Optical Characterization of Black Appliques

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ABSTRACT

For some stray light applications, it may be advantageous to use a black applique rather than a conventional black coating. Appliques consist of a free-standing sheet of black material and an adhesive or other means for attaching the applique to a substrate. In this paper the optical scatter in the visible and infrared of black appliques from Battelle, Dupont, Edmund Scientific, Energy Science Laboratories, Inc. (ESLI), Rippey and Rodel is reported and compared to Martin Black. The Rippey and Rodel appliques are sold as polishing cloths for the semiconductor industry, whereas the ESLI applique was originally developed as a low sputter yield coating. The Battelle applique consists of a carbon loaded polyurethane film with a surface which is heat molded into a micro-grooved pattern. The ESLI applique consists of high aspect ratio fibers mounted in an adhesive base and was the blackest applique of all those investigated. For an incidence angle of 10°, a scattering angle of 45° and a wavelength of 632.8 nm, the BRDF of the best ESLI applique was 3×10^{-4}, compared to 1×10^{-3} for the best Battelle applique, 1.5×10^{-3} for a representative Martin Black sample and 1.8×10^{-3} for the Edmund applique. The Battelle applique is quasi-diffuse due to its surface microstructure, with a higher BRDF ($2\text{-}5 \times 10^{-3}$) at scatter angles less than 15°. For a wavelength of 10.6 μm, an incidence angle of 7.5° and a scatter angle of 45°, the BRDF of the ESLI coating (1×10^{-3}) was slightly higher than Martin Black (8×10^{-4}), with the Battelle applique exhibiting strong dependencies on scatter angle and groove orientation. In the 2-14 μm spectral range, the directional hemispherical reflectance of the ESLI coating at a 20° incidence angle is below 0.45% and only weakly dependent on incidence angle to 60°. In-plane and cross-plane BRDF measurements at 3.39 μm are reported on a 'biased' ESLI coating which is designed for use at near grazing incidence. In-plane BRDF measurements at wavelengths of 0.6328 and 10.6 μm are reported for most of the appliques studied.

Keywords: Stray light, black coatings, BRDF, BSDF, reflectance, directional hemispherical reflectance, Martin Black

1. INTRODUCTION

Optical engineers can choose from a variety of black paints, etched surfaces, and black coatings when attempting to reduce stray light in optical systems. For some applications it may be advantageous to use a freestanding sheet of black material, which will be referred to as an applique in this paper. Appliques offer several advantages compared to conventional black surfaces. First, the scattering levels of the appliques investigated are lower than that of most commonly used diffuse black paints. Because appliques are manufactured in a factory under controlled conditions, in principle they offer better optical reproducibility than manually applied paints. Second, the durability of many appliques is good, with the optical scatter properties unaffected by light handling. Third, most appliques are produced with non-vacuum processes, and thus tend to be cheaper to produce than exotic (non-paint) black coatings. As an example, the flocked applique from Edmund Scientific offers comparable visible BRDF levels to Martin Black for 1% of the cost. Fourth, with the proper choice of adhesive, appliques can be removed and upgraded as blacker surfaces become available. Fifth, appliques can be applied to virtually any surface. Unlike many conventional black coatings, appliques do not require that a surface be exposed to wet chemical baths or vacuum environments during the coating process. Finally, in contrast to many paints and to coatings produced with wet chemical methods, most appliques are environmentally benign to both the end user and the manufacturing workforce. Appliques can be applied in a shirtsleeve environment while concurrent tasks are being conducted.

Appliques do have a number of disadvantages. First, appliques tend to have lower laser damage thresholds (LDT) than conventional black coatings. The lower LDTs are probably due to the low melting temperature of many applique materials and the low thermal conductance of the applique, adhesive and related interfaces. The appliques reported on in this paper that are made of polymeric materials can be easily damaged with a 2W focused CO_2 laser beam. The flocked fiber appliques reported on in this paper did not show any signs of laser damage after exposure to the same CO_2 laser. This could be due to two factors: the relatively large surface area (10-50 x base area) of the fibers and the use of fiber materials with high laser damage thresholds. The second disadvantage of appliques regards their outgassing properties. It is expected that the appliques fabricated entirely from polymeric materials may not pass the NASA specifications for total mass loss (TML) and collected volatile condensable materials (CVCM). Many of the polymeric materials used are porous with a significant water fraction by weight. The components of one of the flocked appliques studied in this paper are reported to have passed both the TML and CVCM specifications, however no tests on the applique itself have been conducted. Tests of the TML and CVCM for many of the appliques reported on in this paper are in progress. Finally, the polymeric appliques are expected to have lower peak temperatures and atomic oxygen exposure limits than many conventional black coatings. In this paper, optical scatter data is reported on a number of black appliques which are commercially available or in an advanced development stage.

2. EXPERIMENTAL SETUP

Bi-directional reflectance distribution function (BRDF) measurements were performed on both a modified TMA[2] in-plane scatterometer ('CASI' model) at the Naval Research Laboratory (NRL) and a custom TMA full hemispherical scatterometer at Wright Laboratory. The laser sources for the in-plane scatterometer operate at 0.6328, 3.39 and 10.6 μm; for the hemispherical instrument the sources operate at 0.325, 0.6328, 1.06, 3.39, and 10.6 μm. For the in-plane instrument, the sources are s-polarized. The hemispherical instrument has full linear polarization control over the incident and detected radiation at all wavelengths. Both scatterometers are operated in a HEPA filtered environment Instrument signature scans and measurements of diffuse standards were performed on a regular basis, and all samples were cleaned before measurements. None of the data reported in this paper is cosine corrected. Data from the NRL CASI instrument were previously included in a 10.6 μm BRDF round-robin[3].

The directional hemispherical reflectance (DHR) measurements were performed at Wright Laboratory with a Bomem DA3 FTIR and a diffuse gold integrating sphere. The integrating sphere uses a center mounted sample stage which can be rotated from 0 to 60 degrees relative to the incident beam. Larger incidence angles are possible by increasing the sample size or decreasing the spot sizes.

Samples of black appliques were obtained from Battelle[4], Dupont[5] (black Kapton and black Tedlar), Edmund Scientific[6] (black flocked paper). Rippey[7] (UltraPol V), and Rodel[8] (40 Film). For comparison purposes, samples of Martin Black[9], Martin Black Enhanced, EAS-Orlando Black[10] and Lord Corporation[11] Z302 glossy black paint were also obtained. The ESLI flocked fiber sample (#CLSA96/1) was fabricated directly on an aluminum substrate and is the blackest sample of approximately 10 that were studied. An applique version of the ESLI coating is expected to be available in the near future. All samples were stored in HEPA filtered enclosures when not in use.

3. OPTICAL RESULTS

The in-plane BRDF of several Lambertian appliques is shown in Figure 1 for an incidence angle of 10 degrees and a wavelength of 632.8 nm. The data shown are for samples of Rodel's 40 film, Rippey's UltraPol V, Edmund's flocked paper, Martin Black, and Energy Science Laboratory's CLSA96/1 flocked coating. Prior to characterizing the samples, the BRDF of a Labsphere Spectralon sample was measured. The measured BRDF value of 0.30 ± 0.01 is in good agreement with the expected value of 0.31. The measured BRDF value for Martin Black agrees well with literature values[12].

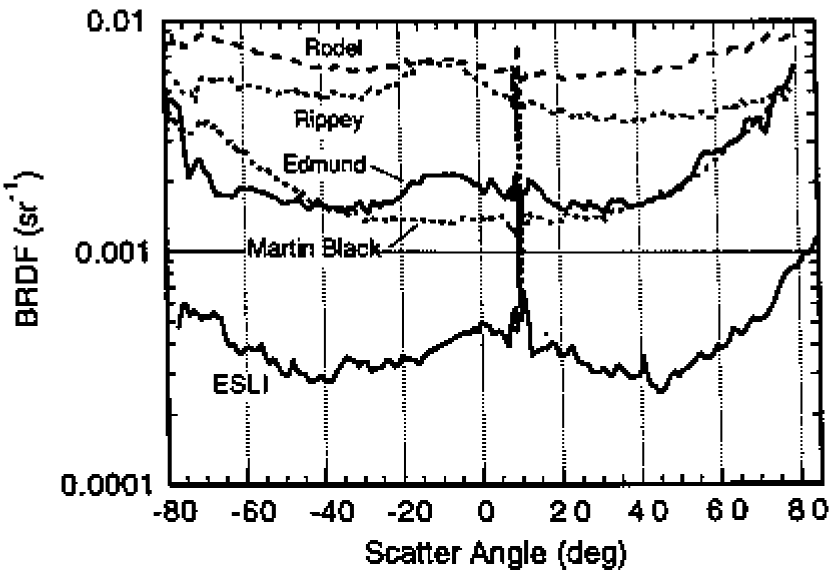

Figure 1. Visible BRDF of assorted diffuse appliques compared to Martin Black for an incident angle of 10 degrees and a wavelength of 632.8 nm.

Note that the inexpensive (~$l/ft^2) flocked paper from Edmund Scientific has a BRDF comparable to Martin Black. The EAS Orlando black sample (not shown) had BRDF values comparable to Martin Black with slightly higher values (2x) for small scattering angles (<30°). No data was taken within ±5° of the retroreflection direction due to blocking of the incident laser beam by the scatterometer's detector. Consequently the data is shown as a straight line in this angular range. Even with this loss of data, one can see that both the Edmund and Rippey samples show signs of near-retroreflection scatter. The ESLI sample was the blackest of all the diffuse blacks measured, with a corresponding hemispherical reflectance of less than 0.15%. The BRDF of most of the appliques studied is not significantly affected by lightly touching the surface of the coating.

The BRDF of several non-diffuse appliques is shown in Figure 2 for an incidence angle of 5 degrees and a wavelength of 632.8 nm. For comparison, the BRDF of a sample of Lord Corporation's Z302 glossy paint is also shown. Note the azimuthal asymmetry in the Battelle applique depending on the groove orientation. For scatter angles > 20°, the Battelle applique has a BRDF of approximately 1-2 x 10^{-3}.

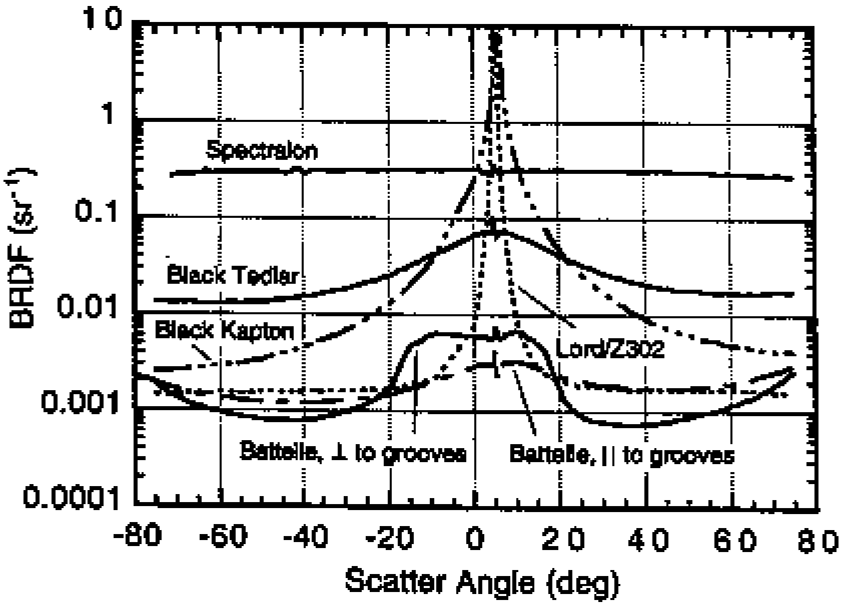

Figure 2. Visible BRDF of Battelle, Dupont and Lord samples versus scatter angle for an incident angle of 5 degrees and a wavelength of 632.8 nm.

The infrared BRDF of the Edmund, Rodel, Rippey and ESU appliques is shown in Figure 3 for an incidence angle of 7.5 degrees and a wavelength of 10.6 µm. Prior to these measurements, an Infragold Plus sample from Labsphere was characterized. For near normal incidence, the Infragold Plus BRDF value at a scattering angle of 45° was 0.26 ± 0.02, which agrees well with the expected value of 0.30. The BRDF values measured for an EAS Orlando sample (not shown) were very close to the values observed for the Edmund and Rodel appliques. The BRDF values observed for the Martin Black and Enhanced Martin Black samples are consistent with those in the literature.[12] The large near specular scattering of the Enhanced Martin Black sample is inconsistent with previous data on this coating and is thought to be due to an irregular or damaged sample. For a 45° scattering angle, the BRDF of the ESLI CLSA96/1 coating is similar (1-1.5 x 10^{-3}) to the Martin Black sample (8 x 10^{-4}). BRDF values below 1 x 10^{-3} have been observed for parts of the ESLI sample; however, the values shown in Figure 3 are representative of the bulk of sample CLSA96/1.

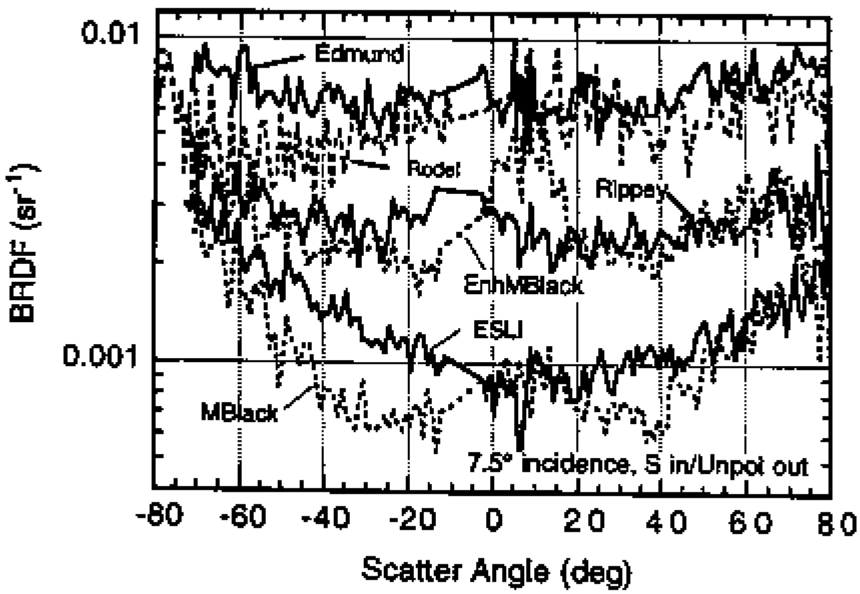

Figure 3. Infrared BRDF of selected appliques versus Martin Black and Enhanced Martin Black for an incident angle of 7.5 degrees.

The infrared BRDF of the Battelle micro-grooved applique, black Kapton applique and Lord Corporation Aeroglaze Z302 coating is shown in Figure 4 for an incidence angle of 7.5 degrees and a wavelength of 10.6 μm. The Battelle microstructure (see §4) leads to different scattering properties when the plane of incidence is aligned parallel to or perpendicular to the groove axis. Note the broad peak in BRDF values for scattering angles < 10 degrees when the plane of incidence is perpendicular to the grooves. These higher BRDF values may be caused by multiple reflections in the microgroove.

The uncorrected infrared directional hemispherical reflectance of the ESLI carbon fiber coating is shown in Figure 5 for several incident angles. Note that the DHR increases slightly with wavelength and is only weakly dependent on incident angle out to 60 degrees. For the thermal infrared (8-12 μm), the mean and standard deviation of the DHR is 0.45 ± 0.20% for an incident angle of 20°, 0.78 ± 0.20% at 40° and 1.9 ± 0.3% at 60°. For a 30° incident angle, the zero offset in the thermal infrared of the FTIR - integrating sphere system was measured to be < 0.20%.

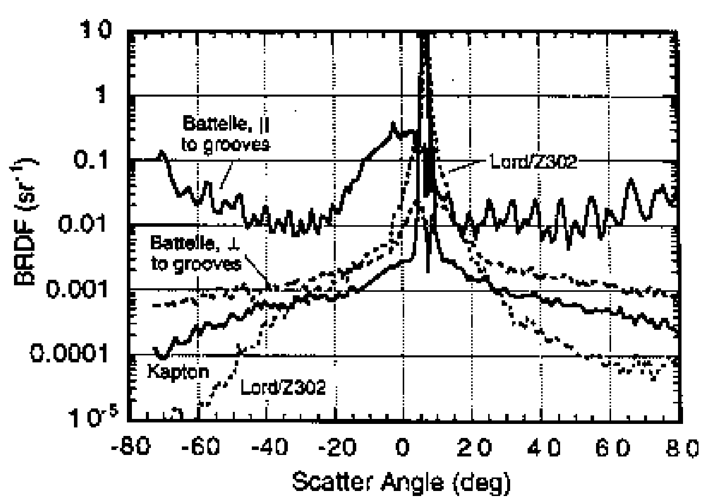

Figure 4. Infrared BRDF of non-Lambertian appliques and Lord Corporation Z302 coating versus scatter angle for an incident angle of 7.5 degrees and a wavelength of 10.6 μm

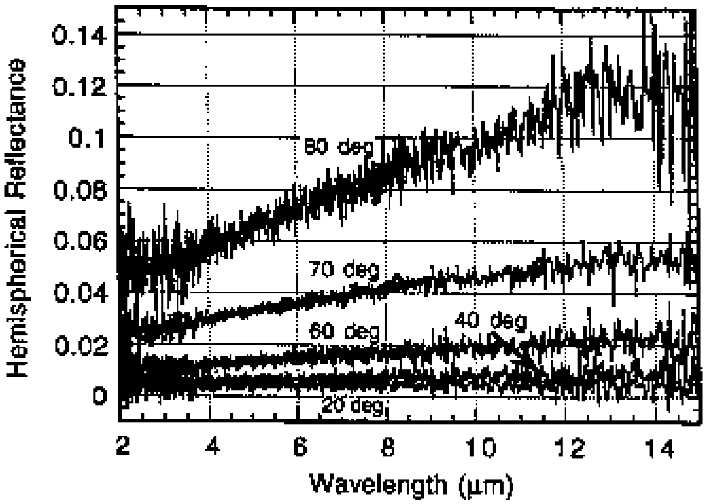

Figure 5. Infrared directional hemispherical reflectance of ESLI sample CLSA96/1 from 2-15 microns for incident angles of 20, 40, 60, 70 & 80 degrees.

For some applications, it may be desirable to have lower reflectance values near grazing incidence than the values shown in Figure 5. For those cases ESLI has developed a 'biased' coating in which the axes of the carbon fibers are tilted relative to the base plane of the coating. One of these biased coatings was evaluated at 3.39 µm on the Wright Laboratory hemispherical scatterometer. The fibers were biased at approximately 30-35° from the normal to plane of the substrate. The in-plane, unpolarized BRDF of the biased ESLI coating is shown in Figure 6 for incidence angles of 40, 60 and 80 degrees. The cross-plane unpolarized BRDF of the same sample is shown in Figure 7 for the same incidence angles of 40, 60 and 80 degrees. Tie asymmetry of the cross-plane scans was caused by either a waviness in the sample or the incident beam not being perfectly perpendicular to the lay of the fibers. The cross-polarized, cross-plane measurements at a 40 degree incidence angle (not shown) were below the noise floor of the scatterometer (2 x 10^{-4} sr^{-1}) for small scattering angles. Hence, these data were assumed to be zero when calculating the unpolarized BRDF. The 40° and 60° data are consistent with a DHR of less than 1%; the 80° data suggest a DHR of less than 3%.

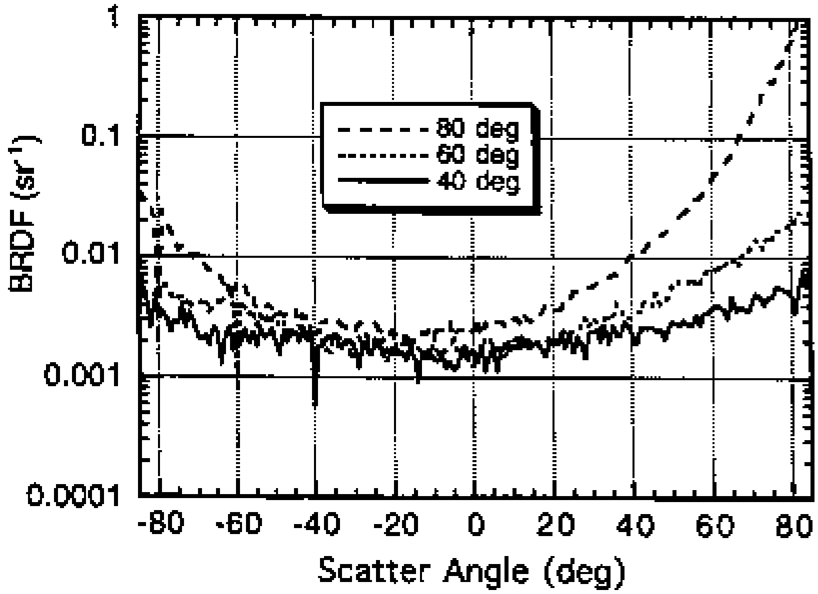

Figure 6. In-plane unpolarized BRDF versus scattering angle for a biased ESLI sample at λ = 3.39 μm and incident angles of 40, 60 & 80 degrees.

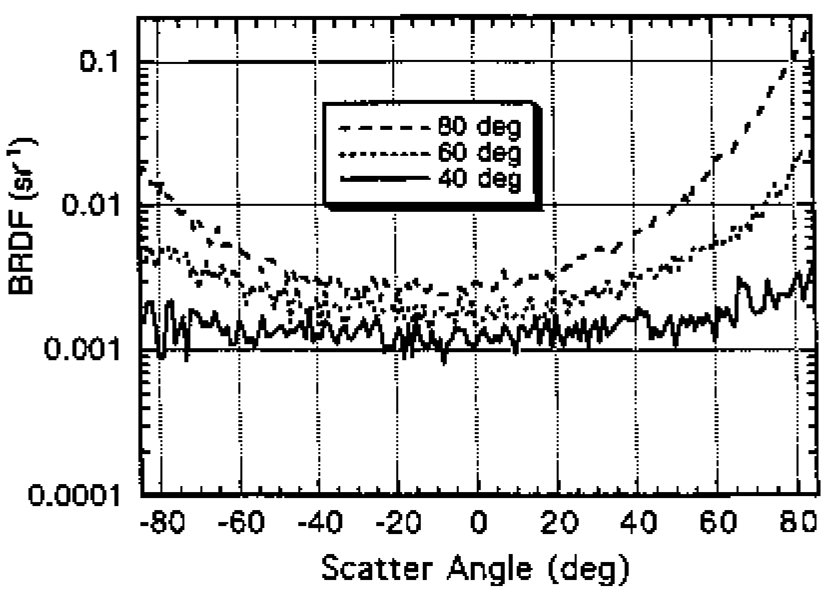

Figure 7. Cross-plane unpolarized BRDF for a biased ESLI sample for λ = 3.39 μm and incident angles of 40,60 & 80 degrees.

4. SURFACE MICROSTRUCTURE

The microstructure of the appliques studied was characterized with a Hitachi S-800 field emission scanning electron microscope (SEM). The surface structure of the ESLI, Edmund, Rodel, Rippey and Battelle appliques is shown in Figure 8a-e at a magnification of 150x. For comparison, the Martin Black sample reported on in this paper is also shown in Figure 8f. All of the appliques utilize cavities to trap incident radiation. Note that the mean cavity sizes of all of the appliques are significantly larger than the corresponding dimension for Martin Black. This suggests that the appliques studied may remain diffuse at significantly longer wavelengths than Martin Black. The ESLI and Battelle coating processes permits tailoring of the coatings' characteristic dimensions, thus enabling one to tune the coating for a specific wavelength range. The period of the grooves in the Battelle sample is approximately 100 μm. Tilted micrographs of the Battelle sample (not shown) show that the included angle in the groove is less than 35°. The peaks of the Battelle sample's ridges have a flat plateau approximately 6 μm wide. At grazing incidence, this plateau renders the coating semi-glossy in the cross-groove direction. The coating remains black at grazing incidence when the sample is viewed along the groove direction.

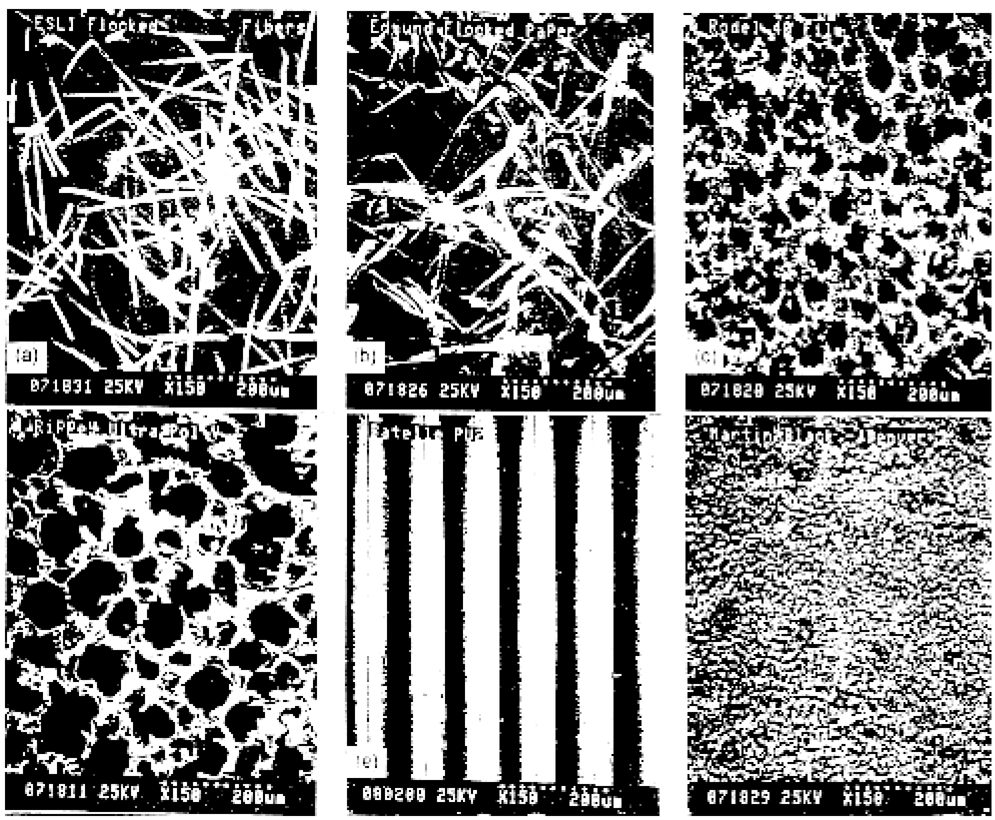

Figure 8. Scanning electron micrographs of samples from a) ESLI, b) Edmund (flocked paper), c) Rodel (40 film), d) Rippey (UltraPol V), e) Battelle and f) Lockheed Martin (Martin Black). All samples were photographed at a magnification of 150x.

5. ACKNOWLEDGEMENTS

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