

Figure 33: Enlarged view of the area marked 2 in figure 31 shows coarse grains as compared to those shown in figure 32, X10.

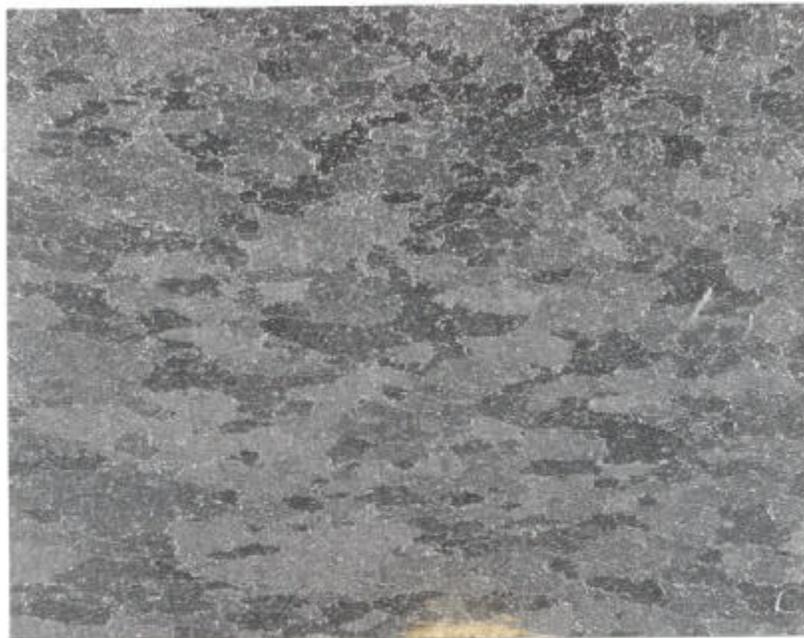
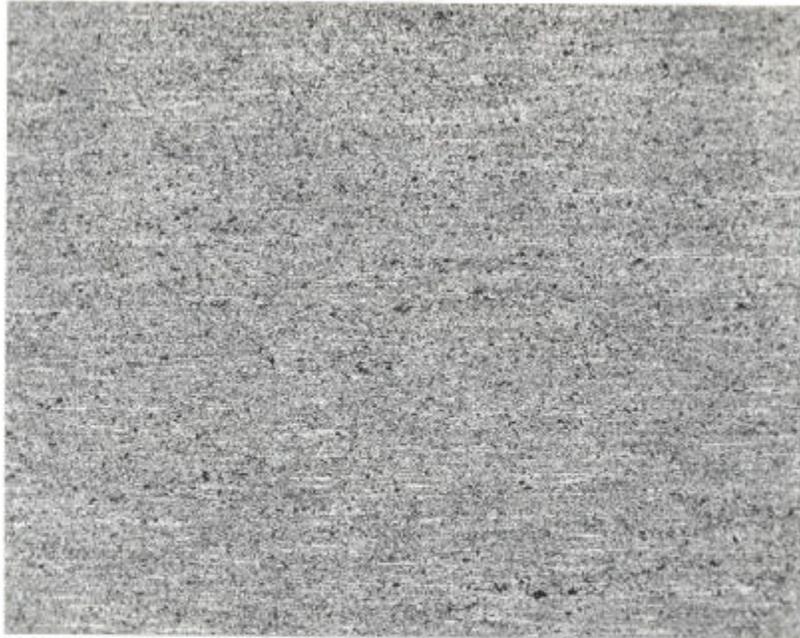
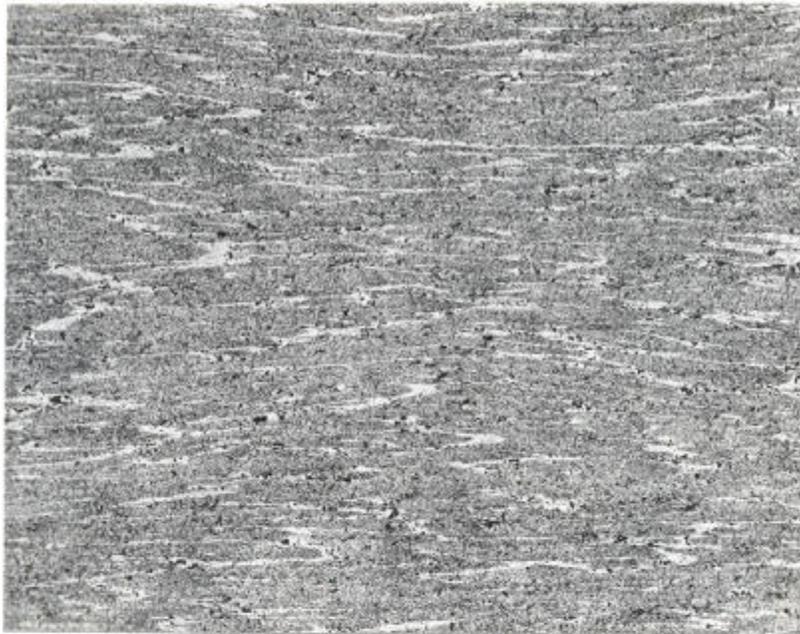


Figure 34: Enlarged view of the area marked 3 in figure 31 shows coarse grains as compared to those shown in figure 33, X10.

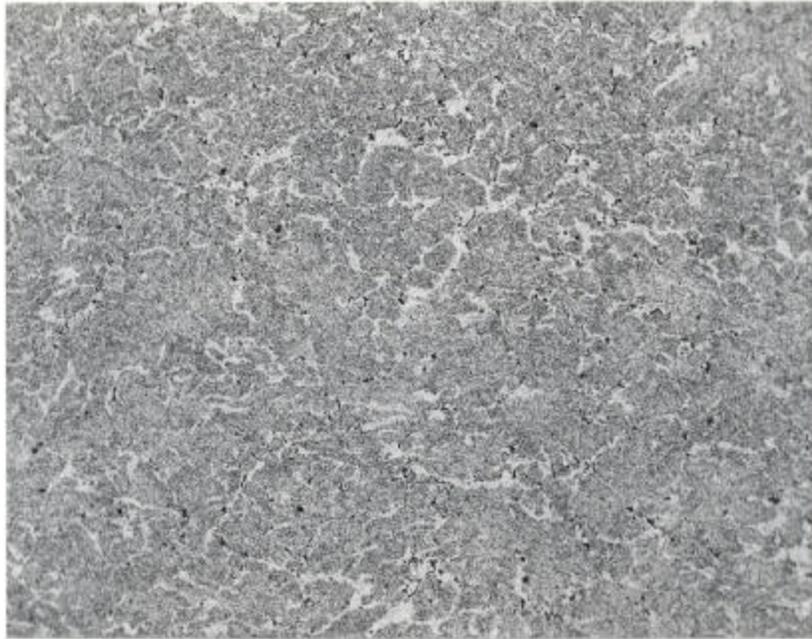


A

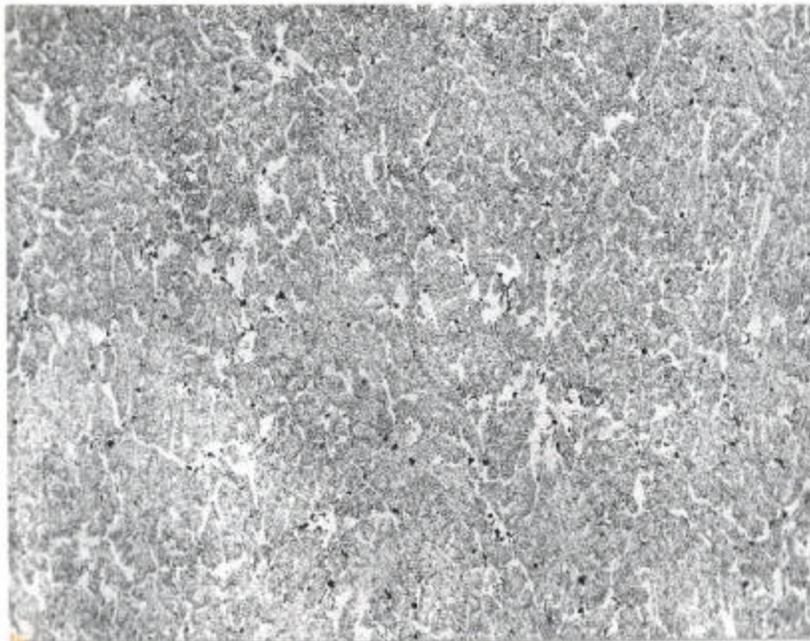


B

Figure 35: Photomicrographs showing microstructures near the inside surface (A) and near the outside surface (B). Extrusion flow lines are apparent. Grain size near the inside surface appears to be much finer as compared to that near the outside surface, X100.



A



B

Figure 36: Photomicrographs showing microstructures near the inside surface (A) and near the outside surface (B). No extrusion flow lines are present. They exhibit extensive grain growth in the area. Some precipitate particles can be seen along grain boundaries and inside grains, X100.

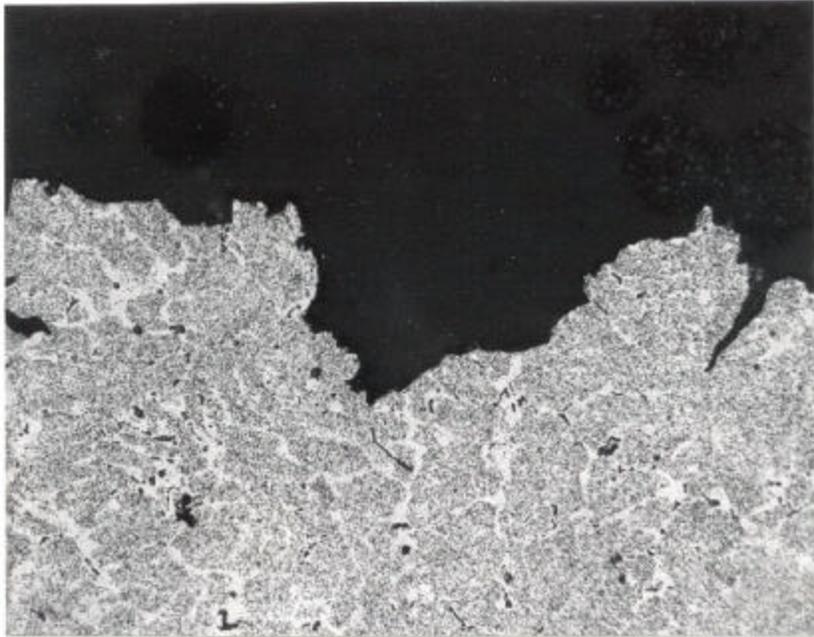


Figure 37: Photomicrographs showing the microstructure along the main crack front. It indicates a mixed mode fracture consisting of intergranular and transgranular failure, X200.

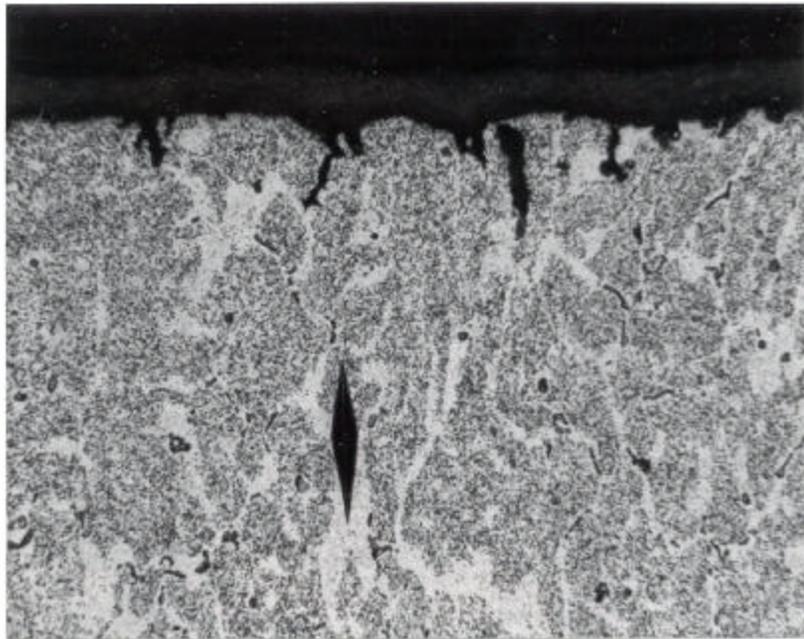


Figure 38: Photomicrograph exhibits a Knoop microhardness indentation showing its half diagonal located in the white region larger than the other half in the dark region, X300.

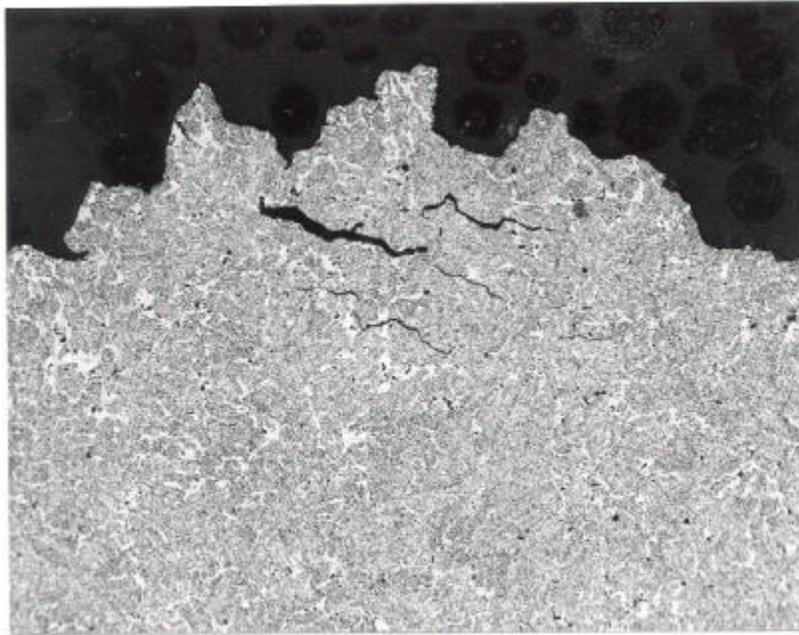


Figure 39: Photomicrograph shows internal cracks running parallel to the main crack front, X100.



Figure 40A: SEM micrograph exhibits the spot on a precipitate particle which was analyzed using EDS, X3390.

EG&G Ortec System 5000  
Spectrum Plotting Program  
Printplot V02.05

Sample ID: J9509.002;Mount #8150-Particle

Energy Range: 0 - 20 keV 10 eV/ch Hi Res

Preset: Off

Real Time: 388.37 Sec. Live Time: 50.08 Sec.

88% Deadtime 44373 Counts/Second

Cfs 4K

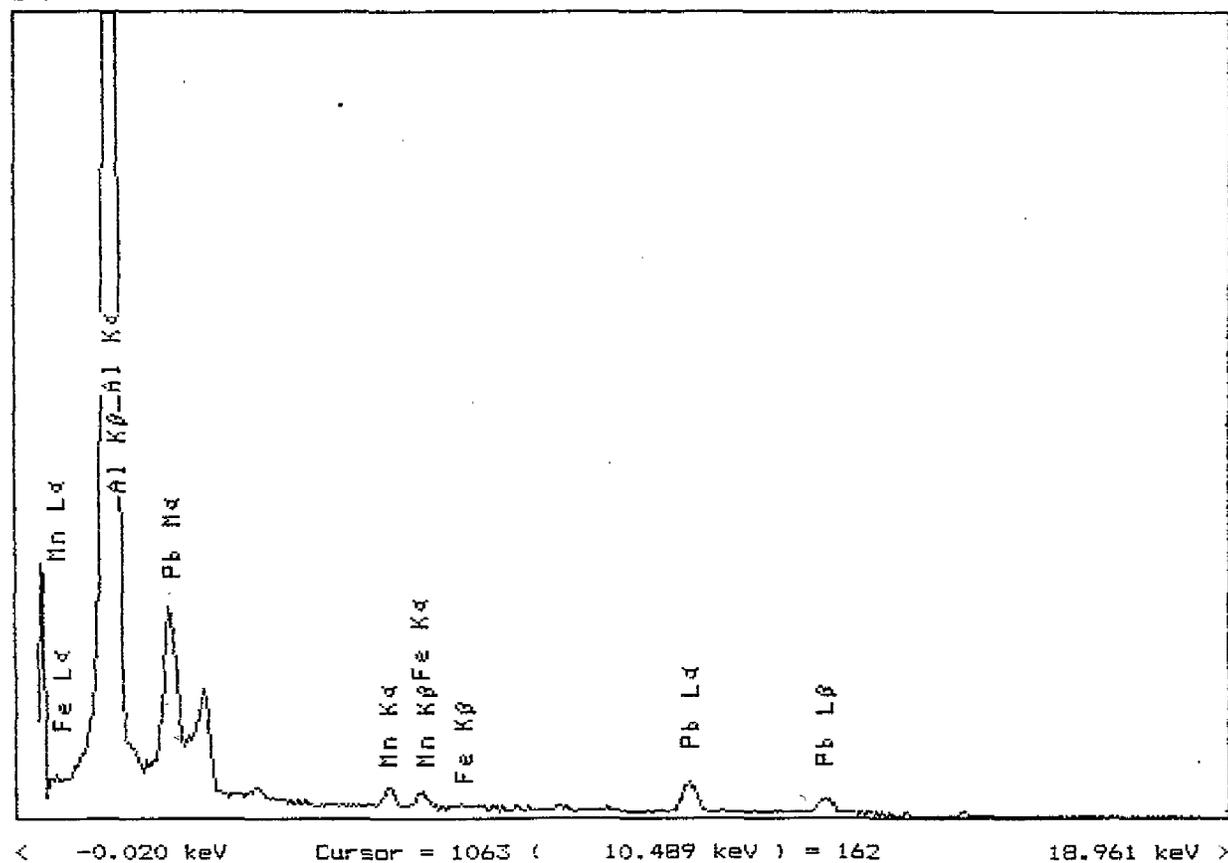


Figure 40B: EDS spectrum of the spot shown in figure 40A indicates the presence of Pb, Mn, and Fe.

EG&G Ortec System 5000  
Spectrum Plotting Program  
Printplot V02.05

Sample ID: J9509.002; Mount #8150-Particle

Energy Range: 0 - 20 keV 10 eV/ch Hi Res

Preset: Off

Real Time: 345.28 Sec. Live Time: 50.10 Sec.

85% Deadtime 44623 Counts/Second

Cfs 4K

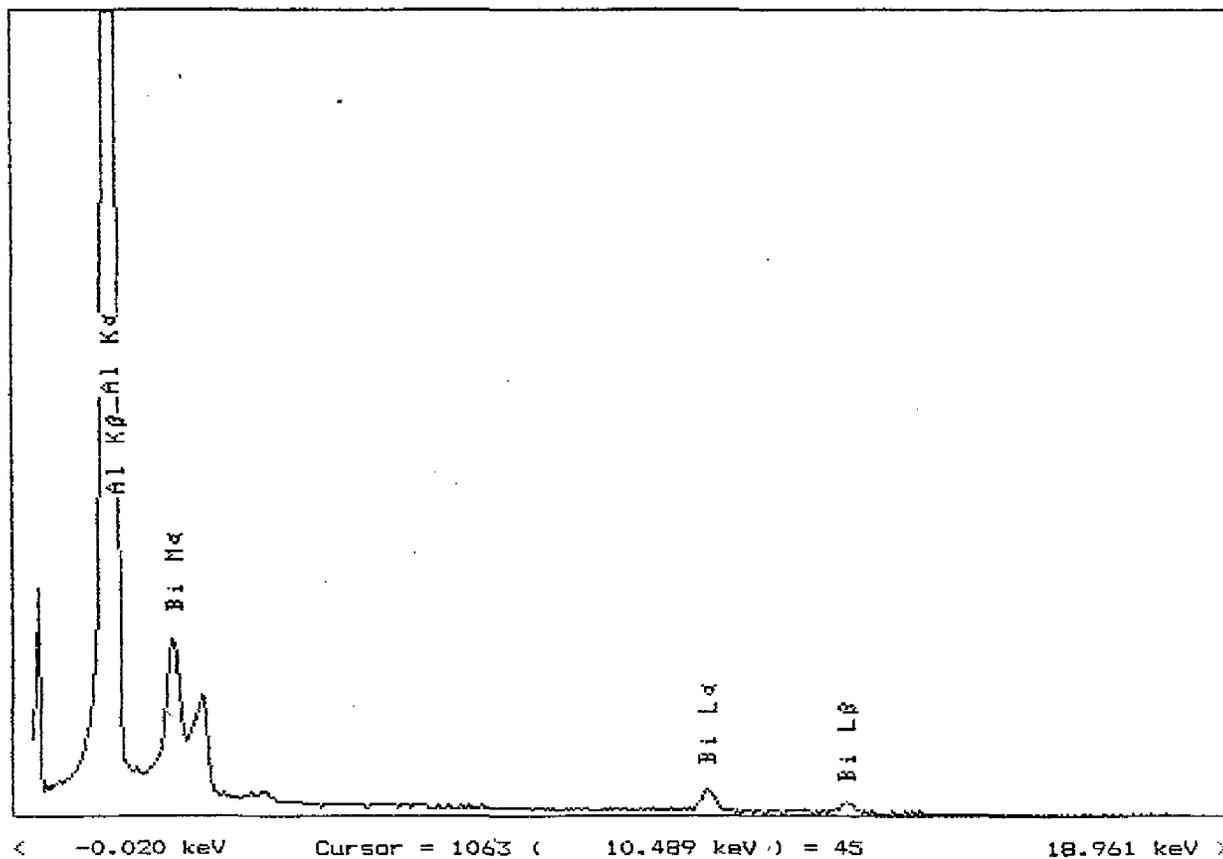


Figure 40C: EDS analysis of another spot on the same mount as in figure 40A detected appreciable presence of Bi.



Figure 41A: SEM micrograph showing the spot on a precipitate particle which was analyzed using EDS, X5850.

EG&G Ortec System 5000  
Spectrum Plotting Program  
Printplot V02.05

Sample ID: J9509.002;Mount #8152-Particle

Energy Range: 0 - 20 keV 10 eV/ch Hi Res

Preset: Off

Real Time: 286.00 Sec. Live Time: 50.12 Sec.

83% Deadtime 43068 Counts/Second

Cfs 4K

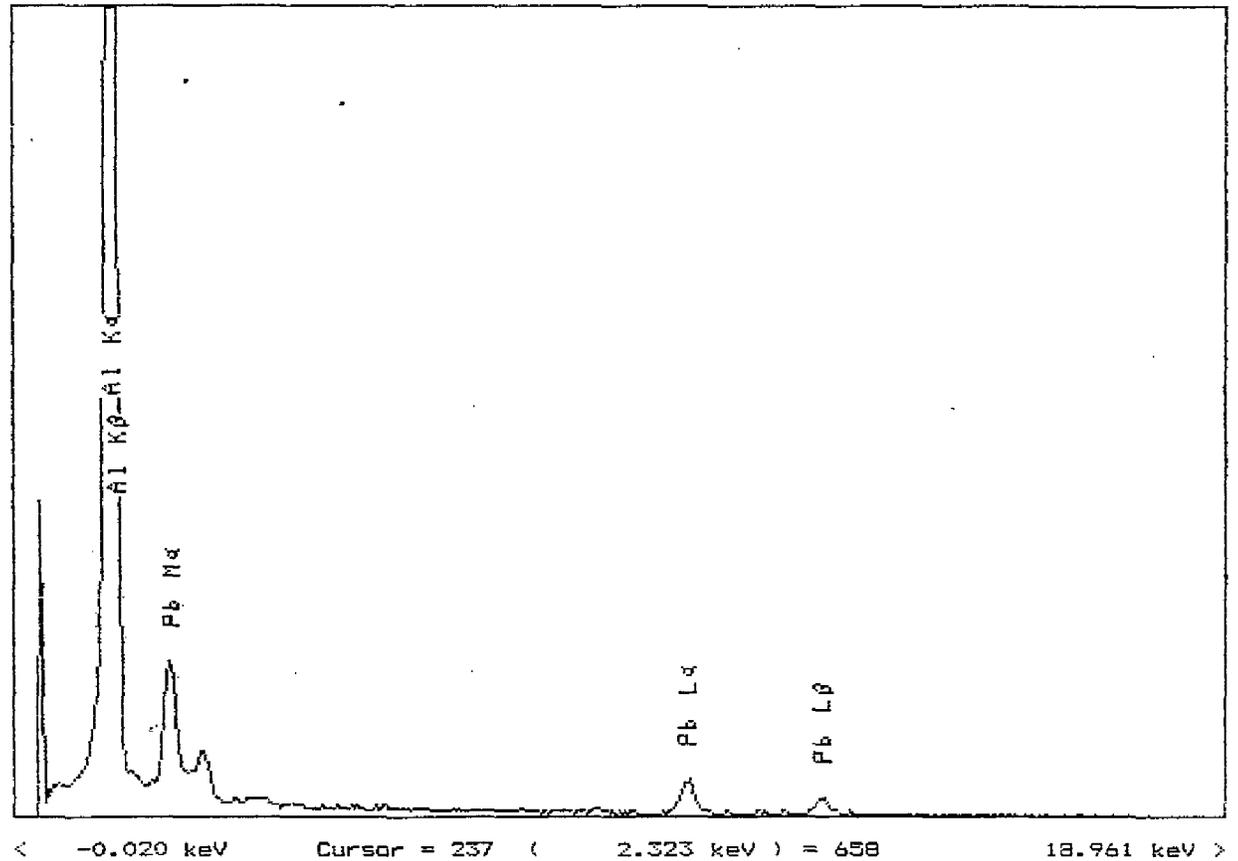


Figure 41B: EDS spectrum of the spot shown in figure 41A shows the presence of Pb.

Appendix I



Appendix II

## Lead Analysis Procedures

ARTECH uses a Buck Scientific Model 200 A Atomic Absorption Spectrometer (AAS) with flame aspiration for the analysis of lead in metals. The metal is digested using standard procedures (nitric acid being the most common digestion to avoid the low solubility of lead chloride). Standard solutions of lead in a matrix matched solvent (use of acid at the same concentration as the metal digestion to eliminate effects from variations in the viscosity of the solutions) are made from NIST traceable standard solution concentrates. The typical standards are 5, 10 and 20 parts per million in solution.

The procedure used for the analysis is as specified in EPA 7420. A lead hollow cathode lamp is used as the light source. An acetylene-air flame is used. The 283.3 nm line is used. Calibration by "Methods of Addition" is used for any sample which has a possible interference due to materials in the sample.

Chemical interferences with lead can include the presence of chromate, which causes a precipitation of lead chromate, the presence of sulfide which precipitates lead sulfide, and the presence of high levels of chloride which precipitates lead chloride. Any precipitation of a metal in a digested sample removes that metal from the solution and thus from the analysis.

## Bismuth Analysis

ARTECH uses a Buck Scientific Model 200 A Atomic Absorption Spectrometer (AAS) with flame aspiration for the analysis of bismuth in metals. The metal is digested using standard procedures (nitric acid being the most common digestion to avoid the low solubilities of some metal chlorides). Standard solutions of bismuth in a matrix matched solvent (use of acid at the same concentration as the metal digestion to eliminate effects from variations in the viscosity of the solutions) are made from NIST traceable standard solution concentrates. The typical standards are 5, 10 and 20 parts per million in solution.

The procedure used for the analysis is as specified in the Perkin-Elmer "Cookbook", method Bi 1. A bismuth hollow cathode lamp is used as the light source. An acetylene-air flame is used. The 223.1 nm line is used. Calibration by "Methods of Addition" is used for any sample which has a possible interference due to materials in the sample.

## Tin Analysis Procedures

ARTECH uses a Buck Scientific Model 200 A Atomic Absorption Spectrometer (AAS) with flame aspiration for the analysis of tin in metals. The metal is digested in an appropriate acid, typically nitric acid. Standards of 20, 50, and 100 parts per million are prepared from NIST traceable concentrates. The flame is set with a fuel rich mixture to give maximum absorption at a known tin concentration in the standard. If the amount of tin in the sample is below 20 parts per million in solution, the sample is spiked with a known amount of tin and the reading corrected for this spike.

The procedures used for tin are from the Perkin-Elmer "Cookbook, Method Sn 1. A tin hollow cathode lamp at 12.0 mA, a slit of 2A, and a wavelength of 2863A is used for the analysis.

#### Specific Procedures, Aluminum Cylinder Analysis

A one gram sample of metal turnings, removed from the core of the sample and solvent washed to remove traces of cutting oil and fingerprints, was dissolved using 25 mL of water and 10 mL of 3N Nitric acid. The resulting solution was taken to 100 mL total volume with deionized water. This solution was analyzed using the standard AAS procedures for lead, bismuth, and tin. The samples were found to be comparable in absorption to the 5 ppm lead standard solution (for lead), comparable to the 5 ppm bismuth standard (for bismuth) and were found to be slightly less than the 20 ppm tin standard (for tin). The expected precision of these measurements are 5% of value.