LA-UR -91-3437

LA-UR--91-3437 7--11

DE92 002524

Los Alamos National Laboratory is operated by the University of California for the United States Department of Energy under contract W-7405-ENG-36.

TITLE: EVALUATION OF ENVIRONMENTALLY SAFE CLEANING AGENTS FOR DIAMOND TURNED OPTICS

AUTHOR(S): L. A. Theye, R. D. Day, J. Weinrach, S. Seiffert and R. Schubert

SUBMITTED TO: 1st Int'l Congress on Environmentally Conscieous Manufacturing, Santa Fe, NM
Sept. 18-20, 1991

EVALUATION OF ENVIRONMENTALLY SAFE CLEANING AGENTS FOR DIAMOND TURNED OPTICS

Lonnie A. Theye, Robert D. Day, Jeffrey Weinrach
Los Alamos National Laboratory
Los Alamos, NM 87545

Rudolf Schubert
Bell Communications Research
Red Bank, NJ 07701

Stephen Seiffert
Benchmark Environmental Corporation
Albuquerque, NM 87110

Background and Need:

Precision machining of metal surfaces using diamond turning has increased greatly in popularity at LANL in recent years. Similar techniques are used extensively to manufacture metal mirrors for use in laser applications. The diamond turned surfaces are easily damaged, making the selection of a cleaning agent very critical.

These surfaces have been traditionally cleaned using Trichloroethane (TCA) to remove residual oil remaining from the machining process. The TCA was then removed with an ethanol rinse, leaving a residue free surface. Recently, however, TCA was pronounced environmentally unsafe. Consequently, we are searching for an environmentally safe cleaning agent for these diamond turned metal optics.

The concern with using alternative solvents is the potential for residual surface films that produce reflectivity changes related to a combination of wavelength, surface coverage, film thickness and dielectric properties. Therefore, we have initiated a program for testing the effectiveness of a variety of environmentally safe solvents used to clean diamond turned optical surfaces.

Our basic test plan consists of comparing a number of environmentally safe solvents against the TCA/ethanol cleaning system. We have identified twelve candidate solvents, but have only been able to perform a partial test on one of them to date. This paper discusses the results obtained to date using this solvent known as P F [1].

Experimental Procedure:

Three different materials have been used for the tests. They are: Oxygen free high conductivity (OFHC) copper, electrolytic tough pitch (ETP) copper and 6061 aluminum. These materials were selected because they are currently used for many diamond machining applications. Test specimens were cut from a single piece of bar stock into blanks 3/4 inch in diameter and 1/8 inch thick.

The surfaces to be tested were finished using a single-point diamond tool with a tool nose radius of approximately 3mm. The machining conditions were adjusted so that a 1 υm/rev feed was obtained.

A fixture was made to simultaneously machine nine blanks. Clean mineral oil was used as a cutting fluid. After removing the blanks from the machine they were stored in a mineral oil bath until they could be cleaned. The mineral oil plus any particulates generated by the machining process must be cleaned from the surface.

The cleaning procedure consisted of squirting cleaning solvent onto the oily surface and then air-drying with pressurized clean air. The fluid coming off of the surface during the cleaning process was collected for subsequent chemical analyses.

The following techniques are used to evaluate each solvent's effectiveness:

A. Bidirectional Reflectance Distribution Function (BRDF) scatter measurements
B. Ellipsometric analysis.
C. Observation of the surface using a Nomarski microscope.
D. Artificially aging for the equivalent of ten years using those solvents which provide the best surfaces based upon the three techniques listed above. After aging, an Auger analysis will be performed to determine surface contamination film thickness.

BRDF Measurements:
The BRDF is a measure of the amount of light scattered by a surface away from the normal angle of reflection. It is sensitive to both particulates and surface films and is a good measure of the surface performance in a typical application. For the purposes of this study, the BRDF measurements provide a functional criterion against which to evaluate the solvents. These measurements are made at a wavelength of 0.6328 mm and at off axis scattering angles between 0.5^{o} and 60^{o}. These correspond to a range of surface wavelengths between 0.73 μm and 73 μm.

Ellipsometry Measurements:
Ellipsometry is a sensitive technique used to measure surface film thicknesses of less than one monolayer. Therefore it is a very good quantitative method for determining how well a solvent removes surface oils. By using data obtained from the chemical analyses performed on the collected rinses along with a mathematical model, ellipsometric measurements can be used to determine the type and amount of surface contaminant. An advantage of this technique is that it works in air as opposed to vacuum.

Nomarski Microscope:
According to Bennett and Mattsson [2] the Nomarski (or Differential Interference Contrast) microscope is the most sensitive instrument for observing surface cleanliness next to the human eye. It is a good method for recording the surface condition on film, but since it produces a qualitative analysis it does not provide a numerical comparison of results.

Artificial Aging and Auger Analysis:
Long term performance of an optic is often an important requirement. Since the cleaning solvent affects surface chemistry, it was decided to artificially age the specimens after cleaning them. The aging is performed in a chamber with an atmosphere containing high concentrations of corrosive chemicals normally present in our environment. For example, a typical corrosive atmospheric mixture will be 10-ppb hydrogen sulfide, 10-ppb chloride, and 200-ppb nitrogen dioxide with a relative humidity of 70%[3]. The Auger analyses performed after aging determines the surface contamination thickness as a function of the solvents used.

Data Analyses Obtained Thus Far:

To date BRDF measurements have been made on three cleaning systems:
1) trichloroethane with an ethanol rinse
2) PF with an ethanol rinse, and
3) PF with no rinse.

Figures 1 through 6 are plots of these data for ETP copper and 6061 aluminum. (The results for the OFHC copper were very similar to those obtained for the ETP copper and therefore are not presented.) An examination of these plots reveals that the amount of scatter produced from the surface cleaned with the PF/ethanol system was similar to that of the TCA/ethanol system for both copper and aluminum substrates. The surfaces that were cleaned using PF without the ethanol rinse produced much more scatter than the surfaces cleaned with the TCA/ethanol system. The peak observed at approximately 40 degrees on the copper data is caused by diffraction effects from the machining marks.

Aging and Auger Analyses Results:

The copper and aluminum were aged for 10 days in an Accelerated Aging Chamber. Auger analyses were performed on each sample after the tests to determine the elements present in the contamination layer and to determine the contamination layer thickness. Figure 7 shows a typical Auger Spectrum taken on one of the copper samples. Figures 8 through 10 are the depth profiling data for the ETP Copper samples with a sputtering rate of 100 angstroms per minute. Figures 11 through 13 are the depth profiling data for the 6061 Aluminum samples with a sputtering rate of 200 angstroms per minute. From these data it has been concluded that there is no significant differences between the cleaning procedures for either material with regard to future corrosion.

Ellipsometric measurements have been delayed because of instrument repair.

Summary:

We discussed the plan to test the cleaning capability of a number of environmentally safe solvents used on diamond turned metal optics. Partial results have been obtained on three cleaning systems, indicating that a suitable replacement for TCA can be found.

Acknowledgement:

This work was funded by the U.S. Department of Energy under contract W-7405-ENG-36, for the University of California.

The authors wish to express their appreciation to Sandia Systems, Albuquerque, NM for taking the BRDF data.

References:

1. P F is a registered trademark of P-T Technologies of Clearwater, Florida.

2. Bennett, Jean M. and Mattsson, Lars, Introduction to Surface Roughness and Scattering, Optical Society of America, Washington, DC, 1989, p. 30.

3. Schubert, R., "A Second Generation Accelerated Atmospheric Corrosion Chamber," American Society for Testing and Materials Special Technical Publication 965, pp. 374-384.

BRDF Measurements on ETP Copper

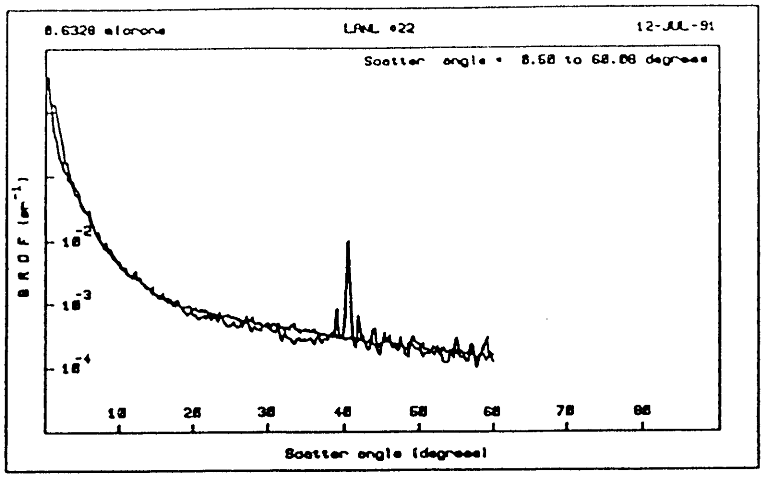

Fig. 1. ETP Copper with TCA and Ethanol Rinse

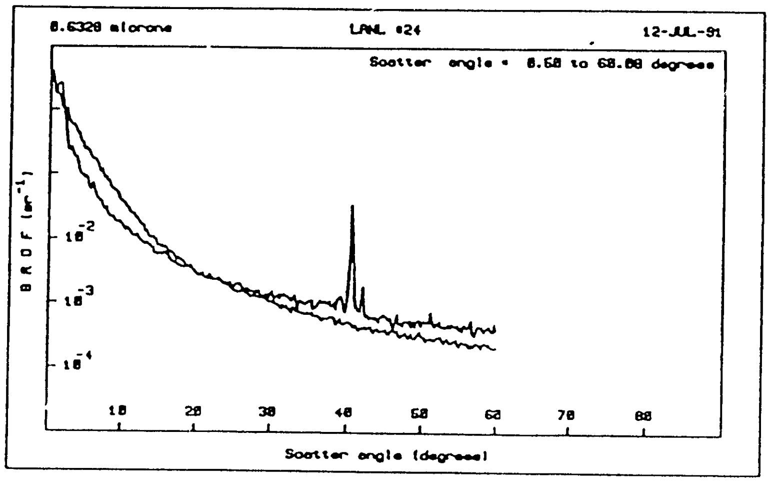

Fig. 2. ETP Copper with PF Rinse

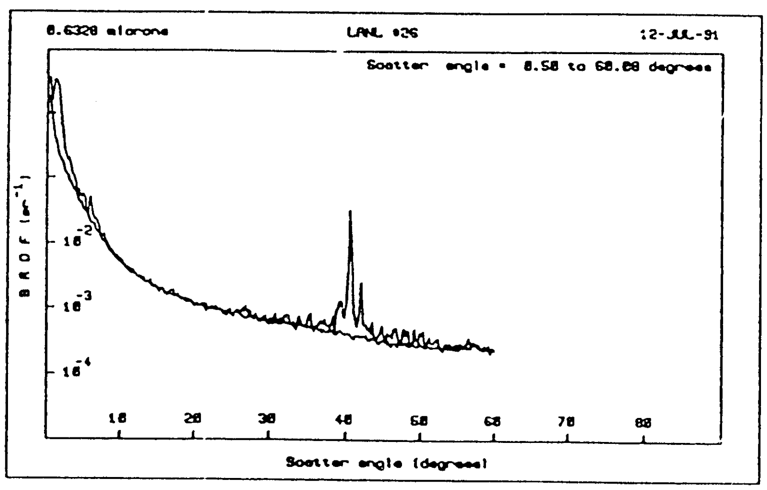

Fig. 3. ETP Copper with PF and Ethanol Rinse

BRDF Measurements on 6061 Aluminum

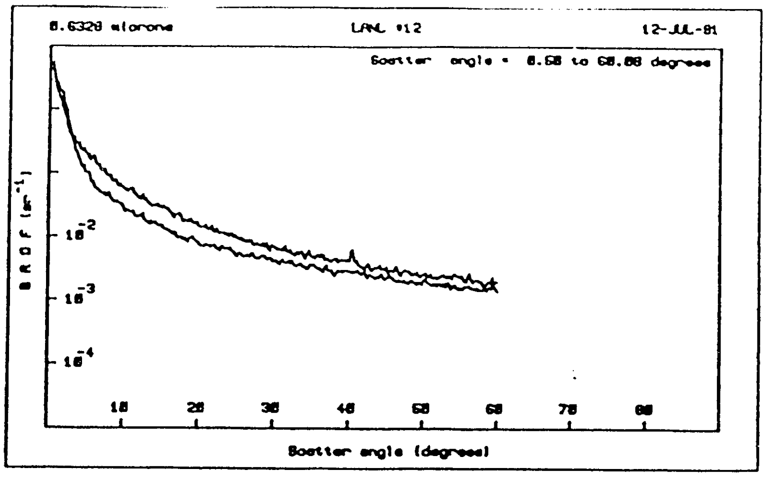

Fig. 4. 6061 Aluminum with TCA and Ethanol Rinse

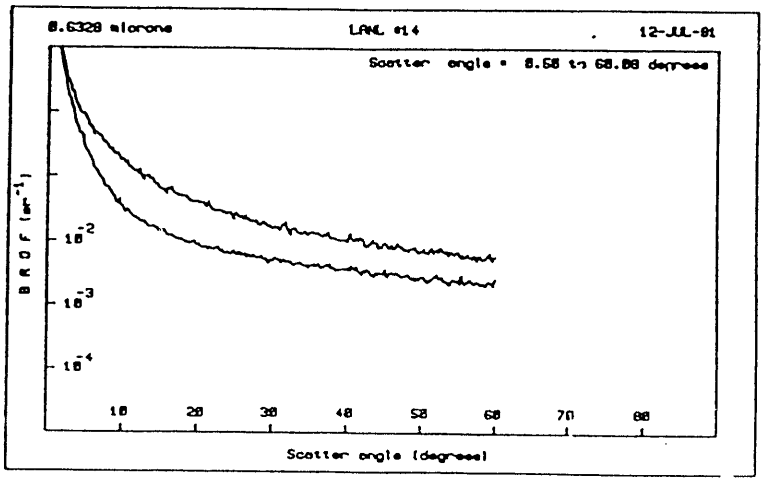

Fig. 5. 6061 Aluminum with PF Rinse

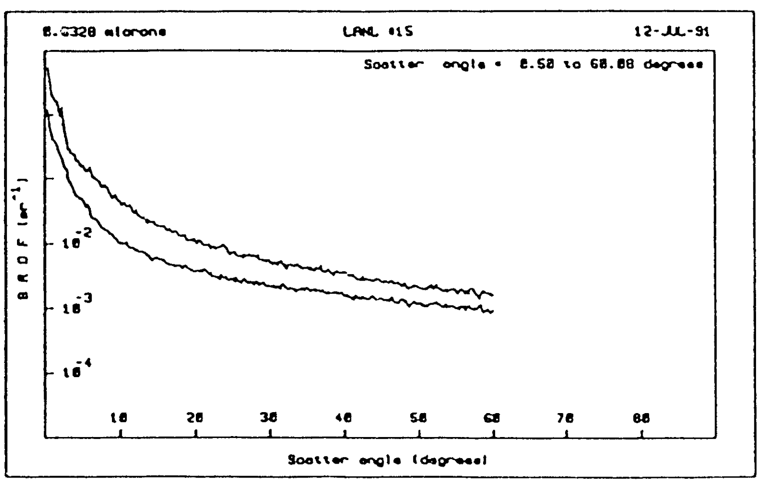

Fig. 6. 6061 Aluminum with PF and Ethanol Rinse

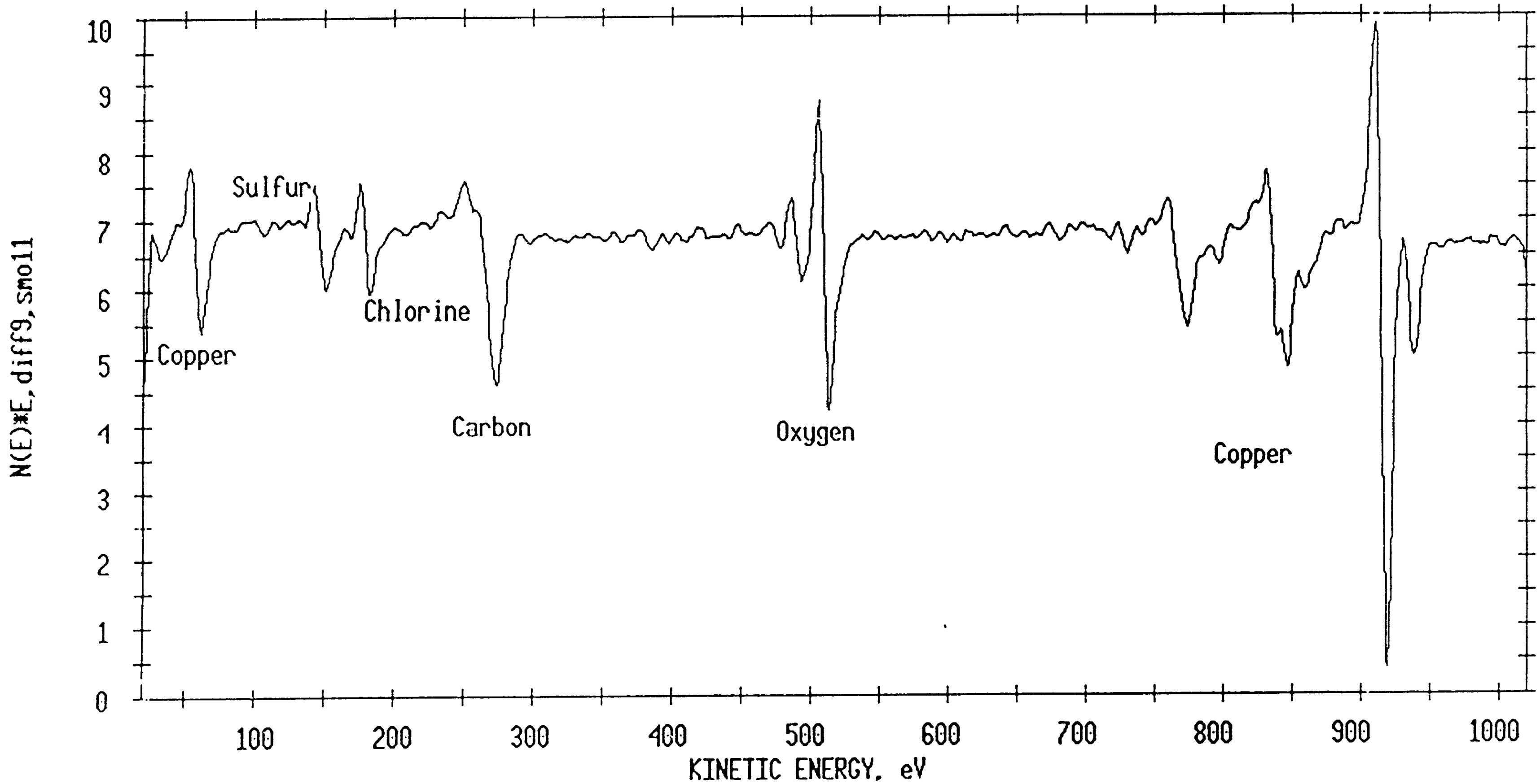

Fig. 7. Auger Spectrum for Artifically Aged ETP Copper

Depth Profiles for ETP Copper
Sputtering Rate is 100 A/Min

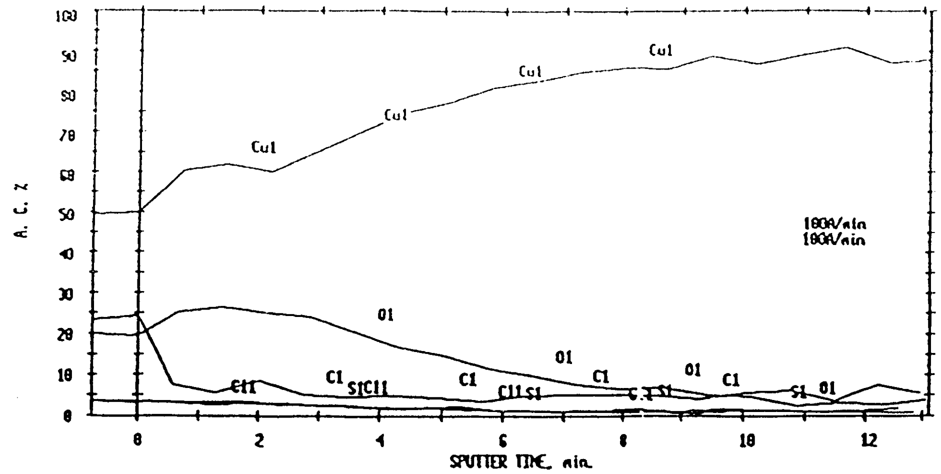

FIG. 8. TCA ETHANOL

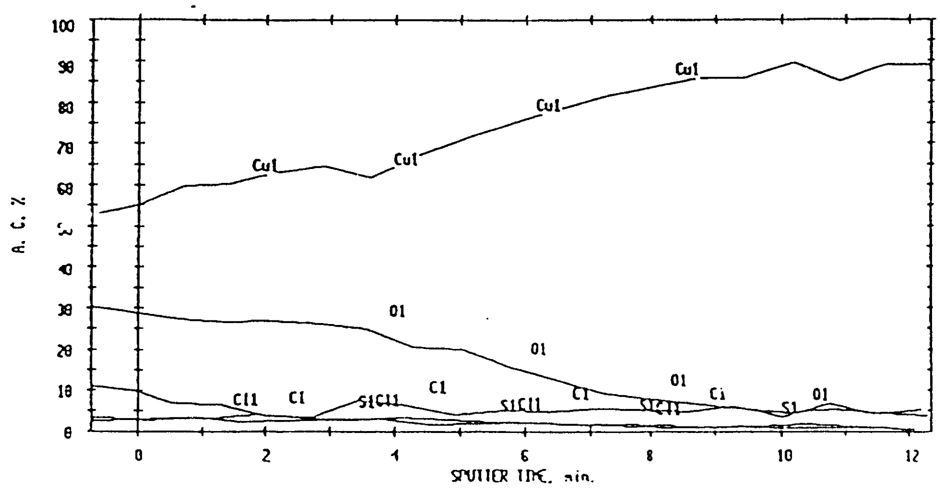

FIG. 9. PF/ETHANOL

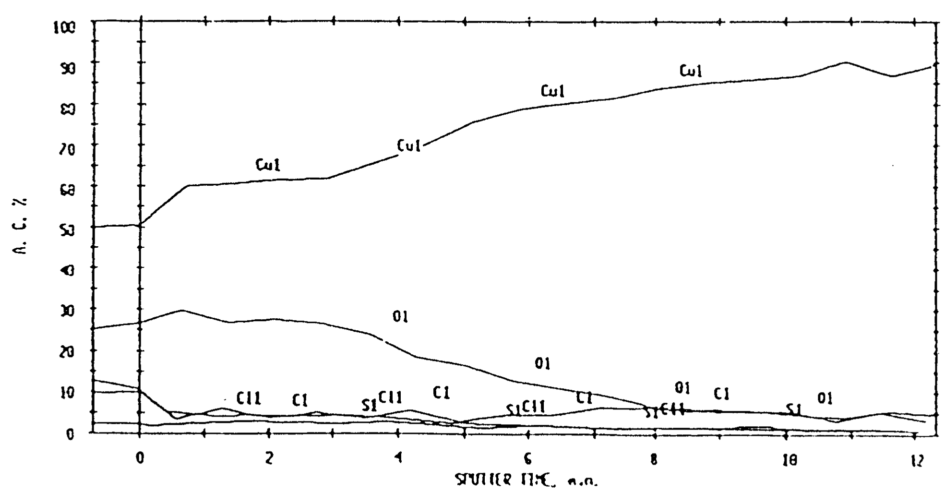

FIG. 10. PF/ONLY

Depth Profiles for 6061 Aluminum
Sputtering Rate is 200 A/Min

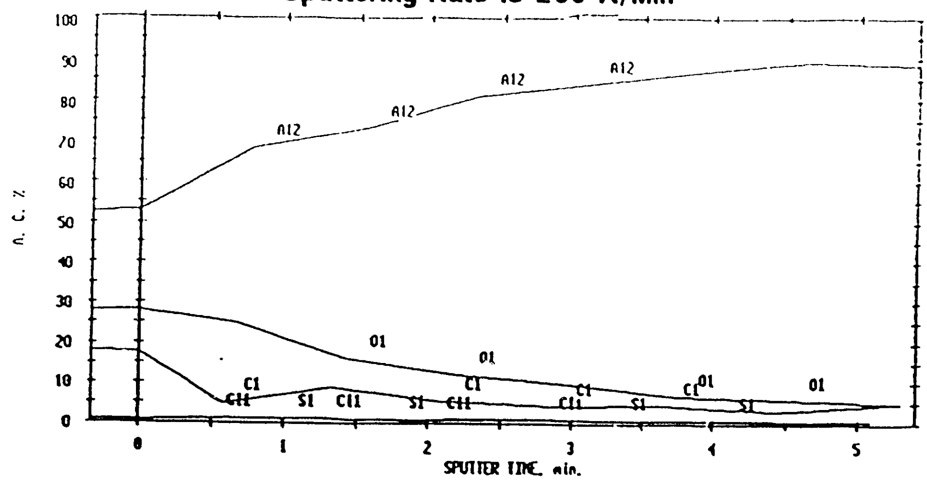

FIG. 11. TCA/ETHANOL

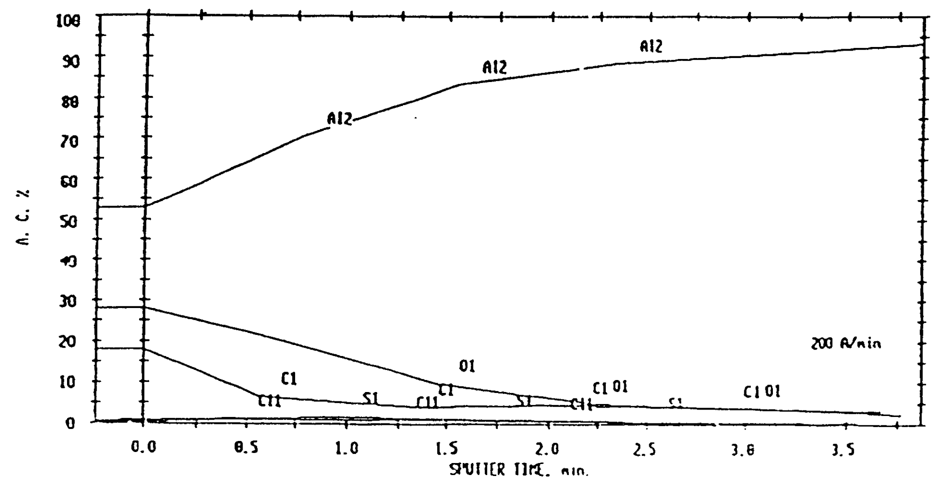

FIG. 12. PF/ETHANOL

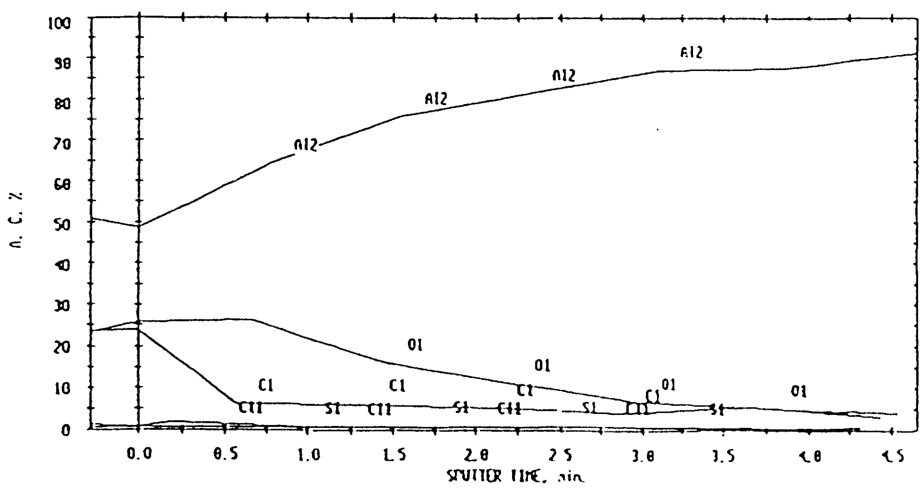

FIG. 13. PF/ONLY

END

DATE FILMED

12/09/91