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Analysis of the Effect of Surface Modification on Polyimide Composites Coated With Erosion Resistant Materials

Tchinga Ndalama and Deidre Hirschfeld
New Mexico Institute of Mining and Technology, Socorro, New Mexico

March 2003

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Tchinga Ndalama and Deidre Hirschfeld
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Analysis of the Effect of Surface Modification on Polyimide Composites Coated With Erosion Resistant Materials
Annual Report PS-03-12

Tchinga Ndalama and Deidre Hirschfeld
New Mexico Institute of Mining and Technology
Socorro, New Mexico 87801

The aim of this research is to enhance performance of composite coatings through modification of graphite-reinforced polyimide composite surfaces prior to metal bond coat/ hard topcoat application for use in the erosive and/or oxidative environments of advanced engines.

Graphite reinforced polyimide composites, PMR-15 and PMR-II-50, formed by sheet molding and pre-pregging will be surface treated, overlaid with a bond coat and then coated with WC-Co. The surface treatment will include cleaning, RF plasma or ultraviolet light-ozone etching, and deposition of SiO_x groups. These surface treatments will be studied in order to investigate and improve adhesion and oxidation resistance.

The following panels were provided by NASA-Glenn Research Center(NASA-GRC):

- Eight compression molded PMR-II-50; 6×6×0.125 in.
- Two vacuum-bagged PMR-II-50; 12×12×0.125 in.
- Eight compression molded PMR-15; 6×6×0.125 in.
- One vacuum-bagged PMR-15; 12×12×0.125 in.

All panels were made using a 12×12 in. T650-35 8HS (3K-tow) graphite fabric (Amaco Fabrics and Fibers Co., Austell, GA). A diamond-wafering blade, with deionized water as a cutting fluid, was used to cut PMR-II-50 and PMR-15 panels into 1×1 in. pieces for surface tests. The panel edges exhibiting delamination were used for the preliminary surface preparation tests as these would be unsuitable for strength and erosion testing. PMR-15 neat resin samples were also provided by NASA GRC.

Surface profiles of the as-received samples were determined using a Dektak III Surface profile measuring system (Veeco Metrology Group, Santa Barbara, CA). Two samples of compression molded PMR-II-50 and PMR-15, vacuum-bagged PMR-II-50 and PMR-15 were randomly chosen for surface profile measurement according to ANSI/ASME B46.1, (American Society of Mechanical Engineers, New York, NY). Prior to each measurement, the samples were blasted with compressed air to remove any artifacts. Five 10 mm-long scans were made on each sample. The short and long wavelength cutoff filter values were set at 100 and 1000 μm, diamond stylus radius was 12.5 microns. Table 1 is a summary of the arithmetic average roughness (R_a) and waviness (W_a) for the composite surfaces.

Table 1: Arithmetic Average Roughness and Waviness for Graphite Reinforced PMR Composites

Sample	R_a (μm)	W_a (μm)
Compression molded PMR-II-50	0.46	4.70
Vacuum-bagged PMR-II-50	2.11	6.30
Compression molded PMR-15	0.31	0.54
Vacuum-bagged PMR-15	2.22	4.73

Contact angles of 0.03ml deionized water on the sample surfaces and Fourier-Transform Infrared (FTIR) spectra were used to characterize the effect of surface treatments. Additional tests were conducted on PMR-15 neat resins to study the effect of Kapton liners on steel dies used during molding. Parameters for plasma spraying zinc were also investigated.

Cleaning and Plasma Etching

Contamination is an inhibitor to adhesion between materials. Surface integrity must be maintained by appropriate handling during processing or by cleaning processes that remove contaminants from oxidation, the atmosphere, or processing residues. Cleaning can be as simple as washing in water or other solvents, light chemical etching to remove thin layers of the bulk material, or can be as complex as dry processes like plasma or laser assisted removal of organic contaminants [1,2].

Sixteen samples of vacuum-bagged PMR-II-50 were used in an experiment to investigate the effectiveness of cleaning method. Contact angles of 0.03 ml deionized water on the sample surfaces were used to characterize the effect of cleaning method (Table 2). Plasma etching was conducted using a Biorad RF Plasma Barrel Etcher Model PT 7100 (Biorad, London, UK). Samples were etched in argon plasma for 2 minutes, under a pressure of 2 mbar at a radio-frequency power rating of 60 Watts.

Results showed that plasma etching had a significant effect on contact angle (Fig. 1).

Table 2: Surface treatments on PMR-II-50 samples prior to contact angle measurements

Treatment	Samples
No treatment	2
Ultrasonic cleaned in methanol for 10 minutes	2
Ultrasonic cleaned in acetone for 10 minutes	2
Ultrasonic cleaned in methanol for 5 minutes then in acetone for 5 minutes	2
Plasma etched	2
Ultrasonic cleaned in methanol for 10 minutes then plasma etched	2
Ultrasonic cleaned in acetone for 10 minutes then plasma etched	2
Ultrasonic cleaned in methanol for 5 minutes then in acetone for 5 minutes and then plasma etched	2

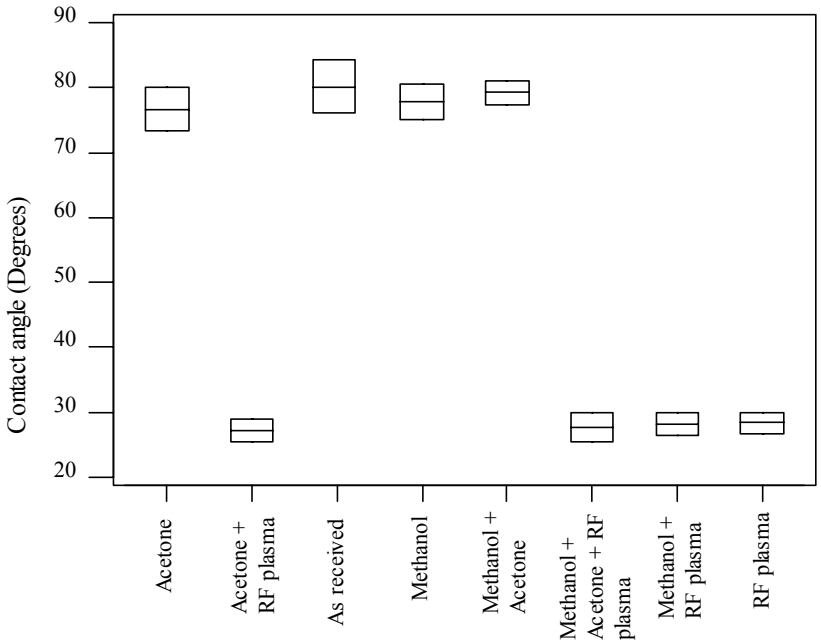


Figure 1: Effect of cleaning/etching on contact angle of vacuum-bagged PMR-II-50.

Similarly, sixteen pieces of vacuum-bagged PMR-15 were used to investigate the effect of cleaning method on contact angle (Table 3). Among the treatments, RF plasma etching had a significant effect on contact angle (Fig. 2).

Table 3: Surface treatments on vacuum-bagged PMR-15 samples prior to contact angle measurements

Treatment	Samples
As received	2
Ultrasonic cleaned in methanol for 10 minutes	2
Ultrasonic cleaned in acetone for 10 minutes	2
Ultrasonic cleaned in methanol for 5 minutes then in acetone for 5 minutes	2
Plasma etched for 2 minutes	2
Ultrasonic cleaned in methanol for 10 minutes then plasma etched	2
Ultrasonic cleaned in acetone for 10 minutes then plasma etched	2
Ultrasonic cleaned in methanol for 5 minutes then in acetone for 5 minutes and then plasma etched	2

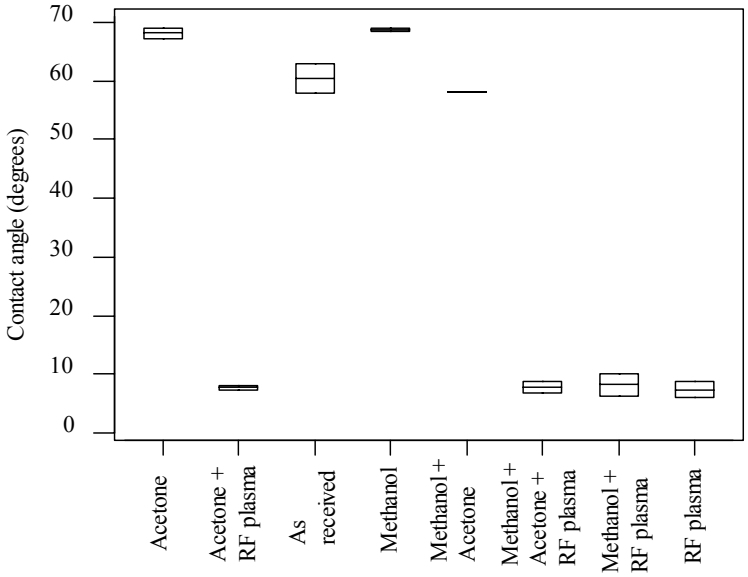


Figure 2: Effect of cleaning/etching on contact angle of vacuum-bagged PMR-15.

Ultraviolet light/Ozone (UV/Ozone) Etching

A dual-purpose UV/Ozone reactor utilizing a low-pressure mercury lamp was constructed for UV/Ozone etching and UV induced chemical vapor deposition (Fig. 3). Vacuum-bagged PMR-II-50 samples were used in preliminary experiments to determine suitable ranges for UV/Ozone etching parameters. Prior to etching, samples were ultrasonically cleaned in acetone for 10 minutes, and then heated at 180 °C for 2 hours. The effect of distance between the UV lamp and sample surface on contact angle was investigated. Samples were UV/Ozone etched for 10 minutes under an oxygen flow rate of 50ml/min. Results showed that lower contact angles were achieved with a shorter distance (Fig. 4).

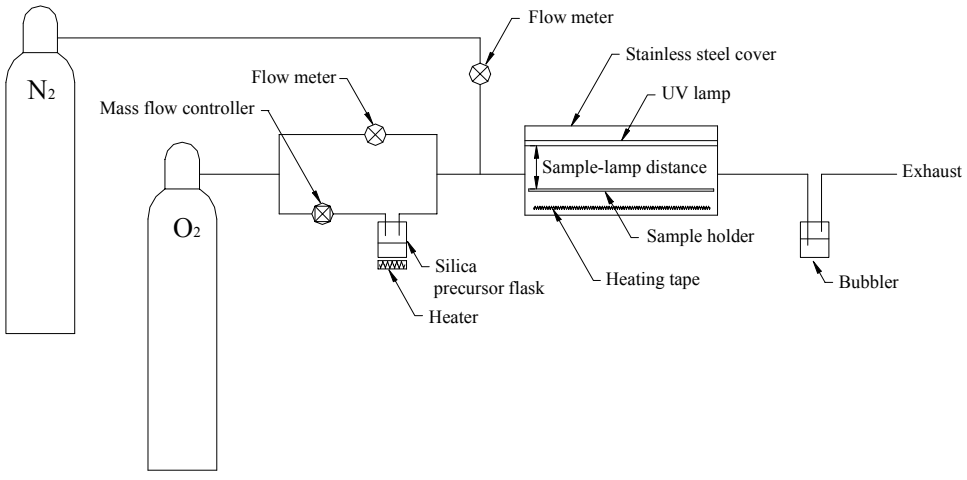


Figure 3: UV/Ozone Reactor.

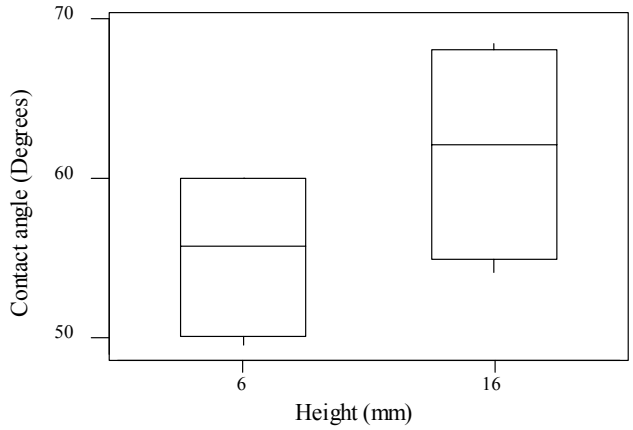


Figure 4: Effect of distance between PMR-II-50 sample and UV lamp.

A 2-level factorial experiment was used to study the effect of exposure time and flow rate during UV/Ozone etching. Sample-lamp distance was set at 6 mm. Results showed that UV/Ozone etching has a statistically significant effect on the contact angle of PMR-II-50 composites, and contact angle decreased with increase in oxygen flow rate (Fig. 5).

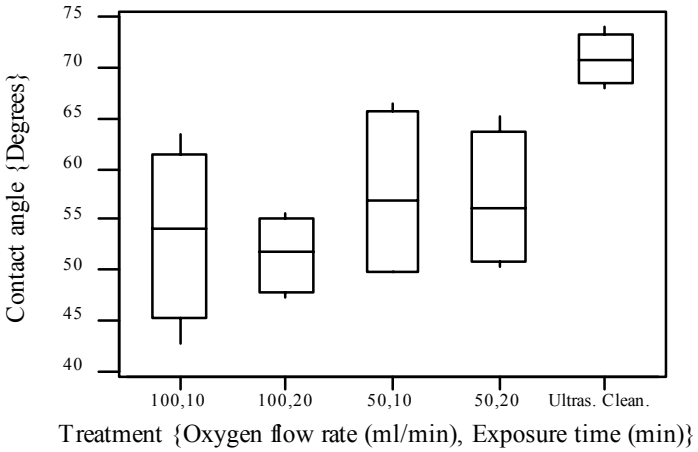


Figure 5: Effect of UV/Ozone etching parameters on contact angle of PMR-II-50.

Based on the results of the preliminary experiments, a three level factorial experiment was conducted to determine the effects of etching factors. Sample-lamp distance was set to 6mm. Results showed that oxygen flow rate of 120 ml/min and exposure time of 24 min, produced the smallest contact angle of 50°.

Further experiments were conducted to explore the effect of extended UV/Ozone treatment time on contact angle. In these experiments, oxygen flow rate was set at 120 ml/min. Results showed that contact angle decreases with increasing exposure time (Fig. 6). Additional experiments are being conducted to optimize etching.

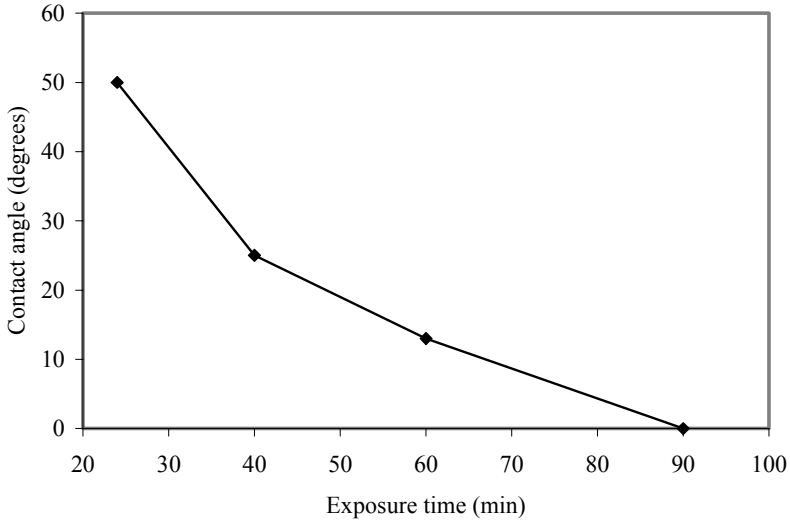


Figure 6: Effect of exposure time on contact angle of UV/Ozone etched PMR-II-50 composites.

In preliminary experiments using PMR-15, vacuum-bagged samples were ultrasonically cleaned in acetone for 10 minutes and heated at 180 °C for 2 hours. The effect of distance between the UV lamp and sample surface on contact angle was investigated. Samples were UV/Ozone etched for 10 minutes with an oxygen flow rate of 50ml/min. Results showed that low contact angles were achieved with short etching distance (Fig. 7).

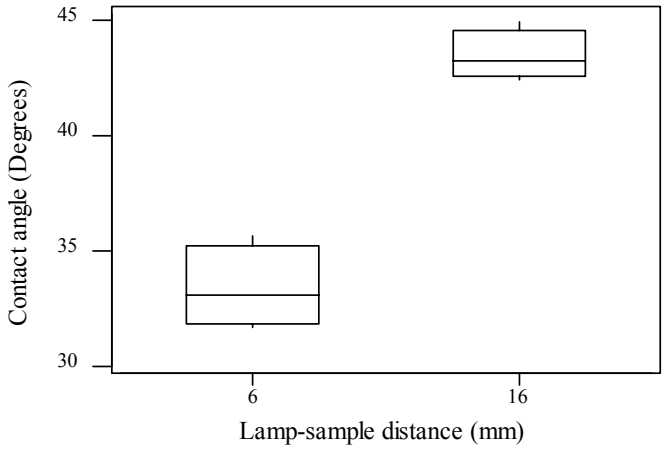


Figure 7: Effect of distance between PMR-15 sample and UV lamp.

A 2-level factorial experiment was used to study the effect of exposure time and flow rate during UV/Ozone etching. Results showed that UV/Ozone etching has a statistically significant effect on the contact angle of PMR-15 composites, and contact angle decreased as both oxygen flow rate and exposure time increased (Fig. 8).

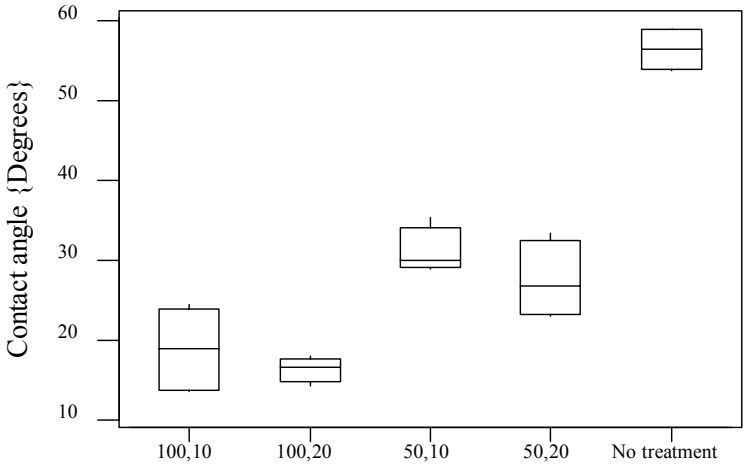


Figure 8: Effect of UV/Ozone etching parameters on contact angle of PMR-15.

Based on the results of the preliminary experiments, a three level factorial experiment was conducted to explore etching factors. Results showed that oxygen flow rate of 120 ml/min and exposure time of 24 min, yielded the lowest contact angle (Fig. 9).

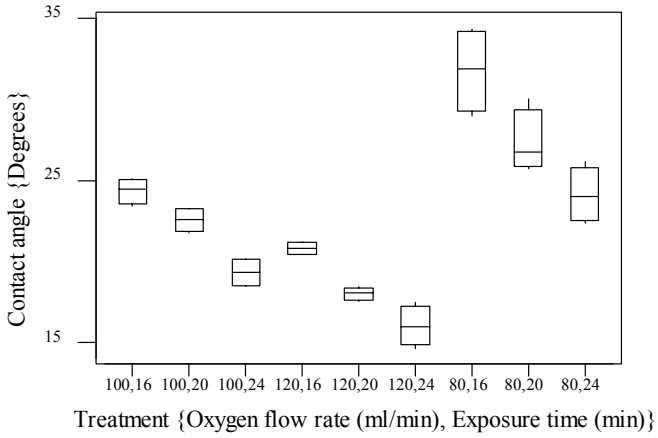


Figure 9: Effect of UV/Ozone etching parameters on contact angle of PMR-15.

Results on experiments conducted to study the effect of longer UV/Ozone etch time on the contact angle of PMR-15 showed that contact angle decreases with increasing exposure time (Fig. 10). Results indicated that exposure time of 90 minutes are required to achieve contact angles similar to RF plasma etching.

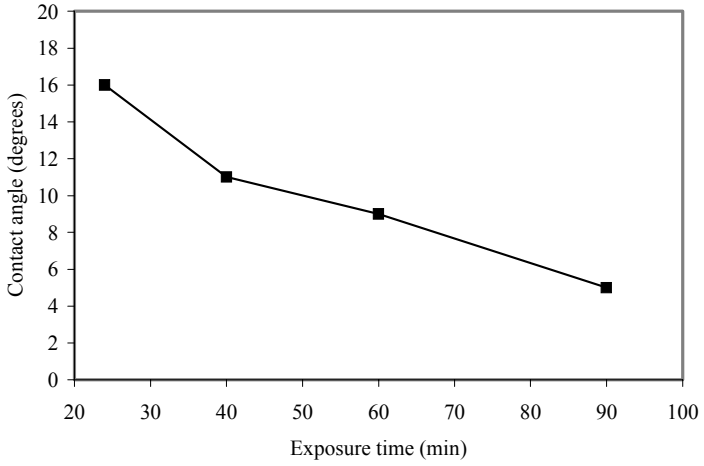


Figure 10: Effect of UV/Ozone exposure time on contact angle of PMR-15. Oxygen flow rate was 120 ml/min.

Plasma etching performance of an inductively coupled RF plasma reactor in the Chemical Engineering Department, New Mexico Institute of Mining and Technology, NM, was compared to that of the Biorad Barrel Etcher. The RF plasma reactor has the advantage that it can be used for chemical vapor deposition in addition to etching. It was found that samples etched using the RF plasma reactor in the Chemical Engineering Department yielded nearly 0° contact angles at 10-minute treatment time on both PMR-15 and PMR-II-50. Treatment parameters for the RF plasma reactor were: 10 minutes exposure time, 20 ml/min oxygen flow rate and 20–50 Watts RF power. On the other hand, samples etched in the Barrel Etcher yielded contact angles <30° for PMR-II-50 (Fig. 1) and <10° for PMR-15 at 2 minutes of treatment time (Fig. 2).

An Avatar 360 FTIR system with an Omni Sampler ATR attachment (Thermo Nicolet, Madison, WI) was used to obtain the FTIR spectra of selected UV/Ozone and RF plasma etched composites. There was good correlation between the FTIR spectra of the as-received PMR-II-50 and PMR-15 composites, ultrasonically cleaned PMR-II-50 and PMR-15 composites, and spectra of PMR-II-50 and PMR-15 resins in published literature [3–5]. The spectra of the as-received composites were used for comparison with the spectra of the etched composites. Both PMR-15 and PMR-II-50 composites that were UV/Ozone etched for 90 minutes under an oxygen flow rate of 120 ml/min, and those that were RF plasma treated for 10 minutes under an oxygen flow rate of 20 ml/min, were selected for FTIR analysis. The FTIR spectra of as-received PMR-15 and PMR-II-50 composites were compared to those of the surface treated composites to study the effect of surface treatment (Figures 11 and 12). Increased absorption in the 900 to 1100 cm^{-1} and 1150 to 1300 cm^{-1} ranges of infrared wavelength indicates a general increase in C-O-C moieties on the surface of PMR-II-50 and PMR-15 composites, respectively [6]. Additional evaluation using XPS is being conducted at the Center of Micro-Engineered Materials, University of New Mexico, Albuquerque, NM, and at NASA GRC.

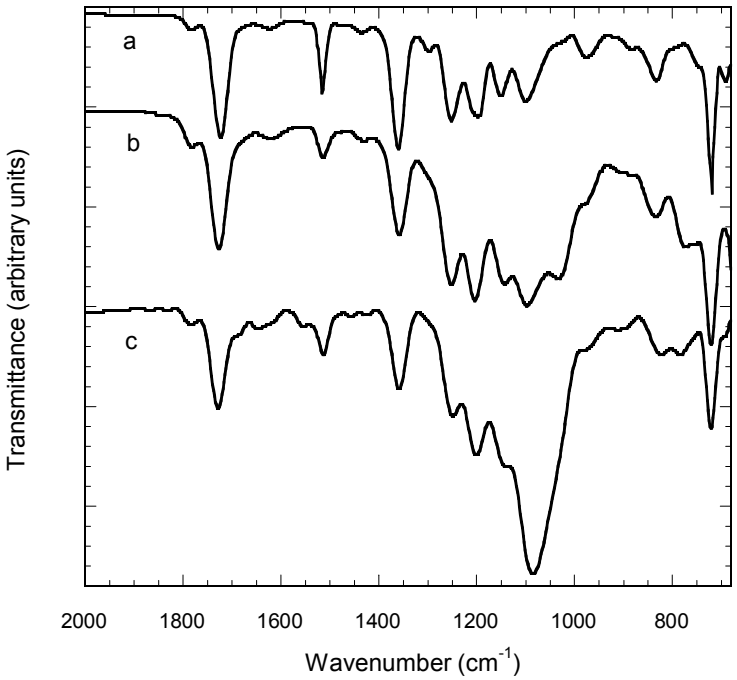


Figure 11: FTIR spectra of (a) as-received PMR-II-50 composite, (b) UV/Ozone etched PMR-II-50 composite and (c) RF plasma etched PMR-II-50 composite.

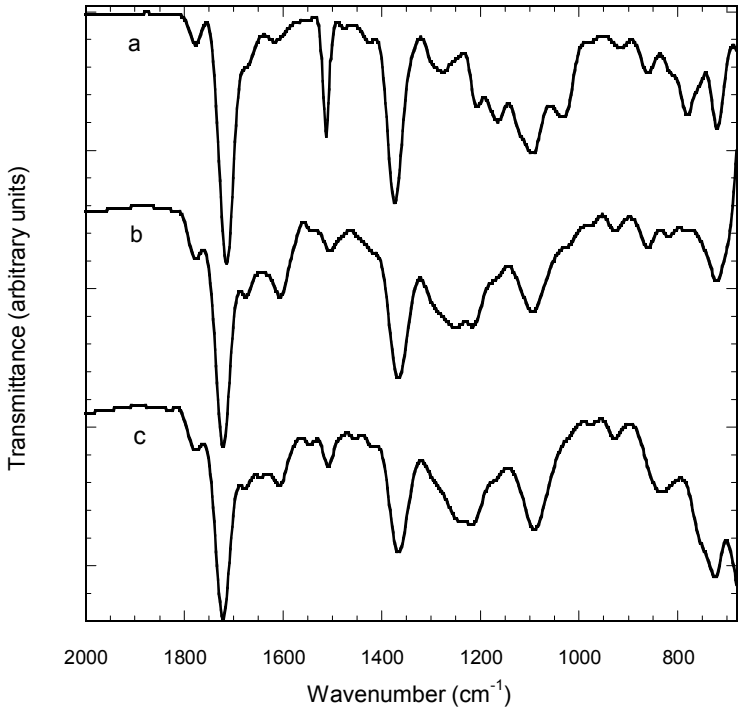


Figure 12: FTIR spectra of (a) as-received PMR-15 composite, (b) UV/Ozone etched PMR-15 composite and (c) RF plasma etched PMR-15 composite.

Chemical Vapor Deposition

An inductively coupled RF plasma reactor in the Chemical Engineering Department, New Mexico Institute of Mining and Technology, was used for plasma-enhanced chemical vapor deposition (PE-CVD). Tetraethoxysilane (TEOS) and Tetramethoxysilane (TMOS) were used as silica precursors in order to compare their effectiveness for SiO_x deposition. It is known that factors such as: RF power, substrate temperature, reactant flow rates and reactor pressure affect the deposition of SiO_2 films [7]. Experiments were conducted to investigate the effect of silica precursor temperature and oxygen flow rate on SiO_x deposition, because these factors could be controlled precisely. Because both TEOS and TMOS lose internal energy when subjected to low pressure, a heater was used to warm the silica precursors to enhance vaporization. A TMOS temperature of 4 °C was chosen because at this temperature, all frozen liquid was molten. Another higher temperature of 20 °C was chosen to study the effect of silica source temperature. Temperatures chosen for TEOS were 20 and 40 °C, for the same reasons. The flow rate of the silica source vapors was not determined. Oxygen flow rate was set at 20 ml/min and at 100 ml/min.

PMR composite samples were ultrasonically cleaned in acetone for 10 minutes, then dried at 180 °C for 2 hours to volatilize the cleaning agent. The composite samples were next placed in the reactor and exposed to a 40-millitorr-oxygen atmosphere. An RF plasma was then lit for 10 minutes to etch the surfaces prior to the CVD process. A mixture of silica precursor vapor and oxygen was then introduced into the chamber for SiO_x deposition. At the end of each deposition run (45 minutes), the vapor and oxygen supply to the chamber was shut off, and plasma power was switched off. FTIR spectra and contact angles of the films were obtained.

A dual-purpose UV/Ozone reactor (Fig. 3) was used for ultraviolet light induced chemical vapor deposition (UV-CVD). Prior to chemical vapor deposition, the PMR composites were ultrasonically cleaned and dried, as in the previous PE-CVD experiments, then UV/Ozone etched for 24 minutes with an oxygen flow of 120ml/min. During deposition, a mixture of oxygen and silica precursor vapor was bled into the reactor for 45 minutes. Distance between lamp and sample was 6mm. Silica precursor temperature was set at 70 °C because oxygen gets saturated with TEOS vapor at this temperature and standard atmospheric pressure [8]. The same temperature was also used for TMOS. In the UV-CVD experiments, oxygen flow rate and substrate-temperature were varied to investigate their effects on SiO_x deposition. FTIR spectra and contact angles of the films were obtained.

To evaluate the films deposited on PMR-II-50, FTIR and contact angle data of the films were compared with that of the as-received PMR-II-50 composite. For FTIR spectra, the amplitude of the peak due to Si-O-Si asymmetric stretching at 1030 to 1090 cm^{-1} was compared to (a) the absorption peaks of the substrate, at 1724, 1516, 1368 and 723cm^{-1} (Fig. 13); and (b) the peaks due to impurities such as Si-OH deformation at 890 to 950cm^{-1} and due to Si-C rocking at 750 to 840 cm^{-1}.

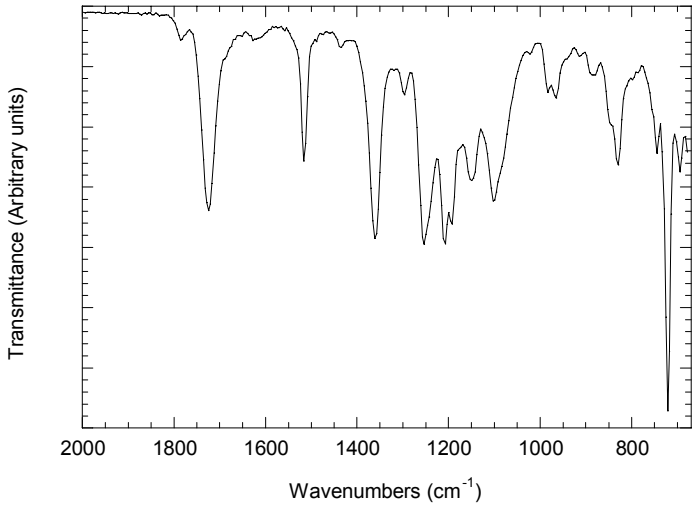


Figure 13: FTIR spectrum of as-received PMR-II-50 composite.

A clearly distinguishable Si-O-Si peak at 1030 to 1090 cm^{-1}, with minute peaks due to substrate and impurities indicated good film quality and coverage, as shown in Figures 14 and 15. On the other hand, a Si-O-Si peak, with strong peaks due to the substrate and impurities in the spectrum, indicated poor film quality and coverage, as shown in Figures 16 and 17. This was confirmed by contact angle data, as shown in Fig. 18, where PE-CVD yielded films with lower contact angles than UV-CVD.

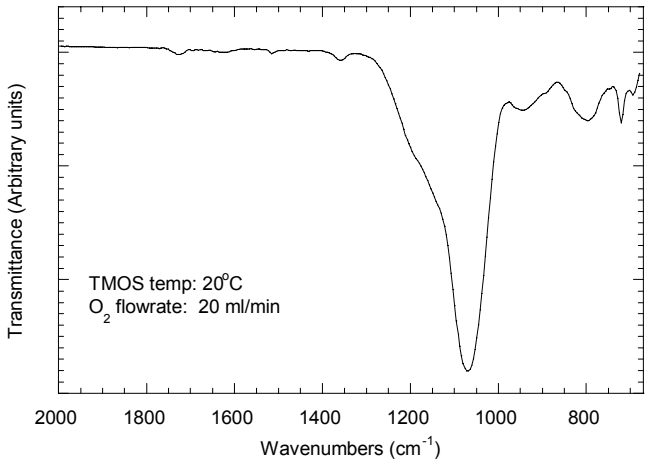


Figure 14: FTIR spectrum of SiOx film on PMR-II-50 by PE-CVD using TMOS.

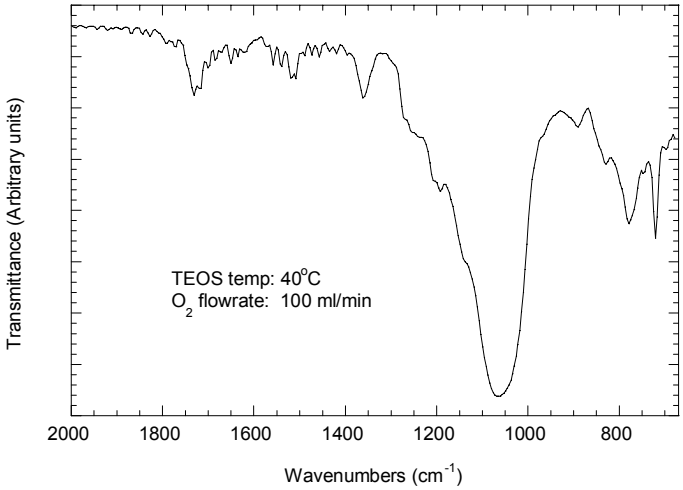


Figure 15: FTIR spectrum of SiOx film on PMR-II-50 by PE-CVD using TEOS.

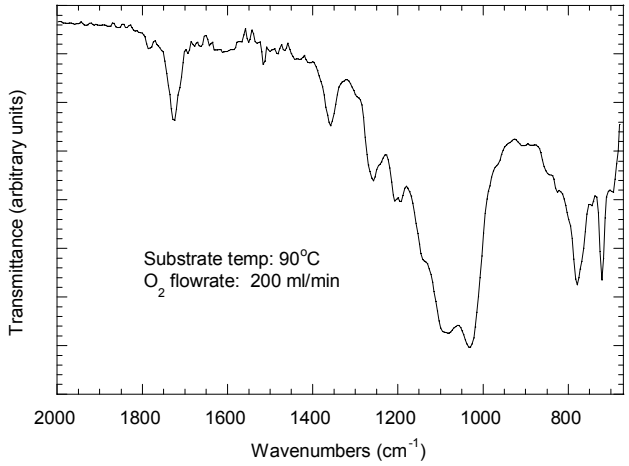


Figure 16: FTIR spectrum of SiOx film on PMR-II-50 by UV-CVD using TMOS.

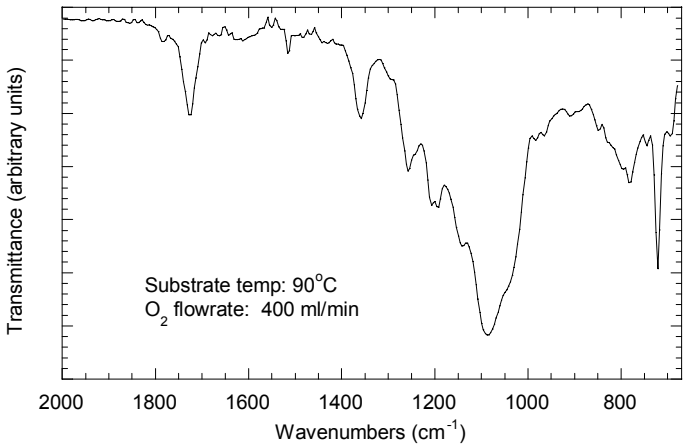


Figure 17: FTIR spectrum of SiO_x film on PMR-II-50 by UV-CVD using TEOS.

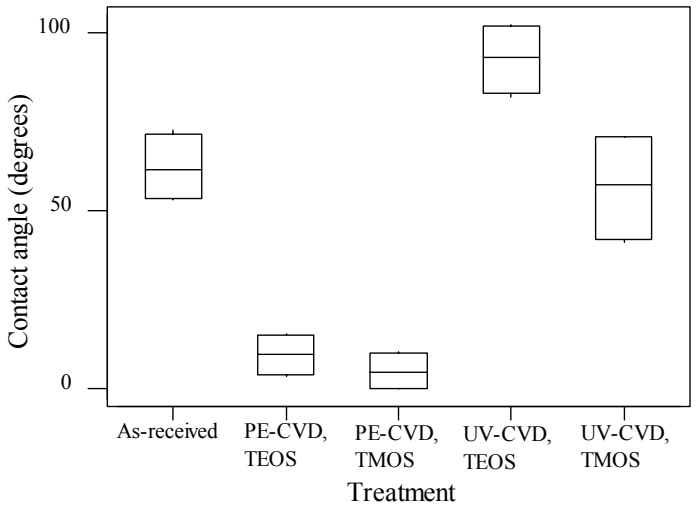


Figure 18: Effect of CVD treatment on contact angle of SiOx films on PMR-II-50. Treatment conditions are the same as specified in FTIR diagrams.

The same strategy used to evaluate SiO_x films deposited on PMR-II-50 was applied to PMR-15. FTIR spectra of as-received PMR-15 (Fig. 19) was compared to that of the deposited films (Figs. 20–23). Generally, SiO_x films deposited by PE-CVD were of better quality(Figs. 20, 21) than films deposited by UV-CVD (Figs. 22, 23). Contact angle measurements confirmed this observation (Fig. 24).

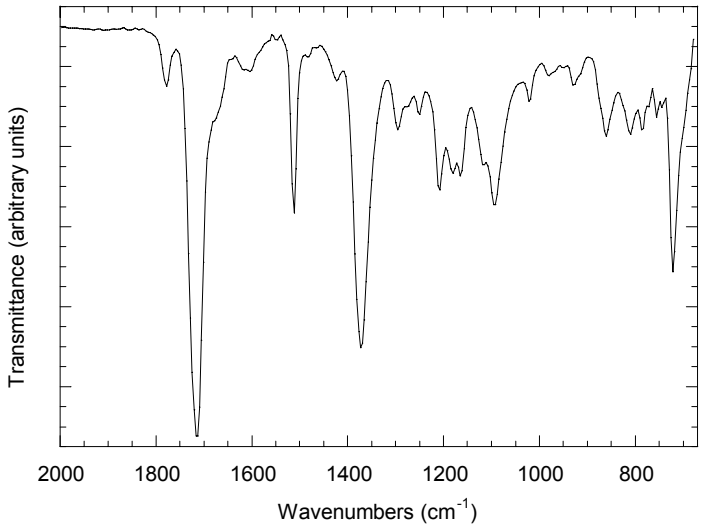


Figure 19: FTIR spectrum of as-received PMR-15 composite.

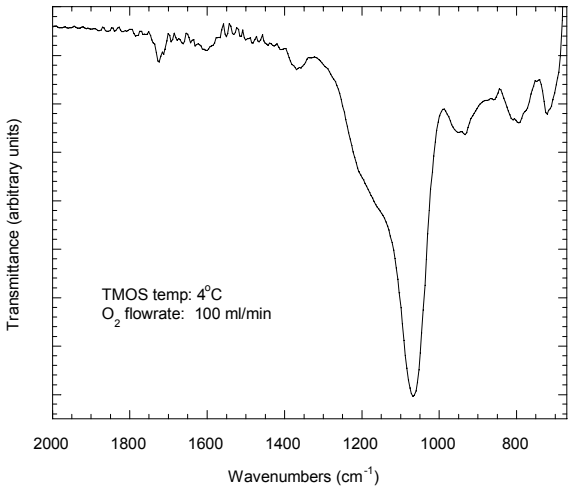


Figure 20: FTIR spectrum of SiOx film on PMR-15 by PE-CVD using TMOS.

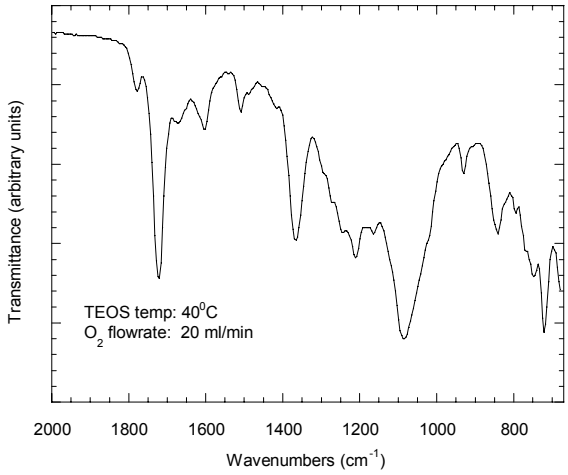


Figure 21: FTIR spectrum of SiOx film on PMR-15 by PE-CVD using TEOS.

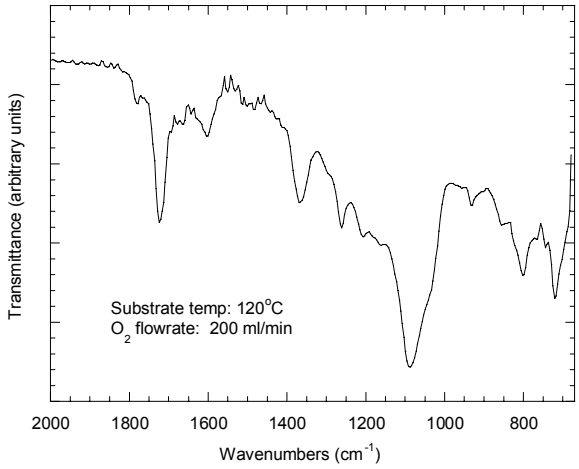


Figure 22: FTIR spectrum of SiOx film on PMR-15 by UV-CVD using TEOS.

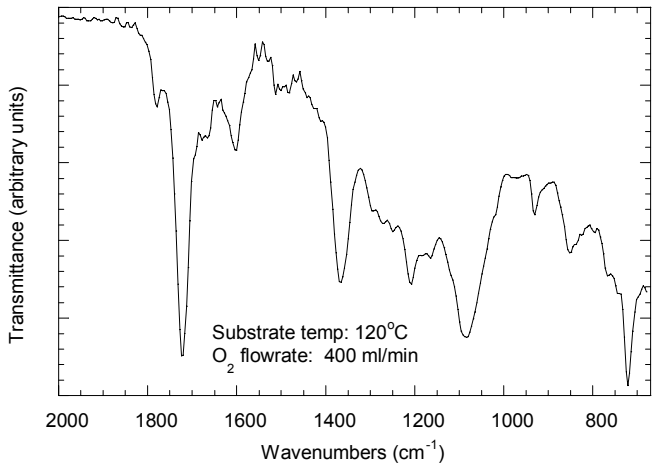


Figure 23: FTIR spectrum of SiOx film on PMR-15 by UV-CVD using TMOS.

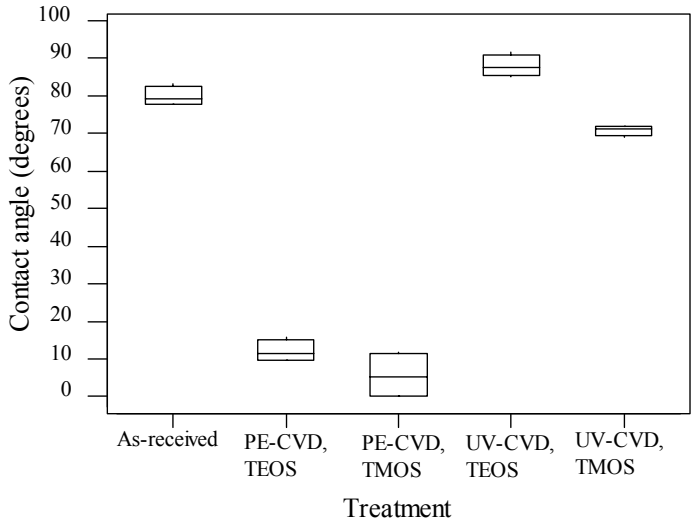


Figure 24: Effect of CVD treatment on contact angle of SiO_x films on PMR-15. Treatment conditions are the same as specified in FTIR diagrams.

Contact angle data showed that TMOS yielded better films than TEOS on both PMR-15 and PMR-II-50, because CVD treatments with the former yielded lower contact angles than the latter. It is known that hydrocarbons have a low surface energy [6]. It is proposed that the presence of $-OC_2H_5$ groups in the films deposited by UV-CVD contributes to a low surface energy, resulting in large contact angles. This may be due to incomplete decomposition and hydrolysis of the silica precursors and the polysiloxane layers, respectively. Average thickness of PE-CVD deposited SiO_x film was 1.4 μm, according to profilometry results. XPS analysis of the treated samples is currently underway at NASA-Glenn Research Center and will elucidate the effect of deposition parameters used.

Effect of Kapton Liners on Molding PMR-15 Neat Resins

Contact angle measurements and FTIR spectra were obtained for PMR-15 neat resins that were compression molded using steel dies with or without Kapton liners. Samples were not treated in any manner before testing. Contact angles and FTIR spectra of the two materials were different (Figs. 25–27). It is likely that some Kapton adheres to the polymer surface.

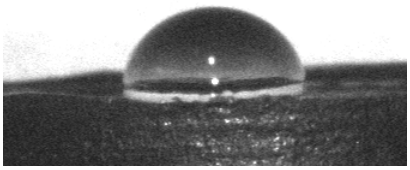


Figure 25: Sessile drop of DI water on PMR-15 pressed with Kapton Liner. Contact angle: 94°.

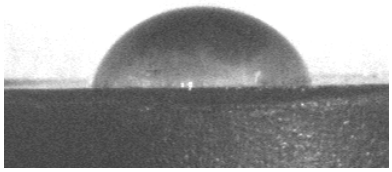


Figure 26: Sessile drop of DI water on PMR-15 (No Kapton). Contact angle: 83°.

In summary, the major differences in FTIR spectra are:

- 1502 cm^{-1} peak more intense on Kapton than the 1513 cm^{-1} peak without: Benzene rings.
- 1247 cm^{-1} peak sharp on Kapton: C-O-C
- 1114 cm^{-1} peak sharp and intense on Kapton: C-O-C
- 819 cm^{-1} peak very intense on Kapton

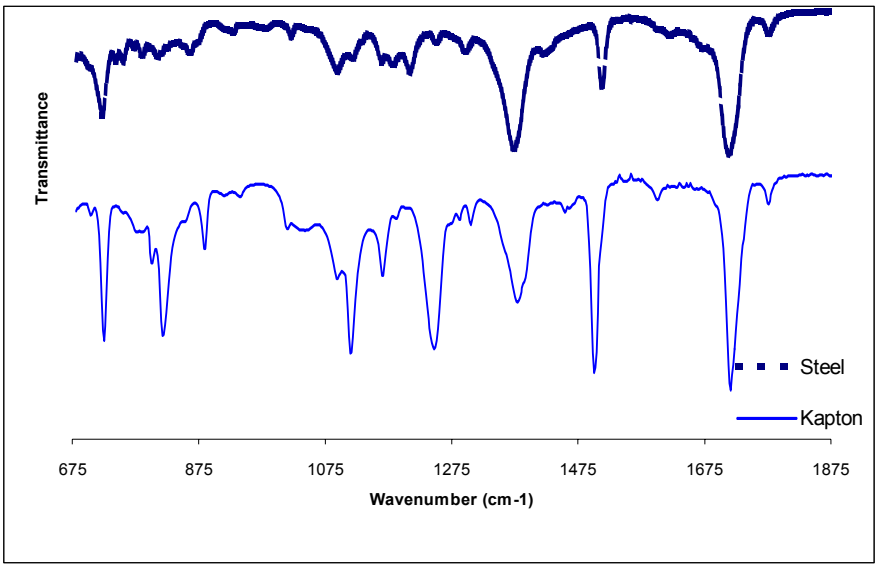


Figure 27: FTIR spectra of as-received compression molded PMR-15 resin samples. Solid Line is with Kapton Mold Liner and Dashed without.

Plasma Sprayed Coatings

Experiments were conducted to investigate plasma spray parameters for zinc coatings. Aluminum substrates were used for the preliminary experiments. Cross sections of zinc coatings were examined using light microscopy for porosity and thickness. Photomicrographs of the cross sections were used to calculate porosity using SCION IMAGE™ software (Scion Corp., Frederick, MD). Average porosity of 1.4 percent was achieved for zinc on aluminum (Fig. 28). Because the polyimide composite substrates have different thermal properties than aluminum, it was expected that spray parameters would be different. Preliminary plasma spray experiments conducted using composite substrates yielded zinc coatings that were less than 2 percent porous (Figs. 29,30). Table 4 is a summary of the parameters used to plasma spray the coatings presented. Plasma spraying is being further optimized to achieve denser coatings.

A preliminary investigation on the use of indentation hardness testing to optimize bond strength in terms of etching, deposition, and minor modification of plasma spray parameters has been conducted. Hardness indents using a Brinnel indenter were made on zinc coated PMR and debonded regions were measured using the profilometer. The standardization of the indentation technique is underway and should provide a screening method to characterize adhesion using a minimum sample size. After optimizing the indentation hardness adhesion, fracture toughness and conventional bond strength measurements will be used to confirm the results.

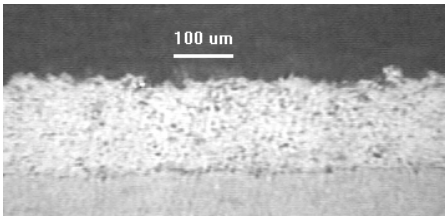


Figure 28: Zinc coating on aluminum exhibiting average porosity of 1.4 percent.

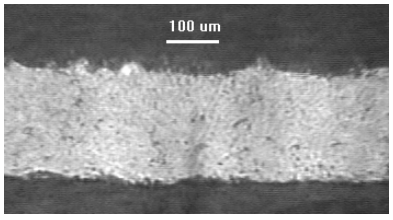


Figure 29: Zinc coating on PMR-15 composites exhibiting average porosity of 1.6 percent

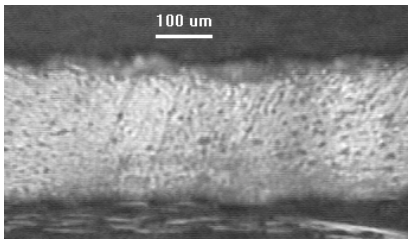


Figure 30: Zinc coating on PMR-II-50 composites exhibiting average porosity of 1.6 percent.

Table 4: Plasma spray parameters for zinc coatings

	Zinc on aluminum	**Zinc on PMR composites**
Parameters	*1.4% porous*	*1.6% porous*
Arc Amperage (A)	600	600
Arc gas(psi)	100	100
Auxiliary gas(psi)	45	55
Powder gas(psi)	40	40
Powder feeder rev (rpm)	2	2
Gun velocity (mm/s)	523	523
Powder nozzle stand off (mm)	13	13
Substrate stand off (mm)	80	80
Gun passes	80	80

Accomplishments

The following tasks and milestones have been achieved in the course of the project:

- literature review of surface modification was conducted and continues to be monitored
- an Omni Sampler ATR accessory for a Nicolet Avatar 360 FTIR (Thermal Nicolet, Madison, WI) were purchased
- modification of PMR composite surfaces by RF plasma and UV/Ozone etching was investigated and optimized using contact angle and FTIR analyses
- modification of PMR composite surface by RF plasma and UV induced chemical vapor deposition of silica films was explored using contact angle and FTIR analyses
- the effect of Kapton liners, used during the processing of PMR-15, on surface chemistry was determined by contact angle and FTIR analyses
- a Dektak III profilometer (Veeco Metrology Group, Santa Barbara, CA) and isolation table were purchased

Future Work

XPS analyses of the SiOx films is underway at NASA-GRC, which will elucidate the chemical composition of SiOx films effect of deposition parameters. Further XPS analyses will be conducted at the Center for Micro-Engineered Materials, University of New Mexico, Albuquerque, NM, to characterize chemical changes on the surfaces of plasma and UV/Ozone etched composites.

Preliminary zinc bond coats on polyimide composites have been applied by atmospheric plasma spraying, using the standard sample preparation procedure followed at NASA-GRC and will provide a control for comparisons. Zinc and other metallic bond coats will be applied to surface modified PMR composites. The effect of surface modification on adhesion will be characterized by indentation testing, fracture toughness and bond strength. The last phase of the research will involve applying the WC-Co topcoat and performance testing (thermal cycling, oxidation and erosion testing) to optimize surface modification.

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Tchinga Ndalama and Deidre Hirschfeld

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New Mexico Institute of Mining and Technology
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13. ABSTRACT ***(Maximum 200 words)***

The implementation of polymer composites in the erosive/oxidative environment of advanced engines requires the use of hard protective coatings. It has been proposed to utilize surface modification techniques to improve the adhesion of the coating system. Experiments on graphite fiber reinforced polyimide composites, PMR-15 and PMR-II-50, overlaid with a bond coat and then coated with WC-Co to optimize surface treatment, bond strength, erosion resistance, and thermal stability are underway. Effectiveness of cleaning techniques and SiO_x deposition using both RF plasma and UV-ozone methods were determined using contact angle measurements and Fourier Transfer Infrared spectroscopy (FTIR). Two sources of silica, tetramethoxysilane (TMOS) and tetraethoxysilane (TEOS) were used as precursors. Results indicate that RF Plasma etching is the most effective cleaning method for both composites and that TMOS coatings yield the lowest contact angles.

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