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LOW ENERGY X-RAY SPECTRA MEASURED WITH A MERCURIC IODIDE ENERGY DISPERSIVE SPECTROMETER IN A SCANNING ELECTRON MICROSCOPE

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ABSTRACT

A mercuric iodide energy dispersive x-ray spectrometer, with Peltier cooling provided for the detector and input field effect transistor, has been developed and tested in a scanning electron microscope. X-ray spectra were obtained with the 15 keV electron beam. An energy resolution of 225 eV (FWHM) for Mn-K_α at 5.9 keV and 195 eV (FWHM) for Mg-K line at 1.25 keV has been measured. Overall system noise level was 175 eV (FWHM). The detector system characterization with a carbon target demonstrated good energy sensitivity at low energies and lack of significant spectral artifacts at higher energies.

INTRODUCTION

Mercuric iodide x-ray spectrometery has been advanced considerably in the last several years (1-10). The exceptionally low room temperature leakage current which is possible with HgI_2, combined with the good transport properties of electrons, have formed a good basis for high energy resolution x-ray detectors capable of operation at room temperature or slightly below. Because there is no need for the cryogenic coolant and its associated vacuum cryostat, the design of compact and lightweight detection system is possible. This simplicity and considerable size advantage can be important for terrestial applications as well as for space exploration.

Mercuric iodide detectors have previously demonstrated 300 eV (FWHM) energy resolution for the 5.9 keV Mn-K_α line from an Fe-55 source and 245 eV (FWHM) for the 1.25 keV Mg-K line with both the detector and preamplifier operated at room temperature (1-3). The sensitivity of HgI_2 detector have also been shown for ultra soft x-ray below 1 keV. The characteristic x-ray peak of oxygen at 523 eV was clearly resolved from noise (9). With the input field effect transistor (FET) cooled to its optimum temperature of approximately 140° K and the HgI_2 detector at room temperature, a resolution of 175 eV (FWHM) for 1.5 keV (Al-K) has been demonstrated (7). These latter resolution figures are comparable to those achieved with commercial Si(Li) detector systems. Currently work is underway to achieve comparable HgI_2 resolution values with the preamplifier input FET cooled using a thermoelectric (Peltier) cooler.

Work on laboratory research prototype version of the Scanning Electron Microscope and Particle Analyser (SEMPA) instrument at The Jet Propulsion Laboratory (JPL) provided motivation for development of a minature, lightweight and low power HgI_2 x-ray spectrometer for scanning electron microscope (SEM) (11,12). The SEMPA instrument is proposed as one of the instruments for a Mariner Mark II mission to rendezvous with a comet in the 1990's (13).

As proposed, the spacecraft would travel with the comet for several years while periodically performing analyses to provide information on the composition and character of the comet dust. The SEMPA instrument would collect solid comet dust, image individual grains and collect x-ray spectra for quantitative determination of Na and key elements with higher atomic number, expected to be in the minerals and rocks. The total actual analytical time for imaging and x-ray analysis is expected to be about 1000 hours.

The requirements of such an interplanetary mission place severe constraints on the selection of analytical instruments, including the choice of an x-ray detector for SEMPA. The use of a Si(Li) detector would require the use of a costly radiative cooler to achieve the required near liquid nitrogen temperatures. There is evidence that a Si(Li) detector system can be operated at degraded performance level with thermoelectrical cooling, however high electrical power and heat dissipation capacity would be needed (14,15). Therefore the use of a HgI_2 detector was identified as a good choice to minimize power consumption and weight of the SEMPA instrument since the preamplifier input FET and detector would clearly need to be cooled using only small thermoelectric coolers.

A series of experiments have been conducted using a mercuric iodide energy dispersive x-ray spectrometer installed in the JPL SEMPA research prototype instrument. These are continuing experiments designed to study and improve such factors as obtainable x-ray energy resolution, effects of detector positioning and proximity to the target, and optimization of thin filters in front of the detector to eliminate unwanted backscattered electrons.

EXPERIMENTAL SET UP

An outline drawing of the SEMPA target chamber is presented as Figure 1. The detector and first stage of field effect transistor preamplification are inside of the flanged housing (to the left in the views) and thus extend into the microscope vacuum. The vacuum flange and electrical feedthroughs lead to the subsequent stages of amplification which are housed externally.

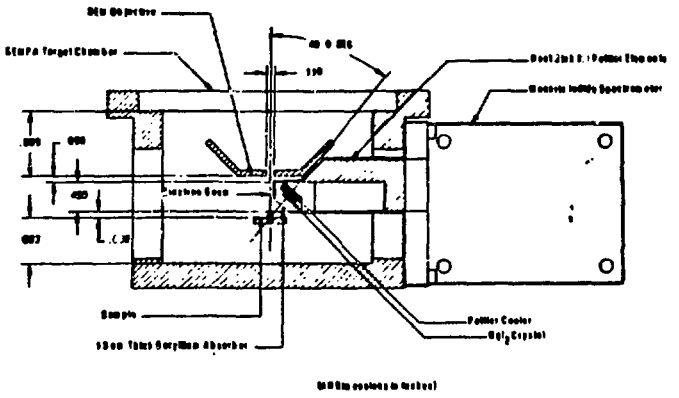

Figure 1. Cross sectional view of SEMPA instrument target chamber

The mercuric iodide detector and associated pulsed light feedback preamplifier are outlined. The detectors in these experiments had 2 to 5 mm^2 active area and were mounted on a single stage Marlow MI 1021 electrical Peltier element so the detector could be slightly cooled to the temperature of about 0° C.

The detector angle relative to the electron beam was 40° and the distance between the target and the HgI_2 detector plane was about 10 mm. The solid angle of collection was 0.02 to 0.05 steradians. The input FET was cooled with a three stage Peltier element Marlow MI 3026 to approximately - 40° C. This is still not cold enough for optimum noise reduction for this type of silicon transistor (2N4416), that temperature being about -120° C. The total power supplied to the detector-preamplifier system including both Peltier coolers was about 3 Watts.

A metal shield formed from thin stainless steel covered the front end assembly containing the detector and first stage FET within the target chamber. A thin section of beryllium was placed between target and detector to absorb unwanted backscattered electrons. The construction allowed for easy replacement of absorbers. Experiments were performed with two different thicknesses of beryllium sections of 12 μm and 8 μm.

The x-ray spectra were obtained with 15 keV electron beam provided by SEMPA instrument. The current of the beam was kept below few nanoampers. The metal targets used were high purity polished standards. The carbon target was a commercial, unpolished, SEM sample mounting stub. Electrical signals from detector-preamplifier system were fed to a Canberra 2020 amplifier utilizing a 12 us shaping time constant. No pileup rejection or reset blanking was used. The pulse height analyzer was a Tracor TN 1242 in the combination with TN 4000 system in the standard configuration as for Si(Li) detectors. The standard Tracor programs were used for peak identification and energy resolution calculations.

X-RAY SPECTROSCOPY

Figure 2 shows the spectrum obtained from a copper target. Three lines Cu L (0.93 keV), Cu K_{α} (8.05 keV) and Cu K_{β} (8.90 keV) are clearly visible. The L line of copper is seen with it's intensity diminished by an 8 μm beryllium absorber and due to the detector carbon entrance electrode. The energy resolution of the K_{α} line is 230 eV (FWHM). Figure 3 shows the K_{α} and K_{β} lines manganese at 5.90 keV and 6.49 keV, respectively. In this case energy resolution is 225 eV (FWHM) for the Mn K_{α} peak. Figure 4 shows the K line of the spectrum obtained from magnesium target. The 195 eV (FWHM) energy resolution is the first obtained below 200 eV using Peltier coolers. Also shown in Figure 4 is a pulser peak. A measurement of the electronic noise level of the mercuric iodide spectrometer made by the pulser method indicated a value of 175 eV (FWHM).

All measured x-ray peaks show excellent symmetry. The shape and intensity of background counts is typical for electron excitation. Within acquired counting statistics, I or Hg escape peaks (ΔE-3.9 keV and 2.2 keV, respectively) are not visible in the spectra. This is expected from the fact that the escape peaks would be from iodine L and mercury M levels which have very small fluorescence yields (16). The energy resolution of the x-ray peaks are significantly improved over values obtained in the previous initial experiments in the scannings electron microscope (8).

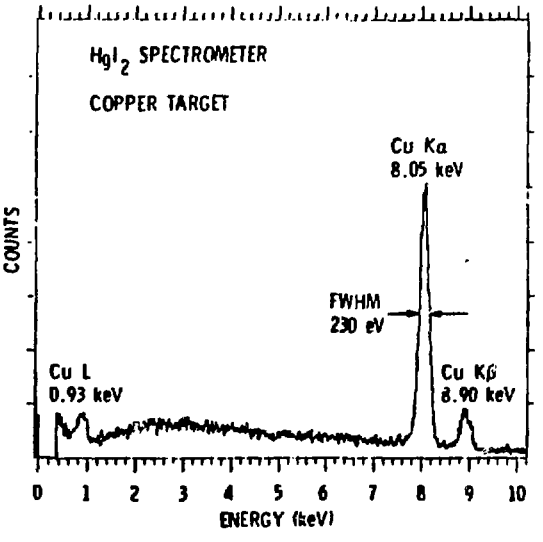

Figure 2. X-ray spectrum obtained from copper target

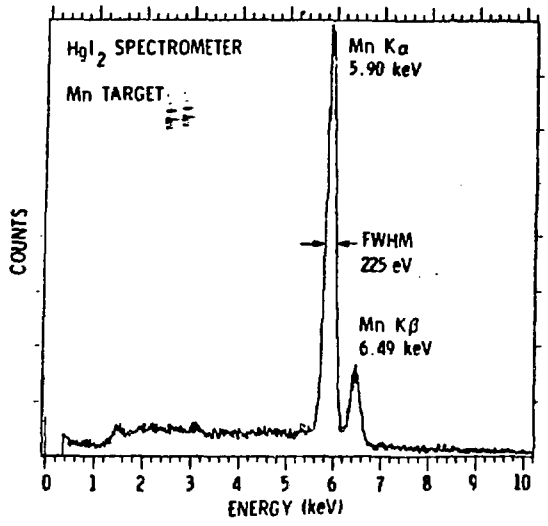

Figure 3. X-ray spectrum obtained from manganese target

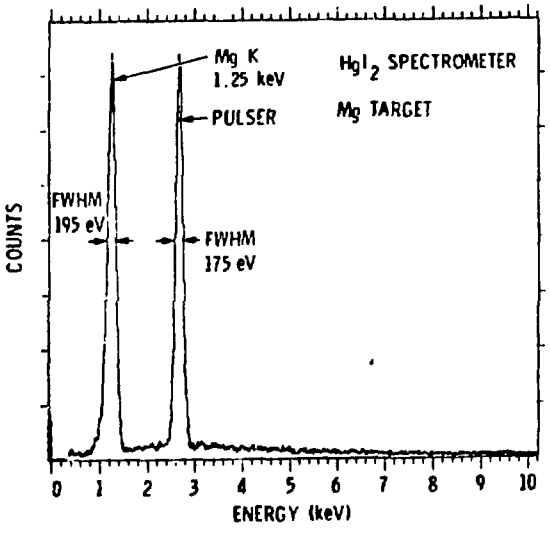

Figure 4. X-ray spectrum obtained from magnesium target. (Electronic pulser indicates system noise)

The Fano factor calculated using energy resolution 230 eV (FWHM) for 8.05 keV (Cu-K_α line) and pulser width 175 eV (FWHM) gives a value of 0.12. This value is similar to the lowest previously reported for HgI_2 (7) and also close to the experimental value of Fano factor obtained for silicon. A lower measured value for the Fano factor reflects a lower contribution from the trapping phenomena in the x-ray spectra and indicates that electronic noise is limiting factor in the obtainable energy resolution.

DETECTOR CHARACTERIZATION WITH A CARBON TARGET

Every energy dispersive detector system creates artifacts in an x-ray spectrum that are characteristic of the detector rather than the excited target. The most obvious of these are limited efficiency for very high and low energies because of the presence of absorbing windows and contacts, and finite detector thickness. More subtle, but always present to some degree are escape peaks, absorption edges and secondary fluorescence peaks due to the crystal properties and materials. One convenient way to partially characterize an SEM detector system for energy response and artifacts is to observe the spectrum generated by a carbon target. The carbon characteristic K line is at 0.282 keV so the spectrum above that energy is entirely Bremstrahlung continuum if no heavier elements are present. The shape of the energy spectrum of the continuum leaving the sample is calculable, so any observed differences are due to the detector. Figure 5 shows the spectrum from a carbon target measured for one of the HgI_2 detectors used in experiments. The spectrum contains 1.6×10^6 counts. The shape of the spectrum above 2 keV is the smooth shape expected from 100% efficiency detection of continuum x-rays. Below 2 keV the detected intensities are greatly reduced by absorption in the 12 μm thick Be window and detector front carbon contact. Calculation using tabulate a mass absorption coefficients indicates that 50% of the detection efficiency decrease was due to the Be window and 50% due to the carbon entrance electrode. The detector's net efficiency at 1 keV is about 10%. The only other spectral artifact detectable above 1 keV in this spectrum is the "peak" at 1.75 keV. This may have been due to secondary fluorescence of Si in the silicone rubber near the detector active area, or due to Si in the carbon target. There are no Hg or I lines or absorption edges that could produce a peak at this location. The largest absorption edge jump should be due to Hg-M at 2.4 keV, but no such feature is observable in this detector. This indicates a very thin HgI_2 deadlayer. An approximate computer model of the continuum and the effect of absorbers and deadlayer has been created. The model confirmed the discrete energy hand calculation of the absorber effect, and

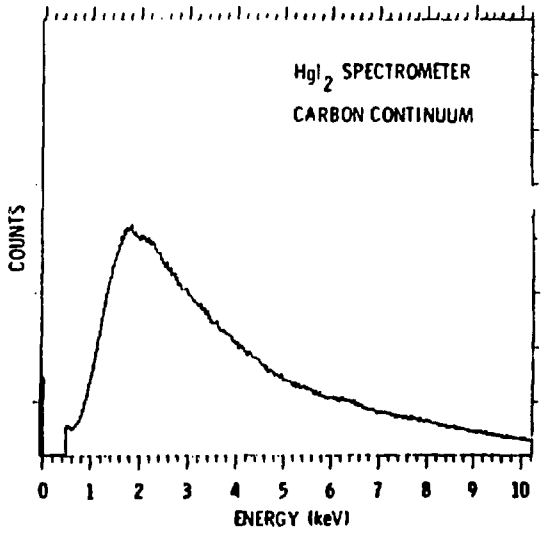

Figure 5. X-ray spectrum obtained from carbon target

indicates that the presence of even 50 nm of HgI_2 deadlayer would significantly alter the continuum from that observed. The computer model for HgI_2 detectors, and deadlayer-absorber simulation will be the subject of a future paper.

DISCUSSION AND CONCLUSION

The experimental results have demonstrated that a mercuric iodide detector system can be successfully used in a compact, power-limited scanning electron microscope. The energy resolution achieved, from 195 eV for Mg-K (1.25 keV) to 230 eV for Cu-K_α (8.05 keV), is adequate for many applications.

The excellent symmetry of the x-ray peaks obtained and the low value of Fano factor indicate good electron collection in the detector and that the influence of trapping phenomena is small. The Fano factor for HgI_2 reported in this paper is similar to the experimental value obtained for silicon. This means that energy resolution values achieved for HgI_2 detectors are mainly limited by electronic noise due to the preamplifier input field effect transistor. More efficient cooling of the input FET closer to its optimal temperature or replacement with a lower noise device than the 2N4416 transistor and would bring the energy resolution of the HgI_2 system closer to the performance obtained from cryogenically cooled silicon spectrometers.

Characterization of HgI_2 detectors with a carbon x-ray target indicates lack of evidence of artifacts in the x-ray spectra due to absorption edges, secondary fluorescence, and escape peaks related to mercury and iodine elements. Detection efficiency in the low energy region (below 2 keV) is limited by the thickness of beryllium absorber and detector carbon entrance electrode. There is no evidence of any inherent deadlayer in HgI_2. By elimination of the beryllium absorber (which is not used for any cryostat housing purpose) and using a thinner evaporated metal entrance electrode (described in ref. 9) a simple, non-hermetic, true windowless detector system should be possible with the addition of magnetic or electrostatic backscattered electron "filter". Moreover, non-hermetic construction allows for detector placement closer to the sample with potentially increased geometrical efficiency.

Compact HgI_2 spectrometers will be extremely valuable for space exploration x-ray analysis because of their reduced weight and power requirements. On earth they could reduce the cost of x-ray fluorescence analytical equipment as well as make these instruments truly hand-portable and convenient for many applications.

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