OBSERVATIONS OF MECHANICALLY POLISHED KCl SURFACES USING SCANNING ELECTRON MICROSCOPY

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13. ABSTRACT

The surfaces of polished KCl samples, prepared by different mechanical polishing techniques, were examined using a scanning electron microscope and electron microprobe. An evaluation of the general quality of the polish and of the chemical composition of foreign matter found on the surface was completed. A qualitative analysis using an energy dispersive system was performed on all samples. Quantitative microanalysis was also completed. The results of these analyses and their implications are discussed in this report.

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Illustrations

Observations of Mechanically Polished KCl Surfaces Using Scanning Electron Microscopy

I. INTRODUCTION

The need for laser window materials with good optical and mechanical properties has generated great interest in producing high quality surfaces. The surface of a potential window material should be sufficiently smooth and polished to accept a protective or antireflective coating. It should also be free from scratches and foreign matter, both of which enhance local electromagnetic fields and act as scattering or absorbing centers. Various mechanical polishing techniques have been developed in attempts to obtain a highly polished surface and to improve the absorption characteristics of the material. However, most of these techniques still produce scratches and deep gouges in the surface. Moreover, remnants of polishing material are sometimes found on the sample after polishing and thorough rinsing have been completed. The problem of obtaining a good mechanical polish on KCl (and similar materials) is particularly complicated since KCl is hygroscopic and soft. The need to evaluate the quality of finish on such a substance motivated this study.

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2. EXPERIMENTAL PROCEDURES

The purpose of this study was to evaluate the surface condition of the mechanically prepared samples for quality of the polish and surface cleanliness; that is, to what degree is the surface free of foreign materials. To accomplish this evaluation, an AMR-900 scanning electron microscope and an ARL EMX-SM electron microprobe were employed. The scanning electron microscope was equipped with an energy dispersive X-ray analysis system[1] which could be used for quick qualitative evaluations of surface constituents. The electron microprobe was used to measure quantitatively the composition of the surface. A random selection of scratched areas on the surface was made to characterize a typical surface in its most unfavorable conditions.

Five different preparation methods were used on samples of single-crystal KCl. These preparation techniques are shown in Table 1.

Table 1. Preparation Techniques on Samples of Single-Crystal KCl

Sample	Polishing Method
1	600 grit SiC
2	SiC, 1μ Al_2O_3
3	SiC, 1μ Al_2O_3, Linde A
4	Same as 3, then Linde B
5	Same as 4, then MgO

Each crystal was lapped in slurry of kerosene and 1000μ grit SiC until all large surface defects were removed. Then the sample was immersed in kerosene and ultrasonically cleaned. Following this, the polishing methods listed in Table 1 were completed. Alcohol was mixed with the polishing compound and the sample was polished in the mixture until dry. After the samples were polished through their respective sequence, they were examined in the electron probe for existence of remnant material on the surface.

The electron probe is an instrument that utilizes an electron beam to excite characteristic X-rays from the elements in the surface of a sample.[2] These characteristic X-rays are then separated by a crystal spectrometer and detected

1. Russ, J. C. (1971) Energy Dispersion X-ray Analysis; X-ray and Electron Probe Analysis, Amer. Soc. for Testing and Materials.
2. Birks, L. S. (1963) Electron Probe Microanalysis, Interscience Publishers.

using sealed or flow proportional counters. The comparison of X-ray intensities from a standard material, after certain matrix corrections have been performed, results in a quantitative chemical analysis of the sample surface.

The scanning electron microscope (SEM) is another electron beam instrument which is chiefly used to examine surface morphology of a sample at high magnification.[3] If an energy dispersive X-ray analysis system is attached, this instrument is capable of doing qualitative chemical analyses. The energy dispersive analysis differs from the wavelength dispersive analysis of the microprobe in that X-ray energy is counted by employing a multi-channel analyzer of channel size ΔE. The number of counts in each channel of width ΔE indicates a relative abundance of the element from which X-rays of that particular energy have originated.

Nonconductive samples must be coated a layer with gold or gold-palladium to prevent charging effects from interfering with the examination of the surface. The conductive layer is usually only a few hundred angstroms thick (100 Å to 300 Å).

3. RESULTS

The results of the SEM and microprobe evaluations of the surfaces investigated indicate none of the mechanical polishing techniques used are really suitable for preparing a surface to accept a protective or anti-reflective coating. In short, foreign matter was found on all samples and numerous scratches persisted. Some preparation techniques were better than others obviously, and the figures in this report indicate the general quality of each surface.

Figures 1 to 5 are SEM micrographs of the surfaces of samples prepared as indicated in Table 1. The quality of each surface is reflected in these micrographs. Our observations have been that those samples with the best surface have been polished through the smallest sized polishing compounds. The SiC sample clearly has the roughest surface finish. Figures 6 to 8 are micrographs of foreign matter found on the surface of sample 5 (final polishing material was MgO). Even after polishing this sample through several stages and different materials, globs of Si or SiC still can be found adhering to the surface. Figures 9 and 10 show an energy dispersive analysis of one of these pieces of foreign matter, showing that, in fact, the material is Si or SiC. It should be noted that Si and SiC appear the same to the Edax system, which cannot detect elements below Na.

The results of other energy dispersive analyses on various samples are shown in the remaining figures. The amount of silicon (SiC) and aluminum (Al_2O_3) is low in most samples, but nevertheless there is evidence that these materials are

3. Thorton, P. R. (1968) Scanning Electron Microscopy, Chapman and Hall Ltd.

still present on the surface. Studies with the microprobe indicate weight percents varying from 0.04 to 23.25 percent of Si, depending on the area investigated.

Cracks, gouges, and areas near the edge of the sample retained polishing remnants the easiest. The aluminum concentration was much lower, varying from 0.02 to 0.66 weight percent for these samples. The largest amount of Mg detected was 2.36 percent by weight on sample 5. These numbers merely indicate the range of values observed with the microprobe. It is particularly apparent that large grit material, such as the SiC, may leave large pieces of material embedded in the surface. These foreign bodies will probably cause havoc when a coat. . . laid down on the surface. Not only will points of stress occur, but also adhesion, optical absorption, thermal conductivity, and other coating properties will be affected by the presence of this "foreign" element in the coating.

In summary, the SEM and electron probe analyses of the surfaces of these mechanically polished samples of single-crystal KCl indicate that mechanical polishing is probably not suitable as a final preparation technique for laser window materials. The general condition of the surface is not sufficiently good to accept coatings with desirable properties such as good optical absorption, thermal expansion, adhesion, etc. This is due to the excessive number of scratches, bumps, gouges, and remnants of polishing material which can still be found on the surfaces of the mechanically polished samples.

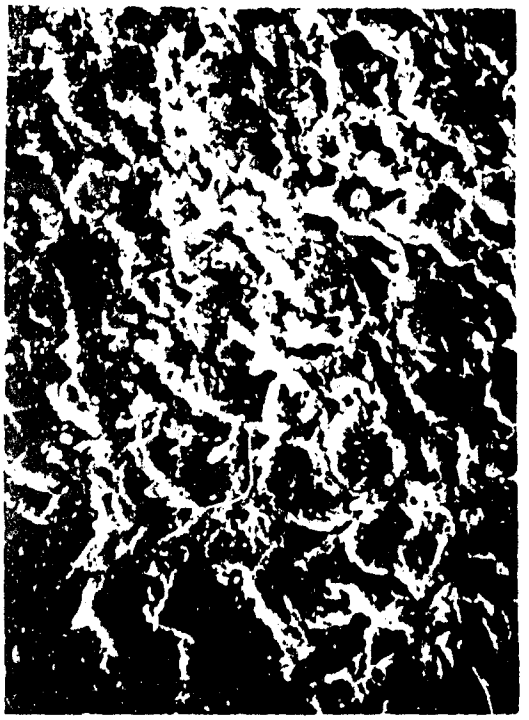


Figure 1. Sample No. 1: Final Polish - 600 Grit SiC - 800×

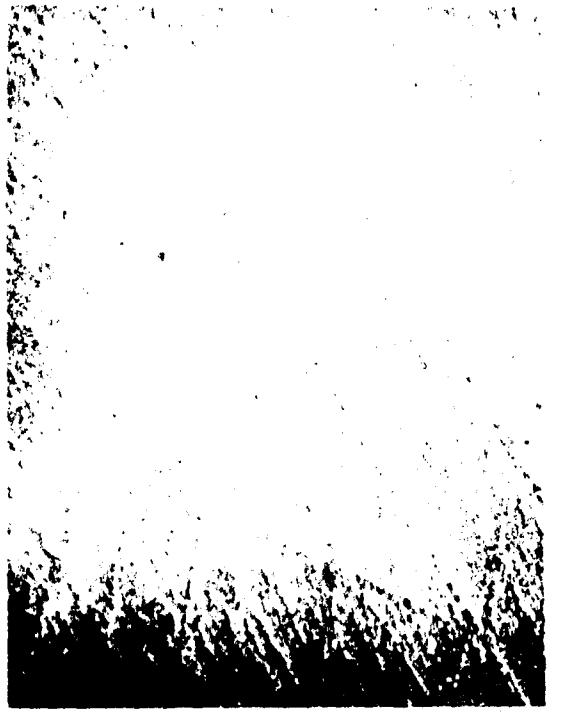


Figure 2. Sample No. 2: Final Polish - 1μ Al_2O_3 - 1680×

Figure 3. Sample No. 3: Final Polish - Linde A - 1680×

Figure 4. Sample No. 4: Final Polish - Linde B - 1664×

Figure 5. Sample No. 5: Final Polish - MgO - 1680×

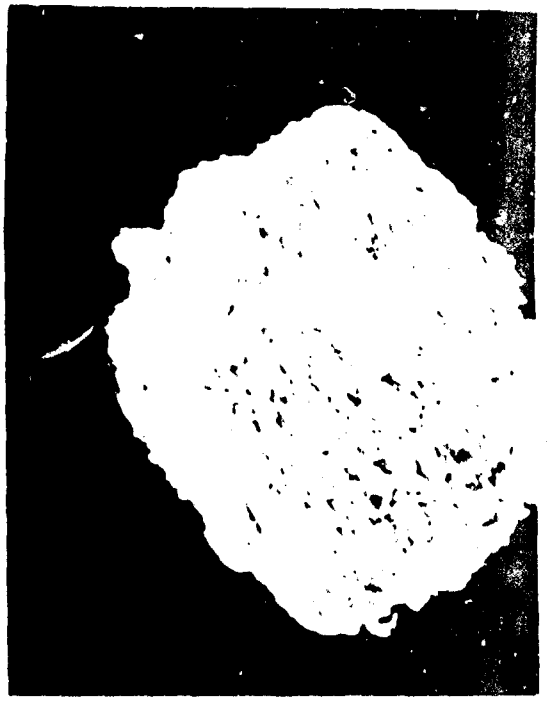


Figure 6. Foreign Matter ("A") on Surface of Sample No. 5 - 840×

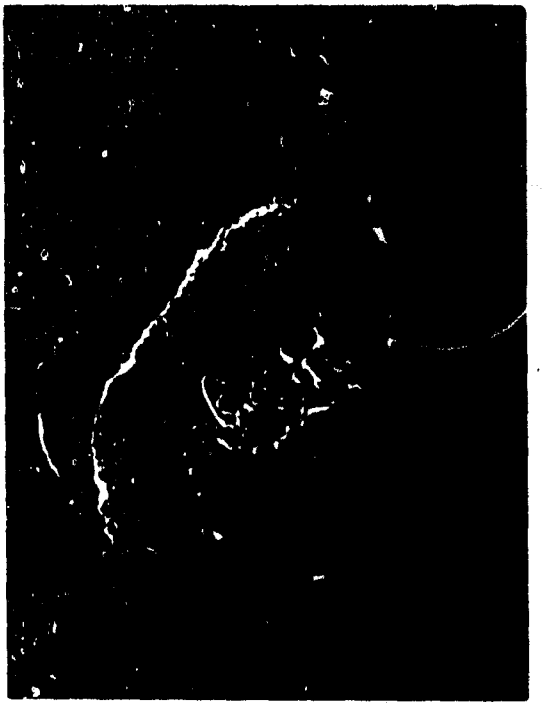


Figure 7. Foreign Matter ("B") on Surface of Sample No. 6 - 168×

Figure 8. Foreign Matter ("B") at 840×

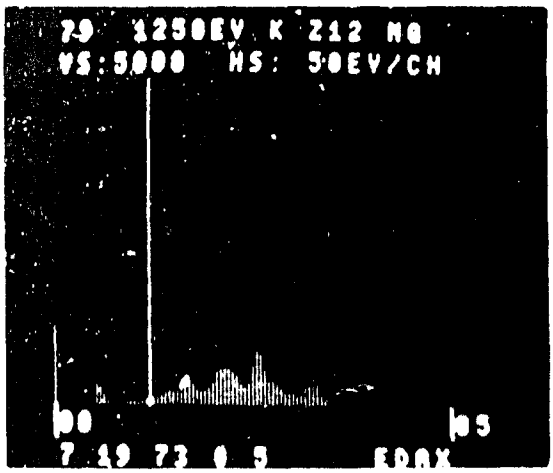


Figure 9. X-ray Analysis of Foreign Matter "B". No magnesium present

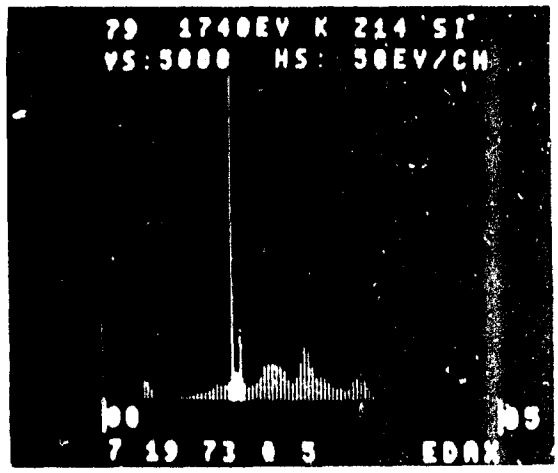
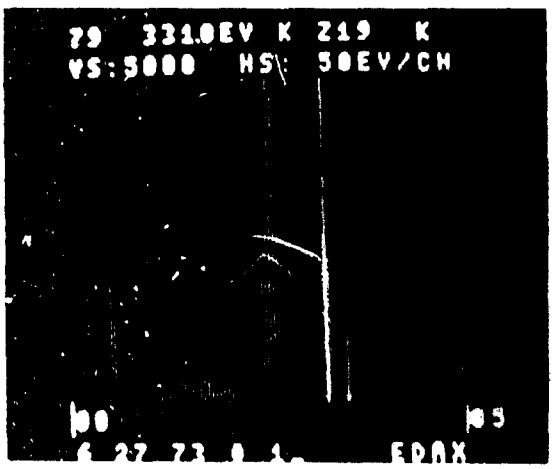


Figure 10. Silicon Lines Identified on Same Spectrum as Figure 9. Other peaks are Au, K, and Cl



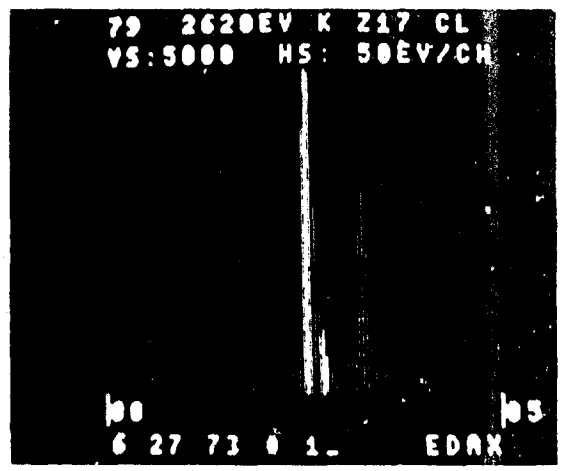
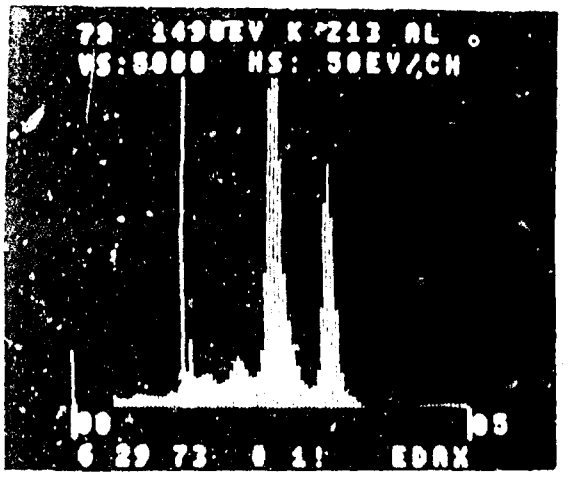
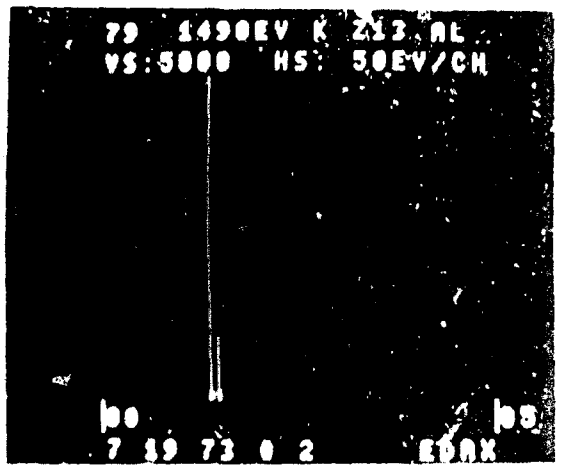
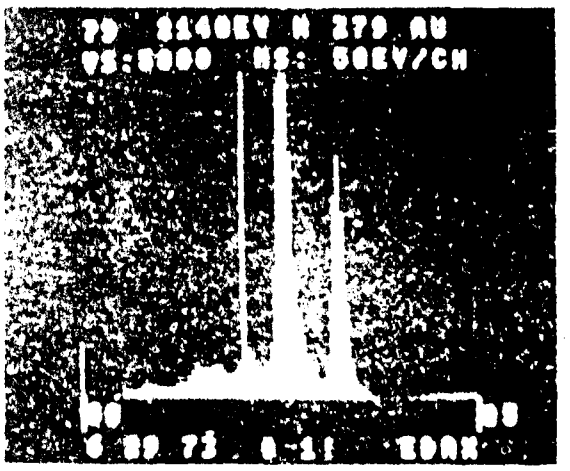


Figure 11. X-ray Spectra of Sample No. 1 with K and Cl Lines Identified







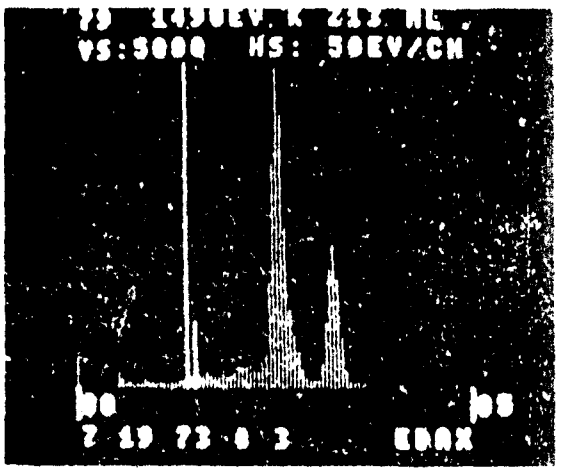
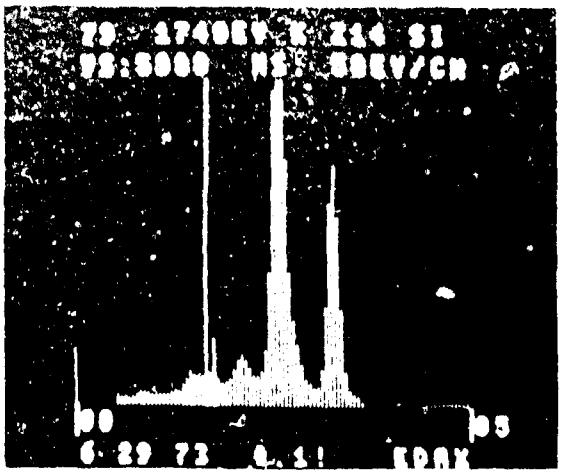


Figure 12. Additional Lines (Al, Au, Si) Identified on Spectrum for Sample No. 1



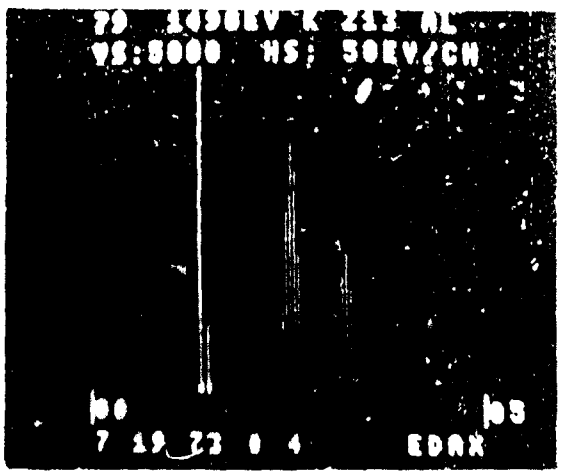
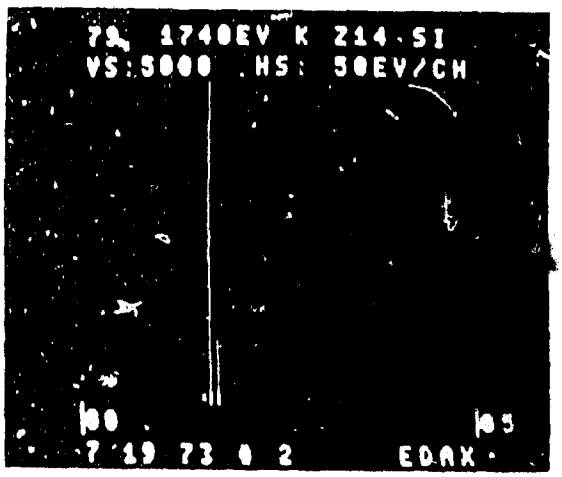
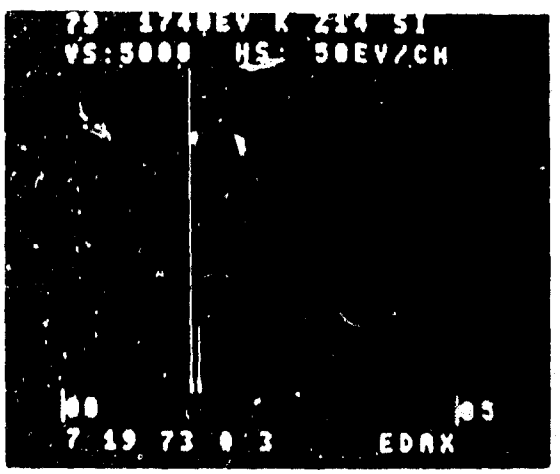


Figure 13. Al Lines Identified on Spectra for Samples No. 2, 3, and 4. Trace amounts of aluminum are present.





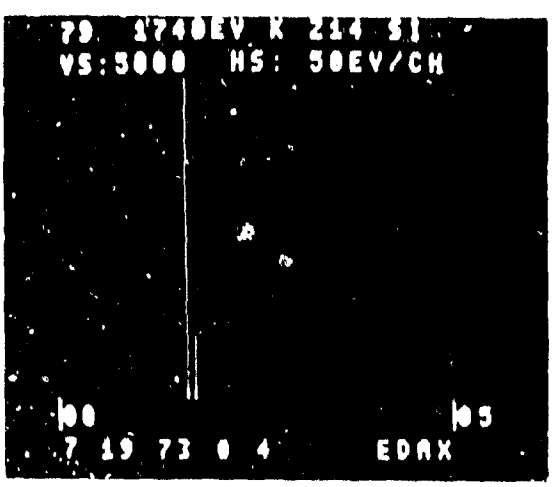
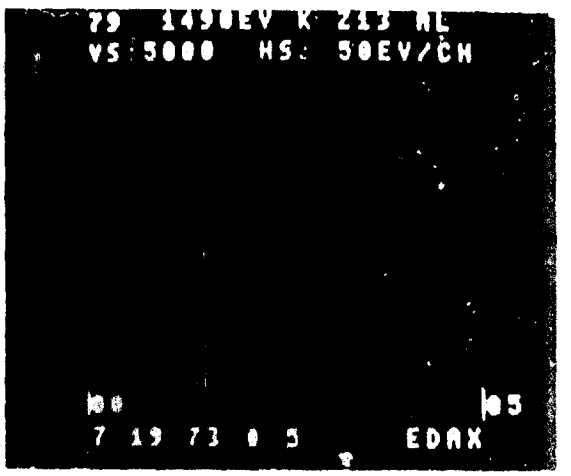


Figure 14. Si Lines Identified on Spectra for Samples No. 2, 3, and 4. Trace amounts of silicon are present



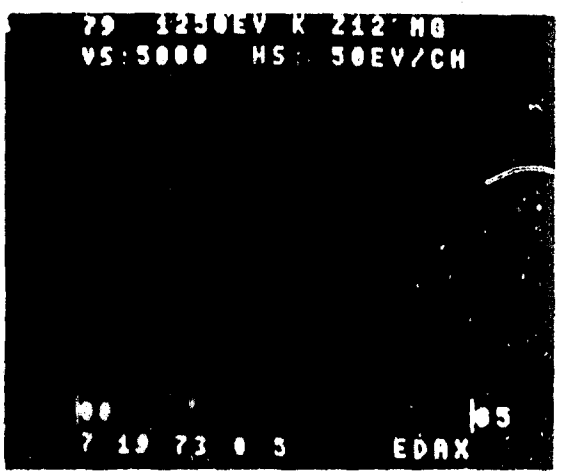


Figure 15. X-ray Analysis of Sample No. 5. Trace quantity of aluminum present and no magnesium