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Integrity Testing Laboratory Inc.

Surface Technologies: An Overview

Integrity Testing Laboratory (ITL) Incorporated is a high-tech, diversified technology-oriented company that was organized to provide consulting, R&D, testing and characterization services to the industrial and scientific communities in Canada and abroad. One of its principal businesses is the development of innovative new surface modification technologies for polymers and composites, in order to add value to existing materials. This paper presents an overview of ITL's existing and developing surface modification technologies:

Photosil™ - the surface treatment originally developed to protect polymer materials from atomic oxygen erosion and polymer degradation often found in low Earth orbiting spacecraft

HydraRelease™ - our core technology for release systems

SulSil™ - highly wettable sheeting surface technology

NanoSil™ - highly water-repellent surface technology

Implantox™ - protective ion implantation technology

Surface Texturing – highly diffuse reflective surface technology

Photosil™ Technology

The Photosil™ process is a surface modification technology which substantially alters the surface structure and chemistry of a polymer. Originally developed to protect polymer materials used in space exploration, Photosil™ is an innovative process with many terrestrial applications protecting polymer materials in a wide range of environments. The process alters a range of surface related properties including: resistance to oxidation and atomic oxygen erosion, wettability, adhesion, and permeability of gases and liquids. The versatile Photosil™ process can be adapted to meet these properties. The Photosil™ process incorporates silicon-containing groups into the sub-surface layer (i.e. up to 1 µm in depth) of the polymer structure. Such silicon surface modification produces the unique properties which differentiate the Photosil™ process.

The Photosil™ process is able to effectively modify a wide variety of polymer and composite materials. These materials range from polyethylene to complex polyimides. The Photosil™ process produces a uniformly graded layer, not a coating.

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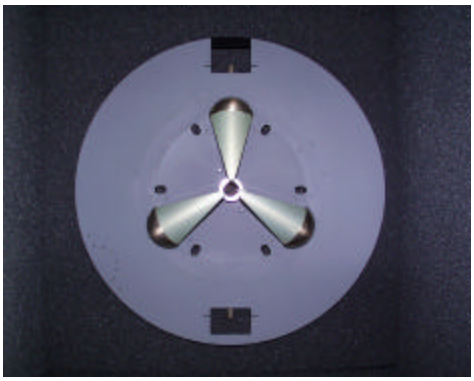
Unlike a coating, the graded layer lacks an abrupt transition boundary and thereby resists cracking and spalling caused by thermal stress, physical stress, and even corrosion. Under certain circumstances, the modified surface structure also has a unique self-healing capability. A key advantage of the Photosil™ process is that, unlike other surface treatments, only a thin layer near the surface is modified, resulting in little or no change to the bulk material properties of the surface material.

The Photosil™ surface treatment significantly reduces, even eliminates the atomic oxygen erosion of polymer materials used in spacecraft applications. The Photosil™ process has shown to provide erosion yield of ~10^{-26} cm^2/at. under fast atomic oxygen exposure, i.e. two orders of magnitude lower than the best polymer materials used presently for space applications, for a variety of materials including: Kapton®, Mylar®, PEEK, polyethylene, PVC, polyamide, graphite reinforced PEEK, epoxy composites, and polyurethane-based paint. The process does not affect the thermal-optical properties or mechanical properties of the treated materials, a critically important factor for space applications.

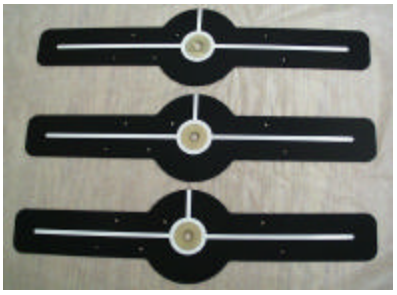
One of ITL's clients is MD Robotics Ltd., designer and builder of the Space Shuttle and the Space Station Remote Manipulator System (RMS) robotic arms, also known as the Canadarms. Photosil™ is employed to protect polymer-surface components of NASA's Canadarm systems, which has been instrumental to the success of many space missions. As well, Photosil™ has been used to treat polymer-surface components of the Special Purpose Dexterous Manipulator (SPDM), a sophisticated evolution of the Canadarm to be installed onboard the International Space Station.

Photosil™-treated Space Components:

Grapple Fixture



Target Plates



The visual results below demonstrate the detrimental effect of atomic oxygen exposure and the protective effectiveness of Photosil™. The unprotected half of the

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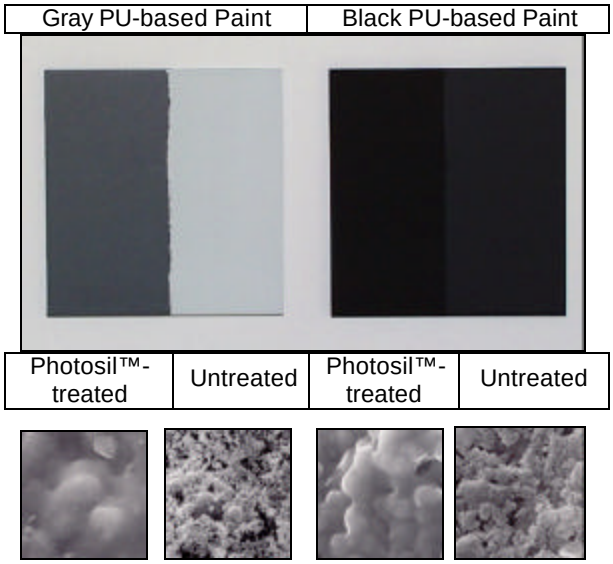
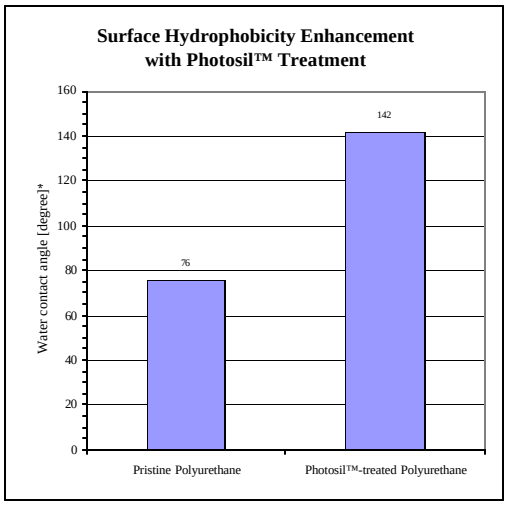
paint exhibited considerable surface erosion while the protected half remained almost intact. The atomic oxygen eroded the polyurethane (PU) portion of the untreated paints, leaving behind the exposed pigments.

O_2 Plasma-exposed Test Coupons



Magnification 2000x

In addition to atomic oxygen erosion protection, the Photosil™ treatment can significantly enhance the degree of hydrophobicity of polymer surface. With the increasing safety concerns in the aviation industry, the industry faces a challenge to prevent the loss in radar sensitivity when aircrafts fly in rain. By varying the silicon chemistry and processing conditions, the adaptable Photosil™ process has shown to create a super-hydrophobic surface which can prevent or minimize radar signal loss by forming water droplets on the radome rather than wetting the entire surface. Contact angle with water as high as 145° has been achieved.



*****Static contact angle was measured using a 6 µL droplet of deionized water with a Krüss G10 instrument (sessile drop method).

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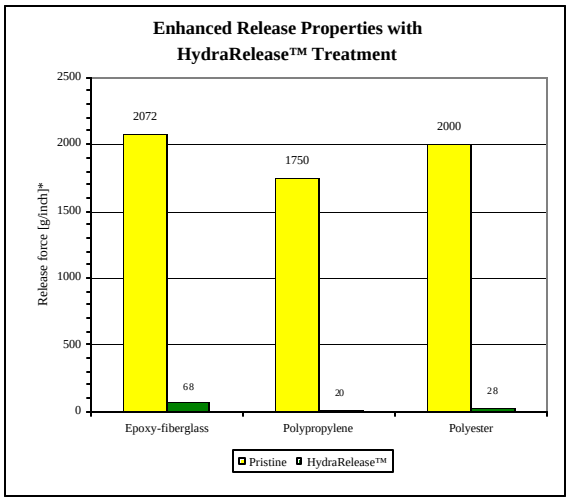
HydraRelease™ Technology

HydraRelease™ Technology is a Photosil™-derived surface treatment, which substantially enhances the release performance of a wide variety of polymer and composite materials. The HydraRelease™ process provides a silicon-enriched surface with permanent release properties, which are suitable for release of pressure sensitive adhesives and tacky industrial materials. An advantage of the HydraRelease™ process is its ability to simultaneously modify the sub-surface region of the original substrate and graft a thin release layer onto the substrate, thus reducing the abrupt transition boundary. Another key advantage of the HydraRelease™ technology is that the treated surface exhibits non-transferring capabilities. In other words, the HydraRelease™ process provides an inert and durable release surface that does not transfer to the surface in contact (i.e. the molded part or adhesive). Potential applications are release surface for molds and tooling materials for plastic/rubber processing and release liner for pressure sensitive adhesives.



*90° peel release test at 300 inches/min with C4704 acrylic-based adhesive tape.

The following example illustrate some of the results of the HydraRelease™ Technology:

A polypropylene (PP)-based film was HydraRelease™-treated. The tables below present the performance characteristics of the treated film. This data clearly indicates that the HydraRelease™ technology can offer remarkably good and stable release properties. The Percent Retain (Subsequent Adhesion) test was conducted to check whether any of the release component is transferred to the adhesive when the tape is peeled from the treated surface. Besides good release properties, HydraRelease ™ process offers superior percent retain, 95%. From this test, one can conclude that there was no considerable loss of adhesive strength, thus suggesting an insignificant amount of release material being transferred to the adhesive. Another measure of the quality of the release surface is Silicon Transfer. This test of high sensitivity

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determines whether the treated film contains loosely bounded silicone particles, which can transfer to other surfaces. By comparing the dot size, there was no reduction in the spreading of the test dye solution, thus indicating no silicon transfer.

	Acrylic-based adhesive tape	
Performance Characteristics	**C4704**	**HD11F4**
Release Force [g/inch-width]* RT Aging (20hr, 10-lb load, 24°C)	20±1	58±7
Release Force [g/inch-width]* Keil Aging (20hr, 10-lb, 70°C)	34±4	89±6
Percent Retain [%]** (Subsequent Adhesion)	95±3	95±2

*Test determines the peeling/release force required to remove a treated liner 1 inch wide from an adhesive tape at a 90° angle and 300 inches/min. speed.
**Test determines the ratio between the force required to remove a tape from a standard stainless steel test panel after its contact with a treated liner, and a force required to remove the same size original tape (no contact with treated liner) from the same test panel. Peel tests are performed at a 180° angle and 12 inches/min speed.

	Pristine PP	HydraRelease™-treated PP
Silicon Transfer Test*		

*Test determines the amount of silicone transferred to the adhesive surface after it has contact with a treated liner. When an alcohol dye solution (0.1% Crystal Violet in i-propanol) is put onto the adhesive side of a tape (3M 810 tape), it will spread. As the silicone contamination increases, the spread decreases. By comparing the change in the dot spread, we have a very accurate measure of the surface silicone.

Some of the major advantages of HydraRelease™ process:

- Simple and easily controlled processing techniques and conditions
- Room temperature vulcanization (RTV)/moisture-curable system
- One-part curing system with fewer chemical components in the formulations
- Available in solvent-free formulations
- Exceptional release performance characteristics

SulSil™ Technology

SulSil™ is a super-hydrophilic coating technology initially developed for anti-fog applications. There are many applications where anti-fogging technology can be used. In addition to the more obvious anti-fogging requirements of bathroom mirrors, glasses and goggles, this technology can be utilized on reflective road signs to prevent dangerous fog buildup. This technology can also be used to prevent dew formation since water has the tendency to sheet over the SulSil™ surface rather than form droplets. This is a useful application for greenhouses where interior high humidity environments often cause droplets containing high levels of bacteria to form and drip down onto the plants below. The sheeting action of SulSil™ causes the water to run down the sides of the greenhouse where it can be collected and properly disposed of.

The SulSil™ coating can be applied to many possible polymer substrates. This technology has been successfully applied to acrylate and polyolefin based materials with static water contact angles of less than 10^o being present on the surface after treatment. By comparison normal polymer materials have static water contact angles of over 70^o.

The following photos demonstrate the advantages of SulSil™ technology on

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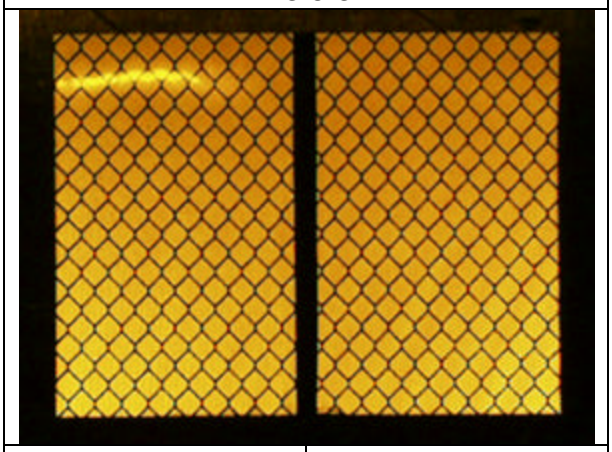
reflective highway traffic sign material. Half of the reflective material was treated with SulSil™ technology and the material then exposed to a dew environment. The non-treated side quickly loses all of its reflective properties while the SulSil™ side remains virtually intact.

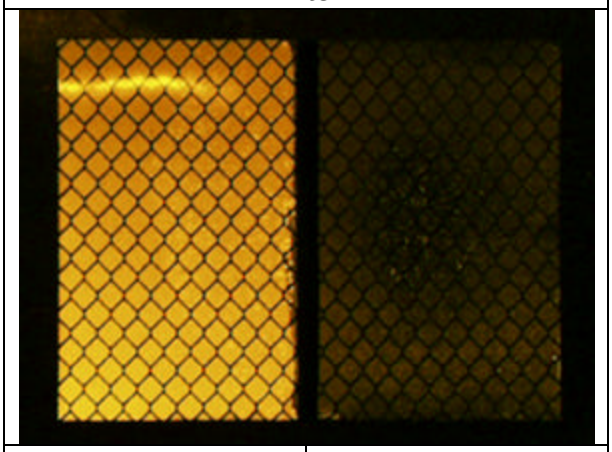
Dew Testing

Before



SulSil™	Non-treated

After



SulSil™	Non-treated

NanoSil™ Technology

NanoSil™ Technology is a new super-hydrophobic surface treatment, which significantly increases the hydrophobicity and water repellency of polymer surfaces. The NanoSil™ process simultaneously modifies the sub-surface region of the original substrate and grafts a highly hydrophobic top layer on the substrate. Therefore, reducing the sharp interface between the substrate and incorporated layer. NanoSil™-treated surfaces can achieve water contact angle as high as 160°. Another unique characteristic of NanoSil™-treated surfaces is its ability to repel water to an extraordinary degree. Unlike other hydrophobic coatings, the coalescence of droplets on the surface into puddles that form rivulets does not occur with NanoSil™-treated surfaces. Water droplets that form immediately run off the treated surface.

A super-hydrophobic surface can be utilized in preventing or minimizing icing, frosting, water adhering, and snow covering. For example, super-hydrophobic surface, which repels water to extraordinary degree, prevents or minimizes the loss in radar sensitivity caused by water filming/sheeting on radome while flying in rain. Similarly, ice formation on airplane surfaces may be alleviated with a super-hydrophobic surface, which inhibits the adhesion of water droplets and causes ice release due to the minimal interfacial contact between the ice and the surface.

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The following examples illustrate some of the results of the NanoSil™ Technology:

Example 1

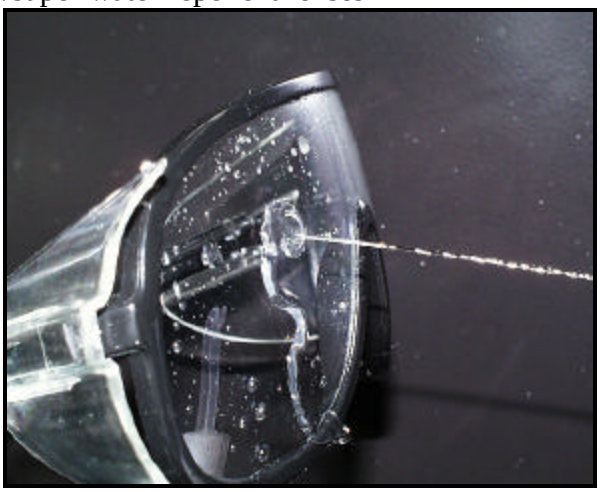
A pressure sensitive adhesive-backed polyurethane (PU) film was NanoSil™-treated, and then laminated on the surface of a polycarbonate lens. Figure 1 below compares the degree of water repellency of a pristine lens and the lens laminated with the super-hydrophobic/super water repellent film. The untreated lens formed partially wetted areas and rivulets indicating low hydrophobicity. However, the laminated lens showed extraordinary degree of water repellency. The water droplets bounced off the surface, and there was a very small percentage of droplets that cover the surface.

Table 1. Water contact angle data

	Pristine PU	NanoSil™-treated PU
Water contact angle*	76°	150°

*Static contact angle was measured using a 6 µL droplet of deionized water with a Krüss G10 instrument (sessile drop method).

Figure 1. Untreated and super-hydrophobic /super water repellent lenses



Untreated lens

Lens laminated with the super-hydrophobic film

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Example 2

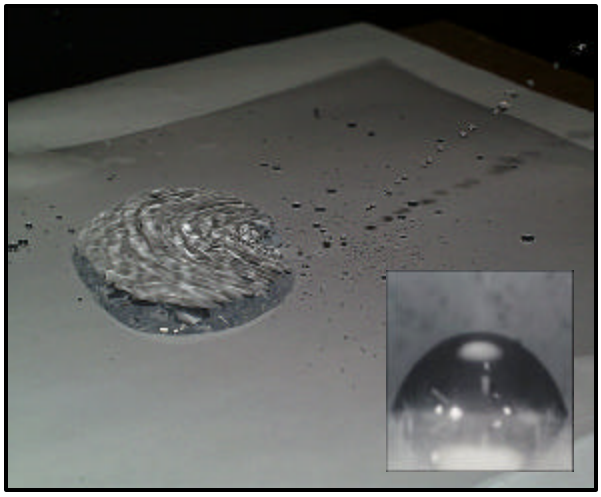
A polyester (PET) film was treated with the NanoSil™ process. The average water contact angle of the treated film was around 155-160° with a tilting/sliding angle of less than 10° (6 µL water droplet). Figure 2 displays the degree of water repellency of a pristine and the super-hydrophobic treated PET film. The water droplets on the pristine film coalesced on the surface into puddles and eventually wet a large percentage of the surface. On the other hand, the treated film exhibited extremely high water repellency. The water droplets bounced off the surface, and there was low water surface coverage.

Table 2. Water contact angle data

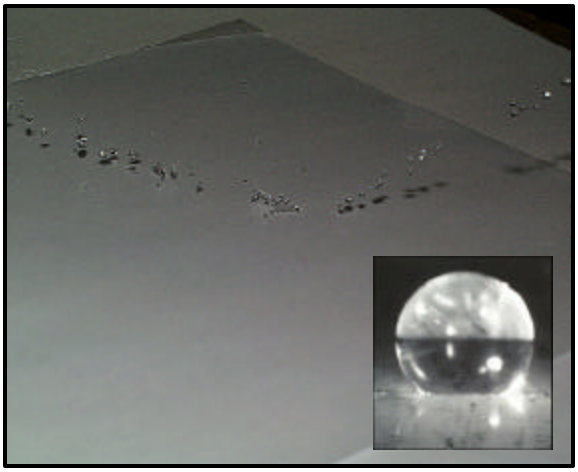
	Pristine PET	NanoSil™-treated PET
Water contact angle*	79°	150-160°

*Static contact angle was measured using a 6 µL droplet of deionized water with a Krüss G10 instrument (sessile drop method)

Figure 2. Untreated and super-hydrophobic /super water repellent treated PET films



Untreated PET film



Super-hydrophobic-treated PET film

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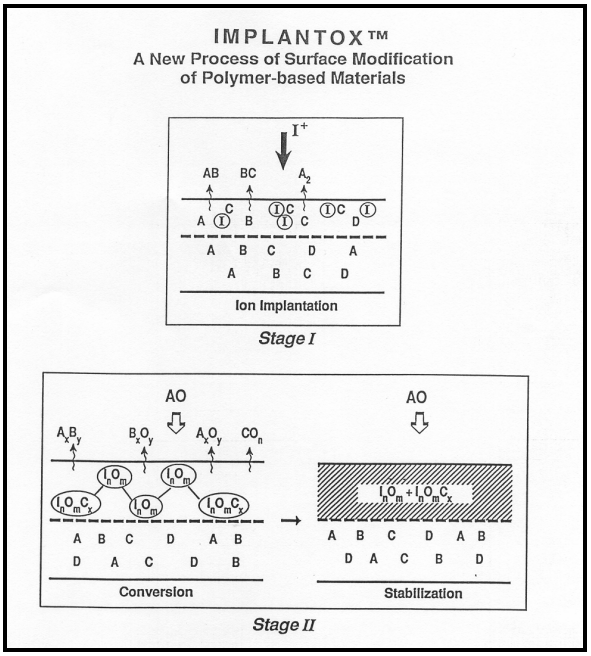
Implantox™ Technology

Implantox™ is an ion implantation technology, which was initially developed toward the atomic oxygen protection of polymers and carbon-based composites in low Earth orbit. Implantox™ greatly increases the erosion and oxidation resistance of polymeric materials. The technology also substantially improves mechanical, electrical, and optical properties. This new method of surface modification is based on high-dose ions implantation of metal or semi-metal combined with a special oxidative post-treatment, which together produce graded oxide(s)-based surface layers. Surface-sensitive properties of the modified layers can be tailored and controlled by the conditions of ion implantation and the type of oxidative post-treatment selected.



A major appeal of the ion implantation approach over conventional inorganic coatings, especially on spacecraft in low Earth orbit, is that the difficulties associated with brittleness, mismatch of coefficients of thermal expansion, and change in surface morphology are mitigated by the creation of a graded surface-modified region. Therefore, problems of adhesion, thermal mismatch, and change in dimensions disappear. Virtually all hydrocarbon polymers and carbon-based composites can be modified by Implantox™ which is a non-thermal process, thus allowing for low melting temperature materials to be treated. Furthermore, the process is absolutely clean and environmentally friendly.

In fact, research studies confirm that the Implantox™ surface treatment has the following benefits:

- Protects polymers and composites from space environment in low Earth orbits
- Increases the oxidation resistance in highly oxidative environments, such as atomic oxygen, ozone, oxygen plasmas
- Improves the durability of polymeric materials and coatings
- Allows tailoring the degree of hydrophobic and hydrophilic properties
- Enables control of optical and thermal-optical surface properties, including refraction, reflection, color, and degree of transparency
- Allows tailoring of surface conductivity to meet specific needs

Based on current data, the ion implantation technique shows excellent promise for the

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protection of polymer-based materials for long-duration space applications. Based on the variety of unique surface structures developed on polymers, the technology may also be suitable for many terrestrial applications.

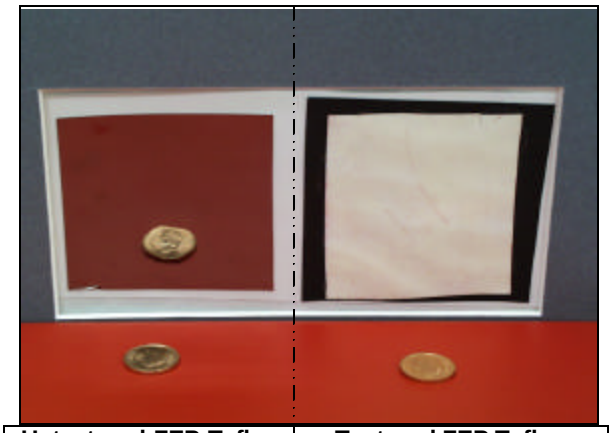
Polymer-based biomaterials and advanced sensors with improved resistance to oxidation and adjustable hydrophilic/ hydrophobic properties may be an outcome of this technology transfer. Advanced gas and liquid sensors for space applications are also possible with this technology. A successful "marriage" of polymer film substrates with graded metal or silicon oxide-based structures is especially interesting for new sensor development.

Surface Texturing Technology

The surface texturing technology is a novel ion-beam process, originally developed to reduce glare and increase diffuse reflection on Teflon surfaces for improved performance of video cameras on Canadian Mobile Servicing Station (MSS) for the International Space Station (ISS).

Silver-backed FEP Teflon is used as a passive thermal control material on the ISS, because of its low solar absorptance and high thermal emittance. However, experiments with this material gave rise to the problem of excessive glare which blinded the optical equipment on the MSS used to located targets attached or in close vicinity to the equipment covered with the Teflon film. The solution to the excessive glare problem was to alter the surface of the Teflon film so that the specular reflection from the film would be reduced substantially to an acceptable level, while maintaining its thermal optical properties.

The novel ion-beam surface texturing process can reduce substantially the specularity of silver-backed Teflon thermal control film by changing its morphological appearance in a controlled manner from a shiny metallic-like surface to a white milky appearance without significantly affecting its thermal optical properties.



Untextured FEP Teflon (Silver coated on the back)	**Textured FEP Teflon** (Silver coated on the back)