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NASA Work Experience

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B.S. CHEMICAL ENGINEERING, PURSUING M.S. BIOMEDICAL ENGINEERING

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Major and Minor Duties

This past summer I had the opportunity to work in the Analytical Laboratories, chemistry section. The chemistry section is responsible for, "Providing support in the investigation of unknown materials (solid, liquid, or gas) and chemical technology development. This support includes performing in-depth chemical analysis of contamination and development of new organic materials" [1]. If a material needs identification or is contaminated with an unknown substance (particle, fiber, liquid, or viscous substance) then various analytical chemistry methods can be used in order to ascertain its identity. Support of projects via chemical analysis allows for trouble-shooting of problem causes and the ability to remove likely sources of contamination. These problems can then be fixed and further progress can be made towards completion of the project goal (for example, using the plant habitat for research on the ISS). I assisted by analyzing samples with various techniques including Optical Microscopy (OM), Polarizing Light Microscopy (PLM), Fourier-Transform Infrared Spectroscopy (FT-IR), Scanning Electron Microscopy with Energy Dispersive Spectroscopy (SEM/EDS), and X-ray Powder Diffraction (XPD).

I have been lucky enough to work on several different samples. These included characterizing fibers pulled from a spacecraft, a white fibrous residue discovered in a jet refueler truck, brown residue from a plant habitat slated for delivery to the ISS (International Space Station), corrosion on a pipe from a sprinkler, and air filtration material brought back from the ISS. I also conducted my own fiber study in order to practice techniques and further my understanding of background concepts necessary for utilization of the microscopes and future particle analysis.

In the fiber study 13 different fiber samples and one hair sample was prepared and analyzed using OM, PLM, and FT-IR. These fibers ranged from natural fibers such as cotton to synthetic fibers such as polypropylene.

In the natural progression of analysis, OM was utilized first to establish the general morphology, color, and number of phases. Morphology is essentially how the fiber or particle looks. For example: color, shape, weave if the fibers are woven, relative size, and birefringence. OM can also be used to ascertain purity, number of phases, and micro-solubility.

Birefringence is the resulting visible light interference colors when polarized light passes through a material with more than one refractive index (is anisotropic). This can be due to crystal structure or if is a strained amorphous material. The amount of birefringence (first, second, etc., order colors) can be used to infer the thickness (such as if the material is oil). The magnitude of birefringence can present as clearly as colorful rainbows or as simply as a slight difference between gray and white-gray (see Michel-Levy Chart in Figure 1).

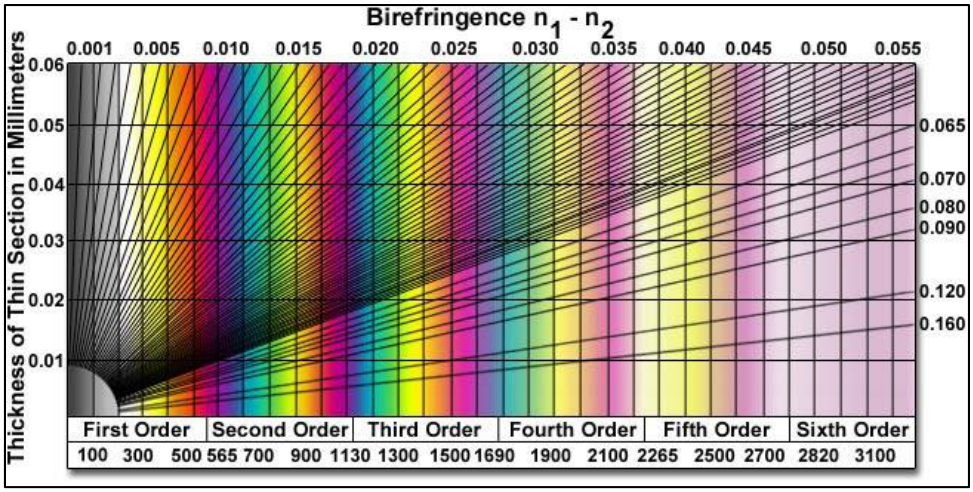

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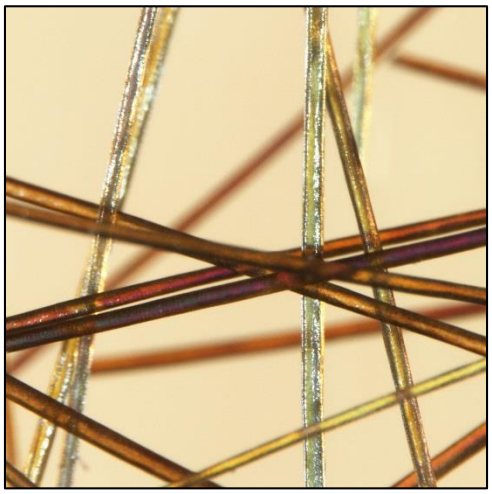
Figure 1. Michel-Levy Birefringence Chart

Along with the diameter of the fiber and additional optical properties of the particle it can be used to determine a possible material identity (see Figure 3) with the use of the Michel Levy Birefringence Chart. Highly birefringent materials can be of such large magnitude that they initially appear almost non-birefringent. By analyzing the material from differing angles, the color changes, as seen in Figure 2. This would not occur in isotropic materials, which indicates it is birefringent.



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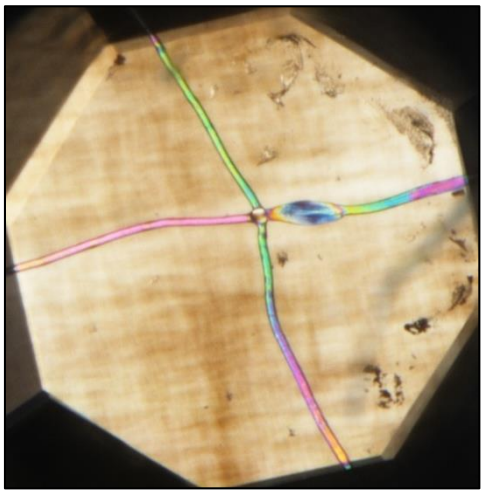

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Figure 2. Human Hair under OM Figure 3. Polyester Fibers Showing Birefringence

OM was also used for initial observations as well as sample preparation to be used with PLM and FT-IR. Once the samples are prepared, PLM can be used to determine the exact optical properties to positively identify a particle or fiber such as refractive index, sign of elongation, birefringence, color, cross-section, size, and morphology. During the initial examination the average fiber diameter was recorded along with some preliminary observations. PLM identification requires the particle to be immersed in a liquid of known refractive index. Mineral oil was used on the sample slides to provide an appropriate medium for observation. One of the fibers, filament triacetate, had a refractive index very close to that of the mineral oil, as the fibers were nearly invisible upon immersion in the oil, as seen in Figure 4.

[5]

Figure 4. Filament Triacetate under OM

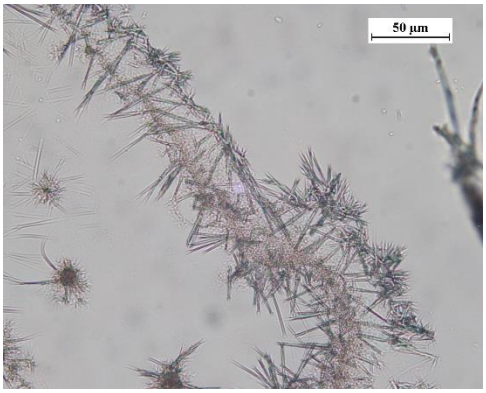
Following PLM observations FT-IR was used to establish the chemical spectra (molecular structure) of the fiber material to further confirm PLM identification. Required daily setup procedures of the FT-IR necessitating the use of liquid nitrogen were also done prior to the equipment's use.

In support of a spacecraft I provided assistance into characterization of particulates gathered from inside metal tubing recently welded onto the craft. The spacecraft should have been "visually clean," where there are either no or very few particles (per specific guidelines). The particle count was well above the threshold, which should not have occurred as welding techniques were used to minimize particle formation. Therefore chemical analysis was done to determine the source of the contamination.

The natural progression of analysis was again utilized by starting the analysis with OM. Once the particles on the stubs were viewed and their approximate locations identified, they were further analyzed via SEM. The plastic particles large enough to be manipulated were analyzed by FT-IR and the type of plastic was determined. I assisted with the formal report compilation for this project as well as some of the microscopic techniques used for the particle identifications. It

was determined that a large portion of the particles were aluminum and titanium alloys consistent with the metal tubing on the spacecraft. The customer determined that an investigation would be done on the welding technique and modifications might be made. However, soft wood particles were also found. It was theorized (by the customer) that a recent change in processing methods allowed wood pallets to come into the room in which the spacecraft was being assembled. Plans were made to discontinue this practice and return to earlier processing protocols. The type of plastic found was common (polystyrene and polyurethane) and may have also been used in shipping materials.

On another project, a white gelatinous residue was found in a jet-refueler tanker during routine service. Multiple instruments were used to facilitate identification of the unknown material. Again, OM was done first. Next a solubility study was executed, wherein the first attempted solvent of nonane showed the crystals had limited solubility in a nonpolar solvent. Methanol, a polar solvent, was used which proved suitable to solubilize the crystals. The solution was allowed to recrystallize and then was prepared for examination under PLM. This examination showed that the crystals were extremely narrow and highly birefringent, which indicated an organic material (see Figure 5).



[6]

Figure 5. PLM of Recrystallized Substance

FT-IR analysis confirmed the material to be an organic amide. SEM analysis was done by mounting a mat of crystals on a bare aluminum (Al) SEM stub. The elemental analysis of the crystals identified the major elements to be carbon, oxygen, and nitrogen, with trace aluminum which was expected since the sample was mounted on an Al sample stub. XPD was utilized for this sample as well.

XPD can be quite useful in assisting identification of unknown crystalline materials. In order to utilize this technique, the particles must be ground below 40 microns, so larger materials may need to be crushed or pulverized in order to use this analytical technique. XPD can be done with as little as 10-50 milligrams of material, but if there is too little material the peaks generated by the x-ray diffraction will not be clear enough to properly identify the material. In addition, the sample must be crystalline, otherwise the only spectra generated will be an amorphous peak that does not give further information. XPD is further limited by the library of spectra it can search, similarly to several of the other techniques, however it can be an extremely useful addition to the other available characterization tools for crystalline materials.

On two smaller projects my mentor and I provided assistance. The first was corrosion on the interior of a small section of pipe from the facility fire sprinkler system that failed. The corrosion was characterized using SEM, and a fungus was found to be present along with the stainless steel corrosion by-products. The second project involved an air purification/filtering material which had previously been aboard the ISS. This material was coated with a metal and it was also examined, first using OM and then SEM, to help determine the coverage of the metal on the material substrate and provide insight on how best to separate the two materials.

Lastly, a liquid and a dried sample on a wipe were received from a payload (a plant habitat) slated for delivery to the ISS. The dry sample was analyzed with OM first, then further viewed under SEM/EDS. The dried liquid was extracted from the wipe and a pH test was done. It was determined that the material was a highly acidic corrosion product. The liquid sample was viewed under OM where it was determined that there was a liquid and solid portion to the sample. A small amount was then fully dried to find nontransparent solids mixed with highly birefringent crystals (see Figure 6). PLM was used to further typify the birefringent crystals. Subsequently, FT-IR followed by XPD and SEM/EDS were done on both materials to allow for more comprehensive identification.

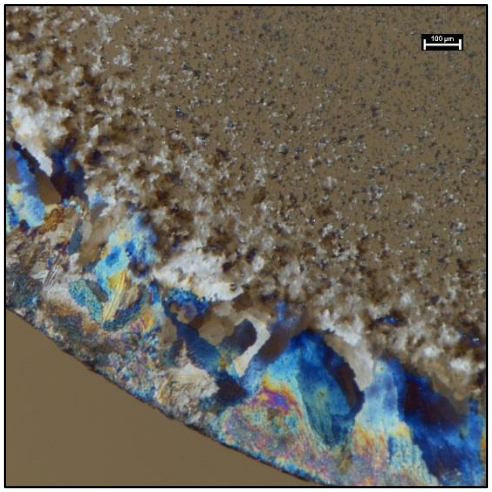


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Figure 6. Birefringent Crystals under OM

I also had the opportunity to participate in diverse work assignments, where I was assigned to work with other branches of the engineering department for 1-2 days each. In the Materials Science branch I participated in the construction of the plant habitat intended for use in research aboard the ISS. Multiple layers of differing composite materials were applied to a frame, and then the entire module was subsequently placed in a vacuum to ensure the layers would properly adhere to one another. During the initial layering process I assisted in applying

heat to one of the layers to enhance its ductility and allow it to conform to the frame. I also helped to ensure adequate depressurization during the vacuum sealing process.

In my other diverse work assignment I was placed in the Testing & Design branch. In order to prevent future failures, mechanical testing (tensile, hardness, etc.) can be done on sample parts (typically metal or composites) to ensure quality of the part or fidelity of the manufacturing technique. In order to do the testing, a significant amount of time was necessary to devote to sample preparation. Prior to the hardness testing the aluminum samples were unpacked and the samples were sanded slightly to ensure that their surfaces were level. Once the hardness tests were completed, the samples were prepared for tensile tests. The sides of the samples needed further sanding to ensure the point of failure during the test occurred at a measureable location. This required several iterations of using subsequently finer and finer sandpaper.

There were also some problems to consider. During the testing process the samples were clamped, which created markings that disguised their labels if they were not sufficiently deep. In addition, a tensile test is done by stretching the sample until it breaks to determine properties of the material such as percent elongation, yield stress, ultimate tensile stress, and stress at rupture. Therefore, once testing was completed, the samples consisted of two pieces rather than one (see Figures 7 and 8). These conditions necessitated adding labeling to the unmarked end of the sample as well as deepening parts of the labeling on the marked end. This was accomplished with a pen etcher which removed small amounts of the material to create markings. Finally, I took measurements of the samples' width and thickness both pre and post-test. I was allowed to assist in ensuring adequate labeling on over 40 samples and assisted with measurements on over 20 samples, which enabled completion of sample testing prior to the end of the week.

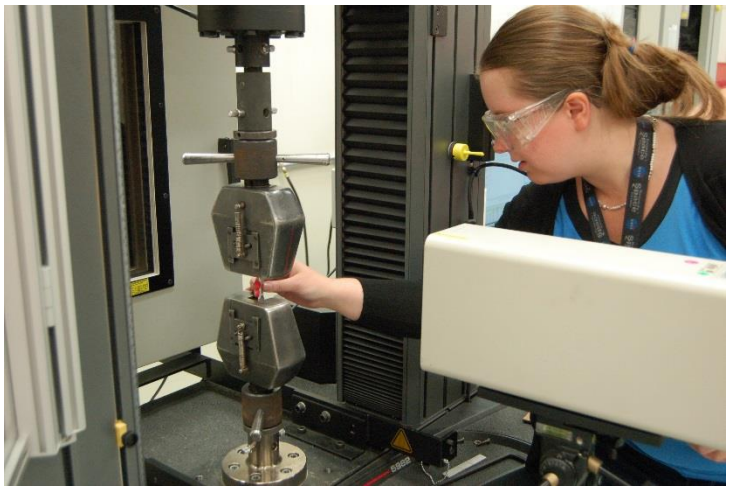

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Figure 7. Sample Placement for Tensile Testing

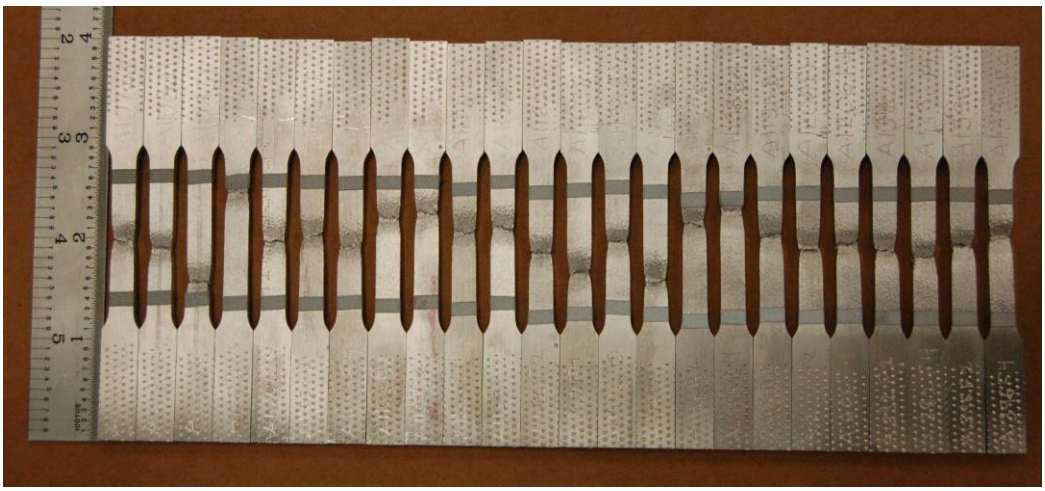

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Figure 8. Samples Post-test

In addition, I have had the privilege to attend multiple tours of the NASA KSC campus, including to the Astronaut Crew Quarters, the VAB (the main area, the Columbia room, and the catwalk), the Visitor Center housing the shuttle Atlantis, the Saturn-V exhibit, the Prototype laboratory, SWAMP WORKS, the Shuttle Landing Facility, the Crawler, and the Booster Fabrication Facility (BFF).

In the near future, an overview of sampling methodologies will be done, as well as further work into how different requirements for sampling as well as sampling history influence the materials used and the quality of results obtained.

Relevance to Coursework

My work directly related to previous coursework, most notably my nanotechnology minor. My class Materials Characterization especially prepared me for using SEM with EDS and also laid background concepts that assisted me in learning to utilize PLM and in sample preparation required for using FT-IR. Other courses were also very vital in preparing me for use of equipment and understanding fundamental concepts. Organic Chemistry 1 prepared me for understanding how to read FT-IR results, as the basics of infrared spectroscopy spectra were covered. Biology and Microbiology classes readied me for using OM as well as the basics of using PLM. My Materials class and laboratory prepared me for understanding the crystal structures and concept of refractive index integral to understanding the images created by PLM. It also allowed me to understand the basics of XPD that was used to analyze crystalline samples. In addition, it further prepared me for my diverse work assignment in the Testing & Design branch as I was already familiar with stress-strain diagrams as well as hardness and tensile tests.

My numerous laboratory courses readied me for laboratory work in general, including General Chemistry Laboratories 1 and 2, Organic Chemistry 1 Laboratory, Physics 1 and 2 Laboratory, Physical Chemistry 1 Laboratory, Phlebotomy Laboratory, CHE Processes Laboratories 1 and 2, General Biology 1 and 2 Laboratories, Materials Laboratory, and Microbiology. My First Aid and Safety course also informed me and provided me with further awareness and ability to utilize emergency-related items such as the AED. I have also completed

several SAChE (Safety and Chemical Engineer Education) safety certifications through AIChE (American Institute of Chemical Engineers), which further assisted me in being aware of possible hazards and in utilizing laboratory equipment in a safe and conscientious way. The courses Nitrogen's Role in Safety and Basics of Laboratory Safety were especially applicable.

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