer of thermal protection system (TPS) material to form a heat-shield. However, a significant overall reduction in the mass of an aeroshell has been projected by using a combination of composite materials and adhesives with high-temperature capabilities that would allow for a thinner layer of TPS material. For a typical application, thermal loads occur in an aeroshell when planetary vehicles perform aeroassist maneuvers that take advantage of atmospheric drag to reduce velocity during entry. The development of fundamental material and adhesive behavior at elevated temperatures is necessary to validate these designs and perform detailed analyses of candidate aeroshell components.

Coupon-scale testing and analyses are being performed on TPS materials, high-temperature composite materials, and adhesives to obtain basic material properties that are essential to the design of a minimum-weight aeroshell. Advancements in the development of several novel TPS materials have provided important parameters for the present system-level studies. Test results by Congdon et al. [1] using these novel TPS materials have been obtained for multiple heating-rate environments on coupon-scale test specimens. Testing of TPS materials under the extreme environments imposed during an aeroassist-type maneuver are relatively standard for TPS systems, and often exceed 800°C. at locations within the TPS near the exterior surface. The determination of basic material properties for high-temperature adhesives rarely occurs for temperatures above 260°C. due to design requirements imposed on previous aeroshell applications and the temperature limitations of most polymer adhesives. Specifically, the most well-known high-temperature adhesive used in the space program, RTV 560, and another frequently-used aeroshell adhesive, HT 424, both have maximum service temperatures of 260°C.

An important objective of the present ISP research is to identify and fully characterize the behavior of an adhesive capable of maintaining a structural bond with a short-duration service temperature between 260 and 340 degrees Celsius, thus allowing a reduction in the thickness for the TPS. While available basic material data and research on high-temperature adhesives is rare, two main sources were used to develop a database of material property information for the high-temperature adhesives: previous and current NASA programs and the open literature. These sources were used to identify both existing test data as well as fabrication and processing information for high-temperature composites and, the focus of the current investigation, titanium.

A survey of vendor supplied data for adhesives used in current and previous NASA programs revealed few adhesives capable of exceeding 260°C. Similar results were found in the open literature as no experimental results or data were located for adhesive joints operating in excess of 260°C. Of note, Dixon et al. [2] studied the effect of including PEEK fibers and powder on the subsequent normal strength of bonded specimens at elevated temperatures using Cytec FM 350. In another report by da Silva et al. [3], investigations were performed on the formation of voids and the resulting performance of adhesively bonded joints at 100 and 200°C. using the high-temperature adhesive Redux 326. Research conducted by Harter [4] on the response of adhesives at moderately elevated temperatures provided a description of their severe nonlinear material behavior as the temperature increases. Finally, a report by Jensen et al. [5] discussed

the processing and lap-shear strength of coupon specimens using the NASA developed polyimide-based adhesive, PETI-8.

The objectives of the present investigation were to fabricate adhesively bonded, titanium lap-shear coupon specimens for ultimate strength testing according to the ASTM D1002 and D3165 standards, document important features of the fabrication methods, conduct lap-shear testing, and select a preferred fabrication method for coupon-scale specimens with high-temperature adhesives. In the remainder of this paper, a description of the titanium processing and specimen configurations, test results, and interpretation of test results is given.

2. TITANIUM SPECIMEN PROCESSING

An original objective of the ISP research was to examine the properties of approximately fifteen different high-temperature adhesives on two substrates, titanium and a polymer matrix composite, at ambient and elevated temperatures. During a literature survey to identify high-temperature adhesives, it was realized that each adhesive manufacturer had recommended a slightly different surface preparation method for use with their adhesive. Performing fifteen different surface preparations for this study was determined to be cost prohibitive. Therefore, a single surface preparation was needed for each substrate material. The details of the work to identify a recommended surface preparation procedure for the titanium substrate are given in this section.

The titanium substrate used was procured as unalloyed titanium sheet (Grade 3) according to ASTM B265, while the planned polymer matrix composite substrate is a carbon fiber/polyimide system using a NASA Langley Research Center (LaRC) developed resin, RP-46. These two substrates were chosen with the intent of conducting elevated temperature testing up to 340°C. Suggested methods for preparing the surface of a titanium substrate has been reported by several researchers [5-10] with two being selected for consideration in the present research investigation. The first method consists of using an anodization process with sodium hydroxide and will hereafter be referred to as sodium hydroxide anodization (SHA). The second method summarized by Clearfield et al. [10] and used by Jensen et al. [5,6] employs the Semco Pasa-Jell™ 107 solution by PRC-DeSoto International, Inc.

Processing using the SHA method consisted of the following steps. A surface degrease of the adherends with an alkaline cleaner, Brulin 815GD. This was followed by gritblasting of the adherends using 120-180 grit Zirclean media from DuPont. Third, the adherends were immersed in a pickling solution for thirty seconds, followed by a deionized water rinse for one to five minutes. The adherends were then anodized for twenty to thirty minutes at ten volts in a NaOH solution. The temperature of the solution was between twenty and thirty degrees Celsius. The anodization was followed by a deionized water rinse for five to twenty minutes and an air dry at 25-60°C. Specific details of the pickling and anodization solutions are given by Clearfield et al. [10]. The SHA method produces a surface morphology which is both micro and macro roughened. Additionally, this method avoids the environmental concerns associated with use of other methods such as chromic acid anodization (CAA). However, several challenges were encountered with the implementation of the SHA method for titanium. Initially, it was difficult

to find a vendor that would perform the anodization treatment and could do so in a reliable manner. Difficulties were also encountered in performing the priming/bonding within the allowable time interval following anodization.

The processing of the titanium using the Pasa-Jell™ 107 solution, hereafter referred to as Pasa-Jell™, was performed according to the following detailed process developed at NASA LaRC. First, the titanium substrate was scrubbed with acetone followed by gritblasting. The titanium was then washed in a methanol bath, and then rewashed. The titanium was allowed to air dry for at least twenty minutes. Next, the substrate was dipped in Pasa-Jell™ with up and down strokes for approximately one minute and then hung in a fume hood while covered with Pasa-Jell™ to allow the substrate to etch for ten minutes. The dipping and hanging step was repeated and then followed by a thorough washing with hot tap water to remove all Pasa-Jell™ solution. The substrate was then cleaned in an ultrasonic bath of distilled water for seven minutes, allowed to dry for five minutes, ultrasonically cleaned in a fresh distilled water bath for ten minutes, and finally air dried for ten minutes. The air dried substrate was then completely dried in an air circulating oven at 100 °C for ten minutes. The surface morphology provided by the Pasa-Jell™ solution is also micro and macro roughened; however, it has been found to be less durable under environmental conditioning than the SHA and CAA methods [10]. Although quite a few steps were required for processing the titanium substrate according to the Pasa-Jell™ procedure, it could be accomplished in approximately half the time of the SHA procedure. This provided additional flexibility for bonding and curing the final specimens.

The final bonding of the titanium substrate occurred within four hours following either the SHA or Pasa-Jell™ surface conditioning for all the coupon specimens fabricated. Vendor-supplied primers were applied and cured/post-cured following the SHA or Pasa-Jell™ surface conditioning. The final step in the bonding process included application of the adhesive followed by the cure and post-cure of the specimen panels per the individual manufacturer instructions. Final machining of the individual panels was necessary to prepare the coupon specimens for testing.

3. PROCESSING STUDY AND TEST RESULTS

A series of processing development tests were conducted on ASTM D3165 and D1002 standard titanium coupon specimens to identify the best combination of surface treatment and specimen geometry. Tests were based on axial lap-shear bond strength. Previous tests conducted on ASTM D3165 specimens obtained from panels bonded with the titanium substrate prepared using the SHA surface treatment procedure and several different high-temperature adhesives had provided unexpectedly-low failure loads. Since a goal of the present ISP research is to screen a matrix of potential high-temperature adhesives on titanium, it was necessary to determine a set of processing conditions that would provide the best bond performance using that material. Therefore, four groups of ASTM D3165 and D1002 standard coupon specimens were fabricated by bonding titanium adherends with a high-temperature modified bismaleimide adhesive from the Loctite Corporation, EA 9673. The EA 9673 adhesive was selected for the processing development tests due to its relatively easy processing requirements and available test data.

Two sets of ASTM D3165 and two sets of ASTM D1002 lap-shear coupon specimens were fabricated using both the SHA and Pasa-Jell™ surface conditioning procedures, described in the previous section, for a total of four sets of specimens. Each set of specimens consisted of three individual specimens except for the ASTM D3165 Pasa-Jell™ set which only had two specimens. A stereoscope with a magnification of 200X and associated digital measurement system was used to determine the thickness of the bondline on each specimen. A typical image obtained from the system is shown in Figure 1. The bondline thicknesses determined using the stereoscope ranged from 0.102 to 0.152 mm.

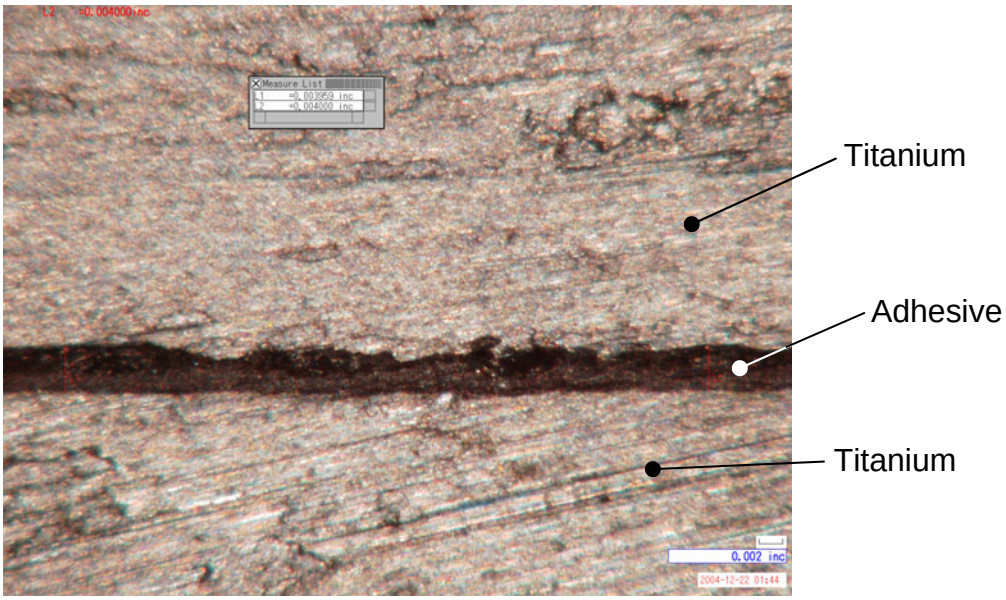


Figure 1. Typical magnified image of ASTM D3165 and D1002 lap-shear coupon specimens with a titanium substrate showing an approximate bondline thickness of 0.102 mm.

Testing of the four sets of lap-shear specimens was performed using a 22 Kip screw-driven tension test machine. All the specimens were loaded in tension using a crosshead speed of 1.27 mm/min at room temperature, approximately twenty-five degrees Celsius, and gripped using hydraulic grips. The lap-shear specimens each had a rectangular cross-section with a nominal width of 25.4 mm and a total length of 190.5 mm. The final failure load and total axial displacement of each specimen was recorded by the data system. The failure loads determined for all four sets of specimens are given in Figure 2. Data provided by the manufacturer of the EA 9673 adhesive indicated an estimated failure load of 4,450 Newtons. The expected failure mode for the lap-shear specimens was a predominantly cohesive failure through the adhesive layer.

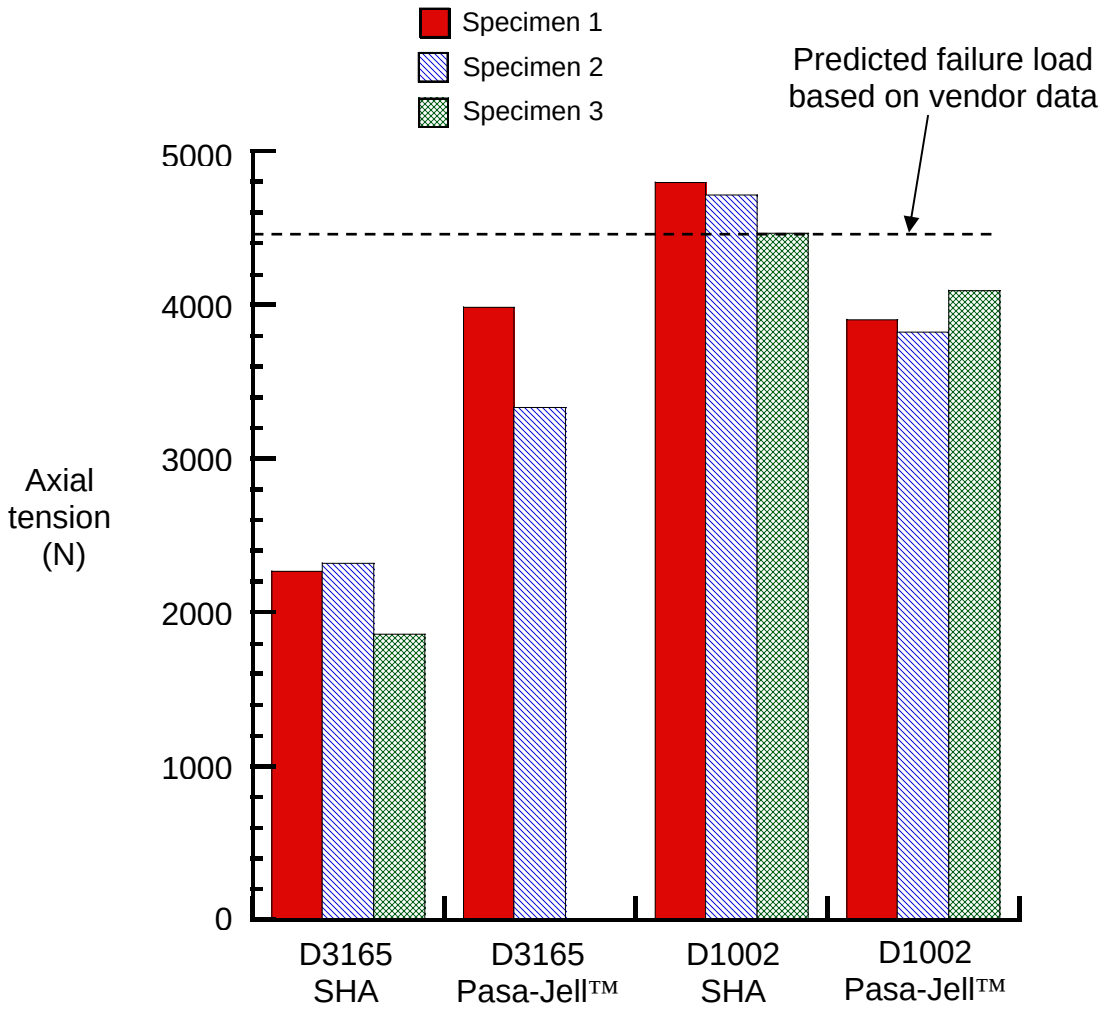


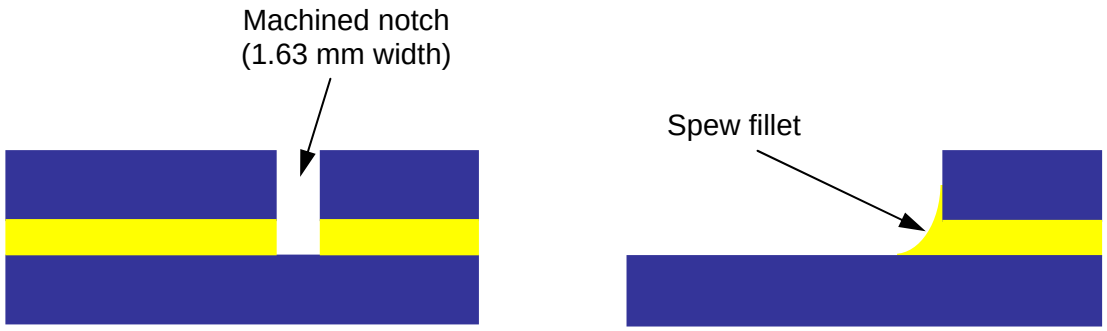
Figure 2. Results of surface conditioned ASTM D3165 and D1002 lap-shear strength tests at room temperature for EA 9673 on titanium.

4. INTERPRETATION OF TEST RESULTS

The experimental results on ultimate bond strength from the ASTM D3165 and D1002 tests conducted on the titanium substrate shown in Figure 2 were mixed. Consistent values were obtained for both the D3165 and D1002 Pasa-Jell™ coupon specimens with mean failure loads of 3661 N. and 3940 N., respectively. However, the SHA coupon specimens provided a large disparity in their values as the D3165 specimens exhibited a mean failure load of 2147 N., while the D1002 results produced more than twice the mean failure load of the D3165 results with an average value of 4660 N. The specimen to specimen variation of failure load showed very little variation between surface preparation method; however, a larger variation was present between the D1002 and D3165 specimen geometries. The difference in failure loads between individual specimens, both SHA and Pasa-Jell™, for the D1002 specimens was approximately 7%, while the D3165 specimens produced a difference in failure loads of 20-25%. Based on these results,

repeatability would be much better for the D1002 lap-shear coupon specimens. Furthermore, the results for the coupon specimens with the SHA surface treatment indicated that the reduction in bond strength was primarily a function of the D3165 processing. All the specimens tested appeared to fail in a primarily cohesive failure mode.

Previous research summarized by Clearfield et al. [10] has shown that the SHA surface procedure produces bonds on titanium that exhibit one of the highest levels of durability and lap-shear strength. Furthermore, the ASTM D3165 coupon geometry has been suggested as an improvement to the older D1002 configuration since the D3165 coupon geometry minimizes initial load eccentricity and reduces bending deformations that lead to large normal or peel strains at the ends of the joint overlap. It was anticipated based on this previous research and the improved coupon geometry that the D3165 coupon specimens would exhibit superior bond strengths as compared to the D1002 specimens. However, a few shortcomings associated with machining of the final specimen geometry were identified for the D3165 coupon specimens. The first factor affecting the bond strength for the D3165 configuration is the lack of a spew fillet at the ends of the overlap region when compared to the D1002 geometry as shown in Figure 3, provided for illustration purposes only (not to scale). This square-end notch feature in the D3165 coupon specimen is a result of post-cure machining that is necessary to construct a single-lap bond region. Significant reductions in single-lap shear bond strength have been identified by Tsai and Morton [11] and Frostig et al. [12] for bonded joints without a well-formed spew fillet. Additionally, the D3165 coupon specimens were fabricated as a panel and machined into individual specimens, which required machine cuts that ran along the length of the specimen. The D1002 specimens did not require machine cuts that ran along the bond region as they were fabricated in slotted or finger panels, Figure 4, that only required machining at two points away from the bond area.



a) D3165 specimen detail b) D1002 specimen detail

Figure 3. Cross-section views of the end regions of the bonded overlap.

One other factor that may have had a negative affect on the bond strength for the D3165 specimens resulted from the gritblasting procedure prior to surface treatment. Although both the D3165 and D1002 specimens received gritblasting prior to chemical etching, the D1002 specimens were only surface abraded in small regions at the ends of the finger panels while the D3165 specimens were abraded as entire panels. As low bond strengths were identified for a few adhesives bonded using the D3165 configuration, it was noted that a slight curvature or

warping of the panel was evident after the gritblasting procedure. The curvature was easily removed during the bonding and curing process; however, small residual strains were present in the completed panels. Final machining of the D3165 coupon specimen configurations appeared to exaggerate this condition as a few specimens fractured and broke during the machining process. Thus, it appears that these residual strains and thermal strains during machining contributed to the lower than expected bond strengths in the D3165 specimens.

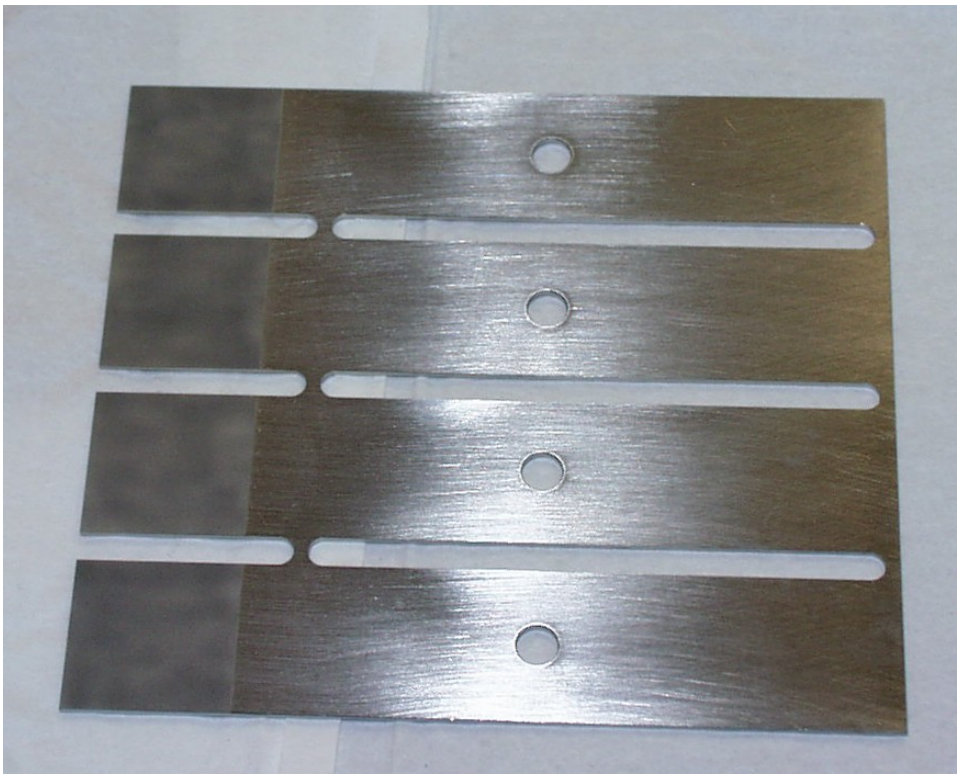


Figure 4. ASTM D1002 titanium finger panel prior to bonding.

5. CONCLUDING REMARKS

A processing study was conducted using titanium ASTM D3165 and D1002 standard coupon specimens to identify the best combination of surface treatment and specimen geometry based on axial lap-shear bond strength due to problems during initial coupon-specimen panel fabrication. The D1002 specimen geometry with the SHA surface treatment resulted in the best mean bond strength from experimental tests compared to the other specimens. The D1002 coupon specimen geometry with SHA surface treatment had a mean failure load of 4,660 Newtons, and was the only combination that exceeded the manufacturer's literature value of 4,450 Newtons.

Several potential factors that could have led to the low bond strengths for the D3165 coupon specimens were identified during fabrication. The two primary factors identified were panel curvature prior to bonding as a result of gritblasting and effects due to final machining of the panels. These difficulties documented during the fabrication process of the D3165 specimens

along with features of the D3165 geometry were identified as factors that contributed to lower lap-shear strength results for the D3165 specimens as compared to the results for the D1002 specimens.

6. ACKNOWLEDGEMENTS

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