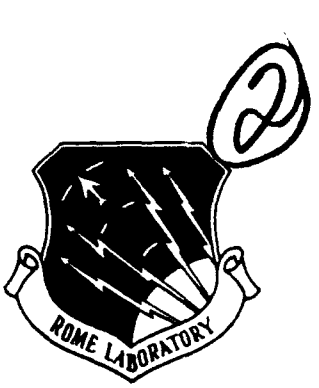

RL-TR-93-109
In-House Report
June 1993

THE APPLICATION OF JET SPRAY AND ION BEAM CONTAMINATION REMOVAL TECHNIQUES TO SAMPLES FROM THE LDEF SPACECRAFT

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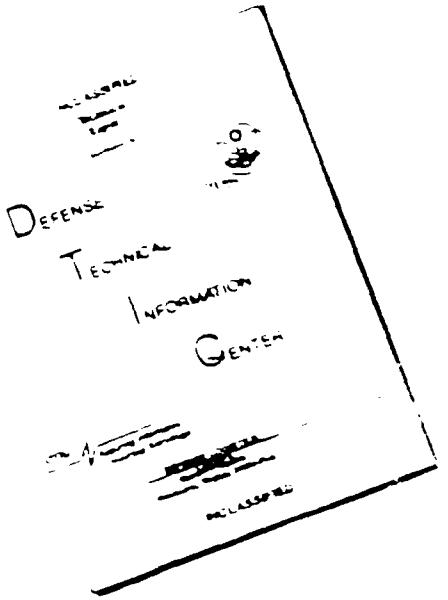

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Two samples from LDEF experiment M0003-4 were analyzed for molecular and particulate contamination prior to and following treatment with advanced satellite contamination removal techniques (CO2 Gas/Solid Jet Spray and Oxygen Ion Beam). The pre- and post-cleaning measurements and analyses will be presented. The Jet Spray removed particulates in seconds. The low energy reactive oxygen ion beam removed 5000 angstroms of photo polymerized organic hydrocarbon contamination in less than 1 hour. Spectroscopic analytical techniques were applied to the analysis of cleaning efficiency including: Fourier Transform Infrared, Auger, X-ray Photo emission, Energy Dispersive X-ray, and Ultraviolet/Visible. The results of this work suggest that the contamination was due to spacecraft self contamination enhanced by atomic oxygen plasma dynamics and solar UV radiation. These results also suggest the efficacy for the Jet Spray and Ion Beam contamination control technologies for spacecraft optical surfaces.

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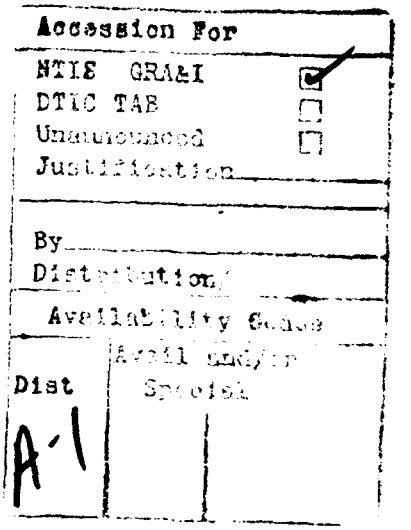
Preface

This paper was presented at the High Power Optical Components Conference held in Boulder CO, in October 1992. This work suggests the importance of the study of the synergistic effects of contamination, radiation, and space plasmas in spacecraft systems.



THE APPLICATION OF JET SPRAY AND ION BEAM CONTAMINATION REMOVAL TECHNIQUES TO SAMPLES FROM THE LDEF SPACECRAFT

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Two samples from LDEF experiment M0003-4 were analyzed for molecular and particulate contamination prior to and following treatment with advanced satellite contamination removal techniques (CO_2 Gas/Solid Jet Spray and Oxygen Ion Beam). The pre- and post- cleaning measurements and analyses will be presented. The Jet Spray removed particulates in seconds. The low energy reactive oxygen ion beam removed 5000 angstroms of photo polymerized organic hydrocarbon contamination in less than 1 hour. Spectroscopic analytical techniques were applied to the analysis of cleaning efficiency including: Fourier Transform Infrared, Auger, X-ray Photo emission, Energy Dispersive X-ray, and Ultraviolet/Visible. The results of this work suggest that the contamination was due to spacecraft self contamination enhanced by atomic oxygen plasma dynamics and solar UV radiation. These results also suggest the efficacy for the Jet Spray and Ion Beam contamination control technologies for spacecraft optical surfaces.

I. Introduction

Today satellite contamination is kept within specification during production, assembly and storage by clean rooms, solvent wipes, inert gas/air purges and vacuum bakeout. Although these techniques have proven acceptable for launching "clean" satellites, (level 1000 typical), the combined effects of the space environment lead to increased contamination levels once deployed [1,2]. LDEF was initially launched with MIL-STD-1246B Level 2000C cleanliness. This is considered clean by industry standards today, but post recovery LDEF analysis showed over one pound of molecular contaminants notwithstanding particulates [3]. LDEF experiments provide a unique window into the contamination effects on a large variety of spacecraft materials all exposed to the same LEO environment for the same amount of time.

In this paper, will be discussed the results of utilizing the CO_2 jet spray and oxygen ion beam contamination removal techniques for the cleaning of LDEF contaminant species. The overall conclusion of the paper is as follows: *Indeed the proper choice of spacecraft materials and prelaunch cleanliness is important, but the physical realities of the space environment necessitate an on-orbit contamination mitigation philosophy which is potentially implementable using the contamination control techniques described herein.*

II. Preclean Analysis

Two solar cell cover glass samples from the LDEF experiment M0003-4 were analyzed in this study. Sample No. L3-IV-4-14-52 was positioned on LDEF tray D9 on the leading edge of the spacecraft. Sample No. T3-IV-4-14-54 was positioned on the trailing edge of the spacecraft in Tray D3. The Leading Edge Sample (henceforth Sample L) was visually different in appearance than the trailing edge sample (henceforth Sample T).

Sample L collected 5000 Angstroms of an organic contaminant film, scattered particulate debris, and two micrometorite craters. Circular polarized optical microscopy showed the presence of many orders of brightly colored Newton's interference rings on sample L, as shown in Figure 1 (magnification = 3.0x). The cover glass sample was stuck to the silicon backing plate by the contaminant film which acted like a glue. This afforded the opportunity to analyze the effects of this photo polymerized contaminant and contamination removal techniques on both the cover glass and crystalline silicon materials. Subsequent microscopic analysis revealed the presence of a subsurface fracture running across the crystalline silicon sample. This defect was deemed responsible for the sample becoming severed in the analysis procedure.

Sample T on the other hand collected only 50 angstroms of a light brown contaminant film and scattered particulate debris. This sample was not "glued " to its silicon backing plate. Sample L was in two parts as can be seen from close examination of Figure 1. As discussed above, sample T was not found to be as heavily contaminated as sample L, and was not fixed to its crystalline silicon backing plate. Figure 2 shows sample T under the same conditions as Figure 1, except that it was positioned above a black microscope ruling disk (central rule = 1mm). Note the presence of particulate debris as evident from the density of optical scatter centers. Figure 3 shows sample T photographed at a magnification of 3.25x positioned upon square graph paper (20 squares per inch). From this figure, the thin brown contaminant film is clearly seen as a contrast difference.

The physical condition of these samples, was anti-intuitive. Since the Leading Edge sample experienced a higher atomic oxygen (AO) fluence than the trailing edge of the spacecraft [4], one would expect a fairly clean-contaminant free surface. It is possible that such a surface would even be slightly eroded due to interaction with the reactive ion flux. During recovery, the AO fluence for sample L was measured to be 8.74×10^{21} atoms-cm^{-2} during the five year mission. The trailing Edge sample was somewhat shielded from this atomic oxygen flux, having an AO fluence of 1.3×10^{17} atoms-cm^{-2}. Intuitively, the author expects this to imply a thicker contaminant deposition on the trailing edge relative to the leading Edge which was not the case for the two samples examined in this work.

A possible pathology for the deposition of the observed contaminant film is as follows. The interaction with the AO flux induced catalysis of the nylon 6:6 and polyacetyl delrin 500 sample holder materials resulting in a plasma sheath of dissociated polymeric species following the sample throughout its orbit. The solar UV photon flux polymerized these species to the substrate surface, while the plasma sheath partially protected the deposited contaminant film from AO etching. This resulted in the 5000 angstroms of the hydrocarbon co-polymer film discussed above. Of course this is mere speculation, and the author has no doubt that several other pathologies can be cogitated for growth of this unexpected contaminant film. The author is still speculating as to the reasons for this contamination density inversion.

II. Contamination Removal

Gas/Solid Jet Spray Technique

The Gas/Solid Jet Spray was used to remove particulate contamination. The CO_2 jet spray is shown in Figure 4. The jet spray has been described in the literature [1,2], but may be simply described as a particle removal process which exploits momentum transfer from incident snow

flakes to particulates adhering to the surface through van der Walls forces (first and second order). The energy/momentum transferred to the adsorbed particle breaks these surface potential forces and the "free" particle is entrained in the gas stream and carried away from the surface. The mixture of solid/gas in this process is very important for the removal of submicron particles [1]. which are not removed by high pressure gas and liquid streams due to the gas/surface boundary layer's "insulating" action.

Ion Beam Technique

The molecular film was removed by reactive ion etching using a beam of oxygen ions and electrons from a Hughes helicon wave source (HWS) shown in Figure 5. The output beam contains oxygen ions and neutral atoms as well as electrons. The HWS also has a UV radiation component. The effects of these species upon contaminant removal is under investigation. The ion cleaning experimental parameters are as follows. The ion energy was varied between 12 and 45 eV (average). The ion flux densities varied between 550 and 1300 $\mu A/cm^2$ (average) as measured by a Faraday cup. The plasma was operated at 165 Mhz with a power of 10 to 20 Watts. The oxygen flow rate was measured to be 10 sccm using an Omega Engineering gas flow meter (FMA-5601). Chamber partial pressures were monitored by a VG Scientific Micromass 560 mass spectrometer to be: Oxygen: 3x10-5 Torr, Water: 3x10-5 Torr, and Nitrogen: 5x10-5 Torr. Other species were present in the chamber registering partial pressures of less than 1x10-8 Torr, and as such were of no consequence to this work.

III. Contamination Removal Analysis

Figure 6a (left) shows a circular polarized light micrograph of a heavily contaminated region of sample L. This Figure is a montage of photomicrographs pasted together in a jigsaw puzzle fashion since the field of view for one micrograph at 13x was too small to contain the entire sample. The brightly colored interference rings indicate the presence of a molecular contaminant film. Figure 6b (right) is the same region of the sample after 21 minutes of treatment with the reactive oxygen ion beam contamination removal device. Figure 7 is a Nomarski photomicrograph of the same region of the sample after an additional 40 minutes of treatment with the ion beam cleaner and a 3 second spray with the CO_2 jet spray.

In one hour of total treatment time, the sample went from being contaminated at levels that the unaided eye could easily discern, to having a contamination level at the Nomarski Microscopy threshold of detection .

Figure 8 is a Dark Filed Micrograph (DFM) of a section of sample L contamination prior to ion cleaning, and Figure 9 is a DFM of the sample after ion treatment. Note the small bright specks on the surface of the sample. These scatter centers were found to be the result of micro-abrasions and not submicron particulates as might be presumed from a casual look at the Figure 8 precleaning data. Higher magnification (50x) Nomarski and DFM photomicrographs of this general region of the sample are shown in Figures 10 and 11, respectively. The sample was masked during cleaning by the cover glass shown on the left. The glass protected the underlying contaminant film from the ion etching process while areas to the right of the mask were cleaned. There is evidence of a large particle just to the right of the mask and what appear to be smaller particles scattered to the right of the mask in the photograph. The DFM of Figure 11 however, suggests the presence of many more particles distributed over the sample. Strong cross lighting and the Nomarski objective illuminated the surface defects of Figure 12. This data was digitized and processed using the

MacPhase digital image processing software [5], which showed that the scatter centers were indeed micro abrasions rather than particles. Typical contour maps for these micro abrasions are shown in Figure 13. The vertical scale is calibrated in gray scale intensity and the horizontal scale in pixels. At the time of the analysis, physical length calibration was not performed, rather a semi-quantitative indicator of surface hole rather than raised prominence was given. It is unlikely that the ion cleaning or jet spray are responsible for these micro abrasions, since these techniques have been applied to several samples of glass in laboratory tests and showed no such effects.

The microcraters are remarkably similar to the space charge induced divots observed in optical materials in separate experiments by the Naval Air Warfare Center (NAWC) [9-10], Vanderbilt University [15] and the Aerospace Corp. [8] on Simulated Space Radiation Experiments. The NAWC work simultaneously presented enhanced-reflectance mirror coatings with protons, electrons, neutrons and UV photons in a cryogenic vacuum environment to simulate the LEO and MEO environments. For this LEO simulation scenario, the conditions were similar to those of the LDEF samples [14] with the exception of the AO fluence; the LDEF samples analyzed by RL were subjected to an AO environment while the NAWC samples were not. This difference notwithstanding, the microcraters observed by NAWC bear remarkable similarity to those of this and ongoing research [12]. Aerospace Corp. work [8], although performed at higher radiation doses than those of the LDEF environment, also shows formation of microvoids as a result of ionizing radiation in dielectric materials.

The geometry of the microcraters observed in these samples has a smooth walled, conical or sometimes cylindrical nature with no apparent radial shock wave induced ripples such as those observed in craters produced by particle impacts. Such craters have been documented not only in LDEF and other spacecraft studies, but also micron-sized particle impacts from ground based impact simulations using rail gun electromagnetic accelerators [13]. **The lack of correlation of ground based radiation induced void-type defects and those due to particle impacts, taken together with the correlation between the LDEF microvoids of this study and ground based radiation induced voids, suggests that micron sized particles of interplanetary dust or space debris are probably *not* the origin of these defects. The interpretation at this time, with the available data is that these submicron voids are due to atomic diffusion (mass migration) due to radiation induced electrostatic charging effects.** At this time, the author is inclined to view radiation induced charge trapping as the phenomena responsible for the microvoids in question, although more research should be performed to test this hypothesis.

The above microvoids appear to be smaller than the typical micrometeorite and space debris impact images reported to date, including those from the Interplanetary Dust Experiment [11]. For example, Figure 12 is a Nomarski photomicrograph (200x) of a micrometeorite impact crater from sample L. The crater is over 200 microns in diameter with sub-surface damage propagating radially outward for another 500 microns. The Nomarski fringes on the localized material fragments and absence of fringes in the center of the crater suggests the possibility of contaminant film growth prior to the impact and very little growth thereafter. The author speculates that this could be due to two possible damage pathologies. The first is that the impact event occurred very late in the mission, when there was a low rate of contaminant offgassing, leaving little time and probability for further film deposition. An alternative view is that the rough crater surface was an insufficient substrate for film deposition. The DFM of Figure 15 illustrates the presence of collateral damage and secondary debris scattered in the vicinity of the impact site. Computer images of the impact crater show local melting and a cylindrical crown due to energy transfer at impact. This morphology was similar to that of the other, larger micrometeorite impact studied on

the same sample. The crater region is shown at lower magnification after treatment with the cleaning techniques in Figure 16. This figure again shows the presence of micro abrasions after contaminant film removal.

Figure 17 is a blue light fluorescence light micrograph of a masked and unmasked section of sample L. The dark (non-fluorescing) side of the micrograph shows the result of removal of 1760 angstroms of molecular film. There is evidence of residual contamination (brightly fluorescing yellow matter) near the mask boundary. The dark brown and bright yellow species indicate the possible presence of more than one contaminant. Other wavebands used for fluorescence light microscopy (green and UV) also indicated a color difference in these regions.

Fourier Transform infrared spectroscopy (FT-IR) was performed with a biorad FTS-40 spectrophotometer. FT-IR indicated that the contaminant film was composed of nylon 6:6 and polyacetal Delrin 500 [6]. These could have been chemically altered by exposure to space radiation. Auger, ESCA and EDX analyses corroborated this identification contaminants [6]. This contaminant most probably was due to the nylon screws and delrin sample holder used to hold the sample in place during the flight. These chemical analyses, when combined with Nomarski microscopy yielded an approximate molecular film thickness of 4500 angstroms. Auger microprobe analysis yielded a 4800 angstrom thickness for the contaminant film. It is important to note that these contaminants were not the predominant contaminants found on LDEF; most other researchers reported species due to silicone based thermal control paints [3]. although Meshishnek also found evidence of nylon and delrin on the M0003 samples in agreement with the author [7].

Secondary electron imaging and EDX were performed in a JEOL JM840 SEM with a Tracor 5500 EDX spectrometer(Be/Li window). The SEM of Figure 16 (sample T) indicates particulate contaminants ranging in size from tens of microns to millimeters in spatial dimension. EDX analysis of these particles identified them as metallic in composition: Copper, Zinc, Tin, Aluminum and Silicon.

IV. Discussion

Results of the application of the jet spray and ion beam techniques to the leading edge sample indicate a removal rate of 83.3 angstroms/minute for the organic hydrocarbons deposited onto these samples. The cleaning rate and efficiency will of course depend upon the particular species deposited, and the experimental parameters associated with the plasma and substrate. Plasma parameters include. ion energy, ion flux density, neutral energy, neutral flux density, electron energy, electron flux density and UV photon energy and intensity. Substrate parameters influencing cleaning rate include temperature, degree of contamination and surface defect topology.

The microvoids observed after removal of contaminants are most likely to be due to atomic diffusion due to radiation induced electrostatic discharge rather than micrometeorite or space debris impacts. The correlations noted in section III notwithstanding however, in general, the synergistic effects of ionizing radiation, micrometeorite impacts, the near satellite plasma energy density and chemistry and spacecraft particulate contamination interactions result in a large number of void-type defects on the order of a micron and smaller in spatial extent.

The above contamination removal techniques have been shown to successfully remove spacecraft contamination and development is underway to build small, lightweight flight qualifiable

contamination removal systems. However, there remains the problem of preventing the removed contaminants from re depositing onto the cleaned surfaces. In response to this problem, the author developed a contamination collection device [1,2]. This contamination collector is capable of collecting and containing both molecular and particulate contaminants throughout the spacecraft operational parameter space (temperature, vibration, radiation, vacuum and micrometorite environments). One embodiment of this device, the Aerogel Mesh Contamination Collector (AMCC - patent pending) is shown in the SEM of Figure 18. In the figure is shown a cross section of the AMCC with collected particulate contaminants of various sizes. In a system, the AMCC would work in conjunction with the jet spray and ion beam removal devices as shown in Figure 19. Here, the reactive ion beam removes organic particles and molecular films as the jet spray removes particles and entrails the removed species into the AMCC's pores.

Contamination Control For Spacecraft Applications

The above contamination control techniques are being developed for autonomous operation in spacecraft applications. These data present the first results of the application of these contamination mitigation technologies to long duration spacecraft exterior surface materials. The cleaning rates and efficiencies obtained are optimistic. This suggests that further LEO contamination control experimentation should be performed in orbital systems such as the Retrievable Payload Carrier (RPC) [6]. In such an experiment, small jet spray and ion beam sources, contamination detectors, contamination collectors, and possibly even contamination sources would be mounted in a RPC pallet which could be re-used for both leading edge and trailing edge missions, and/or several low cost contamination control pallets could be fabricated and flown on several RPC missions in various locations. RPC contamination experiment data would feed into a contamination control system for Space Station Freedom, and other future space system designs.

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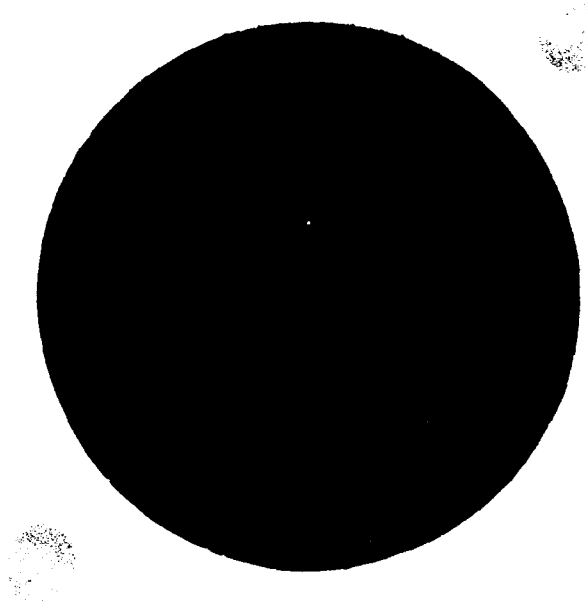

Figure 1 Circular Polarized light micrograph of Leading Edge Sample.

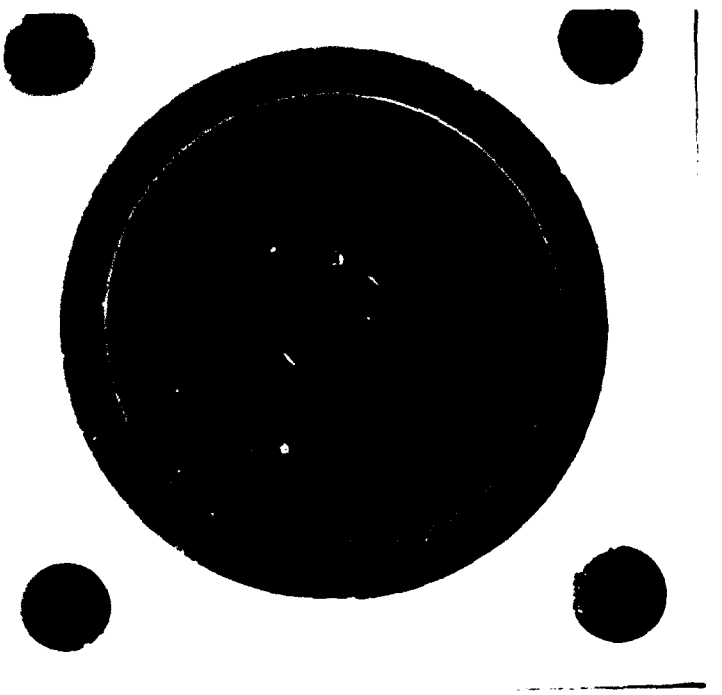

Figure 2 Circular Polarized light micrograph of Trailing Edge Sample.

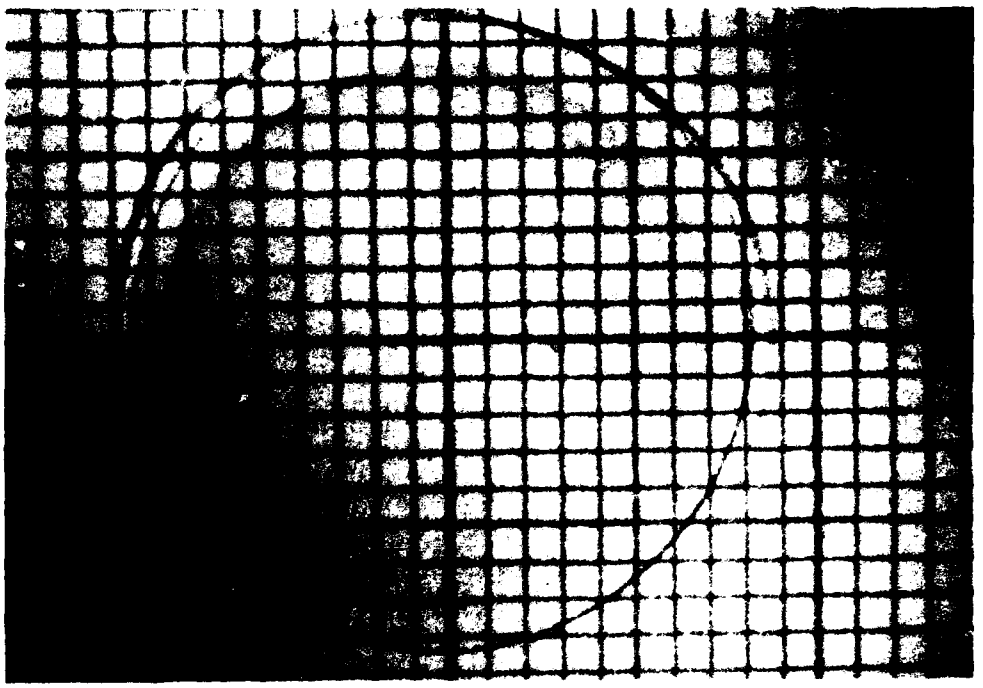

Figure 3 Circular polarized light micrograph of Trailing Edge Sample photographed resting on square graph paper.

Figure 4 The jet spray particulate cleaner in operation.

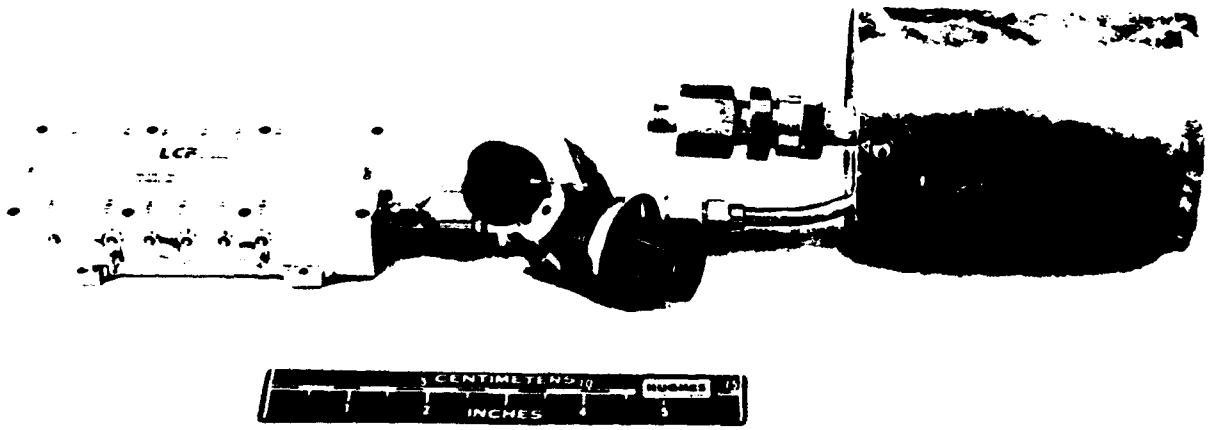

Figure 5 The ion beam Helicial Wave Source. Photo courtesy of Hughes Aircraft Corporation.

Figure 6a (left) Circular polarized light micrograph of a heavily contaminated region of sample L. The brightly colored interference rings indicate the presence of a molecular contaminant film. Figure 6b (right) is the same region of the sample after 21 minutes of treatment with the reactive oxygen ion beam contamination removal device.

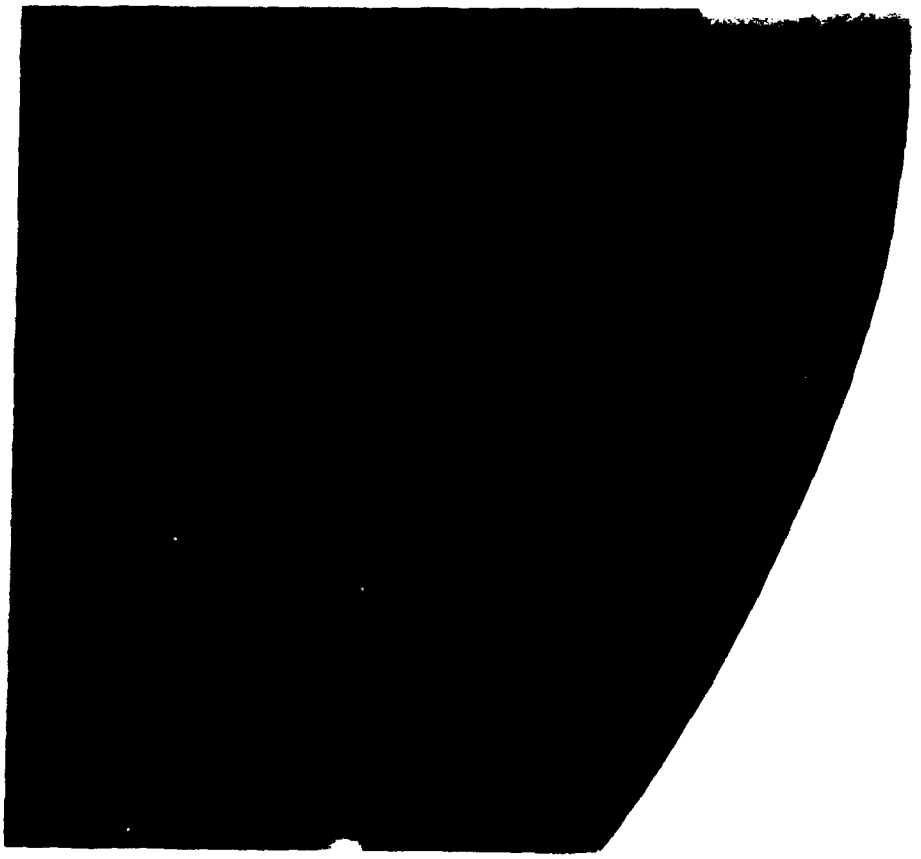

Figure 7 Nomarski photomicrograph of the same region of the sample after an additional 40 minutes of treatment with the ion beam cleaner and a 3 second spray with the CO_2 jet spray.

Figure 8 Dark Field Micrograph (DFM) of a contaminated section of sample L.

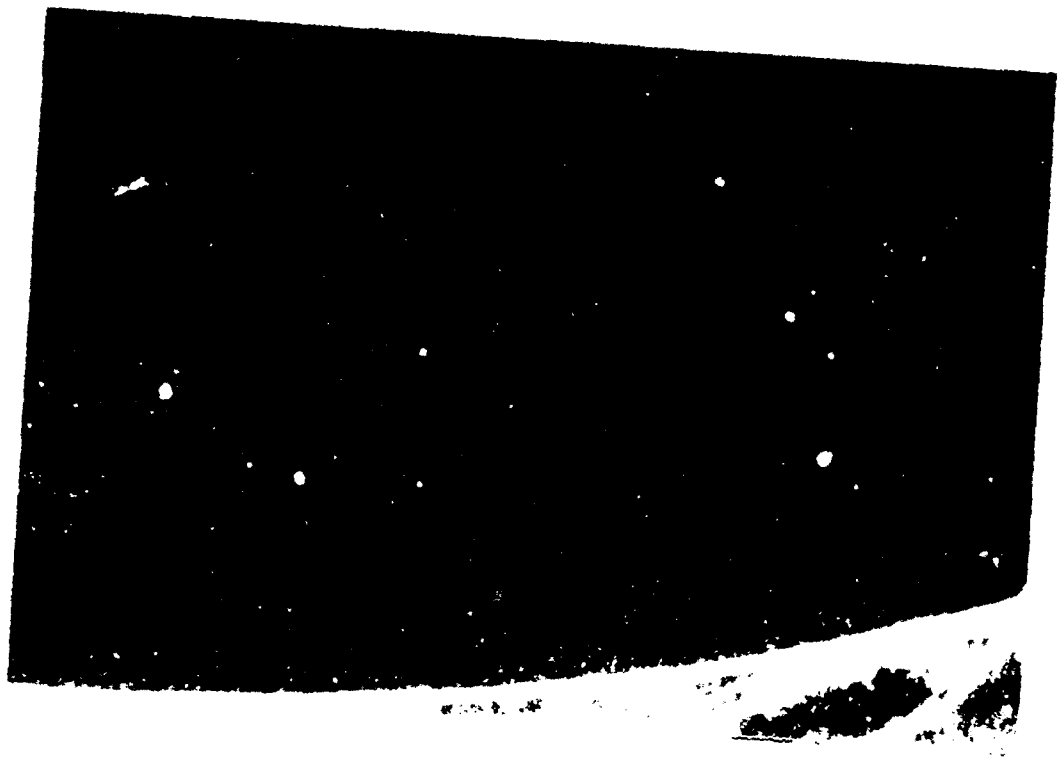

Figure 9 DFM of sample after ion cleaning.

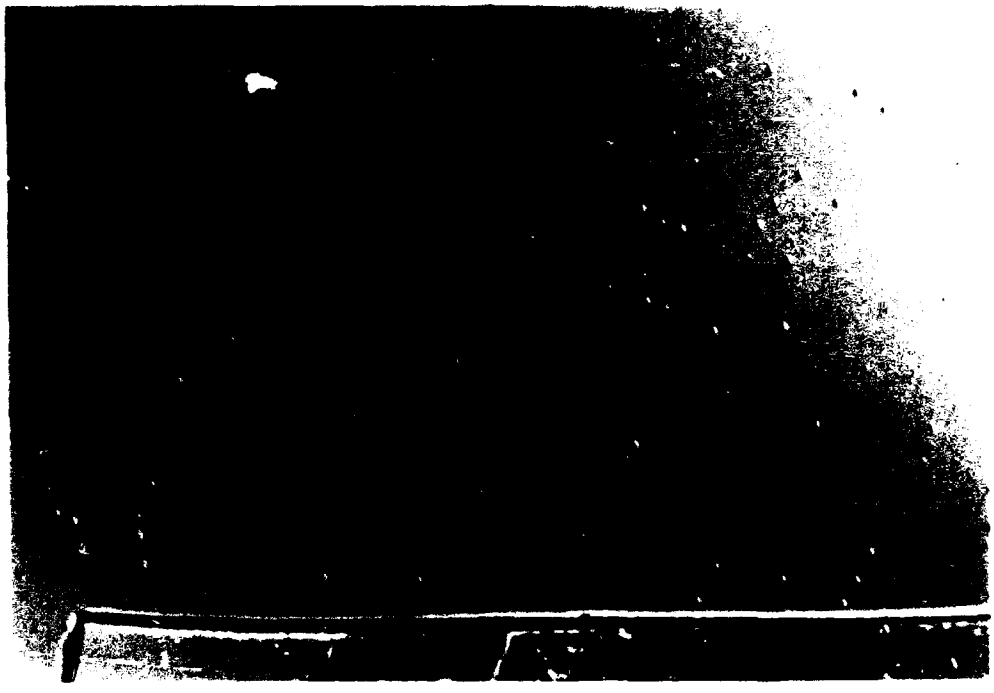

Figure 10 Nomarski photomicrograph (50x) of region of sample L foculsed on cleaned/masked interface region.

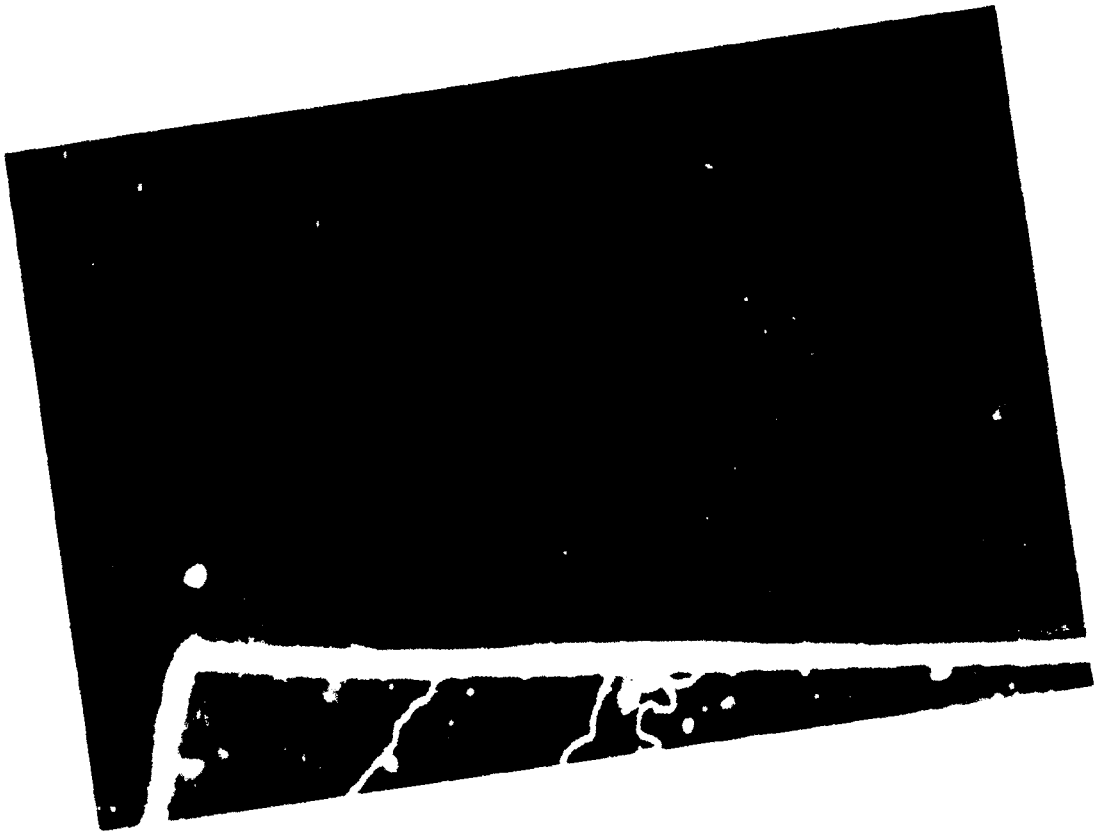

Figure 11 DFM of same region as shown in Figure 8. Bright speckle pattern indicates surface defects.

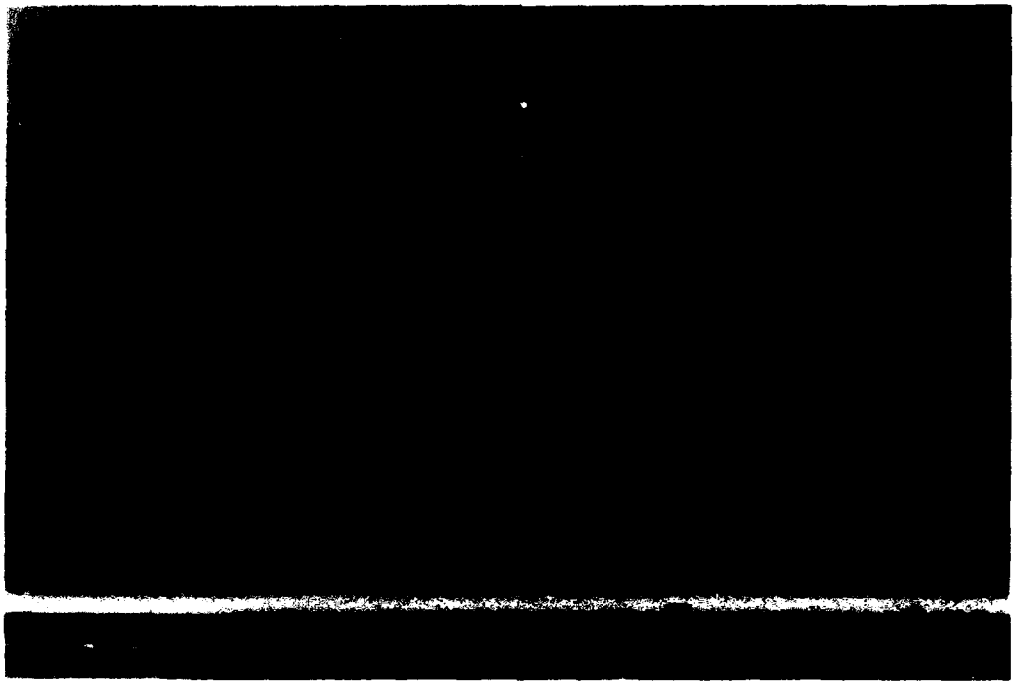

Figure 12 Different Nomarksi setting and lighting condition as shown in Figure 8 illustrating the presence of microabrasions on the surface. These microabrasions correlate with those of the DFM of Figure 9.

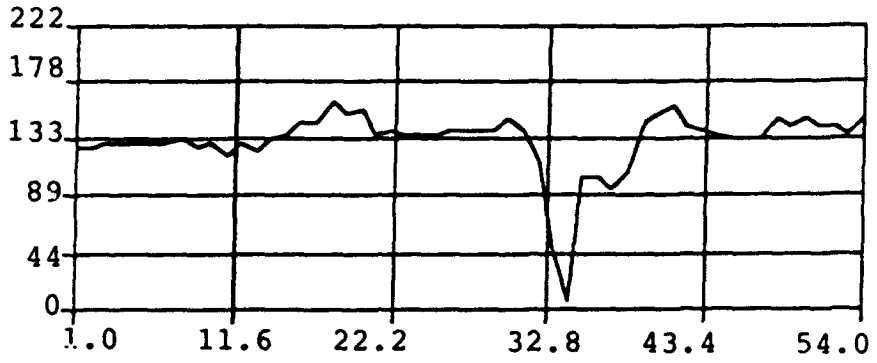

Figure 13 MacPhase profile of typical microabrasion of Figure 10.

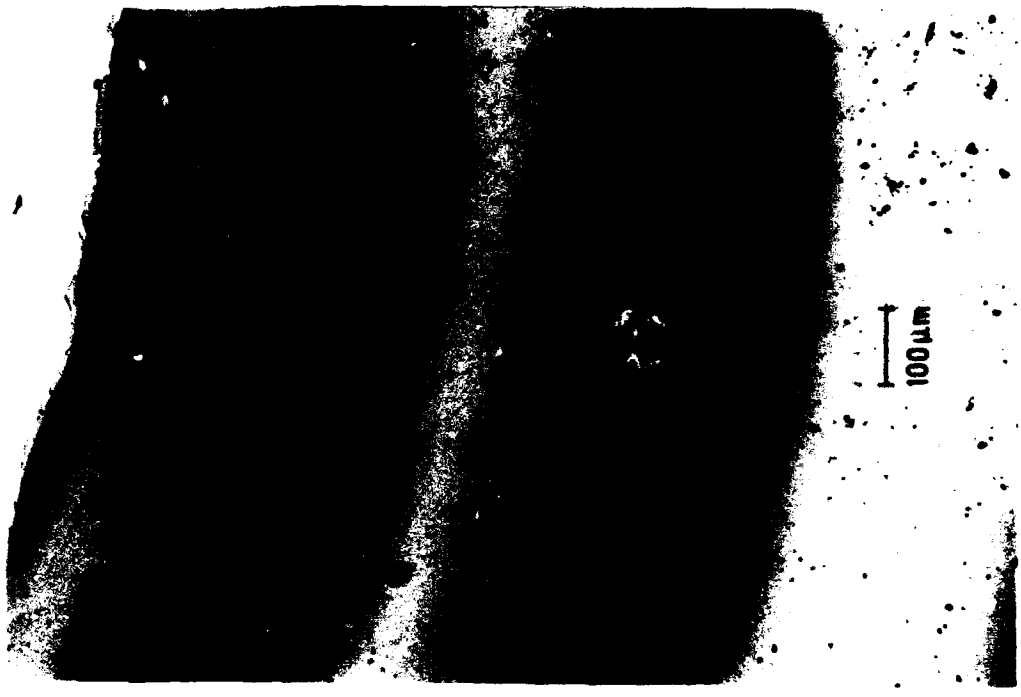

Figure 14 Nomarski photomicrograph (200x) of micrometeorite crater on sample L.

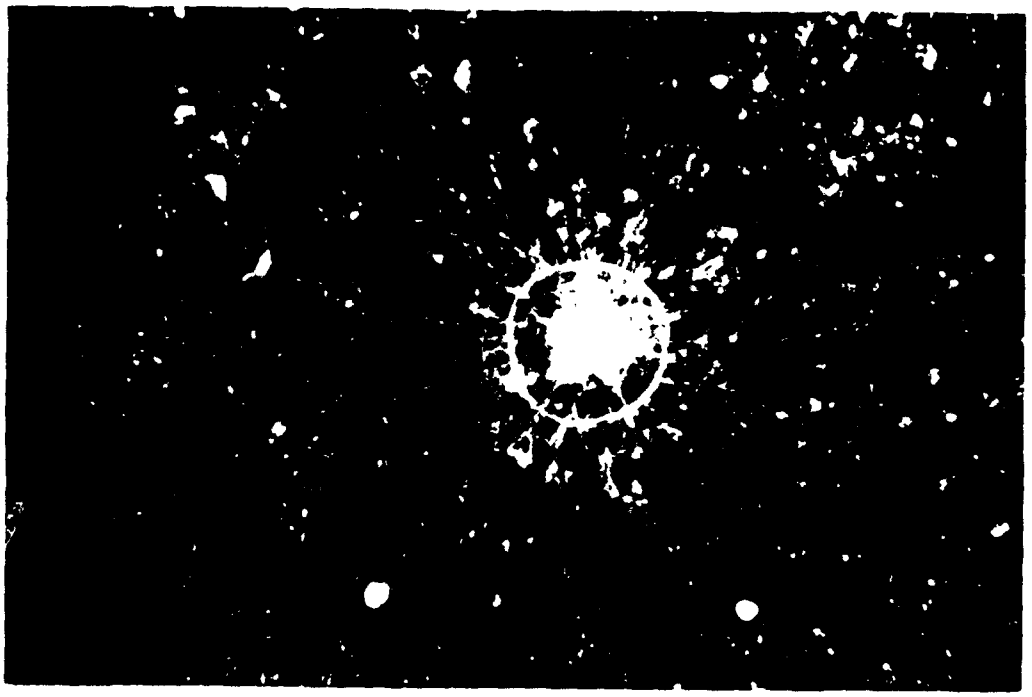

Figure 15 DFM (200x) of micrometeorite crater of Figure 12.

Figure 16 Nomarski Photomicrograph (50x) of crater of Figures 12 and 13 after ion beam treatment. Again note the surface defects which correlate with pattern of the DFM.

Figure 17 Blue Fluroesence light micrograph (FLM) of cleaned/masked sample.

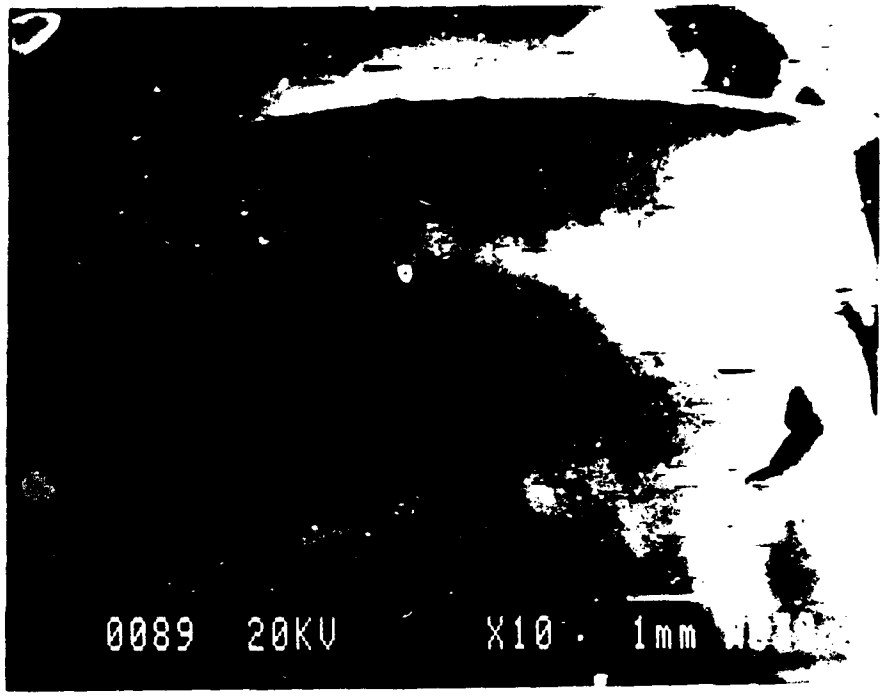

Figure 18 SEM of sampleT showing particulate contamination of varied sizes.

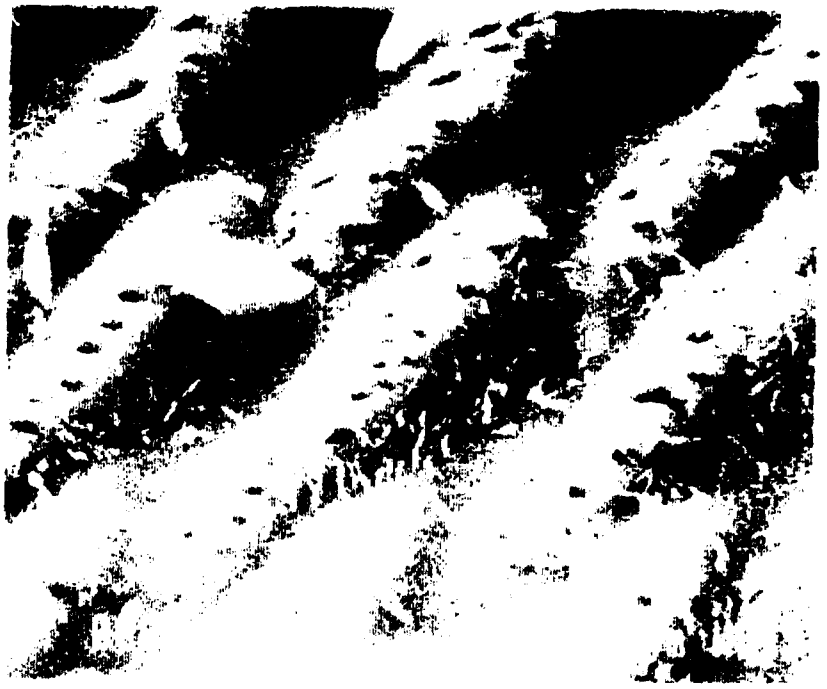

Figure 19 SEM of the Aerogel Mesh Contamination Collector (AMCC patent Pending).

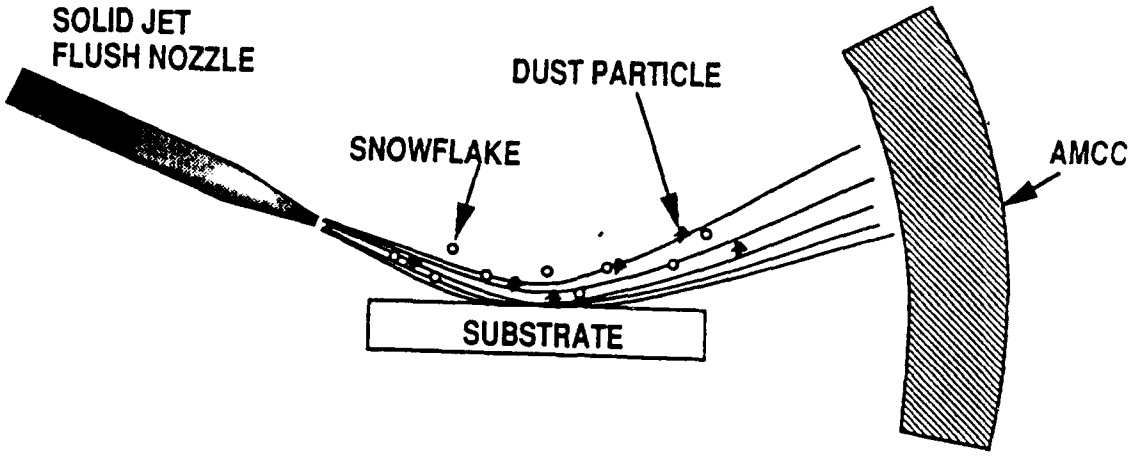

Figure 20 Advanced contamination removal and collection system incorporating the jet spray, ion beam and AMCC.

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