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MATERIALS DEVELOPMENT, ANALYSIS, AND TESTING WITHIN THE STEVEN MULLER CENTER FOR ADVANCED TECHNOLOGY

The proper selection of materials plays an important role in developing an optimum design for a specific application. In selecting materials, many factors must be considered, such as how their microstructure and chemistry affect their engineering properties. At APL, the Materials Laboratory serves as a source of expertise in such matters and offers a broad range of services related to materials science and engineering. The recent move of the Materials Laboratory into the Steven Muller Center for Advanced Technology has enabled it to expand and enhance its capabilities in several areas, including materials evaluation, microscopy, metallurgy, chemistry, composites, and mechanical testing.

INTRODUCTION

The Materials Laboratory at APL was established in 1983 to support the Engineering and Fabrication Branch of the Technical Services Department as well as other APL departments.[1] In January 1991, the Materials Laboratory was incorporated into the Quality Engineering and Materials Group.

The Materials Laboratory offers a variety of services in several areas of materials science and engineering and has supported many APL programs, including Tomahawk, the Topography Experiment, the Ocean Data Acquisition Program, the Energetic Particle and Ion Composition Experiment, the Special Projects Flight Experiment, the Mid-course Space Experiment, Standard Missile, Delta 181, the Expendable Drone, the Hopkins Ultraviolet Telescope, the Polar Beacon Experiment and Auroral Research, and the Bird-Borne Transmitter. It has also collaborated with the Johns Hopkins School of Medicine[2] and the School of Hygiene and Public Health[3] on several programs. The services provided by the Materials Laboratory are described in the following sections of this article.

MATERIALS EVALUATION AND ENGINEERING

Failure Analysis

Failure analysis systematically determines the cause of failure of a component and the sequence of events leading up to the failure. It can be applied to all classes of materials and all applications and has two functions. The first is to identify the root cause of failure of the component, and the second is to allow the engineer to determine how the component functions and how to prevent future failures by changing the design of the component. The detective work is performed in a structured manner to examine the evidence carefully and to reach conclusions. Photographic documentation and evidence must carefully be obtained so as not to destroy the failed part. Various scientific instruments such as the optical microscope, scanning electron microscope, and energy dispersive spectrophotometer may be used for the investigation, in addition to chemical analysis and nondestructive testing techniques.

The following example illustrates the use of the scanning electron microscope. Examination of the fracture surface of a stud arc weld zone of an HY80 steel bolt used in a Navy submarine revealed a columnar martensitic fracture origin (Fig. 1). This microstructure, along with the sustained tensile stress, a corrosive environment, and the passage of time, caused environmentally assisted cracking. The solution to the problem was to substitute a different welding technique that would not produce this microstructure.[4]

Thermal Vacuum System

The thermal vacuum system, shown in Figure 2, is used to evaluate various materials for vacuum applications, such as spacecraft hardware, where material outgassing is of critical importance. Total mass loss and collected volatile condensable materials from outgassing in a vacuum environment can be measured according to Standard Test Method E595 of the American Society for Testing and Materials (ASTM). By using three quartz heaters, thermocouples, and quartz crystal microbalances, the thermal vacuum system can evaluate materials at elevated temperatures and at internal pressures down to 2×10^{-8} torr. The ASTM test is required to qualify the use of any nonmetallic material for spaceflight.[5] Planned uses for the thermal vacuum system include the qualification of materials for the Mid-course Space Experiment and the Vehicle Interaction Program.

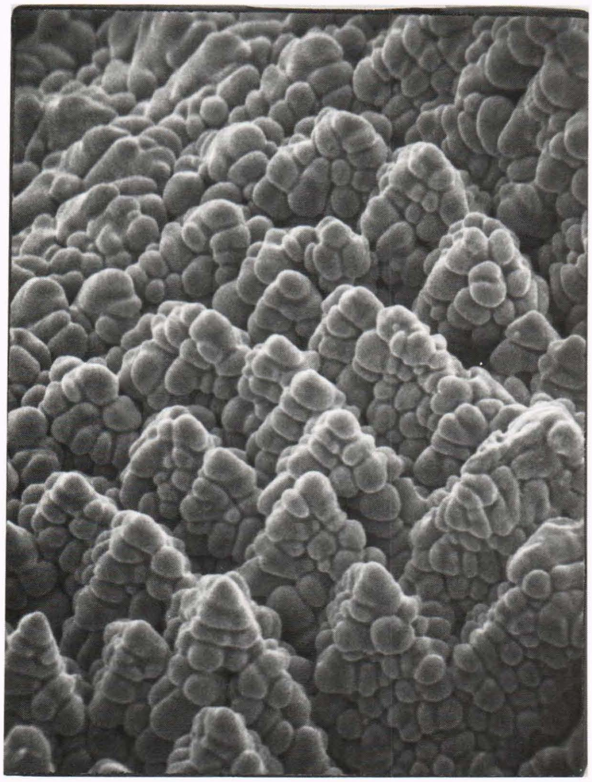

Figure 1. Scanning electron micrograph of a columnar martensitic fracture at the weld zone of an HY80 steel bolt.

Figure 2. The installation of thermocouples on the ten different micro-ovens used during outgassing tests.

Thermal Humidity System

The thermal humidity chamber can test component performance in high-humidity environments or can be used for the processing of materials that require controlled humidity. Polyurethane curing or the bake-out of thermoset inks on aluminum parts that have a chromate conversion coating requires such processing. The chamber can accommodate a test part up to 3 × 4 × 2 ft, and tests can be performed over a temperature range of −18°C to 93°C and a humidity range of 20% to 98%. The chamber is equipped with a programmable, multistep controller and a paper chart recorder to run cycles with both temperature and humidity as variables.

The instrument has been invaluable in testing the rate of moisture transport through the walls of filament-wound, graphite epoxy tubes, and in evaluating the need for moisture barriers for the Tomahawk Composite Canister Launch System.

Nondestructive Testing

Nondestructive testing is used when the presence of internal or external flaws must be determined without rendering the part unfit for use. The Materials Laboratory employs several nondestructive testing methods, which are discussed in the following paragraphs. The methods detect incipient flaws that may cause the unexpected failure of a component.

Liquid penetrant testing is the most widely used nondestructive testing method. It locates and determines the severity of discontinuities that are open to the surface of the component. It is used to test a variety of metallic and nonmetallic materials such as welds, forgings, castings, plastics, and ceramics. Liquid penetrant testing was used to inspect a Monel flange at the Propulsion Research Laboratory, four stainless steel type 17-4 PH parts for the Air Force Inlet Model used by the Propulsion Research Laboratory, and stainless steel type 316 bolts during the development of a testing program for critical fasteners.

During ultrasonic testing, a sample is exposed to high-frequency sound waves to detect and measure surface and subsurface flaws and to determine sample thickness. A novel use of ultrasonic testing by the Materials Laboratory involves testing pressurized helium cylinders on Navy cruise missiles for the Tomahawk program. The testing verifies that helium has not leaked from the cylinders and that the cylinders are still pressurized.

Magnetic particle testing requires that the component first be magnetized. Fine iron particles are then applied to the surface. The particles form a pattern when they are attracted by the magnetic leakage fields created by discontinuities on the surface of the component.

MICROSCOPIC EVALUATION

Optical Metallography

We use metallography for failure analysis,[6] identification of metals and alloys (in concert with chemical analysis), determination of the heat-treatment condition of materials, evaluation of the soundness of weldments, and measurement of the thickness of applied films and coatings. For quality control during the manufacture of printed wiring boards by the Electronic Fabrication Group, we evaluate the APL plated-through-hole process and examine the boards for barrel cracking and plating thickness. Other applications include determining if metallic samples from the Raw Materials Stockroom conform to specifications, evaluating the soundness of coatings, determinating the fiber volume fraction in composite structures, and examining microelectronic circuits for intermetallics and void lines in gold bond joints.

Figure 3 shows an application of metallography to failure analysis. Engineers were testing a new design for the Tomahawk missile's booster rocket when they noted a damaged area on the stainless steel ball in the nozzle. At the request of the Aeronautics Department, we investigated the cause of the problem. The photomicrograph shows a polished and etched cross section of the nozzle ball. The three different microstructures resulted from exposure to elevated temperatures and provided proof that the material had been melted.

Scanning Electron Microscopy

A scanning electron microscope (SEM) microanalytically examines and evaluates materials and electronic manufacturing processes. Our capabilities include secondary electron collection, backscatter or atomic number contrast, metrology, and stereo pairs. The technique is generally nondestructive for conductive samples and has a resolution of 16 nm. One of the main reasons for using the SEM is its high magnification capabilities.[7]

The Materials Laboratory routinely used the SEM and an energy dispersive spectrophotometer for analysis of semiconductors and microelectronic circuits for the Topography Experiment. The SEM inspection is performed according to the Military Standard 883C test method, which allows one to judge the quality and acceptability of incoming components to be used for in-house fabrication of microelectronic devices.

Figure 4 illustrates the use of the SEM for the Nova program. A gold-wire ball bond on an aluminum bonding pad is shown in the figure. A brittle, gold–aluminum intermetallic may form in the wire bond joint when the joint is subjected to temperatures above 150°C. The intermetallic causes a decrease in reliability and possible premature failure because of the lowering of the mechanical strength at the bond.

Image Analysis

The Materials Laboratory can analyze images by using a dedicated Omnimet Image Analyzer or a Macintosh computer equipped with a digitizing board and the IMAGE software program. The Macintosh system is used in a project undertaken for the Johns Hopkins School of Medicine.[8] In that application, flat-mounted retinas are visualized on a Zeiss photomicroscope by using transmitted illumination, and normal and oxygen-exposed capillaries are compared. The extensive vasoconstriction in an oxygen-exposed retinal capillary is demonstrated in Figure 5. For a quantitative measurement of the percent of vascularization in a given field, the image is converted to binary form to discriminate the features of interest. The area fraction or percent of vascularization is determined by analyzing the black and white pixels in the binary image and taking a ratio of black to total number of pixels.

Figure 3. Photomicrograph of 17-4 PH stainless steel. The three different structures resulted from exposure to excessively high temperatures.The top layer is a cast structure produced by exceeding the melting point of the material, which resolidified upon cooling. The middle layer is a transformed structure that resulted from excessive heating above the transformation temperature. The bottom structure is over-aged, tempered martensite.

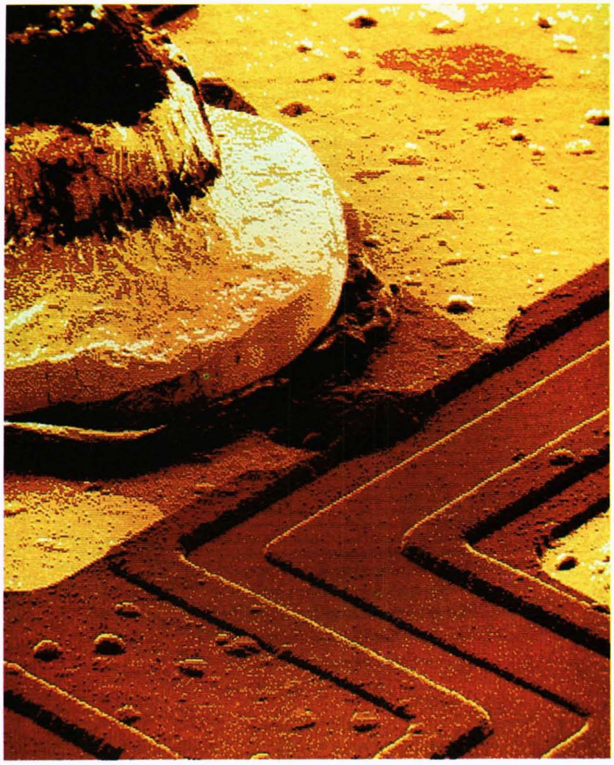

Figure 4. Intermetallic interaction of a gold-wire ball bond (left) with an aluminum metallization layer (yellow). The intermetallic causes brittle failure of the wire bond.

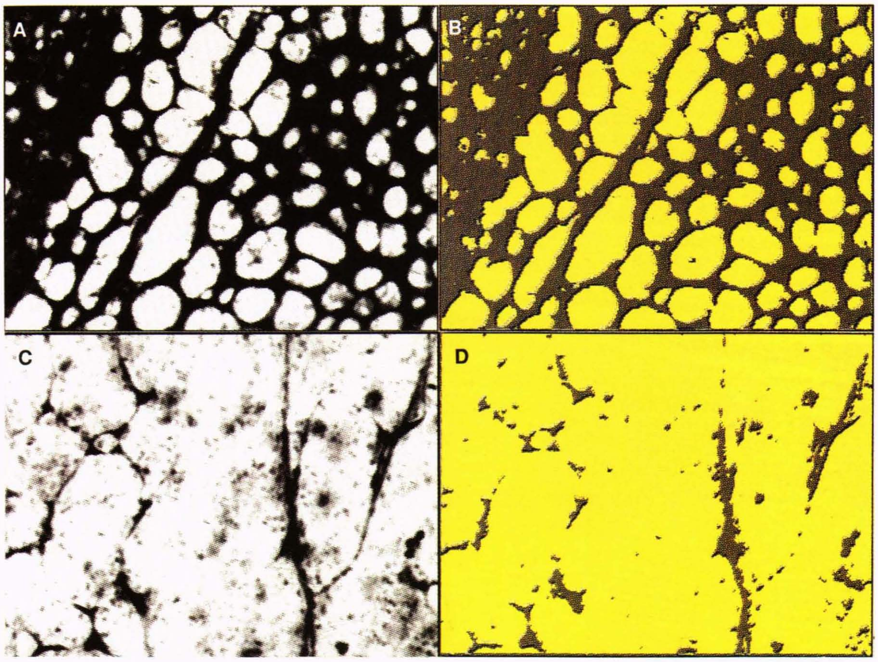

Figure 5. Retinal capillary beds of a neonatal canine. **A** and **B**: Four-day-old normal beds. **C** and **D**: Four-day oxygen-exposed beds. Binary images in parts **B** and **D** generated the area fractions 57% and 8%, respectively.

COMPOSITES

Composite materials can be defined in a variety of ways, from the microscopic to the macroscopic. For our purposes, a composite is a structural material composed of two distinct materials that, when combined, produce a single material with performance exceeding that achieved by either constituent alone. Typically, composites are reinforcing elements such as particulates, flakes, whiskers, or fibers contained in a continuous matrix of a polymer, metal, or ceramic.

Composite materials are desirable in engineering designs because of their many benefits, including directionally tailored properties such as high specific stiffness, high tensile strength, thermal conductivity, electrical conductivity, and thermal coefficient of expansion (can be 0 in one direction); high fatigue strength; creep and stress rupture resistance; and near net-shape fabrication.

The Composites Facility can develop a first-cut design that includes composites by using a software package written for personal computers. Once the idea has been refined, a detailed finite-element model can be developed and analyzed by using ICAN, PATRAN, and NASTRAN software programs.[9]

The Composites Facility can also perform tooling design, process development and documentation, prototype and limited-run fabrication, and prototype evaluation. Our fabrication capability encompasses polymeric matrix composites, both thermoplastic and thermoset, that can be processed with an autoclave or a hot press. The press can achieve 800°F and 100,000-lb clamp force, and the autoclave can achieve 700°F and 250 psi. We can also adhesively fasten most engineering structural materials to themselves or to each other, including materials such as honeycomb or foam core bonding.

Recent projects in composites have included the design and manufacture of a set of six fiberglass/epoxy sunshades for the ultraviolet experiment on Delta 181 (see Figs. 6 and 7), a pair of graphite/epoxy sunshades for the Energetic Particle and Ion Composition Experiment (see Fig. 8), a graphite/epoxy structural member for the Special Projects Flight Experiment, a series of fiberglass/epoxy hydrodynamic fairings for the Ocean Data Acquisition Program MiniSpar (see Figs. 9 and 10), and graphite/epoxy submerged instrument housings for use at external pressures up to 1000 psi.

The Composites Facility will be expanding in the fall of 1991 to accommodate a new, larger autoclave, which will allow processing of a part up to 4 ft in diameter and 8 ft long, at temperatures up to 850°F and a maximum pressure of 300 psi.

CHEMICAL ANALYSIS

Inductively Coupled Plasma Spectrophotometer

The inductively coupled plasma spectrophotometer simultaneously determines the major, minor, and trace

Figure 6. Delta 181 sunshade array. Each single unit protects an ultraviolet imager; each pair protects a spectrophotometer.

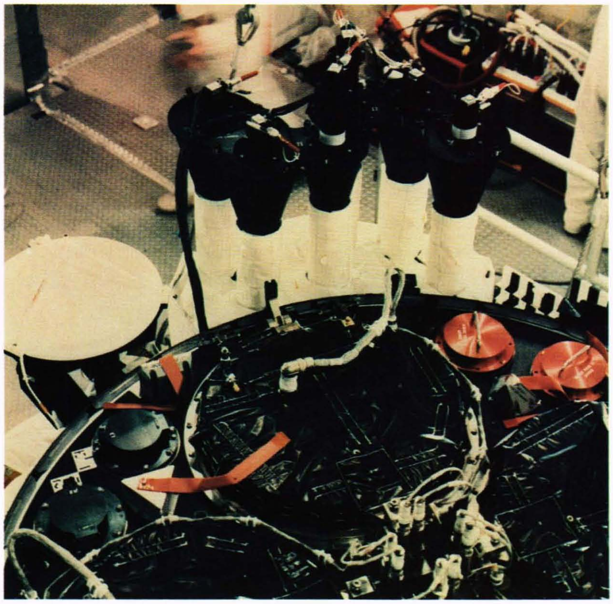

Figure 7. The sunshades installed on Delta 181 with thermal blankets and covers in place.

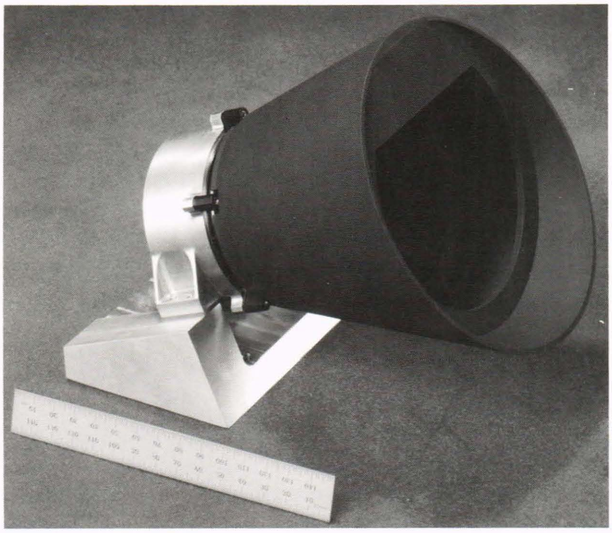

Figure 8. One of the pair of sunshades for the Energetic Particle and Ion Composition instrument. Both incorporate five graphite/epoxy baffles within a graphite/epoxy outer shell.

constituents in solids and liquids. It is also used to analyze the heavy metal content in water to fulfill Environmental Protection Agency requirements.

The inductively coupled plasma spectrophotometer derives its analytical information from atomic emission spectra in the optical region of the electromagnetic spectrum, which includes ultraviolet, infrared, and visible wavelengths. The spectrophotometer operates at the optimal analytical wavelength for each element being determined and each matrix being studied. The software permits the analyst to develop optimal procedures for the analysis being performed and to store the methods on floppy disk for later use.

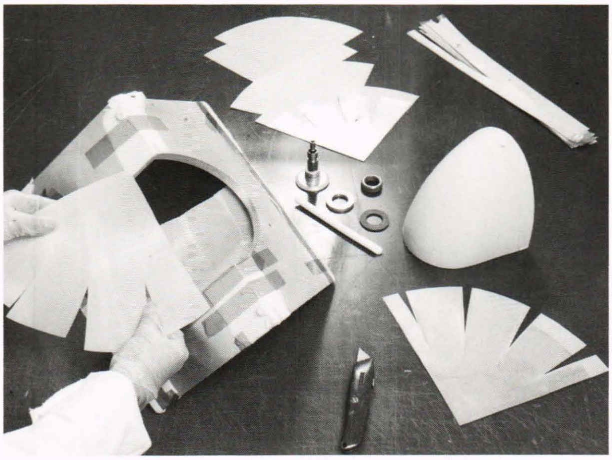

Figure 9. The assembly used to align layers of epoxy-coated glass cloth in a specific orientation for the hydrodynamic fairing shells on a composite tool.

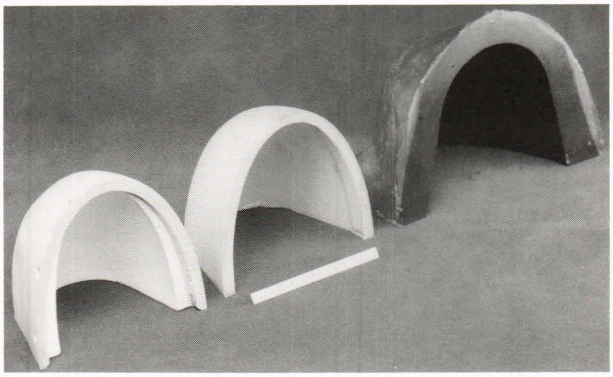

Figure 10. The composite tool (right), a fiberglass/epoxy shell (middle), and a finished hydrodynamic fairing with syntactic foam lining and mounting holes (left).

Energy Dispersive Spectroscopy

The energy dispersive spectrophotometer provides qualitative and semi-quantitative chemical analyses of materials by using the X-rays produced when a sample is placed in the SEM and bombarded with high-energy electrons. Characteristic X-rays with specific energies are analyzed by a photosensitive detector and are used to generate a spectrum of X-ray counts versus energy, as shown in Figure 11. The energy dispersive spectrophotometer detects elements from atomic number 6 (carbon) through 92 (uranium). This test method is generally nondestructive to the sample and has a spatial resolution of 1 μm.

Fourier Transform Infrared Spectrophotometer

The Fourier transform infrared spectrophotometer is used to analyze organic or inorganic compounds in either gaseous, liquid, or solid form. The spectra of three compounds are shown in Figure 12. These spectra serve as fingerprints to identify and distinguish the compounds

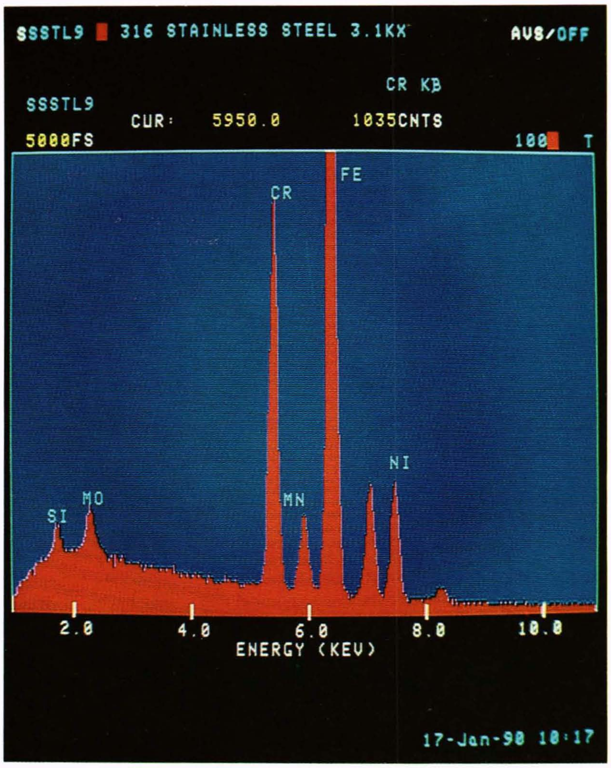

Figure 11. The energy dispersive spectrum of a sample acquired to determine whether the sample was a type 316 stainless steel or a type 304 stainless steel. The presence of molybdenum (Mo) indicated that it was a type 316 stainless steel. The analysis was performed for the Mechanical Fabrication Group.

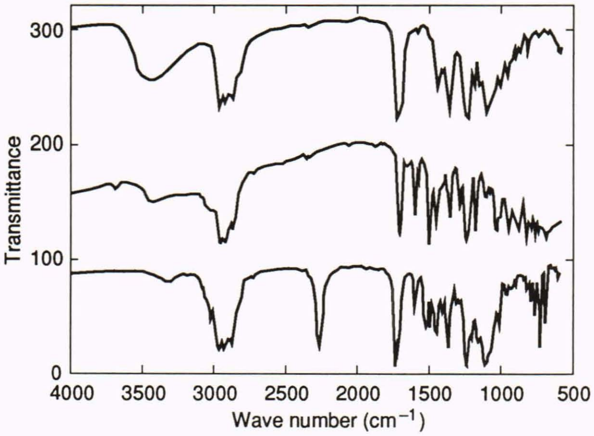

Figure 12. Fourier transform infrared spectra of two uncured primers and a paint that are frequently used on flight assemblies. Chemglaze primer 9924 (top), Chemglaze primer 9922 (center), and Chemglaze paint Z306 (bottom).

from other materials. The most frequently used range of wave numbers for chemical identification is 4000 to 650 cm^{-1} because molecular species have characteristic vibrational, rotational, and absorption energies in this region. The generation of a characteristic absorption (or transmission) spectrum for a sample allows it to be analyzed chemically or identified by comparison with an extensive spectral database.

Ultraviolet–Visible Spectrophotometer

The ultraviolet–visible spectrophotometer, a recent addition to the Materials Laboratory, analyzes organic compounds in a manner similar to that used by Fourier transform infrared spectroscopy but at shorter spectral wavelengths and in a vacuum. The vacuum is required to analyze contamination on witness mirrors (used to collect samples of particulates) at wavelengths less than 200 nm. Analysis at these wavelengths enables us to evaluate contamination on spaceborne ultraviolet optics.

MECHANICAL PROPERTIES

The proper selection of materials is required to optimize designs for specific structures and components. The Materials Laboratory uses tension, hardness, and impact testing to evaluate the mechanical properties of structural materials. The testing is done according to established ASTM and military standard procedures.

Tension Tests

Tension tests determine yield strength, ultimate tensile strength, modulus of elasticity, percent elongation, and percent reduction of area. Our tension testing machines were recently used in establishing a procedure for testing critical fasteners, which is included in the *APL Standard Practices and Procedures* manual. In other recent applications, the mechanical properties of copper–zirconium alloys were tested for the Aeronautics Department to determine if the alloys were suitable for use in the NASA Ames direct-connect combustor test system to be used on the National AeroSpace Plane, and ceramics were tested for the Microelectronics Group for use in a spaceflight application.

Hardness Tests

Hardness is a measure of a material's resistance to penetration. Its significance lies with the correlation of hardness with other engineering properties. Tensile strength, heat treatment or temper condition of metallic alloys, carbon content of steels, and wear resistance of many materials can be approximated simply by measuring the hardness of the material. Rockwell tests are the most widely used measure of hardness. Vickers and Knoop tests measure the hardness of very small areas (individual constituents of the microstructure) of a sample. Brinell tests were the first widely accepted and standardized hardness tests. A recent application of hardness testing was the analysis of a stainless steel ball used in the Tomahawk missile's booster rocket mentioned earlier. The hardness test enabled us to determine the temperature gradient to which the internal structure of the ball was exposed during the test firing of the booster rocket.

Impact Tests

Impact tests determine how a material behaves under rapid loading. Ductile materials such as carbon steels or high-strength low-alloy steels can become brittle under this type of loading. Whereas tension tests are performed under uniaxial stress conditions at a slow rate [on the order of 0.5 in./(in.·min)], impact tests subject the specimen to triaxial stress conditions at a notch at strain rates on the order of 1000 in./(in.·s). These conditions are present at welds, keyways, or places where loading conditions change rapidly.

Performing impact tests at elevated and sub-ambient temperatures allows us to determine the ductile-to-brittle transition temperature. For many materials, a sudden and rapid decrease in the amount of energy that a material can absorb under impact loading may occur over a small temperature range. Thus, materials that are ductile at higher temperatures become brittle at lower temperatures. The transition temperature is governed by the state of stress (uniaxial, biaxial, triaxial) within the material and its ability to deform along certain slip planes at a given temperature.

SUMMARY

The Materials Laboratory continues to be a resource for materials development, analysis, and testing for all APL departments. It has greatly expanded its services to include the fabrication of composite structures up to 4 ft in diameter. Analytical services now include organic analyses by Fourier transform infrared spectrophotometers and vacuum ultraviolet spectrophotometers. Detection and control of molecular and particulate contamination on optical sensors for spaceflight have also been strengthened considerably by the acquisition of modern instrumentation and engineering expertise. In addition, we have been expanding our expertise in failure analysis of electronic components and structural materials from such diverse sources as submarines, surface ships, missiles, and spacecraft.

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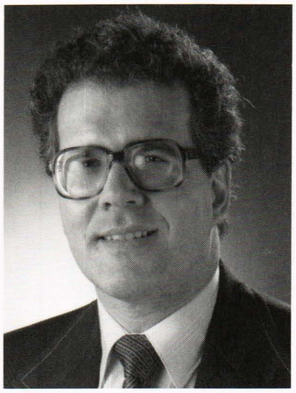

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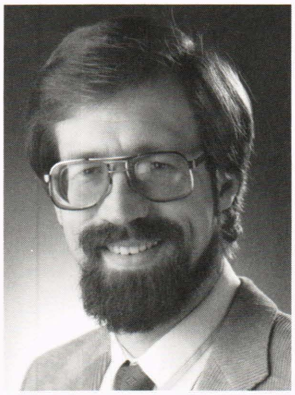
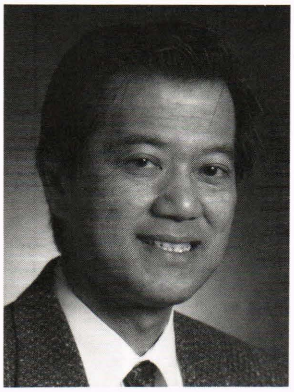


PAUL J. BIERMANN, a member of APL's Senior Professional Staff, received his B.S. in materials engineering from Rensselaer Polytechnic Institute in 1980. He has twelve years of experience in the fabrication and testing of advanced composite materials. Since joining APL in 1986, Mr. Biermann has been responsible for the Composites Design and Fabrication Facility and has conducted research and development programs in a variety of composite applications, including underwater and spacecraft structures. He is a member of the Society for the Advancement of Materials and Process Engineering, ASM International, and the Society for Experimental Mechanics.



O. MANUEL UY is the Section Supervisor of the Materials Laboratory and was appointed to APL's Principal Professional Staff in 1985. He specializes in the chemistry and physics of materials, space reliability, electronic packaging, and gaseous and particulate contamination of space sensors. Presently, Dr. Uy is Principal Investigator for the Contamination Experiments of the Mid-course Space Experiment and Science Investigator for the Vehicle Interaction Program/Particle Monitoring Experiment. Dr. Uy obtained his Ph.D. in chemistry from the Case Western Reserve University in 1967. He is a member of Sigma Xi, the Royal Society of Chemistry, the American Chemical Society, the Society for the Advancement of Materials and Process Engineering, and the International Society for Hybrid Microelectronics.