

**Strengthening of Glass and Pyroceram
With Hydrophobic Coatings**

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FOREWORD

This report describes studies on the application of hydrophobic coatings to soda-lime glass and Pyrocera 9606 to improve strength. The experimental work was carried out from August 2001 to April 2002.

The work was performed in the Chemistry and Materials Division of the Research Department and the Materials Branch of the Airframe Division of the Weapons/Targets Department at the Naval Air Warfare Center Weapons Division. The project was sponsored by the Office of Naval Research through the Airborne Weapons Materials Project, the Anti-Radiation Missile (ARM) Program Office, and the Evolved SeaSparrow Missile) ESSM Project Office.

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INTRODUCTION

The objective of this study was to determine whether significant improvements in strength of soda-lime glass and Pyroceram 9606 could be obtained by applying thin hydrophobic coatings.

Pyroceram 9606 is an important glass ceramic radome material. Its chemical composition is in the cordierite family, consisting principally of SiO_2 , Al_2O_3 , MgO , and TiO_2 . Future demands for systems under design may require continued use of Pyroceram 9606 and possibly exposure to higher service stresses. Pyroceram 9606 is known to be susceptible to sub-critical crack growth at either elevated temperatures (Reference 1) or under stress while exposed to moisture (Reference 2). Even very minor crack growth can significantly reduce the "strength" of Pyroceram 9606. Therefore, tests were performed to see if a coating would increase Pyroceram 9606 strength by blocking or minimizing surface exposure to moist air or water. The tests performed were not intended to comprehensively characterize the effect of the coating, but rather to assess the viability of increasing the strength using organosilicon surface coatings.

The initial approach involved coating soda-lime glass slides with several different hydrophobic compounds and then conducting strength tests in flexure. The rationale was to bond a hydrophobic coating to the ceramic surface to retard access of water to the growing crack front. The hope was that this barrier would increase the strength of the glass or glass ceramic by preventing stress corrosion at the growing crack tip when the material is flexed. Glass slides were acid etched to remove surface defects and slightly mimic the outer fortified surface of Pyroceram. (Fortification is a proprietary etching procedure applied to the surface of Pyroceram to increase its mechanical strength.) The coated glass exhibited more than double the strength of uncoated glass at room temperature. Based on this success, the hydrophobic coating was then applied to Pyroceram 9606 bars.

The Pyroceram samples were received in a "fortified" condition that is achieved by treating with an alkali solution to produce a porous surface layer. This treatment removes mechanical surface damage and the porous layer protects the underlying glass ceramic material (Reference 3). Figure 1 is a photomicrograph of Pyroceram with the porous fortification layer. The fortification layer thickness was chosen to be the deepest penetration of any apparent porosity, about 0.17 millimeter (mm).

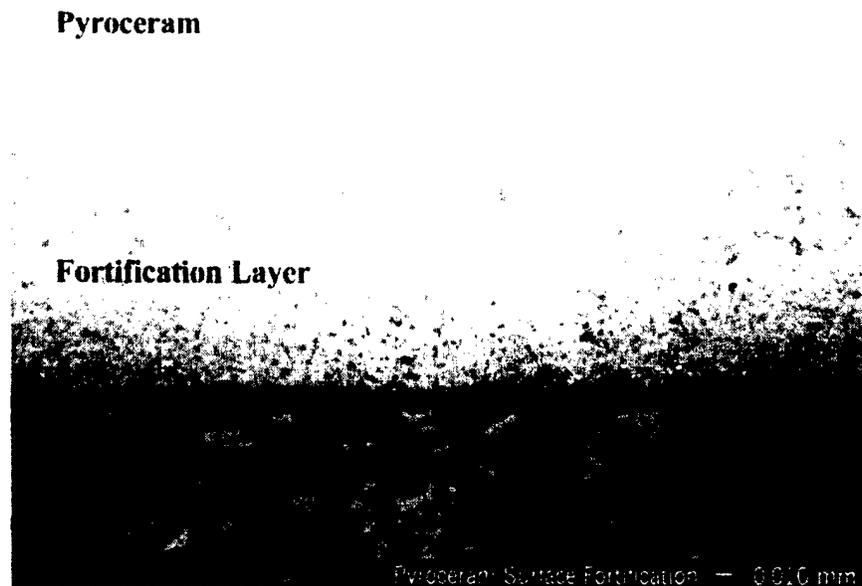


FIGURE 1. Photomicrograph of Cross Sectioned and Polished Pyroceram Showing the Porous Fortification Layer and the Bulk Structure. The dark gray region is the interface between the Pyroceram and a polymer mounting layer at the bottom of the picture.

EXPERIMENTAL SECTION

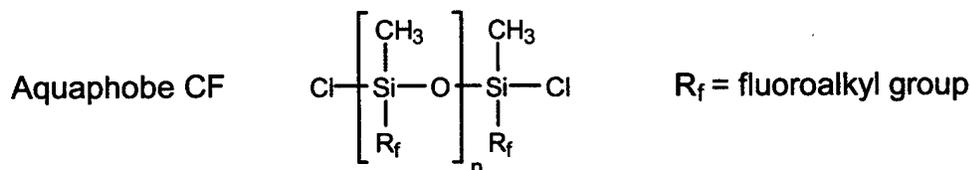
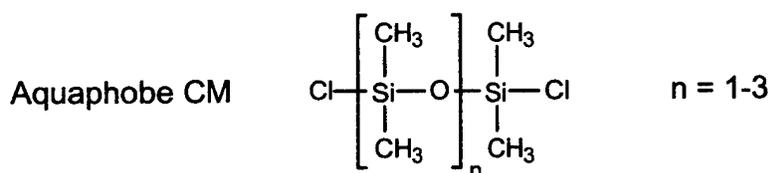
SAMPLE DESCRIPTION AND COATING REAGENTS

Soda-lime glass slides (clear micro slides) were purchased from VWR International. Nominal dimensions are 50.8 x 50.8 x 1.5 mm. The glass slides were subjected to an acid etching procedure to remove mechanical surface damage and, thereby, improve strength (Reference 4). The samples were first rinsed by squirting with ethanol, and then air dried. The samples were then immersed in a solution containing 15% HF and 15% H₂SO₄ (volume %, based on diluting concentrated solutions) for 5 minutes (min), immersed in deionized water for 5 min, squirted with distilled water, and blown dry with argon. The samples were dried in an oven at 105°C for 20 min. The average weight loss due to acid etching was 6.5%, and based on weight loss, the total thickness removed for all slides was in the range of 0.10±0.02 mm. Glass slide dimensions after acid etching were approximately 50.7 x 50.7 x 1.4 mm. Based on weights of the slides, the thickness after etching varies by approximately ±0.07 mm.

Twenty Pyroceram 9606 bars were purchased from Corning. Nominal dimensions are 91.4 x 5.1 x 2.5 mm. The thickness of the fortification layer was measured to be 0.17 mm (see Figure 1).

Four silane-based hydrophobic reagents were purchased from Gelest, Inc.: n-octadecyldimethylchlorosilane, (tridecafluoro-1,1,2,2-tetrahydrooctyl)methyldichlorosilane, Aquaphobe™ CM, and Aquaphobe™ CF (see structures below). Aquaphobe CM is a chlorine-terminated polydimethylsiloxane oligomer, while Aquaphobe CF is a chlorine-terminated polyfluoroalkylmethylsiloxane oligomer. The chlorines react with hydroxy and silanol groups on surfaces to form a chemically bound polydimethylsiloxane "siliconized" surface (Reference 5). Heat curing at 110°C for 15 to 20 min is recommended after dip coating to provide the most effective surface treatment. Parowax Household Wax, a paraffin wax, was used for coatings. McGhan-Nusil D-7040 silicone diffusion pump oil was used for coating.

Chemical structure of the silane compounds:



COATING PROCEDURES

Glass Samples

The Aquaphobe CM and Aquaphobe CF coatings were applied by immersing samples for 10 seconds (s) in a 2 volume % solution of the Aquaphobe reagent in anhydrous heptane (in an open beaker). After dipping, the slides were dried in air and then placed in an oven at 105°C for 20 min. The octadecyldimethylchlorosilane coating was conducted as above, except the immersion time was increased to 1 min to allow more time for reaction. The fluoroalkylmethyldichlorosilane coating was conducted as above with 1-min immersion, except concentration was 1 volume %.

Pyroceram Samples

Pyroceram samples were individually placed in plastic bags and handled only by the ends to minimize the possibility of any mechanical damage. Coating of the Pyroceram samples was conducted as follows:

Samples 1, 3, and 5: Samples were placed together in a flask, which was then sealed with a serum stopper and evacuated through a sidearm stopcock on the flask. A 2% by volume solution (150 milliliters (mL)) of octadecyldimethylchlorosilane in dry heptane was added to the evacuated samples through a double-ended needle. The flask was brought to atmospheric pressure with air, then let stand for 1 hour (hr). The solution was removed and dry heptane added to rinse out excess coating reagent. After 1.5 hr the samples were removed and placed in a beaker to dry in air. After 3 min the samples were placed in an oven at 90°C. The oven temperature was raised such that it had reached 103°C when the samples were removed 24 min later. The samples were placed back in plastic bags.

Samples 7, 8, and 9: Samples were dried in an oven at 112°C for 1.7 hr. Three minutes after removing from the oven, the samples were placed in a tube, which was then evacuated. A 5% by volume solution of octadecyldimethylchlorosilane in dry heptane was added to the evacuated samples. The flask was brought to atmospheric pressure with air, then let stand for 22 hr. The solution was removed and the samples allowed to dry in air for 2 hr. The tube was then capped and left for 3 days. The samples were placed in an oven at 105°C for 0.5 hr. After cooling, the samples were placed back in plastic bags.

Sample 15 was placed in an oven at 105°C for 35 min. After 35 min in air, the sample was immersed in 2% by volume Aquaphobe CM in dry heptane. The sample was removed after 10 min. After 5 min the sample was placed in an oven at 105°C and removed 18 min later. After 27 min the sample was immersed in melted paraffin wax and removed after 4 min in wax. After cooling, the sample was placed back in a plastic bag.

Sample 17 was placed in an oven at 112°C for 24 min. The hot sample was then immersed in 4% by volume Aquaphobe CM in dry heptane and removed after 6 min. After 1 min the sample was placed in an oven at 112°C and removed 12 min later. After 20 min the sample was placed in vacuum oven to dry overnight (104°C, <3 torr). After 21 hr the sample was removed from the vacuum oven and immediately immersed in melted paraffin wax. After about 2 min the sample was removed from the wax and held in air about 30 s to let the wax solidify. The sample was immersed in wax again for about 10 s, removed for about 20 s, immersed again for a couple seconds, removed for about 10 s, and the process repeated for a total of about 5 dips. Then the sample was allowed to cool for about 2 min and dipped quickly in wax (near its melting point of about 60°C) 2 or 3 times to build up a thicker and smoother coating that would cover any cracks in the underlying wax layer. After about 10 min the sample was loaded into the test chamber.

Sample 19 was dried overnight in a vacuum oven (104°C, <3 torr). The sample was removed from the vacuum oven and immediately immersed in melted paraffin wax. The sample remained

in the wax for several minutes while the temperature of the wax was equilibrated to just above the melting point by occasionally warming the top and bottom ends of the test tube containing the wax. Then the sample was removed from the wax using tweezers and held in air a few seconds to let the wax solidify, followed by immersion again for a few seconds, with the process repeated for a total of about 5 dips. Then the sample was allowed to cool in air for about 3 min and dipped in wax very near its melting point twice to build up a thicker coating. After about 10 min the sample was loaded into the test chamber. The wax coating was somewhat thicker (about 1 mm) than for sample 17 and had a considerable thickness gradient along its length. This was due to the wax temperature being closer to the melting point, which led to the wax quickly solidifying when the sample was removed from the molten wax.

Prior to strength testing, several Pyroceram samples were immersed in diffusion pump oil to exclude moisture from the surface. Specific procedures were as follows: samples 13, 14, and 20 were dried in a vacuum oven overnight (104°C, <3 torr), then (after filling the vacuum oven with air) immersed in diffusion pump oil for about 5 min prior to testing; sample 16 was immersed in oil prior to testing (but not dried in the vacuum oven). When the sample was taken out of the oil bath, the oil on the surface was not removed.

STRENGTH TESTING PROCEDURES

Glass Samples

Before testing, the samples were covered on the compressive surface with Scotch™ Packaging Tape (clear, 2-inch-wide tape), then dipped in water for approximately 2 s, then covered on the tensile surface with paper to reduce friction. No attempt was made to differentiate the two faces of the glass samples. For unetched float glass samples, the strength was found to be about twice as high on the surface exposed to the atmosphere during processing (Reference 6). For the etched glass samples studied here, the difference in strength for the two sides should be reduced because etching should remove some of the surface flaws that weaken the surface that was in contact with rollers during processing.

Failure loads were determined using a ring-on-ring equibiaxial flexure test (Reference 7). The tests were performed using an Instron 5500R. Ring diameters were 10.80 and 22.86 mm for the top and bottom ring fixtures, respectively. For the highest strength samples, a limiting factor may have been the bottom ring height of 1.55 mm (the center of the glass plate may have hit the center of the bottom fixture before failure). Most of the tests were run at a constant crosshead speed of 1.27 mm/min. The remaining tests were run at 0.127 or 12.7 mm/min.

The test fixture was shielded to contain glass fragments.

In six tests, the sample did not break as the instrument reached the load limit (nominally 1,000 pounds (lb)) for the setup at the time (see the Appendix), and a larger load capacity (10,000 lb) was used in subsequent tests.

Pyroceram Samples

The Pyroceram samples were tested in air at 65% relative humidity (RH), except for two samples (13 and 20) that were tested under dry nitrogen (<5% RH). Before testing, two samples (5 and 6) were immersed in deionized water for 1 hr, and six samples (7 through 12) were dipped in water for a maximum of 2 s. Two of the samples that were immersed in oil (14 and 16) were tested in 65% RH air with a 5-min exposure to 65% RH air prior to starting the test.

Samples were tested in flexure using four-point bend tests, IAW ASTM C1161 (Reference 8). The outer support span was 40.0 mm and the inner support span was 20 mm. Tests were conducted at a constant load rate (approximately 330 Newtons (N)/min) that corresponds to approximately 5 megaPascals (MPa)/s stress rate for the nominal sample dimension.

For tests 1 through 6, failure initiation was under the same inner roller for all tests. Therefore, the roller faces may not have been parallel (inner to outer), which would cause a higher stress below the single roller. Marks across sample faces at the roller contact locations do indicate decent perpendicularity in that direction. Before test 7 the bend roller alignment was checked and it was verified that all four rollers have "misalignment" tolerances of 0.013 mm or less. Sample 7 also broke under an inner roller. After test 7, a thin layer of Loctite 454 was applied between the rollers and the V-frame (inner span rollers only), which alleviated the alignment problem. Roller misalignment was <0.0025 mm. After test 13 one of the rollers became unglued. Both rollers were cleaned and rebonded as before. For tests 8 through 20, failure locations were random and generally between the inner rollers, indicating acceptable roller alignment.

STRENGTH CALCULATIONS

Glass Samples

Input parameters for the strength calculation (Reference 7) include: load radius of 5.40 mm, support radius of 11.43 mm (measured using a ruler under a microscope), effective disk radius of 30.6 mm (based on a 50.7-mm-square slide), and a Poisson ratio of 0.22 (Reference 9). The effective disk radius was calculated (Reference 10) as the average radius of two circles that fit inside and outside of the square of the glass slide (effective disk radius = 0.25 (square side length) $(1 + 2^{0.5})$). For the deflection calculation, the modulus used was 71.0 gigaPascals (GPa) (Reference 9).

Note that for the six tests where the sample did not break as the instrument reached the load limit for the setup at the time (see the Appendix), the final load (1,180 lb) was included as a regular data point.

Pyroceram Samples

Fracture strength (σ_f) for all Pyroceram flexural test samples was calculated using four-point-1/4 flexure equations as defined in ASTM C1161 (Reference 8). Fracture strength was calculated twice using different sample dimensions: as-measured width and thickness, and fortification layer corrected width and thickness. The latter set of dimensions was determined by subtracting the fortification layer thickness (0.17 mm) from all four sides. Fortification layer thickness was determined using conventional metallographic techniques and optical microscopy. Fortification layer adjusted strength was termed "structural strength." Fracture strength is defined as:

$$\sigma_f = \frac{3PL}{4bd^2} \text{ [MPa]}$$

P is the failure load in Newtons

L is the outer support span in mm

b is the specimen width in mm

d is the specimen thickness in mm

SPECTROSCOPIC EXAMINATION OF COATED SURFACES

Prior to coating the Pyroceram samples, there was a concern about whether the hydrophobic chlorosilane reagents would react and bond effectively to the Pyroceram surface. An attempt was made to detect monolayer coverage of the hydrophobic reagent bonded to glass. Several samples of soda-lime glass slides with hydrophobic coatings were examined by specular and diffuse reflectance Fourier transform infrared spectroscopy (FTIR). No signal from the coating (hydrocarbon or fluorocarbon) was detected. To increase the amount of coating present, the Aquaphobe CF reagent was applied to a fine powder of soda-lime glass that was prepared by grinding a piece of acid-etched glass using a boron carbide mortar and pestle. The coating procedure involved immersion of the glass powder in a 2% solution of Aquaphobe CF in anhydrous heptane for 8 min, then washing three times with heptane (each time removing at least 90% of the liquid) and drying in an oven at 105°C for 20 min. The coating was still not detected by diffuse reflectance FTIR. Because the coating was not detected on glass, it was not possible to compare the amount of coating on Pyroceram to that on glass.

RESULTS AND ANALYSIS

HYDROPHOBIC COATINGS ON GLASS

Four hydrophobic coating reagents were applied to soda-lime glass, as described in the "Experimental Section" of this report. While coating thickness was not measured, for three of

the coatings the thickness should be on the order of a monolayer, because only species that chemically react will stay on the surface due to the volatility of the coating reagent. For the octadecyldimethylchlorosilane compound that is not significantly volatile, the coating thickness is much greater, with the slides visibly cloudy after coating. All of the silane-treated slides exhibited hydrophobic behavior as evidenced by rapid run-off of water from the surface after dipping in water (prior to the strength test).

The strength results for soda-lime glass are listed in Table 1. Data for the individual tests are given in the Appendix, Table A-1. Figure 2 shows representative load versus displacement curves. The octadecyldimethylchlorosilane reagent gave the largest strength increase—more than twice that of the uncoated samples. The other three reagents gave strength increases of 45 to 75% over the uncoated samples. Unetched glass (not treated with acid) had less than a third of the strength of the acid-etched glass, which is consistent with the expected removal of surface flaws by acid etching.

TABLE 1. Strength of Soda-Lime Glass^a Coated With Hydrophobic Silanes.

Coating reagent	Mean strength, MPa	Standard deviation, MPa	Crosshead speed, mm/min	Number of samples	Maximum deflection, mm (calc)
None	626	263	0.127	20	0.411
None	724	442	1.27	20	0.475
None	1040	480	12.7	20	0.682
Octadecyldimethylchlorosilane ^b	1410	990	0.127	20	0.927
Octadecyldimethylchlorosilane ^b	1930	1160	1.27	20	1.270
Octadecyldimethylchlorosilane ^b	2200	1300	12.7	20	1.450
Fluoroalkylmethylchlorosilane ^c	1270	847	1.27	20	0.837
Aquaphobe CF	1040	384	1.27	18	0.681
Aquaphobe CM	1050	542	1.27	19	0.686
None, unetched	208	66	1.27	10	0.137

^a All samples were acid etched unless otherwise noted.

^b This reagent gave a thicker coating after dipping and drying since it is not volatile.

^c Fluoroalkyl = CH₂CH₂CF₂CF₂CF₂CF₂CF₂CF₃.

Figure 3 is a graph of apparent strength (log of failure load) versus crosshead speed (log of strain rate) for the uncoated and octadecyldimethylchlorosilane-coated samples. From the slope of the linear fit of the data, the stress corrosion susceptibility constant, n ($n = (1/\text{slope}) - 1$), was determined to be 8.1 for the uncoated samples and 9.3 for the octadecyldimethylchlorosilane-coated samples. The similarity of these values indicates that the coating did not significantly alter the reaction between water and the glass surface (Reference 4).

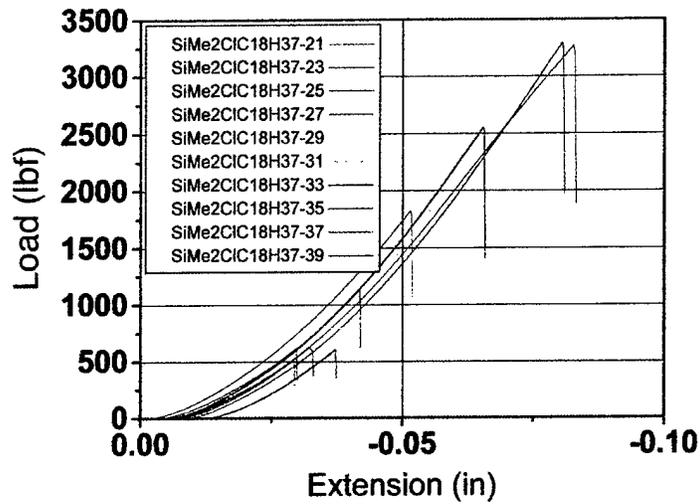


FIGURE 2. Raw Data From Strength Tests on Octadecyldimethylchlorosilane-Coated Glass.

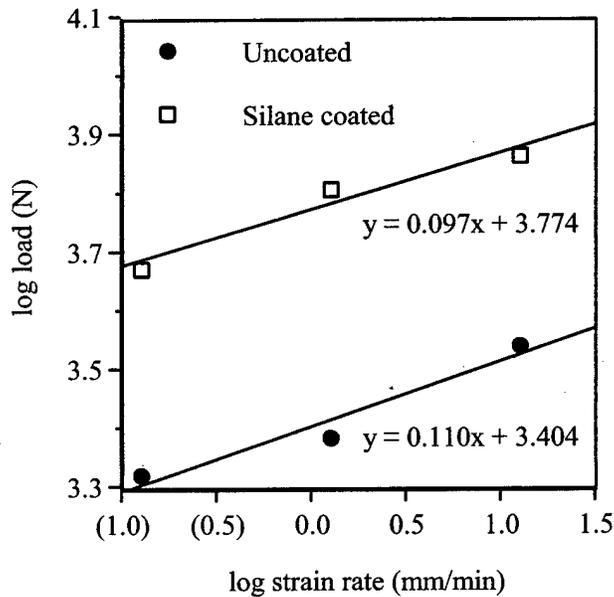


FIGURE 3. Plot of Log of Failure Load Versus Log of Crosshead Speed for Uncoated and Octadecyldimethylchlorosilane-Coated Soda-Lime Glass Slides.

For comparison, Ritter's results (Reference 4) on the same type of glass are: uncoated 2,188 MPa (n = 13.0-16.0), polymer coated 2,237 (silicone), 2,373 (acrylic), and 2,410 (epoxy) MPa (n = 13.8, 17.4, and 15.1). The polymer coatings did not significantly enhance the strength of acid etched glass, but did slightly improve the strength of abraded glass samples. The polymer coating thickness was about 0.08 mm. Ritter's samples were 3-mm-diameter glass rods that

were annealed at 500°C for 1 hr, then acid etched to remove about 0.32-mm depth (compared to 0.05-mm depth removed here from the glass slides), which was said to be adequate to give maximum strength. Ritter's samples were tested in 4-point bending with inner and outer spans of 20.7 and 71.3 mm, respectively. The rods used by Ritter were dip coated, so the polymer was not covalently attached to the glass. In the present work, the first layer of coating is intended to be covalently attached to the glass.

Table 1 lists the mean calculated deflection for each set of samples. The highest mean calculated deflection was 1.446 mm in Table 1, and the highest value calculated for a single test was 2.89 mm (see Table A-1). However, the support ring height was only 1.55 mm, and the maximum extension recorded by the instrument was about 1.8 mm. Because the deflection should ideally be less than half the thickness (i.e., <0.7 mm), the higher strength values may have some added uncertainty.

HYDROPHOBIC COATINGS ON PYROCERAM

The strength results for Pyroceram are listed in Table 2. Additional data for the individual tests are given in Table A-2. The "structural" strength in Table 2 refers to the strength value calculated after subtracting the fortification layer thickness from all four sides.

Because of the small number of Pyroceram samples available, only 1 to 3 samples were treated at a time to look for improvements in strength. Fortified Pyroceram exhibits a high Weibull modulus (19.6 for the data in Reference 1) that allows for meaningful strength comparison despite the very limited number of samples tested in this study. For Pyroceram samples 1 through 6 there is no increase in strength for the coated samples compared to the uncoated samples, unlike what was observed for the soda-lime glass samples. Immersion in water for 1 hr prior to testing (samples 5 and 6) reduced the strength by about 10%. While this long immersion in water should maximize stress corrosion cracking in the uncoated sample and, therefore, also maximize the potential beneficial effect of coatings, it may also degrade the hydrophobic coating. The strengths of samples 1 through 6 are all lower than subsequent samples, because of inadequate alignment of the rollers for samples 1 through 6. Therefore strengths reported for samples 1 through 6 should be used for relative comparison only. The alignment problem was corrected as noted in the Experimental Section.

Application of a thicker coating of the chlorosilane reagent (samples 7 through 9 with "structural" strength of 200 to 230 MPa) still did not noticeably improve strength over that of uncoated samples (samples 10 through 12 with "structural" strength of 204 to 228 MPa). These samples were briefly dipped in water immediately prior to measuring the strength to minimize expected degradation of the coating reagent (which will still contain water-reactive chlorosilane groups in these thicker coatings).

TABLE 2. Strength of Pyroceram Coated With Hydrophobic Silanes and Paraffin Wax.^a

Sample number	Coating, pre-test drying	Test environment	Strength, MPa	"Structural" strength, ^b MPa
1	Chlorosilane, thin	65% RH	111 ^c	158 ^c
2	None	65% RH	107 ^c	151 ^c
3	Chlorosilane, thin	65% RH	105 ^c	150 ^c
4	None	65% RH	107 ^c	152 ^c
5	Chlorosilane, thin	H ₂ O soaked, 65% RH	98 ^c	140 ^c
6	None	H ₂ O soaked, 65% RH	96 ^c	137 ^c
7	Chlorosilane, thick	H ₂ O dipped, 65% RH	140	200
8	Chlorosilane, thick	H ₂ O dipped, 65% RH	154	218
9	Chlorosilane, thick	H ₂ O dipped, 65% RH	161	230
10	None	H ₂ O dipped, 65% RH	143	204
11	None	H ₂ O dipped, 65% RH	160	228
12	None	H ₂ O dipped, 65% RH	145	208
13	None, vacuum dried	"oil", N ₂ , <5% RH	218	308
14	None, vacuum dried	"oil", 65% RH	222	318
15	Aquaphobe CM + thin wax	65% RH	161	229
16	None	"oil", 65% RH	184	260
17	Aquaphobe CM, thick wax after vacuum drying	65% RH	201	287
18	None	65% RH	156	220
19	Thick wax after vacuum drying	65% RH	217	306
20	None, vacuum dried	"oil", N ₂ , <5% RH	218	307

^a See Experimental Section for a complete description of sample coating and test procedures.

^b The "structural" strength is the strength calculated after subtracting the fortification layer thickness from all four sides.

^c These strength values are artificially low due to fixture misalignment and, therefore, should be used for relative comparison only.

In order to determine the maximum strength improvement that could be expected with hydrophobic coatings, sample 13 was tested under very dry conditions (under dry nitrogen after drying the sample in a vacuum oven and immersion in silicone oil). This led to about a 50% improvement in "structural" strength to 308 MPa. Sample 14 was treated similarly but tested under air at 65% RH to see whether a dry atmosphere was required to obtain the high strength. Because the strength remained high (318 MPa), we conclude that the oil is effectively prohibiting moisture from getting to the surface. To determine the role of the vacuum oven drying, this step was skipped for sample 16. The strength dropped to about 260 MPa, which is about midway between the highest strength and the strength of sample 18, which was untreated (220 MPa). Thus, it is important to exclude both surface adsorbed water (e.g., by vacuum drying) and atmospheric moisture.

Because the hydrophobic coatings alone were not effective, we next applied wax coatings by dipping samples in molten paraffin wax. The solidified hydrophobic wax coating should exclude

moisture similar to the liquid oil coating. After applying the Aquaphobe CM coating, sample 15 was dipped once in paraffin wax. The strength was low, 229 MPa, probably due to cracks that form in the wax layer during solidification. To minimize cracks through the wax layer, sample 17 was dipped several times in wax, including a couple of dips at temperatures close to the wax melting point. In addition, sample 17 was subjected to vacuum oven drying prior to wax coating. The strength improved to about 287 MPa. A third sample (19) was coated with wax without first applying the Aquaphobe CM coating. In addition, several dips in wax were conducted at temperatures very close to the wax melting point. The strength improved slightly, to about 306 MPa, indicating that this thick wax coating was equally as effective as the silicone oil.

CONCLUSIONS AND DISCUSSION

A hydrophobic coating of octadecyldimethylchlorosilane on soda-lime glass slides led to doubling or more of the strength (as shown in Table 1 and Figure 2). The increase in strength is attributed to a reduced role of stress corrosion cracking that is promoted by moisture at the surface. Similar hydrophobic treatments were not effective on Pyroceram bars. This may be due to poor coverage of the hydrophobic coating because of different surface chemistry, or possibly due to effects of the porous fortification layer that may be difficult to penetrate by the hydrophobic reagents. Thick wax coatings on dry Pyroceram surfaces were successful at improving the strength by about 40%, similar to the effects of dipping in silicone oil.

Future work might examine thinner wax or polymer coatings on Pyroceram and the effects of elevated temperatures that missile domes experience. Aging studies would also need to be performed to test for possible degradation of the hydrophobic layers. A more rigorous approach to wax treatment would involve immersion of an evacuated sample in molten wax to force the wax into the pores of the fortification layer in Figure 1 while air and moisture were excluded. Ultimately, the highest hydrophobicity may be obtained by first attaching a fluorocarbon to the Pyroceram surface and then filling the pores with a fluorocarbon wax.

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Appendix

RESULTS OF INDIVIDUAL STRENGTH TESTS OF SODA-LIME GLASS

TABLE A-1. Tabulated Results of Individual Strength Tests of Soda-Lime Glass—Data Grouped by Coating and Crosshead Speed.

Uncoated, 0.127 mm/min	Failure load, lb	Failure load, N	Strength, MPa	Deflection, mm
Uncoated-4-38	229.40	1020.4	305.01	0.2002
Uncoated-4-28	279.87	1244.9	372.12	0.2443
Uncoated-4-40	306.03	1361.3	406.90	0.2671
Uncoated-4-30	314.56	1399.2	418.24	0.2746
Uncoated-4-26	320.48	1425.6	426.11	0.2798
Uncoated-4-10	325.81	1449.3	433.20	0.2844
Uncoated-4-20	334.68	1488.7	444.99	0.2921
Uncoated-4-6	339.57	1510.5	451.50	0.2964
Uncoated-4-8	378.23	1682.5	502.90	0.3302
Uncoated-4-24	384.63	1710.9	511.41	0.3358
Uncoated-4-12	420.99	1872.7	559.75	0.3675
Uncoated-4-32	430.95	1917.0	573.00	0.3762
Uncoated-4-2	446.91	1988.0	594.22	0.3901
Uncoated-4-14	498.71	2218.4	663.09	0.4353
Uncoated-4-16	566.39	2519.4	753.08	0.4944
Uncoated-4-34	600.85	2672.7	798.90	0.5245
Uncoated-4-18	757.45	3369.3	1007.11	0.6612
Uncoated-4-22	780.22	3470.6	1037.39	0.6811
Uncoated-4-36	836.41	3720.5	1112.10	0.7301
Uncoated-4-4	869.19	3866.4	1155.68	0.7587
Mean	471.07	2095.4	626.33	0.4112
Standard Deviation	197.81	879.9	263.00	0.1727

TABLE A-1. (Contd.)

Uncoated, 1.27 mm/min	Failure load, lb	Failure load, N	Strength, MPa	Deflection, mm
Uncoated 2-43	196.48	874.0	261.24	0.1715
Uncoated 2-58	240.85	1071.4	320.24	0.2102
Uncoated 2-60	262.20	1166.3	348.62	0.2289
Uncoated 2-55	282.65	1257.3	375.81	0.2467
Uncoated 1-4	309.61	1377.2	411.66	0.2703
Uncoated 1-27	323.68	1439.8	430.37	0.2825
Uncoated 2-49	328.43	1460.9	436.68	0.2867
Uncoated 1-7	339.18	1508.7	450.98	0.2961
Uncoated 2-40	353.50	1572.4	470.02	0.3086
Uncoated 1-18	365.87	1627.5	486.46	0.3194
Uncoated 2-37	461.19	2051.5	613.20	0.4026
Uncoated 2-31	505.81	2250.0	672.53	0.4415
Uncoated 1-21	557.95	2481.9	741.86	0.4870
Uncoated 1-24	583.25	2594.4	775.49	0.5091
Uncoated 2-52	687.67	3058.9	914.33	0.6003
Uncoated 1-15	690.67	3072.3	918.32	0.6029
Uncoated 1-12	760.70	3383.8	1011.43	0.6640
Uncoated 2-46	1090.86	4852.4	1450.42	0.9522
Uncoated 1-9 (didn't break)	(1180.27)	(5250.1)	(1569.30)	(1.0303)
Uncoated 2-34	1366.21	6077.2	1816.53	1.1926
Mean	544.35	2421.4	723.77	0.4752
Standard Deviation	332.41	1478.6	441.97	0.2902
Uncoated, 12.7 mm/min	Failure load, lb	Failure load, N	Strength, MPa	Deflection, mm
Uncoated-3-18	346	1539.1	460.04	0.3020
Uncoated-3-2	368	1636.9	489.30	0.3212
Uncoated-3-14	368	1636.9	489.30	0.3212
Uncoated-3-12	402	1788.2	534.50	0.3509
Uncoated-3-38	433	1926.1	575.72	0.3780
Uncoated-3-28	441	1961.7	586.36	0.3850
Uncoated-3-40	594	2642.2	789.79	0.5185
Uncoated-3-6	624	2775.7	829.68	0.5447
Uncoated-3-16	654	2909.1	869.56	0.5709
Uncoated-3-10	663	2949.2	881.53	0.5787
Uncoated-3-34	710	3158.2	944.02	0.6198
Uncoated-3-4	766	3407.3	1018.48	0.6687
Uncoated-3-30	909	4043.4	1208.62	0.7935
Uncoated-3-36	964	4288.1	1281.74	0.8415
Uncoated-3-24	993	4417.1	1320.30	0.8668
Uncoated-3-32	1089	4844.1	1447.94	0.9506
Uncoated-3-22	1109	4933.1	1474.54	0.9681
Uncoated-3-20	1262	5613.7	1677.97	1.1016
Uncoated-3-8	1424	6334.3	1893.36	1.2430
Uncoated-3-26	1508	6707.9	2005.05	1.3164
Mean	781.35	3475.6	1038.89	0.6821
Standard Deviation	361.01	1605.9	480.00	0.3151

TABLE A-1. (Contd.)

Octadecyl SiMe ₂ Cl, 0.127 mm/min	Failure load, lb	Failure load, N	Strength, MPa	Deflection, mm
SiMe ₂ CiC18H37-3-3	359	1596.9	477.33	0.3134
SiMe ₂ CiC18H37-3-9	438	1948.3	582.37	0.3823
SiMe ₂ CiC18H37-3-23	477	2121.8	634.22	0.4164
SiMe ₂ CiC18H37-3-27	512	2277.5	680.76	0.4469
SiMe ₂ CiC18H37-3-37	519	2308.6	690.07	0.4530
SiMe ₂ CiC18H37-3-35	523	2326.4	695.39	0.4565
SiMe ₂ CiC18H37-3-25	526	2339.8	699.37	0.4592
SiMe ₂ CiC18H37-3-7	653	2904.7	868.24	0.5700
SiMe ₂ CiC18H37-3-15	695	3091.5	924.08	0.6067
SiMe ₂ CiC18H37-3-1	896	3985.6	1191.33	0.7821
SiMe ₂ CiC18H37-3-29	934	4154.6	1241.86	0.8153
SiMe ₂ CiC18H37-3-5	953	4239.2	1267.12	0.8319
SiMe ₂ CiC18H37-3-31	1027	4568.3	1365.51	0.8965
SiMe ₂ CiC18H37-3-17	1090	4848.6	1449.27	0.9515
SiMe ₂ CiC18H37-3-13	1197	5324.5	1591.54	1.0449
SiMe ₂ CiC18H37-3-33	1227	5458.0	1631.43	1.0711
SiMe ₂ CiC18H37-3-11	1430	6361.0	1901.34	1.2483
SiMe ₂ CiC18H37-3-21	1977	8794.1	2628.64	1.7258
SiMe ₂ CiC18H37-3-19	2816	12526.2	3744.18	2.4581
SiMe ₂ CiC18H37-3-39	2982	13264.6	3964.90	2.6030
Mean	1061.55	4722.0	1411.45	0.9266
Standard Deviation	744.87	3313.3	990.39	0.6502
Octadecyl SiMe ₂ Cl, 1.27 mm/min	Failure load, lb	Failure load, N	Strength, MPa	Deflection, mm
SiMe ₂ CiC18H37-1	319.82	1422.6	425.24	0.2792
SiMe ₂ CiC18H37-31	509.05	2264.4	676.84	0.4444
SiMe ₂ CiC18H37-23	608.02	2704.6	808.43	0.5308
SiMe ₂ CiC18H37-27	617.83	2748.2	821.47	0.5393
SiMe ₂ CiC18H37-35	626.62	2787.3	833.16	0.5470
SiMe ₂ CiC18H37-15	688.84	3064.1	915.89	0.6013
SiMe ₂ CiC18H37-7	923.30	4107.0	1227.63	0.8060
SiMe ₂ CiC18H37-17	1101.09	4897.9	1464.02	0.9612
SiMe ₂ CiC18H37-37	1135.68	5051.8	1510.01	0.9914
SiMe ₂ CiC18H37-13	1197.52	5326.8	1592.23	1.0453
SiMe ₂ CiC18H37-3	1349.05	6000.9	1793.71	1.1776
SiMe ₂ CiC18H37-9	1448.68	6444.1	1926.18	1.2646
SiMe ₂ CiC18H37-19	1550.66	6897.7	2061.77	1.3536
SiMe ₂ CiC18H37-5	1691.48	7524.1	2249.01	1.4765
SiMe ₂ CiC18H37-21	1825.05	8118.2	2426.60	1.5931
SiMe ₂ CiC18H37-29	2072.21	9217.7	2755.23	1.8089
SiMe ₂ CiC18H37-11	2227.29	9907.5	2961.43	1.9442
SiMe ₂ CiC18H37-33	2550.63	11345.8	3391.34	2.2265
SiMe ₂ CiC18H37-25	3276.06	14572.6	4355.88	2.8597
SiMe ₂ CiC18H37-39	3295.14	14657.5	4381.25	2.8764
Mean	1450.70	6453.0	1928.87	1.2663
Standard Deviation	874.92	3891.8	1163.30	0.7637

TABLE A-1. (Contd.)

Octadecyl SiMe₂Cl, 12.7 mm/min	Failure load, lb	Failure load, N	Strength, MPa	Deflection, mm
SiMe ₂ CiC18H ₃₇ -2-23	512	2277.5	680.76	0.4469
SiMe ₂ CiC18H ₃₇ -2-7	567	2522.1	753.89	0.4949
SiMe ₂ CiC18H ₃₇ -2-15	602	2677.8	800.42	0.5255
SiMe ₂ CiC18H ₃₇ -2-1	684	3042.6	909.45	0.5971
SiMe ₂ CiC18H ₃₇ -2-39	712	3167.1	946.68	0.6215
SiMe ₂ CiC18H ₃₇ -2-3	960	4270.3	1276.43	0.8380
SiMe ₂ CiC18H ₃₇ -2-31	995	4426.0	1322.96	0.8686
SiMe ₂ CiC18H ₃₇ -2-21	1025	4559.4	1362.85	0.8947
SiMe ₂ CiC18H ₃₇ -2-19	1054	4688.4	1401.41	0.9201
SiMe ₂ CiC18H ₃₇ -2-27	1113	4950.9	1479.86	0.9716
SiMe ₂ CiC18H ₃₇ -2-29	1707	7593.1	2269.64	1.4901
SiMe ₂ CiC18H ₃₇ -2-33	1789	7957.9	2378.67	1.5616
SiMe ₂ CiC18H ₃₇ -2-5	1965	8740.8	2612.68	1.7153
SiMe ₂ CiC18H ₃₇ -2-25	2000	8896.4	2659.22	1.7458
SiMe ₂ CiC18H ₃₇ -2-9	2140	9519.2	2845.36	1.8680
SiMe ₂ CiC18H ₃₇ -2-37	2537	11285.1	3373.22	2.2146
SiMe ₂ CiC18H ₃₇ -2-13	2917	12975.5	3878.47	2.5463
SiMe ₂ CiC18H ₃₇ -2-11	3238	14403.3	4305.28	2.8265
SiMe ₂ CiC18H ₃₇ -2-17	3299	14674.7	4386.38	2.8798
SiMe ₂ CiC18H ₃₇ -2-35	3312	14732.5	4403.67	2.8911
Mean	1656.40	7368.0	2202.37	1.4459
Standard Deviation	978.65	4353.3	1301.22	0.8543
Fluoroalkyl SiMeCl₂, 1.27 mm/min	Failure load, lb	Failure load, N	Strength, MPa	Deflection, mm
SiMeCl ₂ C ₈ H ₄ F ₁₃ -2	223.85	995.7	297.63	0.1954
SiMeCl ₂ C ₈ H ₄ F ₁₃ -24	457.75	2036.2	608.63	0.3996
SiMeCl ₂ C ₈ H ₄ F ₁₃ -36	466.19	2073.7	619.85	0.4069
SiMeCl ₂ C ₈ H ₄ F ₁₃ -40	513.03	2282.1	682.13	0.4478
SiMeCl ₂ C ₈ H ₄ F ₁₃ -20	595.12	2647.2	791.28	0.5195
SiMeCl ₂ C ₈ H ₄ F ₁₃ -8	616.58	2742.7	819.81	0.5382
SiMeCl ₂ C ₈ H ₄ F ₁₃ -14	631.93	2811.0	840.22	0.5516
SiMeCl ₂ C ₈ H ₄ F ₁₃ -28	649.76	2890.3	863.93	0.5672
SiMeCl ₂ C ₈ H ₄ F ₁₃ -12	692.35	3079.7	920.56	0.6044
SiMeCl ₂ C ₈ H ₄ F ₁₃ -10	795.22	3537.3	1057.33	0.6942
SiMeCl ₂ C ₈ H ₄ F ₁₃ -26	802.00	3567.5	1066.35	0.7001
SiMeCl ₂ C ₈ H ₄ F ₁₃ -30	834.54	3712.2	1109.61	0.7285
SiMeCl ₂ C ₈ H ₄ F ₁₃ -16	1034.19	4600.3	1375.07	0.9028
SiMeCl ₂ C ₈ H ₄ F ₁₃ -18	1103.76	4909.8	1467.57	0.9635
SiMeCl ₂ C ₈ H ₄ F ₁₃ -32	1124.60	5002.5	1495.28	0.9817
SiMeCl ₂ C ₈ H ₄ F ₁₃ -34	1182.75	5261.1	1572.60	1.0324
SiMeCl ₂ C ₈ H ₄ F ₁₃ -4	1269.56	5647.3	1688.02	1.1082
SiMeCl ₂ C ₈ H ₄ F ₁₃ -22	1438.15	6397.2	1912.18	1.2554
SiMeCl ₂ C ₈ H ₄ F ₁₃ -6	1512.49	6727.9	2011.02	1.3203
SiMeCl ₂ C ₈ H ₄ F ₁₃ -38	3227.23	14355.4	4290.96	2.8171
Mean	958.55	4263.9	1274.50	0.8367
Standard Deviation	636.81	2832.7	846.71	0.5559

TABLE A-1. (Contd.)

Aquaphobe CF, 1.27 mm/min	Failure load, lb	Failure load, N	Strength, MPa	Deflection, mm
Aquaphobe CF-2-38	246.35	1095.8	327.55	0.2150
Aquaphobe CF-1-5	317.48	1412.2	422.12	0.2771
Aquaphobe CF-1-11	493.97	2197.3	656.79	0.4312
Aquaphobe CF-1-20	556.38	2474.9	739.77	0.4857
Aquaphobe CF-2-50	570.36	2537.1	758.36	0.4979
Aquaphobe CF-2-41	580.03	2580.1	771.21	0.5063
Aquaphobe CF-1-26	661.52	2942.6	879.56	0.5775
Aquaphobe CF-2-47	674.38	2999.8	896.66	0.5887
Aquaphobe CF-2-44	686.28	3052.7	912.48	0.5991
Aquaphobe CF-2-53	913.86	4065.1	1215.08	0.7977
Aquaphobe CF-2-59	921.70	4099.9	1225.50	0.8046
Aquaphobe CF-1-3	940.46	4183.4	1250.44	0.8209
Aquaphobe CF-1-14	942.43	4192.1	1253.06	0.8227
Aquaphobe CF-2-35	988.82	4398.5	1314.74	0.8632
Aquaphobe CF-2-29	1010.60	4495.4	1343.70	0.8822
Aquaphobe CF-1-23 (didn't break)	(1180.26)	(5250.1)	(1569.29)	(1.0303)
Aquaphobe CF-1-17 (didn't break)	(1180.27)	(5250.1)	(1569.30)	(1.0303)
Aquaphobe CF-1-8 (didn't break)	(1180.28)	(5250.1)	(1569.31)	(1.0303)
Mean	780.30	3471.0	1037.50	0.6811
Standard Deviation	288.61	1283.8	383.73	0.2519
Note: Excluded -2-32 and -2-56 (91.9, 78.8 lb).				
Aquaphobe CM, 1.27 mm/min	Failure load, lb	Failure load, N	Strength, MPa	Deflection, mm
Aquaphobe CM-1-2	159.86	711.1	212.55	0.1395
Aquaphobe CM-1-13	362.57	1612.8	482.08	0.3165
Aquaphobe CM-2-30	449.25	1998.4	597.33	0.3922
Aquaphobe CM-2-39	463.07	2059.8	615.70	0.4042
Aquaphobe CM-2-54	512.82	2281.1	681.85	0.4476
Aquaphobe CM-2-33	544.12	2420.4	723.47	0.4750
Aquaphobe CM-1-1	570.00	2535.5	757.88	0.4976
Aquaphobe CM-1-10	617.12	2745.1	820.53	0.5387
Aquaphobe CM-1-22	629.10	2798.4	836.46	0.5492
Aquaphobe CM-2-57	747.99	3327.2	994.53	0.6529
Aquaphobe CM-2-48	777.90	3460.3	1034.30	0.6790
Aquaphobe CM-1-25	848.37	3773.7	1128.00	0.7406
Aquaphobe CM-2-51	874.13	3888.3	1162.25	0.7630
Aquaphobe CM-1-16	902.52	4014.6	1200.00	0.7878
Aquaphobe CM-2-45	911.05	4052.6	1211.34	0.7953
Aquaphobe CM-1-19 (didn't break)	(1180.28)	(5250.1)	(1569.31)	(1.0303)
Aquaphobe CM-1-6 (didn't break)	(1180.32)	(5250.3)	(1569.36)	(1.0303)
Aquaphobe CM-2-36	1229.07	5467.2	1634.18	1.0729
Aquaphobe CM-2-42	1975.24	8786.3	2626.30	1.7242
Mean	786.04	3496.5	1045.13	0.6861
Standard Deviation	407.29	1811.7	541.53	0.3555

TABLE A-1. (Contd.)

Unetched, 1.27 mm/min	Failure load, lb	Failure load, N	Strength, MPa	Deflection, mm
Unetched-10	101.60	451.9	135.09	0.0887
Unetched-4	105.25	468.2	139.94	0.0919
Unetched-7	116.80	519.6	155.30	0.1020
Unetched-3	128.08	569.7	170.30	0.1118
Unetched-2	144.38	642.2	191.97	0.1260
Unetched-6	153.76	684.0	204.44	0.1342
Unetched-9	162.82	724.3	216.49	0.1421
Unetched-5	187.53	834.2	249.34	0.1637
Unetched-8	207.94	925.0	276.48	0.1815
Unetched-1	259.28	1153.3	344.74	0.2263
Mean	156.74	697.2	208.41	0.1368
Standard Deviation	49.88	221.9	66.32	0.0435
Note: no tape or paper on these samples				
Uncoated, first data, 1.27 mm/min	Failure load, lb	Failure load, N	Strength, MPa	Deflection, mm
Uncoated-5	371.74	1653.6	494.27	0.3245
Uncoated-1	386.90	1721.0	514.43	0.3377
Uncoated-3	409.28	1820.6	544.18	0.3573
Uncoated-4	525.70	2338.4	698.98	0.4589
Uncoated-2	643.56	2862.7	855.68	0.5618
Mean	467.44	2079.3	621.51	0.4080
Standard Deviation	115.59	514.2	153.68	0.1009
Note: Since there was no tape or paper on these samples, these data were not averaged in with that of the other uncoated samples.				

Table A-2. Failure Load and Other Data for Pyrocera Tests.

Sample #	Width, mm	Thickness, mm	Load rate, lb/min	Alignment	Failure load, lb	Failure load, N
1	5.159	2.558	7.58	<0.076 mm	28.07	124.9
2	5.116	2.644	8.04	<0.076 mm	28.75	127.9
3	5.136	2.543	7.46	<0.076 mm	26.22	116.6
4	5.149	2.616	7.92	<0.076 mm	28.28	125.8
5	5.174	2.540	7.50	<0.076 mm	24.49	108.9
6	5.151	2.535	7.44	<0.076 mm	23.70	105.4
7	5.149	2.535	7.44	<0.013 mm	34.76	154.6
8	5.103	2.621	7.88	"locktite"	40.54	180.3
9	5.146	2.540	7.46	"locktite"	40.12	178.5
10	5.151	2.540	7.47	"locktite"	35.66	158.6
11	5.169	2.537	7.48	"locktite"	39.90	177.5
12	5.177	2.540	7.51	"locktite"	36.39	161.9
13	5.098	2.647	8.03	"locktite"	58.44	260.0
14	5.141	2.535	7.43	"locktite"	55.07	245.0
15	5.136	2.537	7.43	"locktite"	39.81	177.1
16	5.103	2.631	7.94	2 nd "locktite"	48.67	216.5
17	5.164	2.540	7.49	2 nd "locktite"	50.26	223.6
18	5.110	2.624	7.91	2 nd "locktite"	41.08	182.7
19	5.103	2.631	7.94	2 nd "locktite"	57.36	255.2
20	5.105	2.637	7.98	2 nd "locktite"	57.89	257.5

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