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The Chemical and Physical Laboratory supports VideoDisc

Armed with some of the most sensitive equipment available, the scientists and engineers in this activity aim for better VideoDiscs...through space-age chemical and material analyses.

Abstract: *The Chemical and Physical Laboratory is a multifunctional department interacting with both the VideoDisc manufacturing and VideoDisc development activities to support and troubleshoot the manufacturing of the disc, using the most up-to-date chemical and material analyses.*

The Chemical and Physical Laboratory (C&P Lab) at the Rockville Road plant, where VideoDiscs are made, acts in several roles. These roles are discussed below.

Incoming materials inspection

The VideoDisc process uses a wide range of materials such as PVC resin, plastic additives (stabilizers, lubricants, processing aids), electroplating chemicals, disc-rinsing chemicals, disc lubricant, and adhesives. Each of these materials must be tested to meet the exacting specifications required to make a VideoDisc with high manufacturing yield.

In-process quality control (QC) support

The matrix, rinse-lubrication, and compounding activities require a variety of chemical and physical tests at a high frequency in order to maintain proper process control.

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Manufacturing problem troubleshooting

Unfortunately, no manufacturing operation is trouble-free and occasions will arise where yield or quality will experience serious drops. In many cases, these problems are materials-related, and substantial efforts are required on the part of the lab to define the problem and isolate the cause. In this role the scientist, engineer, and technician act as detectives in solving the mystery. Indeed, many of the microanalytical techniques of forensic science are useful in detecting the small contaminants that can cause large problems in view of the disc geometries and dimensions.

Development project support

While a tremendous amount of effort and resources have been invested to develop the current manufacturing process and materials, optimization will continue for years to come. These projects require a materials characterization support. In addition, the C&P Lab itself must develop new and improved tests with better reliability and improved detection limits as well as tests for parameters yet unspecified.

The VideoDisc manufacturing operation is a high-technology materials- and process-oriented activity that requires a sophisticated approach to materials characterization and chemical analysis. Each of the above areas is essential in either maintaining or improving the manufacturing operation. The Chemical and Physical Laboratory currently services the VideoDisc operation, as well as provides support services to Stylus Manufacturing and Player Engineering.

Laboratory functions

The Chemical and Physical Laboratory can be divided into several functional subgroups that specialize in specific areas of responsibility.

The Chemical Analysis group (Table I lists major equipment) engages in the more classical aspects of chemical analysis. Typical examples include the following tests. The atomic absorption spectrometer is used for trace-metal analysis in resin and additives, for plating-bath compositional analysis, trace-metal analysis in sol-

Table I. Chemical analysis equipment.

Atomic Absorption Spectrometer (Varian).
High-Pressure Liquid Chromatograph (Waters).
Infrared Spectrometer (Perkin-Elmer).
UV/Visible Spectrometer (Beckman).
Particle Size/Distribution Analyzer (HIAC).
Gas Chromatograph (Varian).
Ion Chromatograph (Dionex).
pH/Specific Ion Electrode Meter (Orion).
Karl Fischer Autotitrator.
Sayboldt/Brookfield Viscometers.
Refractometer.

Table II. SEM/x-ray equipment.

Scanning Electron Microscopes (3). a. AMR 1000 (1) b. Cambridge Mark II (2)
Energy-Dispersive X-Ray Analyzer (PGT) for SEM.
Wavelength Dispersive X-Ray Fluorescence Spectrometer (Siemens).
Quadrupole Mass Spectrometer.
Microtome (for carbon-black dispersion).
Optical Microscope.

Table III. Compound testing equipment.

Capillary Rheometers (Instron).
Torque Rheometers (Brabender).
BET N_2 Surface Area Equipment.
Absorptometer.
Particle Size Classifiers (Rotap, Alpine).
Resistivity Measurement Device.

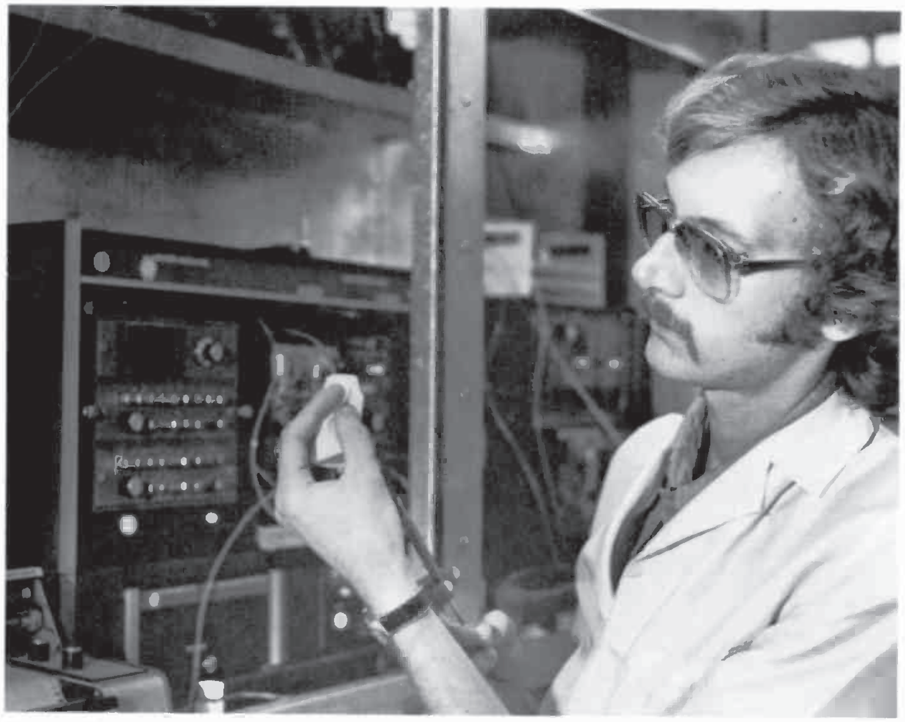

Fig. 1. The high-pressure liquid chromatograph (HPLC) provides an analytical separation technique used in mixture analysis and in determination of molecular weight distributions for resins and disc lubricants.

vents and environmental testing on waste-water analyses. The ultraviolet, visible and infrared spectrometers are used for both qualitative and quantitative analyses of solvents and contaminants in them, organic components in plating baths, and percentage additives in lubricants. The high-pressure liquid chromatograph (Fig. 1) is used for molecular-weight distribution of resins and disc lubricants.

The scanning electron microscope (SEM) and x-ray group (Table II lists equipment) is responsible for monitoring the cutterhead, disc, and stylus geometries. The critical dimensions involved in the VideoDisc system must be measured in microns and angstroms and can be measured accurately only under the SEM. Three SEMs are employed virtually full time, with the bulk of the workload being QC-type monitoring.

The energy-dispersive x-ray analyzer (EDXRA) (Fig. 2) is used in conjunction with an SEM for chemical analysis of microscopic defects in discs, metal parts, styli, as well as microcontamination in raw materials. The x-ray fluorescence spectrometer is used for QC monitoring of lubricant thickness as well as for routine examination of compound for stabilizer additive levels and contamination.

The primary function of the Compound Test Lab (Table III lists equipment) is in the routine monitoring of production compound material for melt viscosity, resistivity and heat stability; as well as characterization of resin, carbon and other compound raw materials. This group provides manufacturing with real-time feedback.

The Physical/Thermal testing lab (Table IV lists equipment) is concerned with determination of melting points, heat capacities, material degradation and weight loss through the use of the differential scanning colorimeter and the thermogravimetric analyzer (Fig. 3). Thermal mechanical analysis (TMA) is used for determination of glass transition temperatures and ther-

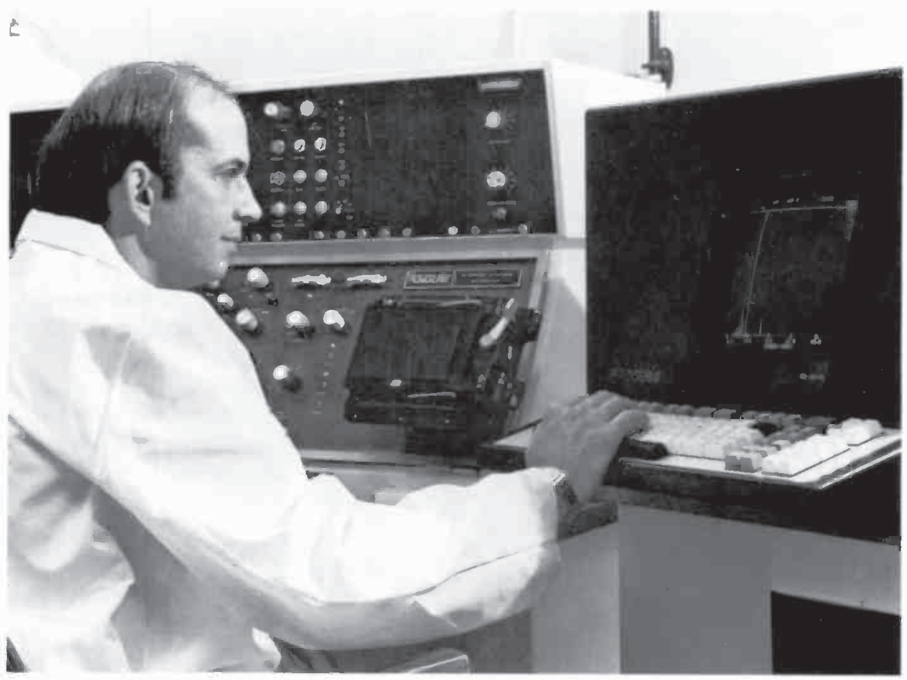

Fig. 2. The scanning electron microscope (SEM) is used in measurement and inspection of the microscopic geometries important to the disc, stamper, cutterhead, and stylus performance. The energy dispersive x-ray analysis (EDXRA) is used to provide chemical analysis of microscopic defects and microcontamination.

Fig. 3. The thermal analysis lab uses differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), and thermomechanical analysis (TMA) to determine such parameters as melting point, heat capacity, glass transition temperature, expansion coefficients; and to monitor such phenomena as thermal degradation of polymers.

mal expansion coefficients. The Rheometrics mechanical spectrometer (Fig. 4) can be applied to studies of mechanical properties such as dynamic viscosity, and elastic modulus as a function of temperature.

Typical problems

As an example of a manufacturing problem encountered by the C&P Lab, we will discuss microcontamination, a specific case.

Fig. 4. The Rheometrics mechanical spectrometer is a versatile device that can subject the test sample to a variety of static and time-varying stresses at different temperatures, while monitoring mechanical response. Dynamic viscosity and elastic modules are two parameters that can be measured.

See Table V for a general summary of some materials-related problems.

VideoDiscs that contain inhomogeneous particulate contaminants near the surface can exhibit undesirable playback effects when the stylus hits the defect. After electrical playback testing has located a defect on a disc, it is examined microscopically. The defect is opened using a surgical knife under an optical microscope and it can be examined by SEM for particle morphology and analyzed chemically by EDXRA (Figs. 5 and 6).

When a number of defects have been cataloged by chemical type, the real work starts. Is the contamination from a process fluid such as deionized water or a chemical rinse treatment? Is it from corroding process equipment? The answers to these questions are uncovered by the team of C&P Lab scientist and process engineer by identifying likely sources of contamination, performing the chemical analyses and matching results to our defect catalog (or fingerprint file). Successful completion of these tasks allows the process engineer to eliminate or reduce the contamination source.

In the case of raw materials, appropriate techniques need to be developed. For example, an appropriate separation technique (sieving, dissolution/filtration, etc.) must be determined for each raw material with analysis of the contamination separated out. After sufficient work

Table IV. Physical/Thermal testing equipment.

Differential Scanning Calorimeter (Perkin-Elmer).
Thermogravimetric Analyzer (Perkin-Elmer).
Mechanical Spectrometer (Rheometrics).
Thermomechanical Analyzer (Perkin-Elmer).
Environmental Ovens.

with the material vendor, an appropriate materials specification can be agreed upon, which will safeguard the disc product quality against contaminants in raw materials.

Additional problems might include such items as disc and stamper staining. This would involve analysis of the stained areas in both stamper and disc. Is it a compound-related stain? If so, is it due to low thermal stability and subsequent compound degradation, or is it due to bleed-out of lubricant or plasticizers due to incorrect weights in formulating the compound lot? If it is a stamper-related stain, what is the nature of the thin film on the metal surface and where did it originate in the matrix process? Again, the team of laboratory analyst and process engineer provides the answers to these questions, which ultimately lead to a solution of the problem.

Table V. Materials and chemicals process-related problems.

Problem	*Origin*
Tracking	Microcontamination (process or raw material).
Staining	Additive bleedout/ Compound degradation/ Stamper thin-film contaminant.
Warping	Compound rheological properties.
Audio/Video	Composite dependent on carbon properties and compound processing.
Carrier Distress	Surface contamination from process.
Metal Parts	
Substrate Machinability	Copper bath parameters/ Bath contamination.
Stress in Nickel Parts	Bath contamination.
Brittle Stampers	Bath contamination.

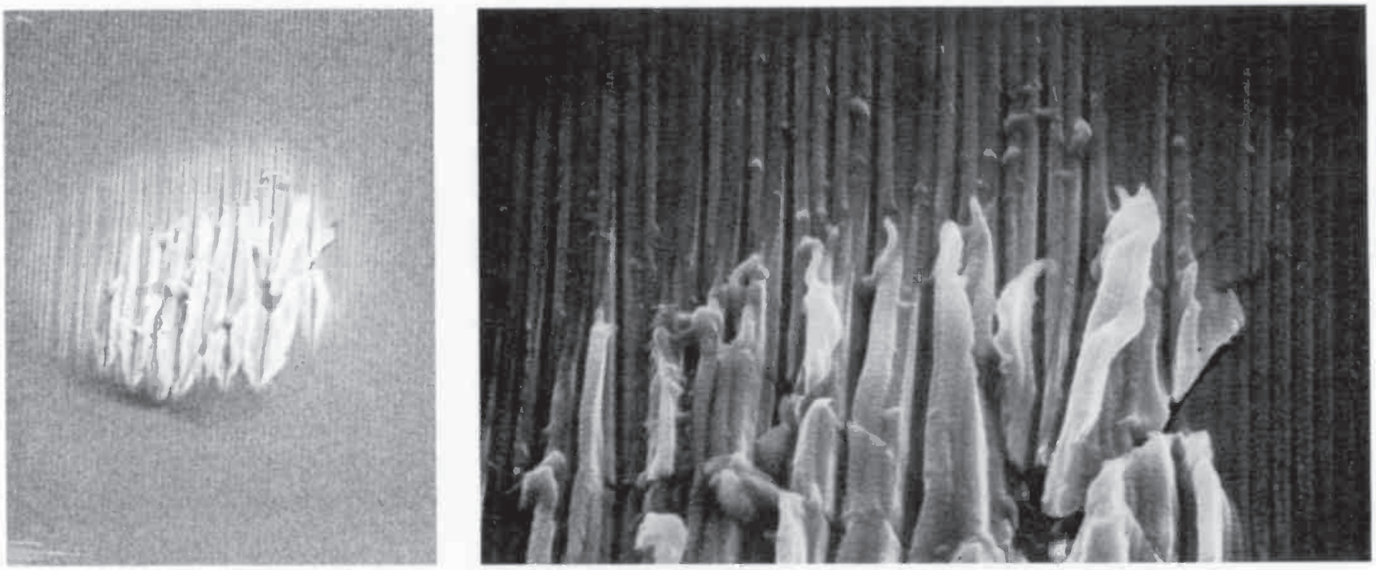

Fig. 5. The SEM can be used to examine carefully the morphology of disc surface defects and to discover what happens when the stylus interacts with such a defect.

Summary

As delineated above, the C&P Lab is a multifunctional department interacting with both the manufacturing and development activities to provide routine support and troubleshooting capabilities to maintain and advance the manufacturability of the VideoDisc. It is the challenge of the chemicals and materials analyst to be expert in the techniques of analysis as well as to understand the intricacies of the manufacturing process in order to be most effective. This challenge will be met by the C&P Lab personnel as the VideoDisc product continues to mature.

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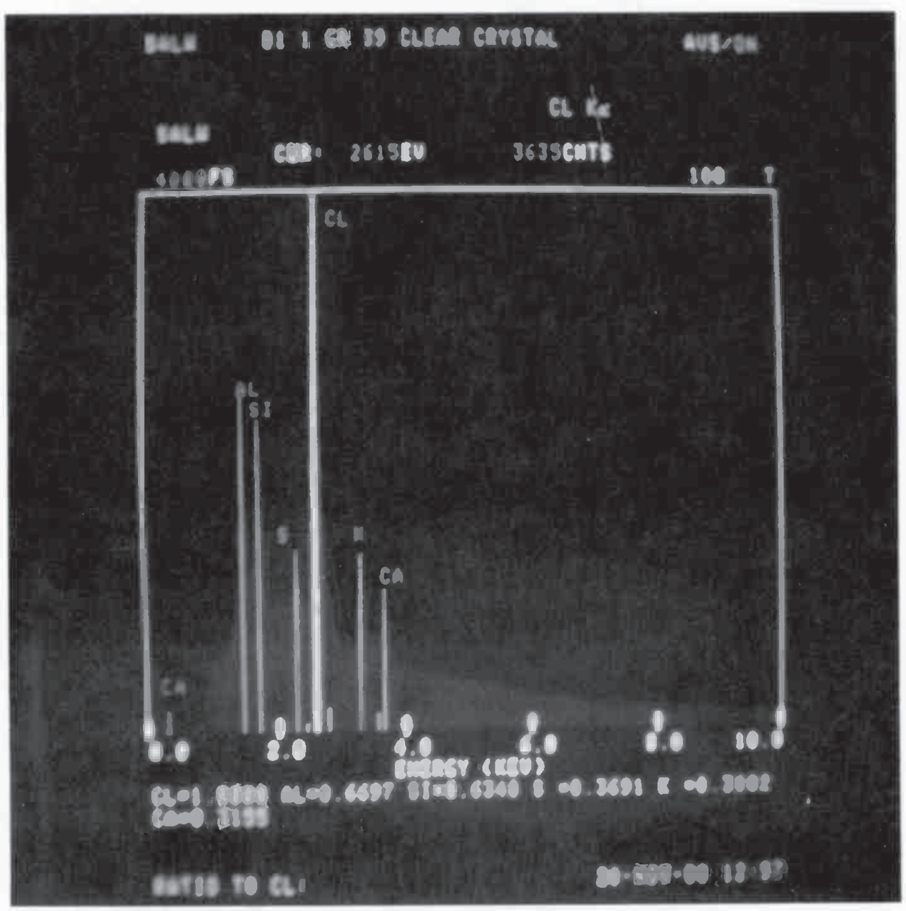

Fig. 6. The EDXRA is used to obtain a simultaneous analysis of chemical elements in a disc defect (Atomic Number 11 and higher). This serves as a fingerprint to be matched up with potential sources to identify and ultimately eliminate the problem.