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PRELIMINARY RESULTS FROM THE CONCAP-II-01 AND EOIM-3 EXPERIMENTS ON STS-46

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SUMMARY

CONCAP-II-01 (Complex Autonomous Payload, first flight) was the maiden flight of "CONCAP-II," a secondary payload, utilizing the Get-Away Special (GAS) carrier system, sponsored by the Consortium for Materials Development in Space (CMDS), at the University of Alabama in Huntsville (UAH). A host of materials such as high-T_c superconductor samples, metal films, polymers, carbons, and semiconducting materials were exposed to a low-Earth orbit (LEO) environment. We have also measured on orbit the changes in the electrical resistance of some of the high-T_c materials plus carbon and silver dosimeters exposed to the space environment. The preliminary results of this experiment are compared with those from EOIM-3, both of which flew on STS-46 and are presented here.

INTRODUCTION

Both CONCAP-II-01 and EOIM-3 (Evaluation of Oxygen Interactions with Materials—third series) payloads flew on Space Shuttle *Atlantis*, STS-46, on July 31, 1992.

The CMDS is one of the Centers for the Commercial Development in Space funded jointly by NASA and private sector companies interested in promoting the commercialization of space. UAH serves as the integrator of CONCAP-II. The EOIM-3 experiment was managed by Johnson Space Center.

The first flight of the facility carried three thin film experiments which investigated high-temperature superconducting materials improvement and materials preparation and longevity in hyperthermal oxygen. The payload contained an electronic controller and data system which monitored and recorded electrical resistance of 24 material samples of diverse composition. The system also controlled a hot-plate at 320 °C, the highest temperature at which materials studies of this kind have been conducted in space. Figure 1 shows a layout of experiment insert plate. The experiments that were flown on the first flight of the facility are briefly described below.

Experiment 1: High-T_c Superconductors

This experiment addresses the improvement and survivability of high-T_c superconductors (HTSC's) in space. In 1987 at UAH, a ceramic material, $YBa_2Cu_3O_{7-x}$, was discovered which attained zero electrical resistance above the normal boiling point of liquid nitrogen (0 K) (ref. 1).

For optimum performance, these materials must be fully oxygenated. With normal methods, this is achieved by sintering at 600 to 700 °C which severely limits the kind of substrates on which thin-film HTSC devices may be deposited. Further, exposure to heat and vacuum causes loss of oxygen, and thus, performance.

Based on recent experiments at Los Alamos Laboratory (ref. 2), it is believed that HTSC's prepared with mediocre superconducting properties may be converted to high-performance materials by processing in hyperthermal atomic oxygen (AO) (1.5 eV) at considerably lower substrate temperatures. The aim of the CONCAP experiment was to process a variety of thin film HTSC materials in the intense 5 eV AO flow encountered by the space shuttle in LEO.

In order to differentiate changes in HTSC film properties which occur during short ambient AO exposure (~10 h) from those produced by 3 to 4 months of environmental exposure, we have measured the electrical resistance of the devices during the flight exposure, using a 4-point contact technique. All devices, which included HTSC thin films and carbon and silver dosimeters, were exposed both at 320 °C and ambient temperatures to determine the effect of temperature on the oxygen-uptake process. These objectives have been satisfactorily achieved on this maiden flight. The hot plate and the controller performed efficiently and we have some interesting results. We are still in the process of analyzing these data.

Experiment 2: Longevity of Materials in Space

This experiment was concerned with the longevity of materials in space. We have exposed a large number of samples (metals, carbons, polymers, semiconductors, and high-T_c superconductors) to the 5 eV AO flow on the ambient plate and are in the process of measuring the degradation, contamination, and changes in the chemical, mechanical, and optical properties using a variety of techniques. We also had 16 samples on the 320 °C hot plate to evaluate the temperature dependence of AO surface reactions. There were identical samples on the EOIM-3 experiment, both on the ambient plate and three different hot plates (60, 120, and 200 °C). The CONCAP-II samples received one-half of the oxygen fluence of those on EOIM-3, and were covered and protected from contamination during all other flight operations by the motorized door assembly (MDA).

SAMPLE PREPARATION

The majority of the metallic longevity samples were deposited using planar diode sputtering and e-beam evaporation on fused silica optical flats, 1 inch and also 1/2 inch in diameter, supplied by Acton Research Inc. These metal films were optically thin in many cases, with thickness in the range of 20 to 230 nm. Polymer and some semiconductor samples were obtained from various sources. Some of the polymer thin films flown on CONCAP-II were prepared by the spin-coating technique. Some of the vitreous carbon samples were prepared in-house and the rest were manufactured by Union Carbide.

The high-T_c superconducting films, typically of the 1-2-3 type, were provided by our co-investigators, Dr. Tony Mogro (General Electric), Dr. Ian Raistrick (Los Alamos), Dr. Hoi Kwok (SUNY), and Dr. C. Y. Huang (Lockheed). These were mounted with AREMCO 569 and 571 epoxy glue in flatpax™ holders supplied by AIRPAX, MD as shown in the figure and the connections were made by thermoacoustic bonding.

Carbon and silver films for oxygen dosimeters were provided by Dr. Renchler (Los Alamos) and Dr. Smith/Ms. Lan (McDonnell Douglas), respectively. Additional films were obtained from Metech, PA. These were mounted in flatpaxes™ using AREMCO 569 epoxy and were bonded thermoacoustically at SCI, Huntsville.

MEASUREMENT TECHNIQUES

X-ray photoelectron spectroscopy and scanning profilometry are used to obtain preliminary results and are described in detail elsewhere (refs. 3 and 4). We intend to use other techniques such as scanning electron microscopy, scanning Auger microscopy, scanning tunneling microscopy/ atomic force microscopy, thin film x-ray diffraction, fourier transform infrared spectroscopy, Raman spectroscopy, and ultraviolet- (UV-)visible reflectance measurements.

RESULTS AND DISCUSSION

Onflight resistance data from carbon and silver dosimeters measured using 4-point technique is presented here briefly. The data on HTSC samples are being analyzed and will be published separately. Figure 2 shows the resistance (Ω) versus mission elapsed time (MET) (in minutes) data measured on the carbon thin film (~5,000 Å) which was mounted on the ambient plate. It is evident from this figure that most of the material was eroded away within ~400 min of exposure to AO in the RAM direction. Figure 3 shows the Ω versus MET data measured on silver thin film (220 Å thick) mounted on the ambient plate. As can be seen from this figure, most of the silver was attacked by AO, forming nonconducting silver oxides. These data indicate that the controller and associated electronics worked as designed.

Postflight visual inspection of the CONCAP-II samples suggested they were cleaner than the EOIM-3 samples which had a whitish appearance at the mask boundary on some samples. It is apparent that CONCAP-II samples were exposed only in orbit and were subsequently covered by the motorized door assembly (MDA) after ~21 h of exposure to AO, unlike EOIM-3 samples which remained exposed to the ambient environment of the shuttle after the mission. The relative levels of contamination on EOIM-3 and CONCAP-II will be compared.

X-ray photoelectron spectroscopy is one of the most surface-sensitive analytical techniques. This technique probes <100 Å at the surface, and it is easy to extract information about surface atomic concentration and chemical states of the constituents from the details of changes in shape and binding energy shifts of the core level peaks. The surface composition information for Cu, Ni, and Cu samples from CONCAP-II and EOIM-3 is tabulated in Tables 1, 2, and 3. Examination of the data in these tables shows that all the longevity samples that are analyzed have Si contamination in the range of 2 to 11 percent. In addition, other contaminants such as Na, Cl, and F plus some N are observed. Carbon content due to ambient hydrocarbon contamination was similar on all the metallic samples. Oxygen is present on all the samples, in the form of oxides on metallic samples and in adsorbed form due to moisture and or CO and CO_2 species on carbon samples.

Figures 4, 6, and 8 compare the X-Ray Photoelectron Spectroscopy (XPS) survey scans measured on Cu, Ni, and C samples from CONCAP-II and EOIM-3. In addition to the substrate peaks, we can clearly see the peaks due to oxygen, carbon, and silicon. Figure 5 compares the high resolution Cu 2p scans measured from the Cu samples. The peak shape and the appearance of "shake-up satellites" suggest the presence of CuO (Cu^{2+}) species in the exposed area of the flight samples (ref. 3). Figure 7 compares the high resolution Ni 2p peaks from Ni samples, the binding energy, and the shape of Ni 2p peaks suggest that Ni at this interface is in NiO (Ni^{2+}) form (ref. 5). Detailed analysis will be presented in a separate paper. There is hardly any difference in the shape of the carbon peaks measured from the CONCAP-II and EOIM-3 samples (Figure 9). It is difficult to differentiate between the carbon from the bulk and the contamination due to residues from extraneous hydrocarbons. Some portion of the measured carbon 1s peaks may be due to this contamination.

The advantage of using stylus profilometry to measure the erosion characteristics on polymers and metals has been amply demonstrated previously (ref. 4). Here we present profilometry results on Cu and carbon samples. Figure 10 shows the stylus profile of the exposed region of Cu thin film from the ambient plate of CONCAP-II. Based on the initial thickness of 570 Å, this film shows an increase in thickness of ~90 Å in the exposed area. Similar increase in thickness was observed on the Cu thin film exposed on the ambient plate of EOIM-3. The Cu thin film exposed to the AO on the 320 °C hotplate on CONCAP-II showed an increase in thickness of ~900 Å (Figure 11). Similarly there was an erosion of ~2 μm on the highly polished vitreous (glassy) carbons exposed on the ambient plate of CONCAP-II as shown in Figure 12. More measurements on EOIM-3 samples are underway.

CONCLUSION

A comparison was made of contamination on samples from CONCAP-II and EOIM-3. The XPS results measured on samples from these two experiments agree reasonably well. Silicon contamination on samples from both the experiments is similar. Similar contaminant levels were observed on samples from the STS-8 flight. The origin of this contamination may be attributed to siloxanes/silicones which converts to silica on being exposed to AO. From XPS data we can deduce that the oxide formed on Cu thin film samples mounted on both the experiments is predominantly CuO in nature. Similarly, we have observed the presence of NiO species on Ni samples.

Scanning profilometry results show that there is an increase in the thickness of ~90 Å on the Cu samples from the ambient plates of CONCAP-II and EOIM-3. However, we observed a step height of ~900 Å on the Cu sample exposed on 320 °C hot plate of CONCAP-II. It would be interesting to compare this result with those from samples on three different hot plates (60 °C, 120 °C, and 200 ° C) of EOIM-3. We have observed an erosion of ~2 mm on the highly polished samples of vitreous carbon from CONCAP-II. Other samples are being currently analyzed.

The operation of the flight experiment facility CONCAP-II on STS-46 has demonstrated its flexibility and usefulness as a rapidly deployable system for obtaining active measurements on materials in orbit. Resistance measurements from HTSC and oxygen-dosimetry devices were recorded during oxygen atom exposure. It is surprising that the Si levels on CONCAP-II samples are, within the scatter of the data, the same as these on EOIM samples, which were exposed to the shuttle bay environment throughout all orbital operations. These included deployment of EURECA and the tethered satellite. It is noted that adsorption of silicon molecules onto surfaces is dependent

on surface temperature and perhaps on surface chemical nature. Large amounts of silicones are used in the treatment of shuttle external surfaces before launch, and it appears that a volatile fraction of these is present in the shuttle gas cloud on orbit. It may be that the ram-beam of AO striking the surfaces of the shuttle bay actively contributes to the silicone molecular density by desorbing bound molecules. We have observed similar effects in terrestrial AO beam facilities.

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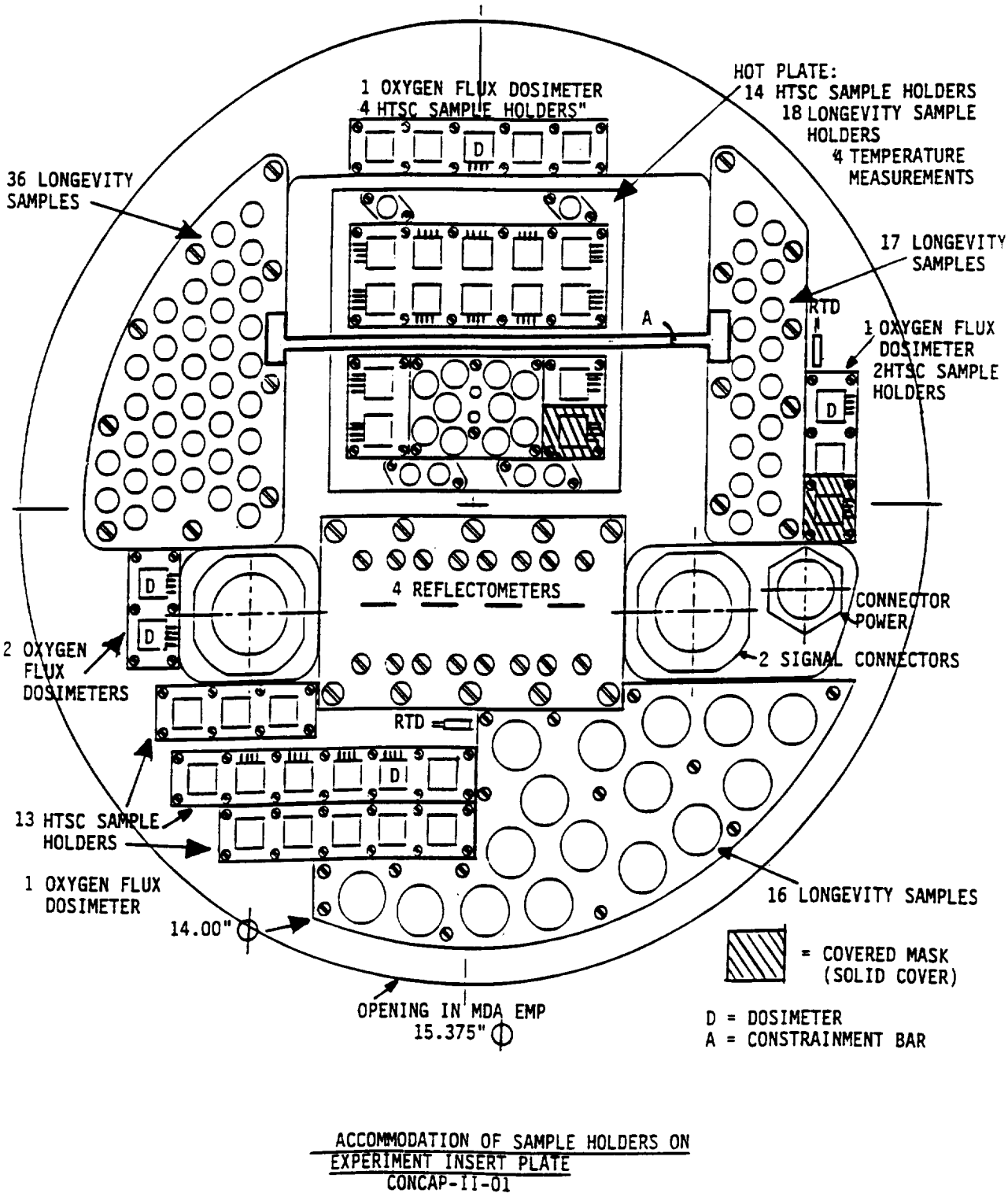


ACCOMMODATION OF SAMPLE HOLDERS ON EXPERIMENT INSERT PLATE
CONCAP-II-01

Figure 1. Layout of CONCAP-II.01 experiment insert plate.

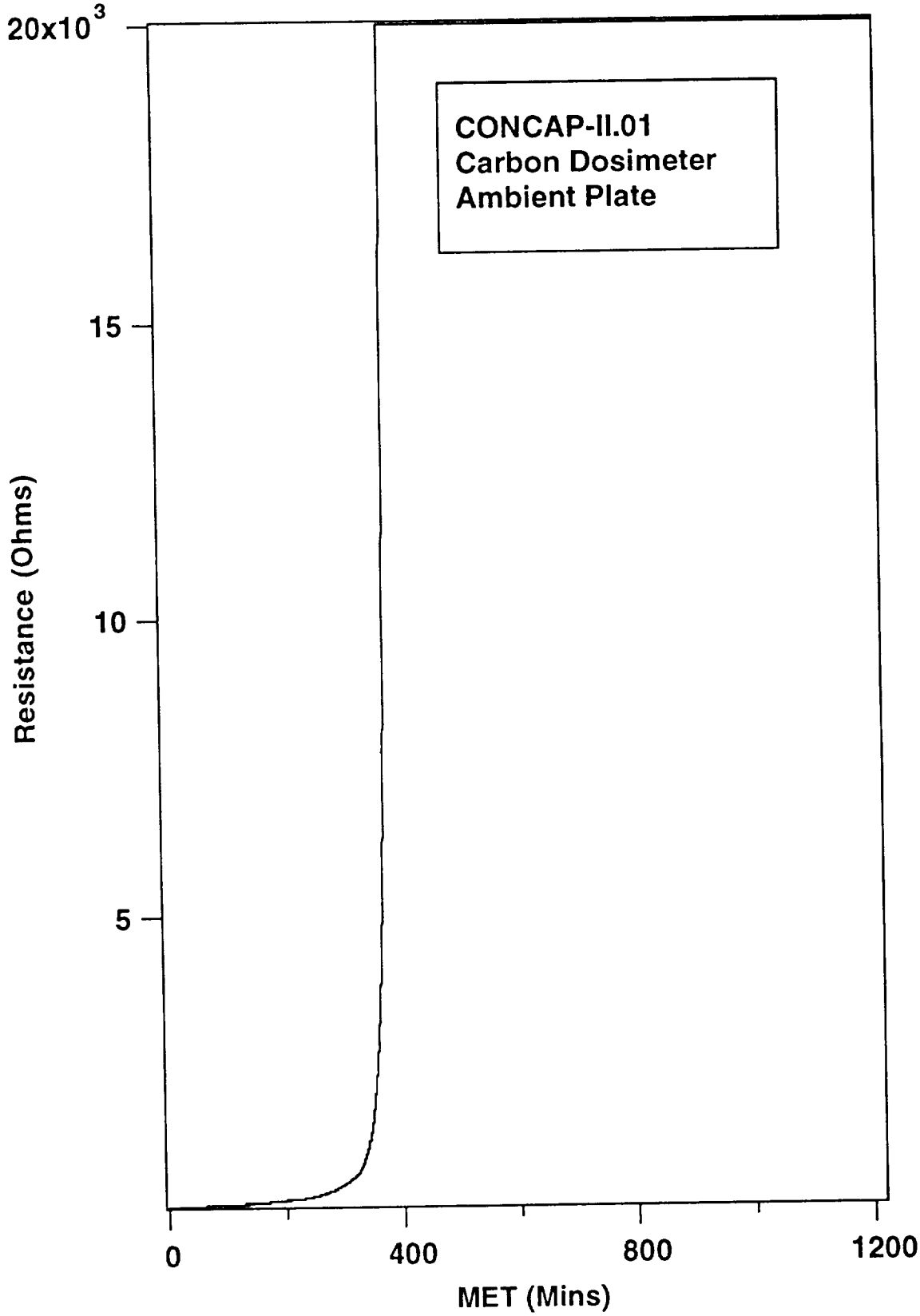


Figure 2. The resistance versus MET time plot of carbon dosimeter from the ambient plate.

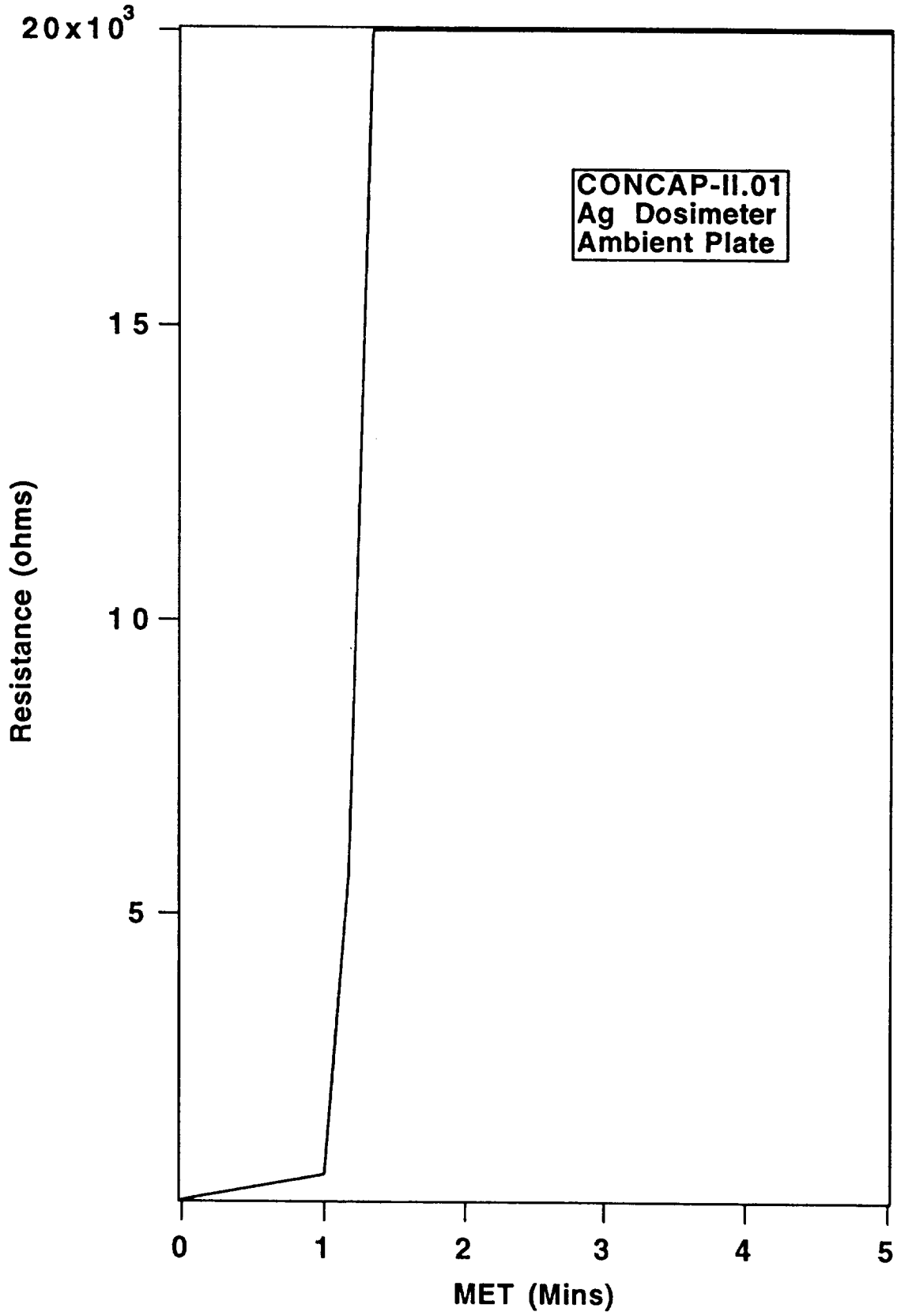


Figure 3. The resistance versus MET plot of silver dosimeter from the ambient plate.

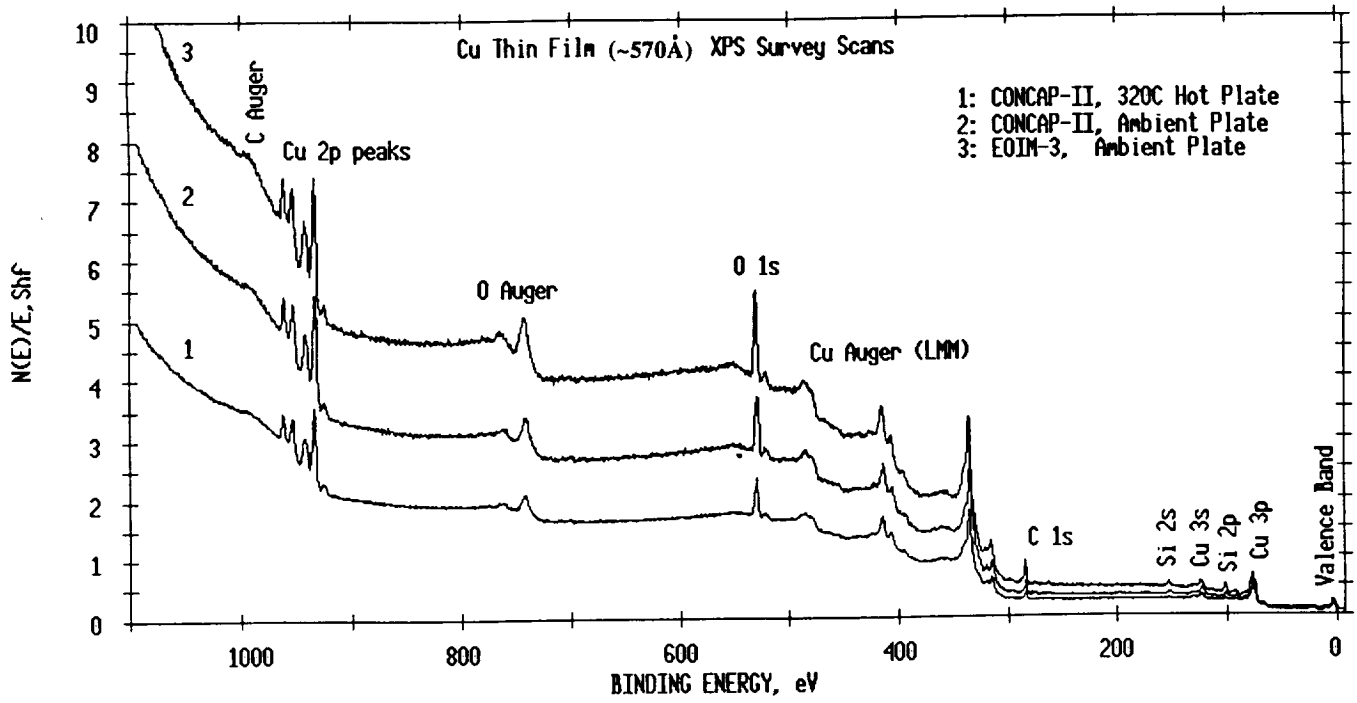


Figure 4. Comparison of XPS survey scans from Cu thin films from CONCAP-II and EOIM-3.

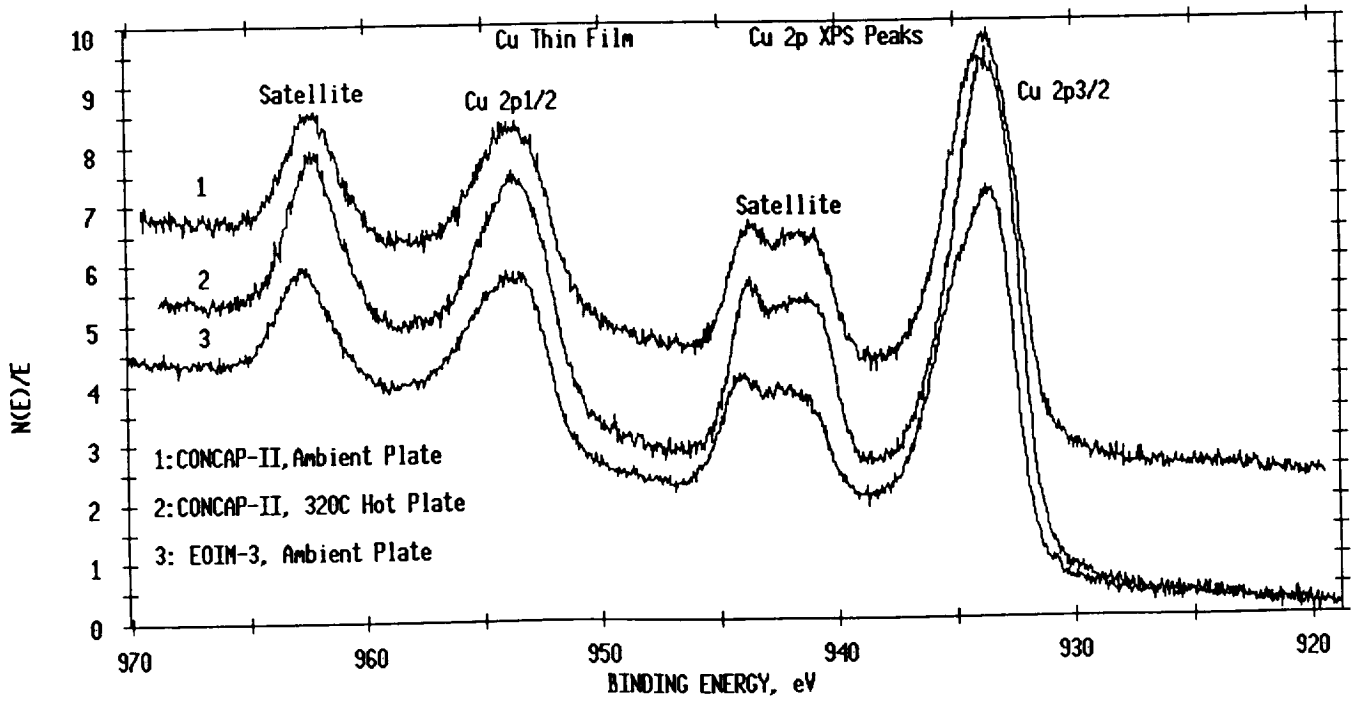


Figure 5. Comparison of Cu 2p core lines from Cu thin films (~570 Å).

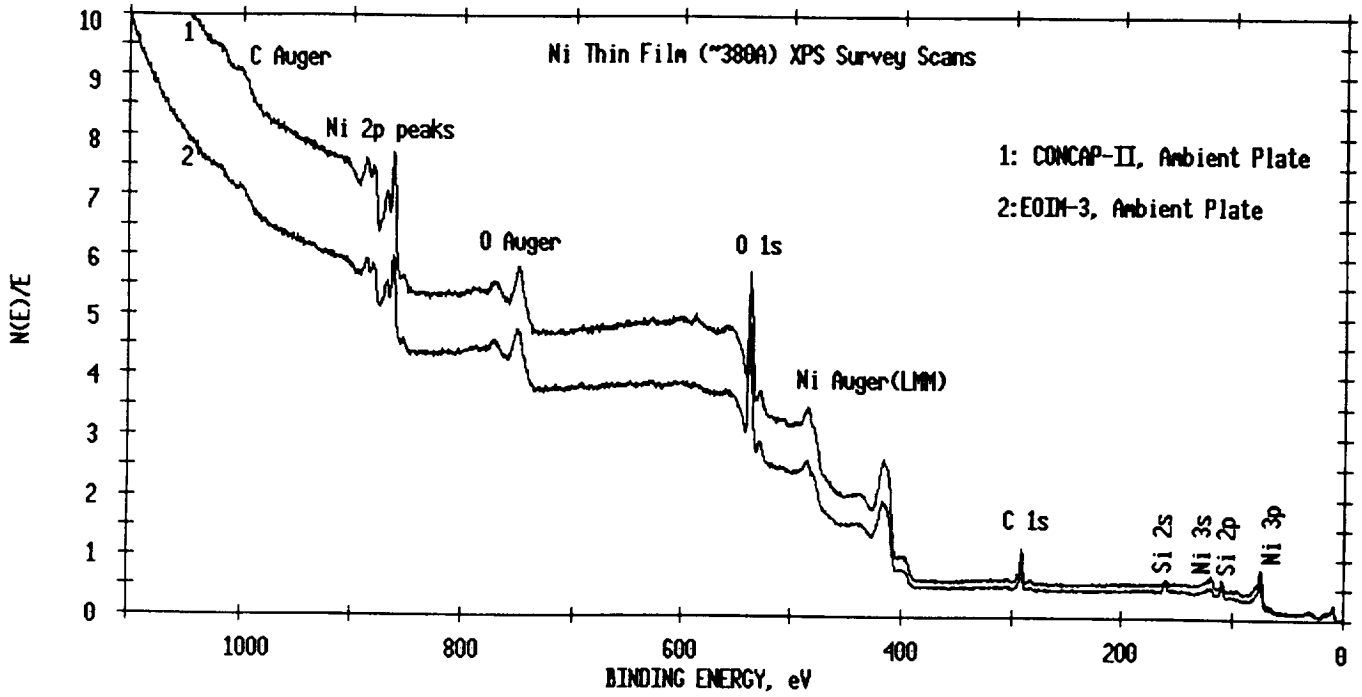


Figure 6. Comparison of XPS survey scans from Ni thin films from CONCAP-II and EOIM-3.

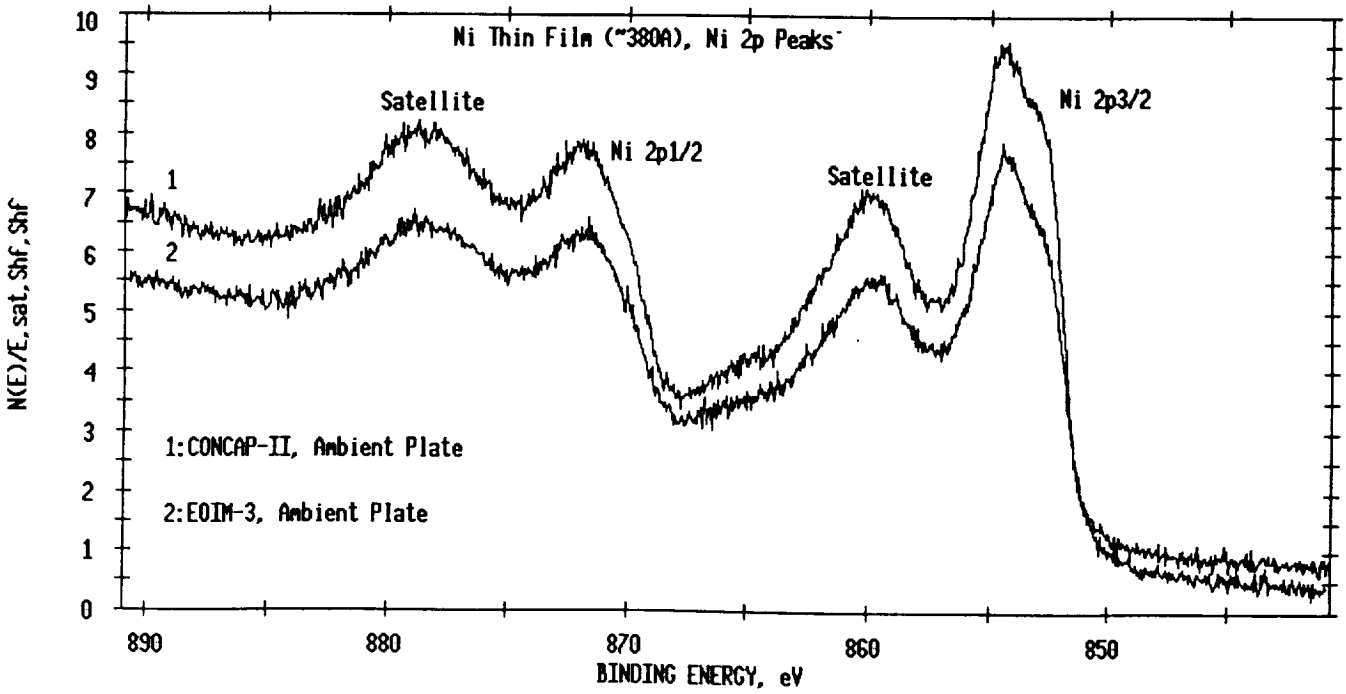


Figure 7. Comparison of Ni 2p core lines from Ni thin films (~380 Å).

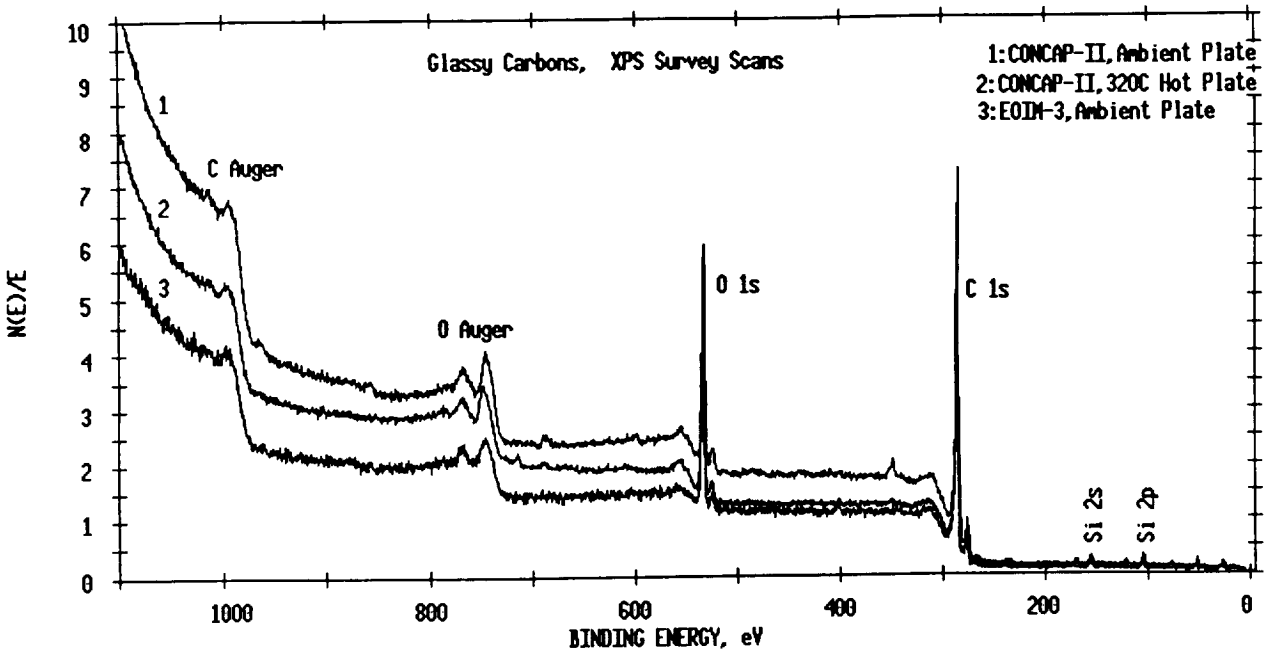


Figure 8. Comparison of XPS survey scans from glassy carbons from CONCAP-II and EOIM-3.

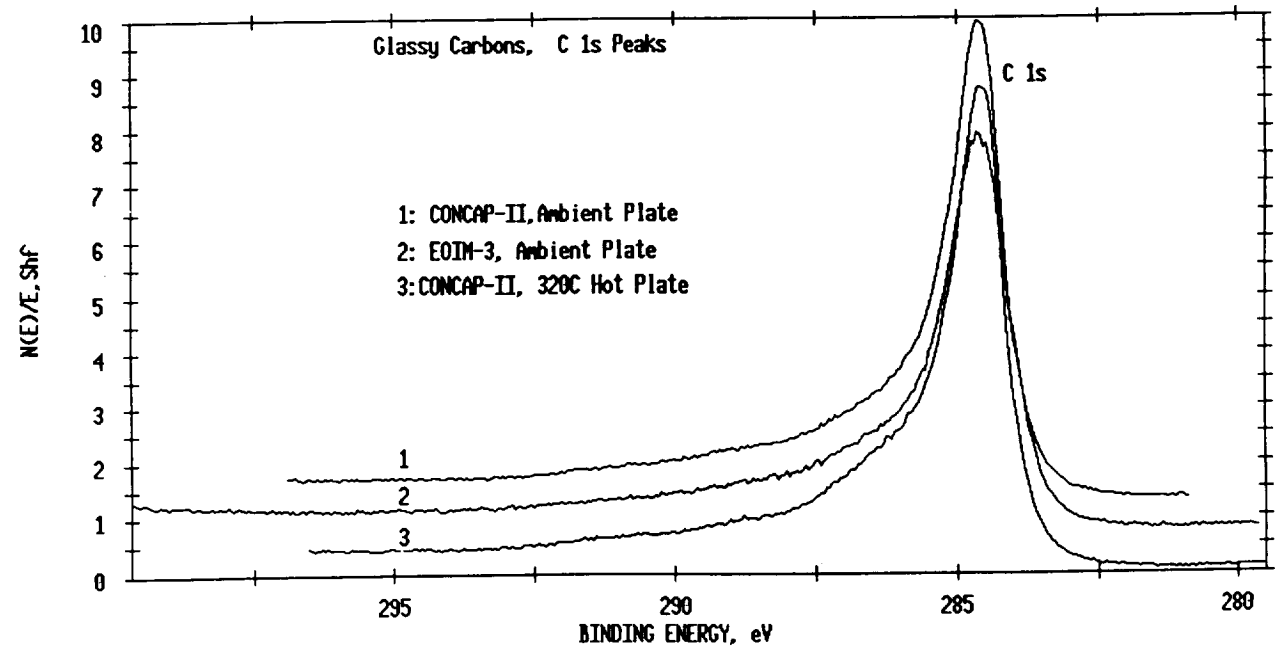


Figure 9. Comparison of C 1s core lines from glassy carbons.

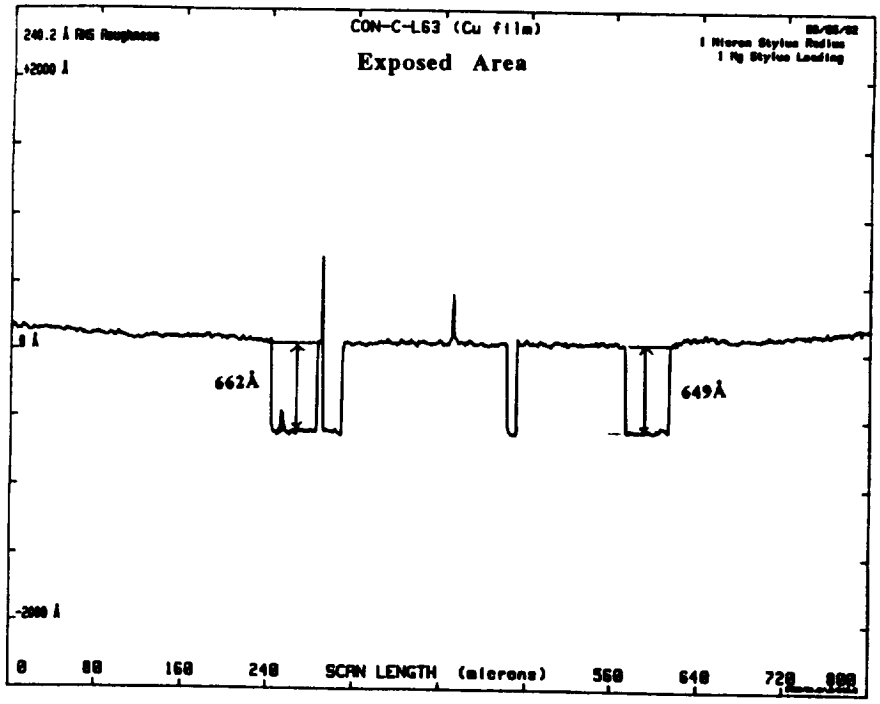


Figure 10. Surface profile of the exposed region of Cu thin film from the ambient plate of CONCAP-II.

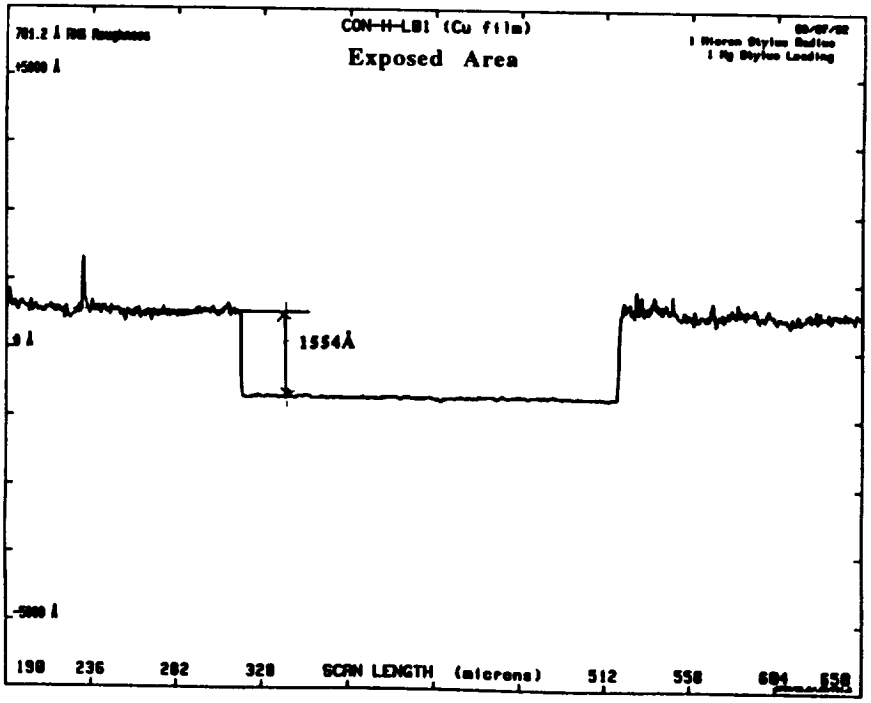
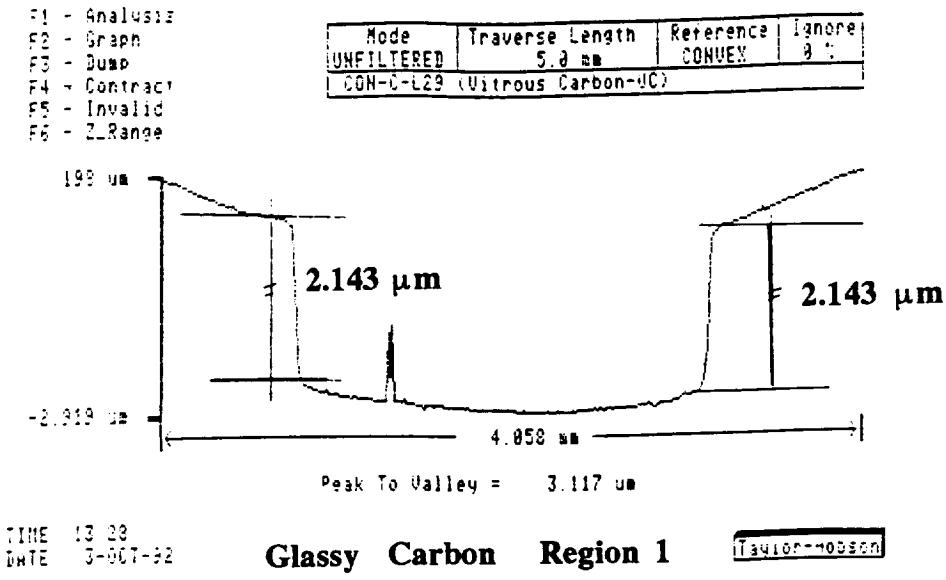


Figure 11. Surface profile of the exposed region of Cu thin film from the 320 °C hot plate of CONCAP-II.



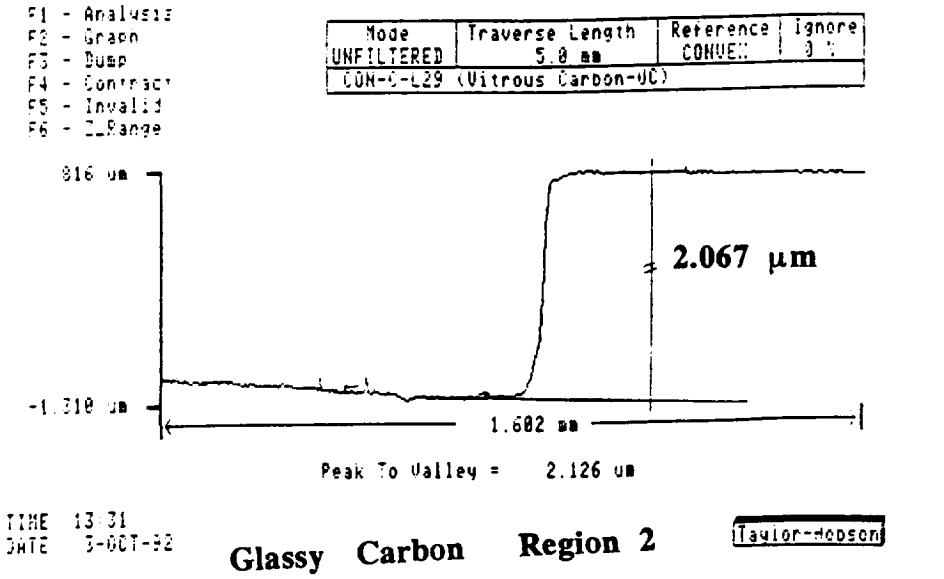


Figure 12. Surface profile of the exposed region of glassy carbon from the ambient plate of CONCAP-II.

Table 1. Atomic concentrations of Cu thin film samples from CONCAP-II and EOIM-3.

XPS Photo Peak	CONCAP-II Ambient Plate (~300 Å)	CONCAP-II Ambient Plate (~700 Å)	CONCAP-II 320 °C Hot Plate (~700 Å)	EOIM-3 Ambient Plate (~300 Å)
Cu 2p A.C in %	12.4	12.8	14.6	21.2
O 1s A.C in %	46.5	47.5	54.3	43.8
C 1s A.C in %	26.5	26.6	22.4	22.4
Si 2p A.C in %	11.4	9.2	6.4	9.9
Na 1s A.C in %	2.0	1.7	1.1	1.0
Cl 2p A.C in %	1.2	2.1	1.2	1.3
F 1s A.C in %	0.0	0.0	0.0	1.3
N 1s A.C in %	0.0	0.0	0.0	0.0

Table 2. Atomic concentrations of Ni thin film samples from CONCAP-II and EOIM-3.

XPS Photo Peak	CONCAP-II Ambient Plate (~380 Å)	EOIM-3 Ambient Plate (~300 Å)
Ni 2p A.C in %	21.0	17.0
O 1s A.C in %	44.2	48.3
C 1s A.C in %	25.3	21.8
Si 2p A.C in %	7.2	10.9
Na 1s A.C in %	0.3	0.0
Cl 2p A.C in %	6.0	0.6
F 1s A.C in %	1.4	1.7
N 1s A.C in %	0.0	0.0

Table 3. Atomic concentrations of glassy carbons from CONCAP-II and EOIM-3.

XPS Photo Peak	CONCAP-II Ambient Plate (1/2-inch Disk)	CONCAP-II 320 °C Hot Plate (1/2-inch Disk)	EOIM-3 Ambient Plate (1-inch Disk)
C 1s A.C in %	71.0	62.8	84.0
O 1s A.C in %	16.0	23.6	12.0
Si 2p A.C in %	2.7	4.7	1.8
Na 1s A.C in %	9.1	0.4	0.2
Cl 2p A.C in %	0.3	0.4	trace
F 1s A.C in %	0.9	0.6	0.3
N 1s A.C in %	1.4	1.1	1.7
Al 2p A.C in %	nd	6.4	nd