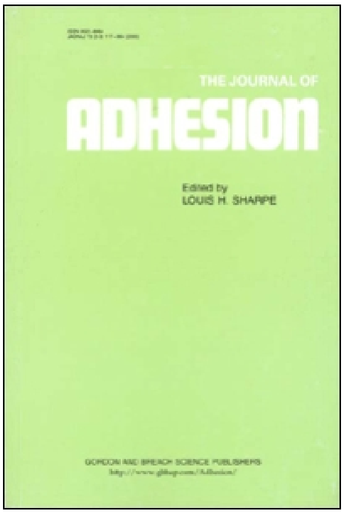

The Journal of Adhesion
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713453635

Adhesive Strength of Rubber Bonded to Al_2O_3 Surfaces: The Role of Chemistry and Morphology

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To cite this Article Browning, J. , Newman, N. , Ghetzler, R. , Mast, M. and Swendson, B.(2001) 'Adhesive Strength of Rubber Bonded to Al_2O_3 Surfaces: The Role of Chemistry and Morphology', The Journal of Adhesion, 75: 2, 229 — 253
To link to this Article: DOI: 10.1080/00218460108029602
URL: http://dx.doi.org/10.1080/00218460108029602

J. Adhesion, 2001, Vol. 75, pp. 229–253
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Adhesive Strength of Rubber Bonded to Al_2O_3 Surfaces: The Role of Chemistry and Morphology

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(Received 10 February 2000; In final form 21 August 2000)

Tensile pull tests were performed on polydimethylsiloxane (PDMS) rubber bonded to single-crystal and ceramic Al_2O_3 substrates prepared using several surface treatments. An accurate and reliable mechanical test method was developed using a frustro-conical geometry to eliminate the influence of tri-axial stress effects at the periphery. Properties of the sample surface were quantified prior to encapsulation using surface science techniques. Chemical cleaning of atomically-smooth sapphire resulted in a significantly larger joint strength (1 MPa) than attained from as-received substrates (0.448 MPa). The introduction of a significant amount of contamination ($\sim$ 25 nm thick) from isopropyl alcohol (IPA) residue resulted in even weaker adhesion (0.241 MPa). Abrasive cleaning of sapphire using fine ($\sim$ 100 micron) Al_2O_3 beads created more 0.5–1.0 micron topographic features and significantly higher joint strengths (1.103 MPa) than for similarly-prepared surfaces using coarse ($\sim$ 350 micron) Al_2O_3 beads (0.621 MPa). The application of a titanate-modified silicone primer resulted in the greatest joint strength encountered ($>$ 1.207 MPa), even when significant levels of surface contamination were present prior to priming. Adhesion to ceramic Al_2O_3 substrates was systematically stronger than to sapphire for as-received samples (0.965 MPa), abrasively-cleaned surfaces (1.034 MPa), and surfaces containing IPA residue (0.827 MPa) as a result of the rougher topography.

Keywords: Adhesion; Interface; PDMS; Rubber; Ceramic; Morphology; Sapphire substrate

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INTRODUCTION

Silicone elastomers are commonly used for encapsulation of microwave power modules (MPM) and other airborne electronic systems to provide environmental protection and structural support for electronic components. Silicones are ideally suited for electronic packaging applications due to their temperature and aging stability, resistance to oxidative degradation, stable dielectric nature, and opacity to infrared radiation [1]. Device reliability does, however, depend upon the formation of strong and uniform elastomer/ceramic interfacial bonds. In order to attain this goal, an investigation has been performed to develop methods to quantify the mechanical performance of interfaces, as well as elucidate the key interfacial properties needed to obtain strong and reliable bonding.

For this study, a wide range of surface chemical and topographical configurations were investigated. Single-crystal sapphire substrates (RMS (root mean square) roughness < 7 Å) were used as a model atomically-smooth surface that is essentially defect free. Because of the flatness and uniformity of the as-received substrates, changes in the surface after sample preparation could be precisely and accurately determined. Chemically-identical polycrystalline Al_2O_3 ceramic substrates (RMS roughness > 580 nm) were chosen because of their high-defect density and because of their practical utility in airborne-electronic systems. The key objective of this work was to understand the critical properties of interfaces that optimize strength, uniformity and reliability of the bond.

EXPERIMENT

To understand the effects degreasing and abrasive cleaning techniques have on maximizing adhesive joint strength, sapphire and polycrystalline Al_2O_3 disk substrates (10.16 mm × 1.39 mm) were prepared using the following techniques:

(1) Degreasing with 99.5% acetone (Fisher, Scientific grade)/ethanol (Pharmco: USP ethyl alcohol)/DI water (A/E/DI) for 2 minutes each in an ultrasonic cleaner at room temperature.
(2) Degreasing with 99.9% isopropyl alcohol (IPA) (Fisher, Scientific grade) for 2 minutes in an ultrasonic cleaner at room temperature.

(3) Plasma cleaning with argon and oxygen (90% – 10%) for several seconds.
(4) Abrasive cleaning using coarse Al_2O_3 beads (250 – 425 μm grit) at 280 kPa for 30 seconds.
(5) Abrasive cleaning using fine (powder) Al_2O_3 beads (80 – 120 μm grit), at 280 kPa for several seconds.
(6) Chemical priming (General Electric, GE SS-4155) applied by brush, then cured in a custom-built condensation chamber at 65% RH and ~40°C (± 1°C) for 1 hour.

Substrate surface morphologies were characterized [2, 3] by Atomic Force Microscopy (AFM) (Digital Instrument's Nanoscope IIIa Scanning Probe Microscope (SPM)), Scanning Electron Microscopy (SEM) (Hitachi S-4500) and Energy Dispersive Spectroscopy (EDS) (Princeton Gamma-Tech, Prism-60).

Prepared substrates were encapsulated to bolts in a casting mold, as illustrated in Figure 1. The polydimethylsiloxane elastomer (Dow Corning; DC-170), is composed of a 2-part reaction mixture having a vinyl-terminated oligomeric dimethylsiloxane, a methyl hydrogen siloxane as a cross-linking agent, and a platinum complex as a catalyst for the hydrosilation reaction [1]. A 50 : 50 ratio was mixed using a two-part epoxy-dispensing gun. This mixture was de-gassed in 100 mT vacuum for 20 minutes prior to encapsulation. The elastomer was subsequently injected into the evacuated mold in ~10 minutes. An adhesive layer thickness of 0.762 mm ± 0.025 (0.030 in) was chosen to insure uniform substrate coverage due to the viscous nature of the elastomer, while minimizing the influence of edge effects on mechanical testing [4].

The frustro-conical geometry shown in Figure 1 was designed to essentially eliminate the tri-axial stress distributions resulting from necking through the production of a radial outward stress component [4 – 6]. This method, therefore, allows for measurement of a more accurate and reliable "pure" tensile strength of the adhesive bond [7]. Finite element modeling [4, 8] of this geometry has shown that this method yields stress concentration at the edge by a factor of ~1.17 (Fig. 2) in contrast to up to ~2.59 for conventional cylindrical adhesive layer configurations.

To validate this approach experimentally, pull tests were performed for a range of adhesive layer thickness (h) while using a substrate of identical radius (a) [9]. The results are compared with the results of

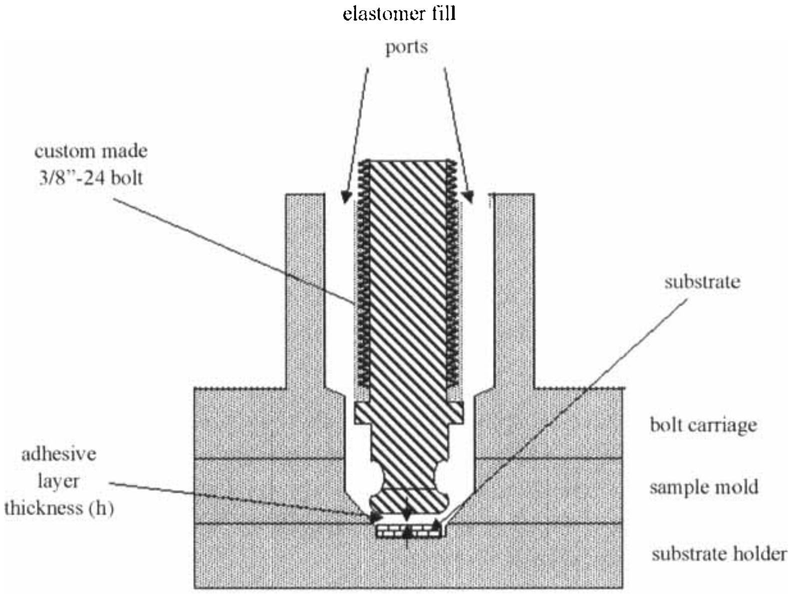


FIGURE 1 Schematic side view of sample encapsulation fixture. When fastened together, the bolt and substrate form a 0.76 mm (0.030 in) channel for the adhesive layer thickness. From Ref. [7].

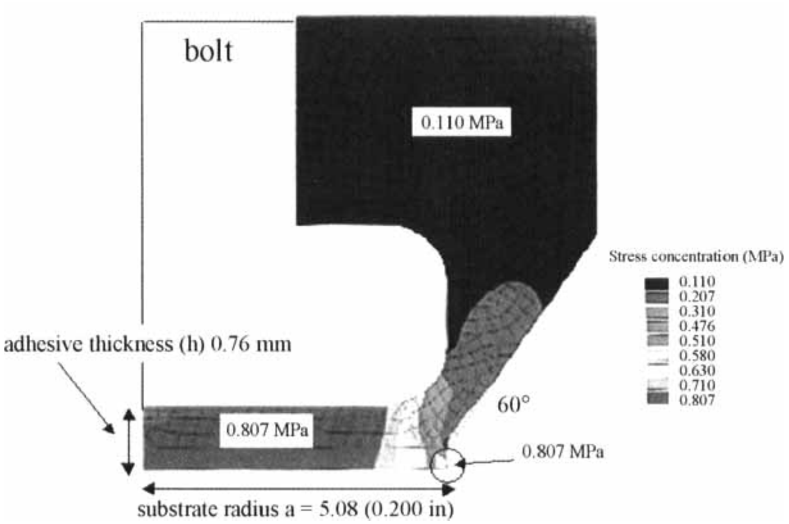


FIGURE 2 Finite element stress analysis at 0.689 MPa (100 psi) for a 0.76 mm (0.030 in) thick frustro-cononical adhesive layer. An edge stress concentration factor of 1.17 occurs for this geometry (Ansys 5.4). (See Color Plate V).

finite element modeling. The modeling results quantitatively predicted the relationship of the adhesive layer thickness (*i.e.*, *a*/*h* ratios), stress–strain relationships, and peripheral failure stress. As shown in Figure 3, higher tensile failures are observed for similarly prepared sapphire substrates (bare – unprocessed) at higher *a*/*h* ratios. The apparent Young's modulus (slope) for each experiment does not increase significantly with thickness for thinner adhesive layers (Fig. 4). Rather, the apparent modulus remained approximately the same (or decreased) for thinner adhesive layers. This work verifies that the calculations accurately model the stress distributions for this geometry and validate the advantages of this test fixture. A more detailed description of this method is reported in Ref. [7].

Polymerization (curing) was performed under atmospheric conditions in two steps: at 65°C for 16 hours then at 100°C for 4 hours [4]. The sample was detached from the mold and a second (3/8)-24 inch bolt (sanded parallel to threading) was affixed to the opposite substrate face using commercial grade Epoxy Patch® resin. The epoxy was set to cure for an additional 24 hours to yield the final sample configuration.

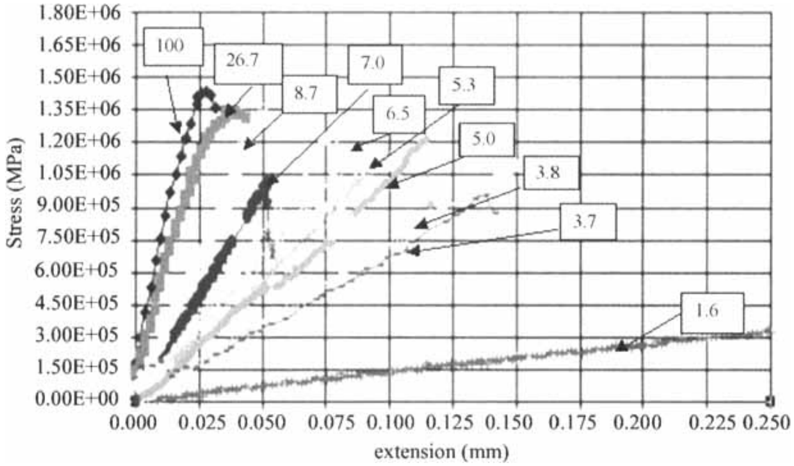


FIGURE 3 Experimental pull test data for different *a*/*h* ratios. Adhesive layer thickness (*h*) was varied between 0.051–3.15 mm while using a substrate of identical radius (*a*) of 5.08 mm (0.200 in). The decrease in maximum adhesive tensile strength as thickness is increased results from an increase in edge stress concentration. (See Color Plate VI).

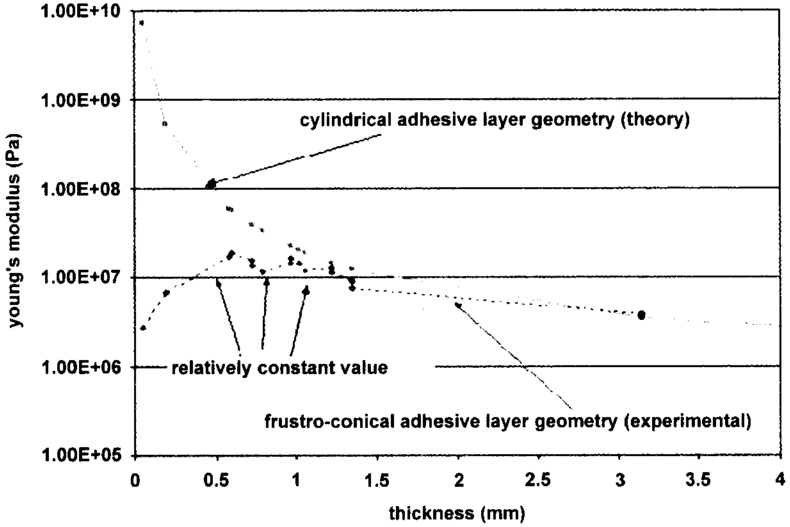


FIGURE 4 Apparent Young's modulus estimations for cylindrical and frustro-cononical adhesive layer thickness. The relatively constant value observed for the frustro-cononical geometry indicates minimal variations in edge stress for different adhesive layer thickness.

The silicone primer (General Electric, SS-4155) consists of a 1-butanol, titanium(4+) salt (5–10%), a tetraethylorthosilicate (10–30%) and an aliphatic hydrocarbon solvent (60–80%) [10]. The final product, an alkoxysilane with a titanium ester bond, was attained through a condensation cure (65% relative humidity (RH) at slightly elevated temperatures (40°C)).

Tensile strength measurements were performed using a Sintech 20/G testing workstation with a 5000 N load cell (testing resolution of 0.002 mm/pulse) at an extension rate of 0.4 mm/sec.

RESULTS

Measurement results on the surface topography of sapphire and ceramic substrates are summarized in Table I. Because of the extreme flatness and uniformity of the sapphire substrates, the influence of sample preparation on the roughness of these samples is readily

TABLE I AFM surface roughness values for Al_2O_3 substrates prepared with various surface treatments

	Sapphire (single crystal)	*Ceramic (polycrystalline)*
Processing technique	*Surface RMS roughness*	*Surface RMS roughness*
bare (as received)	0.54 (± 0.1) nm	580 (± 35) nm
plasma cleaning (Ar and O_2)	0.42 (± 0.1) nm	
acetone/ethanol/DI degreasing	0.34 (± 0.1) nm	563 (± 23) nm
isopropyl alcohol (IPA) degreased	6.43 (± 1.3) nm	573 (± 33) nm
isopropyl alcohol (IPA) degreased: particles visible	23.12 (± 4.2) nm	573 (± 26) nm
coarse glass beading (250 – 425 um) (incomplete processing)	176 (± 63) nm	–
coarse glass beading (250 – 425 um)	160 (± 35) nm	594 (± 42) nm
fine glass beading (80 – 120 um)	50 (± 18) nm	–
fine glass beading (80 – 120 um) and primed	349 (± 23) nm	–
coarse glass beading (250 – 425 um) and primed	356 (± 22) nm	496 (± 23) nm
primed	340 (± 28) nm	480 (± 33) nm

apparent. In contrast, the rough and uneven topography of the ceramic samples makes it more difficult to discern the changes from each surface treatment.

The maximum tensile stress before plastic deformation is used to quantify the bond strength. The force at which failure in adhesion occurred is defined by the occurrence of a sudden change in extension during the stress – strain measurement. Samples prepared using primed surfaces were the only ones found to fail within the elastomer (*i.e.*, cohesively). This occurred at several large tears centralized near the sample center. A summary of the tensile strengths for each substrate is shown in Table II.

MORPHOLOGY

Representative images of sapphire and ceramic substrates are shown in Figures 5 and 6. Individual roughness profiles for all sapphire surfaces are shown in Figure 7.

In general, bonding to unprocessed ceramic substrates gave an adhesive strength twice as large as that to atomically-smooth sapphire.

TABLE II Bond tensile strength data for Al_2O_3 substrate/elastomer systems prepared with various surface treatments

	Sapphire (single crystal)		*Ceramic (polycrystalline)*	
Processing technique	*Tensile strength (MPa) (quantity)*	*Failure type*	*Tensile strength (MPa) (quantity)*	*Failure type*
bare (as received)	0.445 (± 0.015) (3)	in adhesion	0.965 (± 0.029) (3)	in adhesion
plasma cleaning (Ar and O_2)	0.943 (1)	in adhesion	–	
acetone/ethanol/DI degreasing	1.044 (1)	in adhesion	0.993 (1)	in adhesion
isopropyl alcohol (IPA) degreased	1.002 (1)	in adhesion	0.972 (1)	in adhesion
isopropyl alcohol (IPA) degreased: particles visible	0.242 (± 0.069) (3)	in adhesion	0.834 (1)	in adhesion
coarse glass beading (250 – 425 um) (incomplete processing)	0.540 (1)	in adhesion	1.048 (± 0.021) (3)	in adhesion
coarse glass beading (250 – 425 um)	0.627 (1)	in adhesion	1.034 (1)	in adhesion
fine glass beading (80 – 120 um)	1.101 (± 0.010) (3)	in adhesion	–	
fine glass beading (80 – 120 um) and primed	1.217 (1)	cohesive	–	
coarse glass beading (250 – 425 um) and primed	1.232 (0.008) (3)	cohesive	1.234 (± 0.016) (3)	cohesive
isopropyl alcohol degrease and primed	1.222 (1)	cohesive	1.234 (1)	cohesive
primed	1.236 (± 0.006) (3)	cohesive	1.248 (± 0.008) (3)	cohesive

Abrasive cleaning of the polycrystalline substrate caused a small (< 5%), but measurable, improvement in bond strength. In contrast, abrasive cleaning of atomically-smooth sapphire was shown to alter the surface topography and adhesive joint strength significantly. Sapphire substrates abrasively cleaned by coarse (250 – 425 micron)

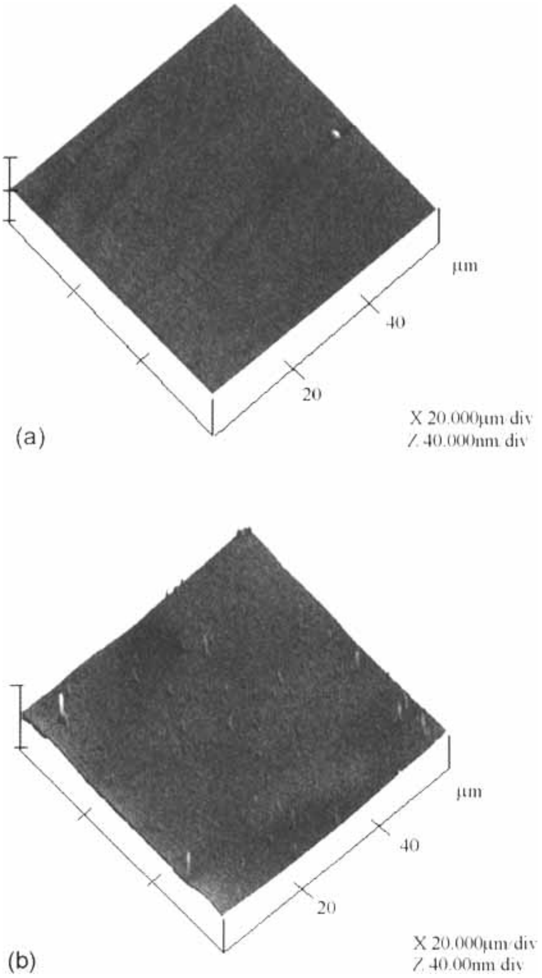


FIGURE 5 AFM and SEM representative data for sapphire substrates. AFM images are for (a) bare (b) plasma cleaned in Ar and O_2 (c) isopropyl alcohol (IPA) degreased (d) abrasive cleaning (e) priming. SEM images are representative of (f) bare (g) abrasive cleaned (h) primed. All images are for 60 × 60 micron area.

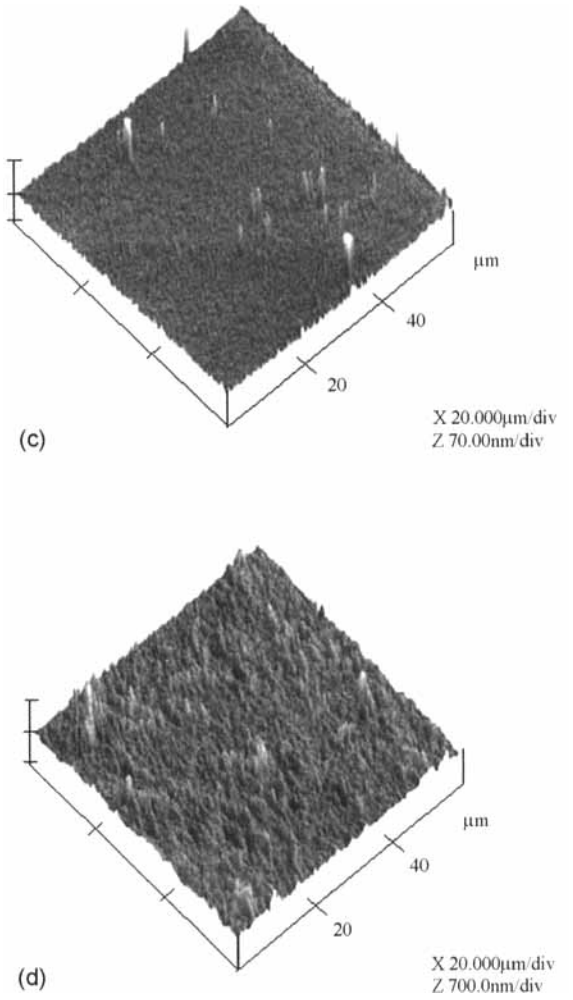


FIGURE 5 (Continued).

and fine (80 – 120 micron) Al_2O_3 beads have RMS (root mean square) [11] roughness of 160 nm (± 35) and 50 nm (± 18) and have adhesive strengths of 0.621 MPa and 1.096 MPa, respectively. When abrasive cleaning was performed for only a few seconds, a similar *overall* RMS roughness was produced, but with a significant standard deviation in

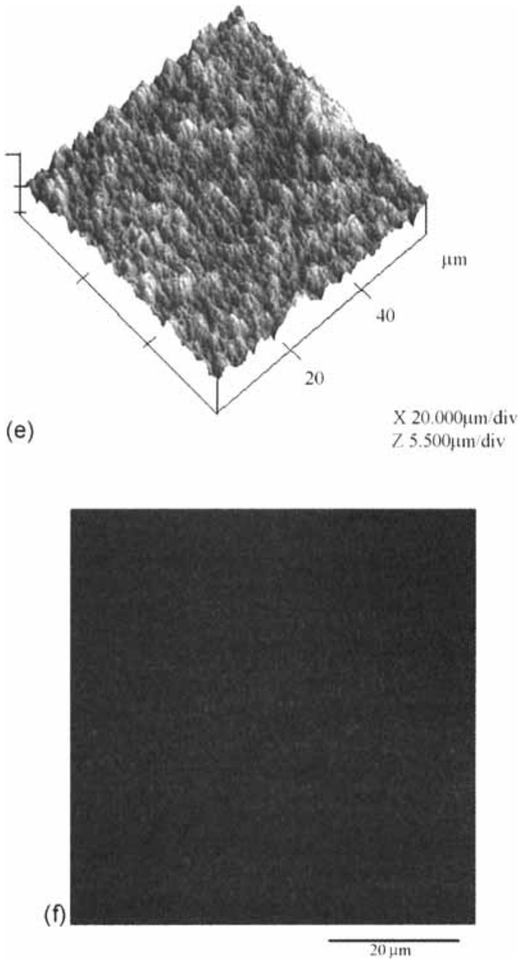


FIGURE 5 (Continued).

the value. These incompletely prepared surfaces where found to fail at 25% smaller forces than for substrates Al_2O_3 beaded for longer times (30 seconds.)

Although surface RMS roughness is generally taken as a "figure of merit" of topography, this value averages overall feature sizes. Power Spectral Density (PSD) [11] determinations from AFM images convey

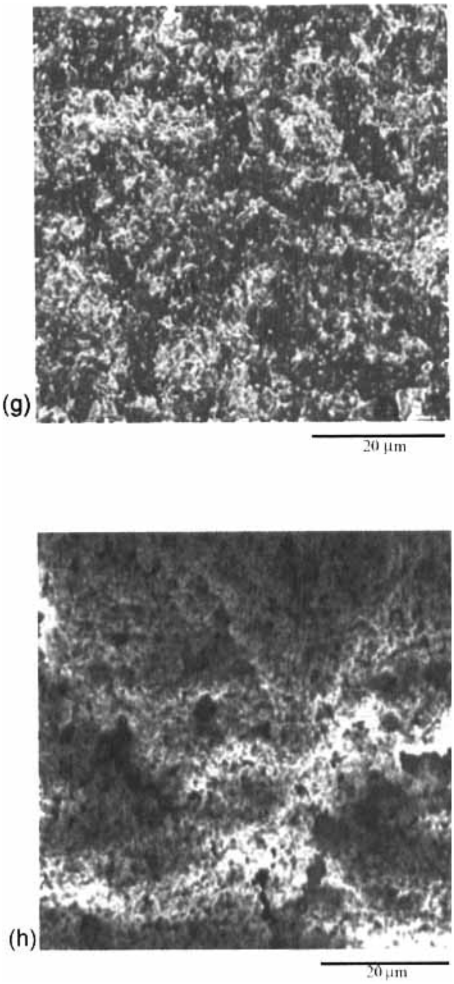


FIGURE 5 (Continued).

detailed information on the density of topographic feature sizes. For 5 seconds of coarse Al_2O_3 beading, a majority of defects are observed at larger feature sizes (5 – 12 microns) and < 2 microns. For 30 seconds of the same treatment, more small (*i.e.*, 3 – 6 micron) features are observed and with a smaller number of overall large features. Fine Al_2O_3 beading produces proportionally more 0.5 – 1.0 micron features than other processes.

The observed changes in ceramic topography resulting from each surface preparation are consistent with the work on sapphire. However, the rough original topography often makes it much more difficult to quantify these changes.

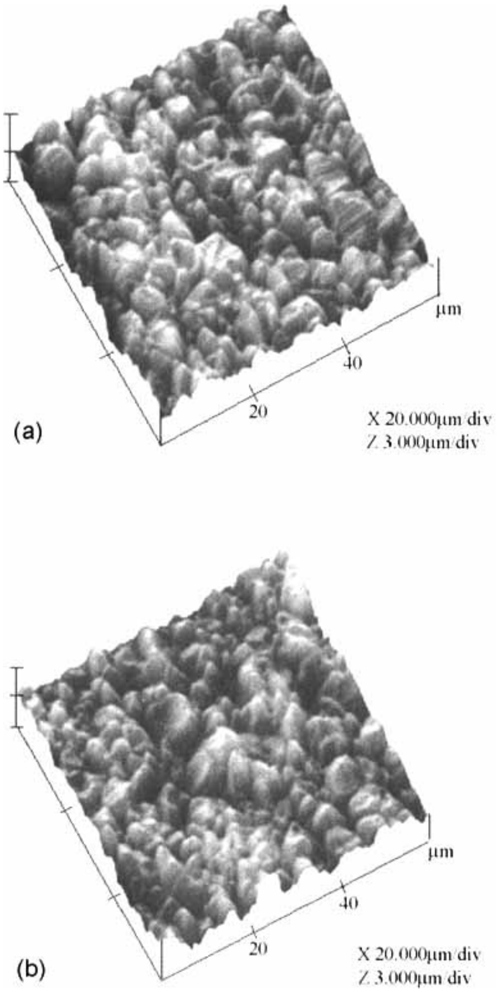


FIGURE 6 AFM and SEM representative data for polycrystalline substrates. AFM images are for (a) bare (b) plasma cleaned in Ar and O_2 (c) isopropyl alcohol (IPA) degreased (d) abrasive cleaning (e) priming. SEM images are representative of (f) bare (g) abrasive cleaned (h) primed. All images are for 60 × 60 micron area.

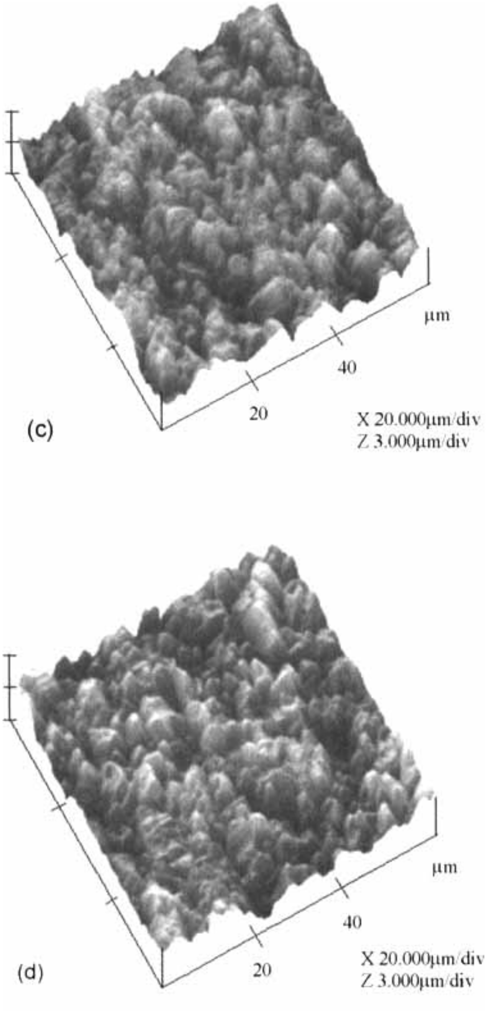


FIGURE 6 (Continued).

CHEMISTRY

IPA residue on degreased sapphire substrates formed an additional ~25 nm surface roughness. AES (Auger Electron Spectroscopy) showed that this layer is predominantly carbon with a few percent

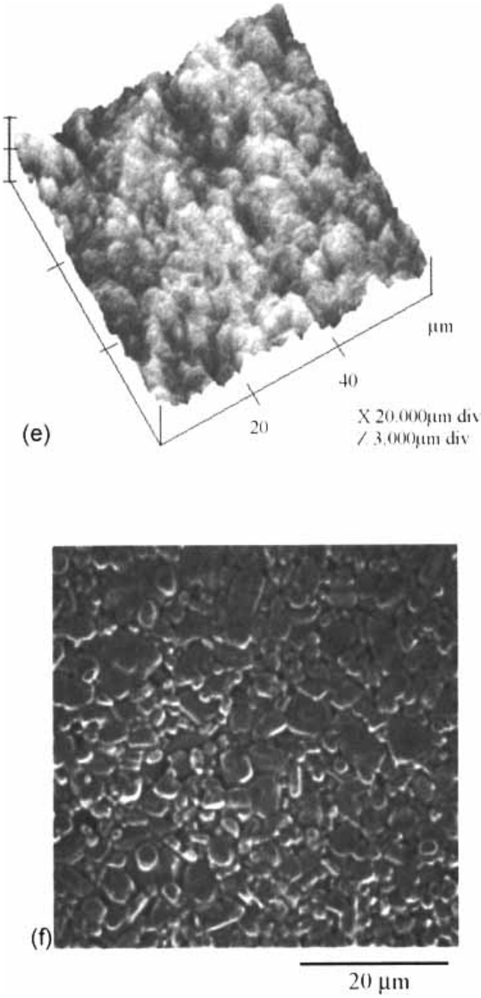


FIGURE 6 (Continued).

Na and Pb. Plasma cleaning removed the IPA residue and did not significantly alter surface roughness.

Acetone/Ethanol/DI degreased substrates indicated a smaller RMS roughness than for bare substrates, indicating that degreasing removed contaminates found on the "as-received" substrates. This technique resulted in a RMS surface roughness of 0.35 nm (± 0.10).

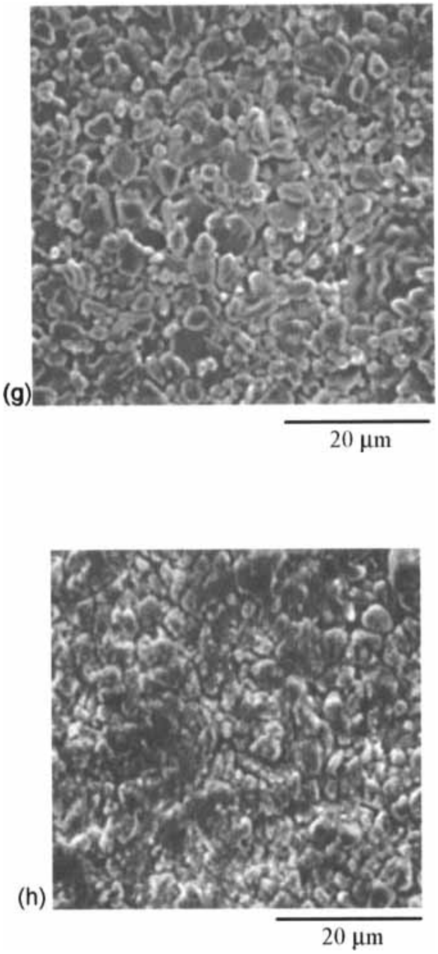


FIGURE 6 (Continued).

Substrate priming was shown to result in a significant increase in joint strength. Cohesive/plastic failure was observed for all primed substrates, regardless of pre-processing techniques. Precipitates in the primer dominate the topography of primed substrates. Polycrystalline Al_2O_3 systems showed a decrease in RMS roughness with application of a primer, while sapphire systems showed an increase

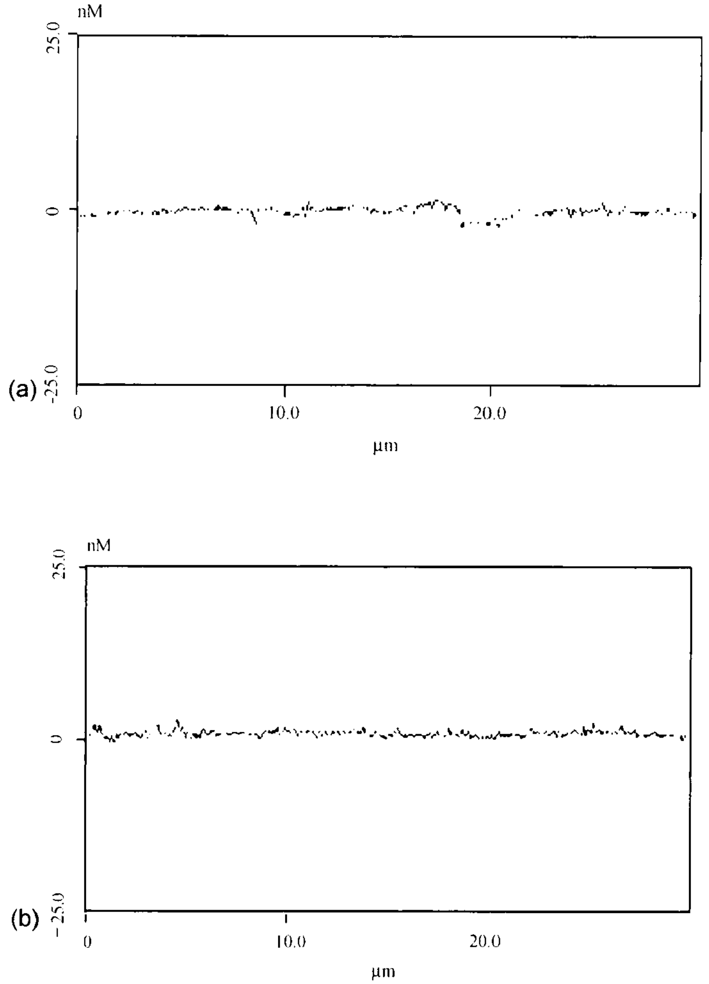


FIGURE 7 AFM section profiles for prepared surfaces on sapphire: (a) bare substrate, (b) plasma cleaned (Ar and O_2), (c) acetone/ethanol/DI degreasing, (d) isopropyl alcohol (IPA) degreasing, (e) fine (80 – 120 um) glass beading, (f) coarse (250 – 425 um) glass beading (g) incomplete coarse (250 – 425 um) glass beading, (h) primed substrate, (i) coarse (250 – 425 um) glass beading and primed. Note changes in *Z* scale.

in RMS roughness of ~1000 times over unprocessed bare substrates. Implications and advantages of priming are discussed in detail below.

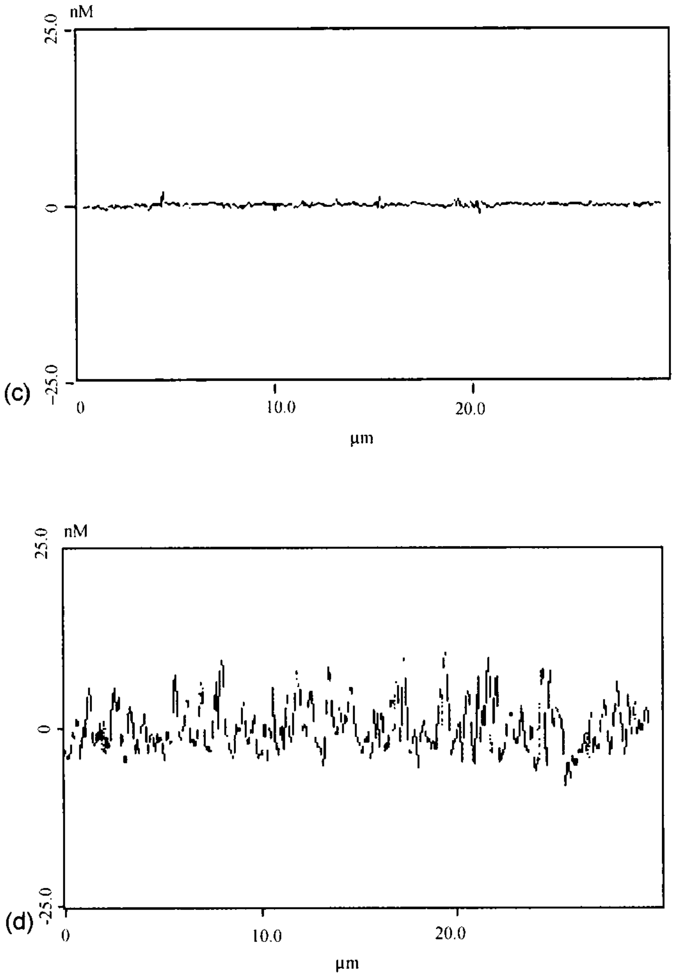


FIGURE 7 (Continued).

DISCUSSION

Topographical Effects on Adhesion

In general, enhanced adhesion following Al_2O_3 beading has been attributed to a combination of improved surface cleanliness and

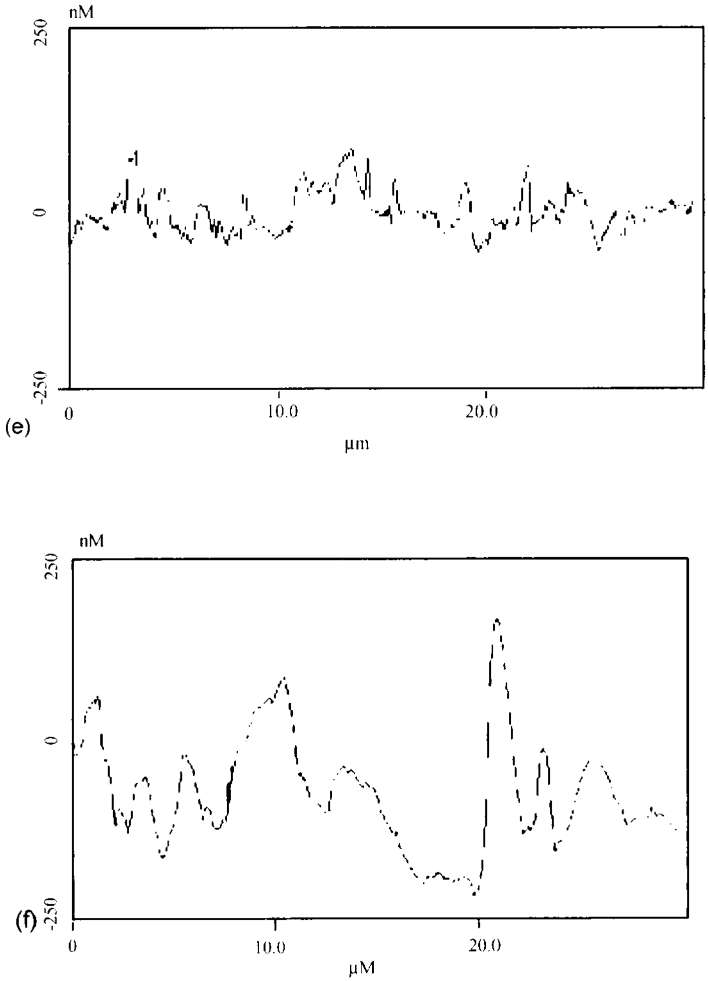


FIGURE 7 (Continued).

increased surface roughness [12, 13]. It is clear that achieving rough surfaces is critical to achieving high interfacial bonding. However, for chemically-identical interfaces, abrasive cleaning with 80–120 micron Al_2O_3 beads was shown to yield higher joint strengths than for similarly prepared substrates with 250–425 micron beads. Failure in adhesion is proposed by Gent [14] to occur at a site of high local stress within the joint. This initial failure will then propagate under the

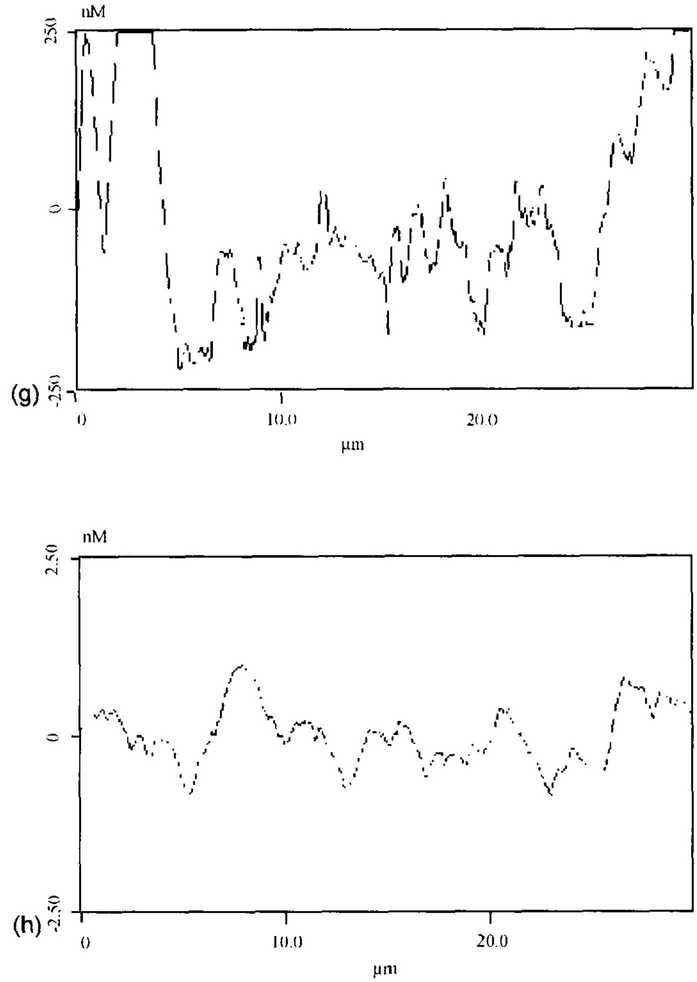


FIGURE 7 (Continued).

influence of the stress field, yielding eventual total interface failure. Smaller features on surfaces reduce the possibility for localized high stress concentrations. For surfaces beaded for short times with Al_2O_3, the large topographical irregularities (features of 5–12 micron and < 2 microns) reduced the joint strength by ~25% over similar surfaces beaded with Al_2O_3 for longer times. For sufficient processing times, surfaces prepared using fine Al_2O_3 beads had proportionally

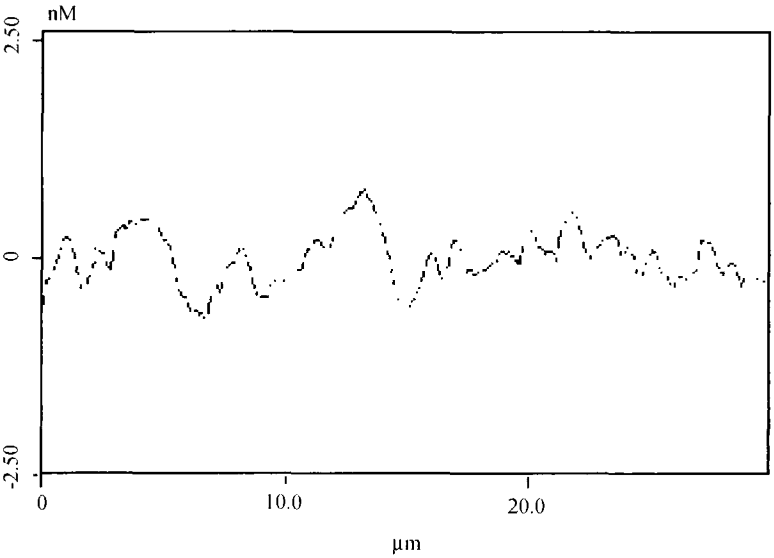
(i) 

FIGURE 7 (Continued).

more 0.5 – 1.0 micron features, while abrasive cleaning with coarse beads resulted in proportionally more large ~2 micron features. Surfaces with more small (*i.e.*, 0.5 – 1.0 micron) features (*i.e.*, cleaned with fine beads) had significantly higher joint strengths (45% improvement) than the surface containing larger features.

Effect of Surface Chemistry

The use of atomically-smooth sapphire substrates allows for the effectiveness of surface degreasing methods to be qualitatively determined. As-received sapphire substrates have an joint strength one-half that of wet or dry processed surfaces, despite having similar RMS roughness. Previous studies have shown [15, 16] that the removal of surface contaminants and weakly-bonded molecular fragments (CO, H_2O) can significantly improve interfacial adhesion. It is widely accepted that plasma cleaning can be an effective technique to remove very thin layers of contaminants from the surface [17]; however, in the absence of rough surface topography, conventional acetone/ethanol/DI solvent degreasing and $Ar:O_2$ plasma cleaning yield similarly effective surfaces for adhesion, as measured by joint strength (0.943 MPa and 1.044 MPa, respectively). The increased surface area and decreased crack propagation velocity due to surface roughness on ceramic substrates [12] make it much more difficult to quantify the role of surface reactivity on the surfaces.

In the case of IPA cleaning, AFM measurements of atomically-smooth sapphire before and after surface treatments proved to be a particularly sensitive method to detect surface residue. AFM of sapphire substrates, after isopropyl alcohol (IPA) cleaning and subsequent air-drying, indicated the presence of an additional surface roughness of ~25 nm over an atomically-smooth untreated surface. AES analysis indicated the residue consisted of ~88% carbon, ~10% Na and ~2% Pb. In mechanical tests, IPA degreasing was found to degrade the joint strength significantly. Failure was found to occur at 50% of the substrate tensile strengths (0.255 MPa). When the IPA was *not* allowed to evaporate off the substrate (minimizing visible particle contamination), joint tensile strength was found to be comparable with A/E/DI degreased substrates. Distilled or ultra pure versions of IPA may eliminate this residue.

Plasma cleaning with Ar and O_2 (90%:10%) gases was found to improve adhesive joint strength to the same level as standard degreasing procedures. Post-plasma cleaning of IPA degreased substrates removed IPA residue, yielding a joint strength identical to plasma cleaning alone.

Effect of Priming

The results of our study indicate that the use of a primer produces a strong bond, even on smooth and contaminated surfaces. The application of a primer results in a new surface morphology whose topography is completely dominated by the primer. The resulting roughness is essentially independent of surface preparation for sapphire substrates and smoother overall for polycrystalline substrates. From Table I, polycrystalline substrates showed a lower RMS roughness following priming and priming following Al_2O_3 beading. Additionally, primed substrates following IPA degreasing were shown to attain a joint strength equal to that of primed substrates that were not degreased.

Substrate priming is known to increase the adhesive strength of a joint [18], as well as to improve the joint's service life. By forming primary interfacial bonds between the substrate and Si—O—Ti coupling agents of the primer, adhesive strength of the joint is improved [19].

Although priming is shown to be the best surface preparation method for achieving strong bonds, some caution should be exercised since, for example, priming of electronic components can have a detrimental effect on device performance and reliability. 10 MΩ TaN resistors (Electro-Films, Inc.) were found to exhibit electrical shorts during device operation if primers were used prior to encapsulation.

CONCLUSION

An investigation of the role of chemistry and topography on the adhesive strength of rubber/refractory-oxide interfaces of poly(dimethylsiloxane) (PDMS) bonded to Al_2O_3 substrates has been performed. Tensile pull tests were performed on poly(dimethylsiloxane) rubber bonded to single-crystal and ceramic Al_2O_3 substrates prepared using several surface treatments. An accurate and reliable mechanical test method was successfully developed which used a frustro-conical geometry to eliminate the influence of tri-axial stress effects at the periphery of the bond. Chemical cleaning of atomically-smooth sapphire resulted in a significant larger joint strength (1 MPa) than that obtained from as-received substrates (0.448 MPa). The introduction of a significant amount of contamination (~ 25 nm thick) from isopropyl alcohol (IPA) residue resulted in even weaker adhesion (0.241 MPa). Abrasive cleaning of sapphire using fine (~ 100 micron) Al_2O_3 beads created more 0.5 - 1.0 micron topographic features and significantly higher joint strengths (1.103 MPa) than for similarly prepared surfaces using coarse (~ 350 micron) Al_2O_3 beads (0.621 MPa). The application of a titanate-modified silicone primer resulted in the greatest joint strength encountered ($>$ 1.207 MPa), even when significant levels of surface contamination were present prior to priming. Adhesion to ceramic Al_2O_3 substrates was systematically stronger than to sapphire for as-received samples (0.965 MPa), abrasively-cleaned surfaces (1.034 MPa), and surfaces with a residue from IPA cleaning (0.827 MPa) as a result of the rougher topography.

Acknowledgements

A portion of this work was supported by Northrop-Grumman Corporation under contract #97211059.

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