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STUDY AND ANALYSIS OF THE FAILED USB DEVICES USED IN COPPER MINE USING ENVIRONMENTAL SCANNING ELECTRON MICROSCOPY AND X-RAY PHOTOELECTRON SPECTROSCOPY

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Abstract - *The article throws a spotlight on the study & analysis of the failed USB devices which was used to store the important parameters such as pH and ion concentration at hourly intervals from a copper mine. These USB devices had failed due to immersion from flooding of acid mine. An outflow of acidic water from a mining site is referred to as Acid Mine Drainage (AMD). The study was done using Environmental Scanning Electron Microscopy (ESEM) in which the electron beam interacts with the surface of the sample to produce images and the analysis was done using Energy Dispersive Spectroscopy (EDS), which can be referred to as one of the important applications of SEM. Further, X-ray Photoelectron Spectroscopy (XPS) was used because it gives the valuable quantitative information from the surface of the sample. High levels of lead and oxygen were found in the USB device using EDS. Iron and copper sulphides were found as the major compounds from the XPS. Alternates to these devices were also found.*

Key Words*: USB device, *Environmental scanning electron microscopy, Energy dispersive spectroscopy, Scanning electron microscopy, X-ray photoelectron spectroscopy, Acid mine drainage

1. INTRODUCTION

USB devices which were used to store the parameters such as pH, ion concentration and other parameters from the sulphide-based copper ore are the samples which will be analyzed. The samples have failed because of the flooding of mine. Overflowing of acidic water from a mining site is referred to as Acid Mine Drainage and therefore, AMD is responsible for the failure of the USB device. Due to this failure, the data stored on the USB stick has also vanished. This lost data can bring the company under huge financial loss. Figure 1 shows the two USB sticks ready to be analyzed using ESEM, EDS and XPS. Green and Blue PCB's are the USB sticks and the two black chips near Green PCB has been fallen somewhere from Green and Blue PCB.

We are using ESEM to analyze our samples because they are insulating in nature and these types of specimen can be analyzed using ESEM without destruction and additional sample preparation procedures. Further, Energy Dispersive Spectroscopy (EDS) will help us in elemental analysis of the failed USB devices. Scanning Electron Microscopy (SEM) could also have been used but that would have conducted the electric current in our sample because it directly uses electron beam for imaging.

Fig -1: Failed USB Devices

We are using X-ray Photoelectron Spectroscopy (XPS) because it is a surface analysis technique which provides valuable quantitative and chemical state information from the surface of the sample. This analysis will also help us identify why the rugged and reliable USB device is important and why it should be used in these mines to store the data instead of normal USB devices.

2. MATERIALS & METHODS

2.1 Environmental Scanning Electron Microscopy (ESEM) & Energy Dispersive Spectroscopy (EDS)

Environmental Scanning Electron Microscopy [1] (ESEM) can be used for examining uncoated biological and industrial materials with an electron beam in a high chamber pressure atmosphere of water vapor. In ESEM, primary electrons are emitted from the gun and they eject secondary electrons from the sample surface. These ejected electrons from the sample accelerate towards the detector. Collisions between the electrons and gas molecules liberate more free electrons.

Positive ions of water vapor neutralize excess electron charge over the sample and controlled pressure reduces surface charging of the specimen. ESEM helps in producing back scattered electron images which helps further in the Energy Dispersive Spectroscopy (EDS).

Figure 2 shows a back scattered electron image formed in ESEM from Green PCB where as figure 3 is obtained from the Blue PCB. Figure 4 shows a back scattered electron image formed in ESEM from the resistor which have been fallen somewhere from the Green & Blue PCB’s. Results from the images have been described under the results section. All the images obtained from ESEM are high-resolution images.

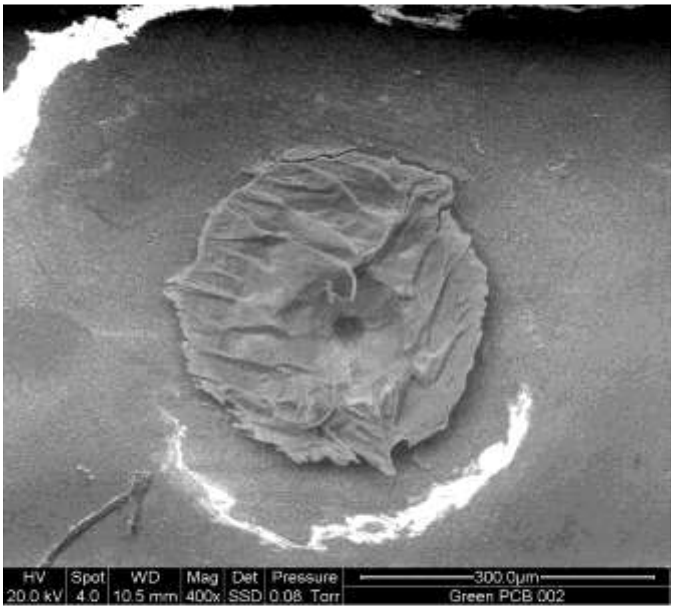

Fig -2: Back scattered electron image of part of Green PCB formed using ESEM

Fig -3: Back scattered electron image of part of Blue PCB formed using ESEM

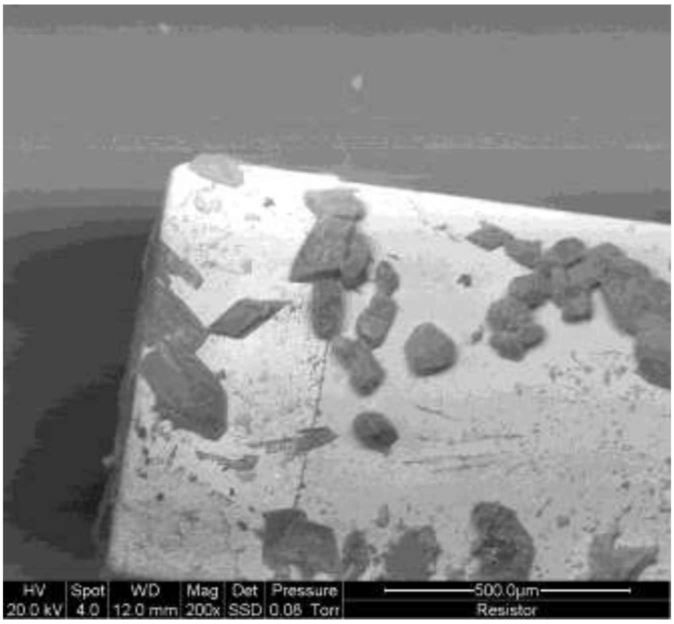

Fig -4: Back scattered electron image of part of Blue PCB formed using ESEM

Energy Dispersive Spectroscopy (EDS) [2] is a chemical microanalysis technique which is used with ESEM for elemental analysis or chemical characterization of a sample. An EDS detector is used to separate the characteristic x-rays of different elements into an energy spectrum and EDS system software is used to analyze the energy spectrum to determine the abundance of specific elements.

2.2 X-ray Photoelectron Spectroscopy

XPS [3] is the most widely used surface analysis technique. XPS provides valuable quantitative and chemical state information from the surface of the material being studied and this data is further used in industrial and research applications where surface plays a critical role in performance.

XPS is typically accomplished by exciting a surface with mono-energetic x-rays causing photoelectrons to be emitted from the sample surface. From the binding energy and intensity of a photoelectron peak, the elemental identity, chemical state and quantity of a detected element can be determined.

3. RESULTS & DISCUSSION

3.1 Environmental Scanning Electron Microscopy (ESEM) & Energy Dispersive Spectroscopy (EDS) Analysis

Figure 2, 3 & 4 shows the back scattered electron image of the Blue & Green PCB and resistor in ESEM. Bright color in the images represent the presence of higher atomic mass where as the darker regions are due to the presence of lower atomic mass. Further, the EDS was done for figure 5 and 6 which gave us the chemical characterization and elemental analysis of the PCB.

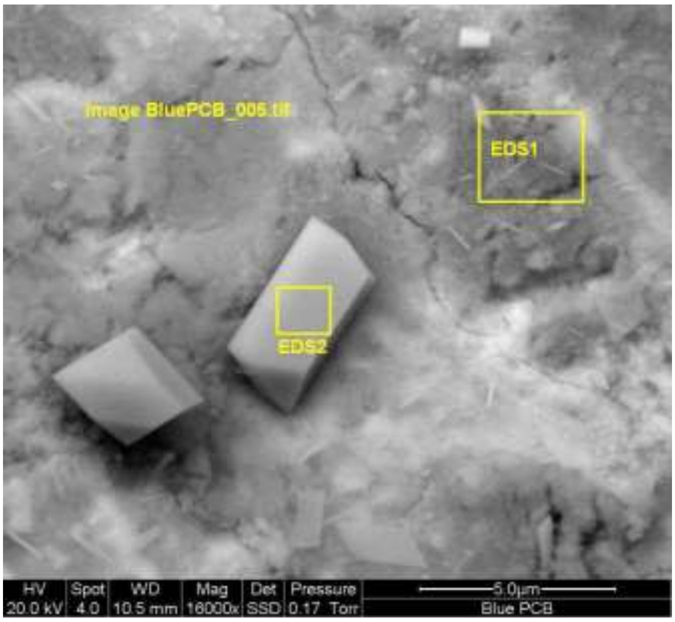

Fig -5: Back scattered electron image of part of Blue PCB formed using ESEM

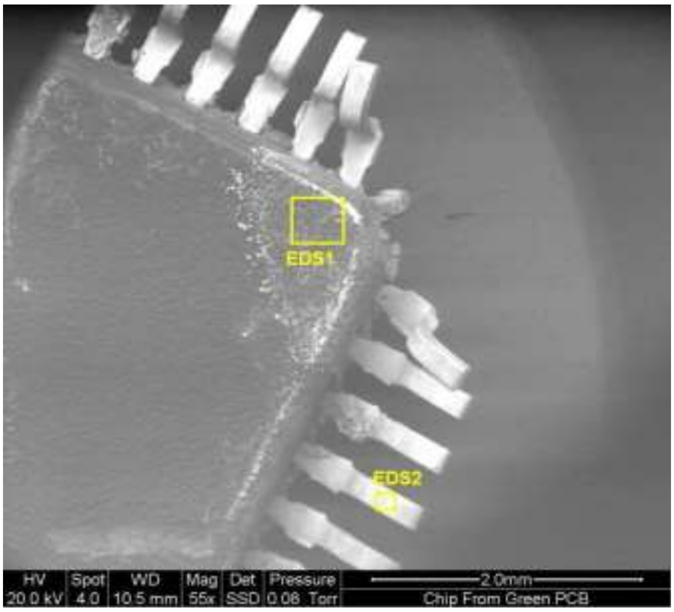

Fig -6: Back scattered electron image of part of Green PCB formed using ESEM

The highlighted areas from the figure 5 & 6 are the areas from where the EDS data was obtained. For figure 5, the EDS data is represented by figure 7 & 8 whereas the EDS data is represented by figures 9 & 10 for back scattered electron image obtained using ESEM in figure 6.

From figure 7 & 8, it can be observed that high amounts of compounds like lead & Sulphur whereas the small amounts of carbon, copper & oxygen were observed. Higher amount of tin was observed on the surface whereas the amount of tin observed on the button like structure was found to be in very small amounts.

From figure 9, it is observed that the higher amounts of silicon are present but the traces of other compounds like Sulphur, nickel and tin were also found. Also, very high amounts of carbon and oxygen were also found in the EDS spectrum.

Figure 10 confirms the presence of higher amounts of tin whereas other elements are also found such as copper, silicon, lead & nickel. Calcium and oxygen were discovered in higher amounts.

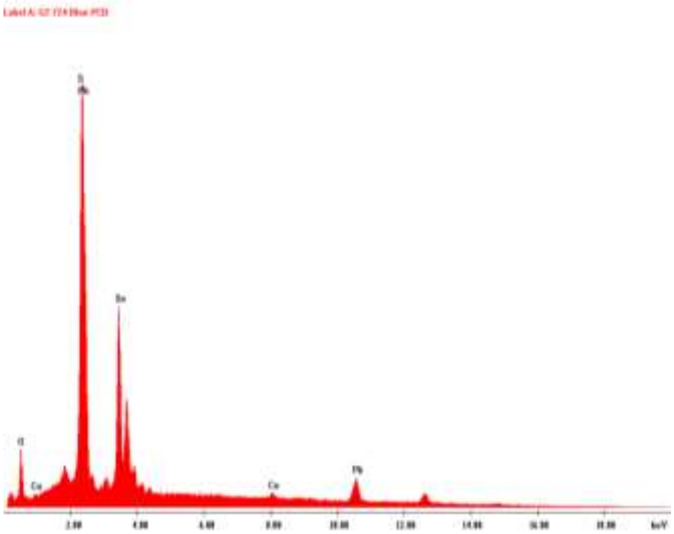

Fig -7: EDS 1 spectrum from figure 5

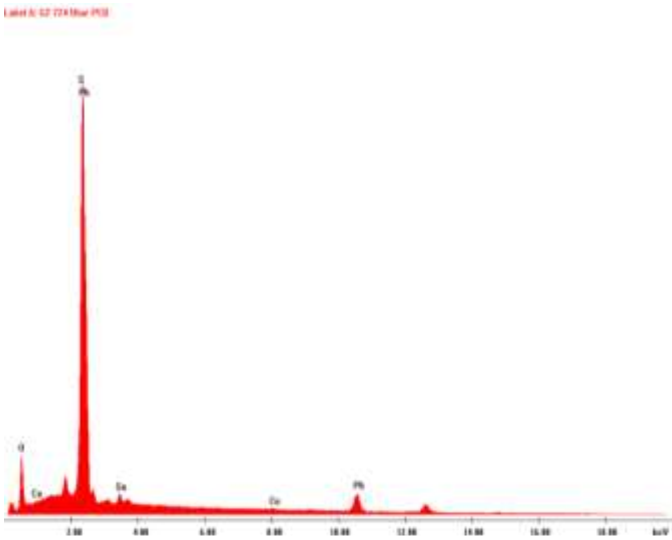

Fig -8: EDS 2 spectrum from figure 5

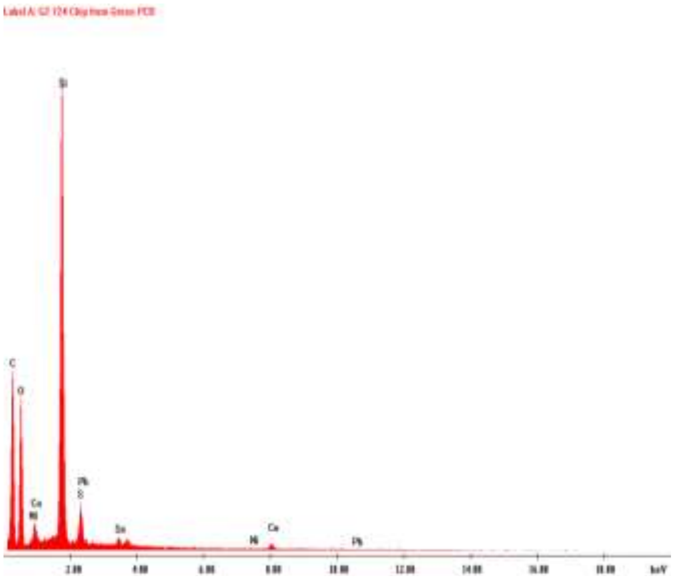

Fig -9: EDS 1 spectrum from figure 6

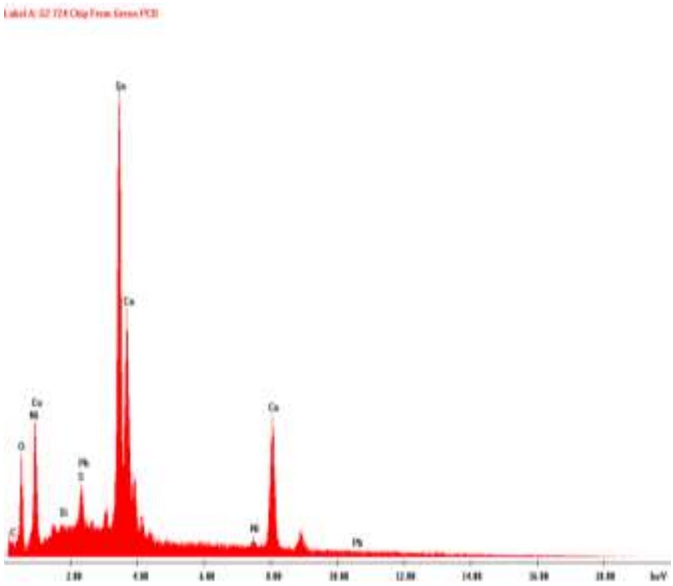

Fig -10: EDS 2 spectrum from figure 6

Therefore, from the ESEM and EDS images it can be concluded that these USB devices were damaged due to the exposure to highly acidic environment as found in the Sulphur based copper ore mine.

3.2 X-ray Photoelectron Spectroscopy Analysis

Kratos Axis Ultra DLD was the instrument used to obtain the XPS images. Al Ka monochromatic beam was operated at 150 W power and the hybrid magnetic and electrostatic lens was used. Survey scans were done at pass energy of 160 eV with step size of 1 eV for 180 seconds. Core level scans were done at pass energy of 20 eV with step size of 0.1 eV for 60 seconds except for carbon and Sulphur.

Casa XPS [4] is the software which is used for the analysis of the XPS. Figures 10 & 11 shows the Figure 10 shows the wide scan of XPS for the Blue PCB and figure 11 represents the wide scan of XPS for the Green PCB.

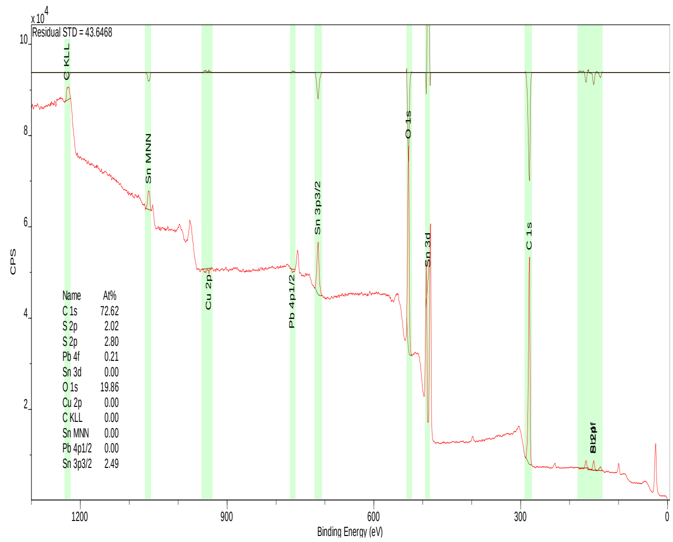

Fig -11: Wide scan of XPS for the part of Blue PCB

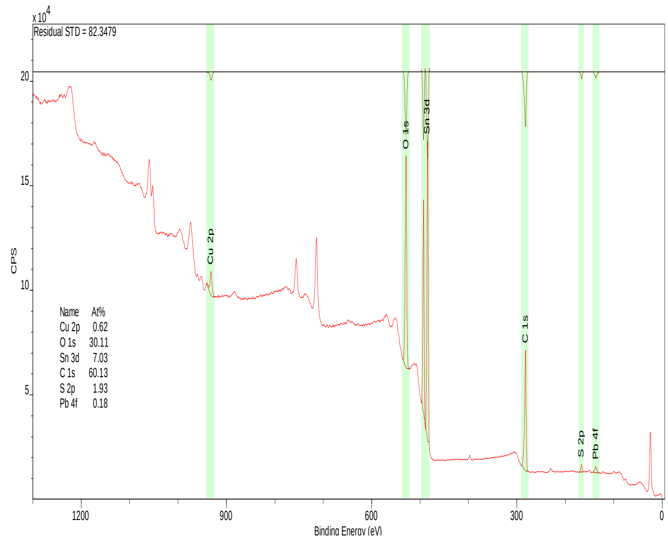

Fig -12: Wide scan of XPS for the part of Green PCB

Table 1 shows the composition of elements present in Blue PCB which is obtained from figure 11. Table 2 shows the composition of elements present in Green PCB which is obtained from figure 12. Figure 13 shows the peak fitting of carbon done with the help of Casa XPS software.

Table -1: Composition of elements in Blue PCB from wide scan of XPS

Name of the Element	Atomic % of the Element
Carbon (C)	72.62
Sulphur (S)	4.82
Lead (Pb)	0.21
Oxygen (O)	19.86
Tin (Sn)	2.49

Table -2: Composition of elements in Green PCB from wide scan of XPS

Name of the Element	Atomic % of the Element
Copper (Cu)	0.62
Oxygen (O)	30.11
Lead (Pb)	0.18
Carbon (C)	60.13
Sulphur (S)	1.93
Tin (Sn)	7.03

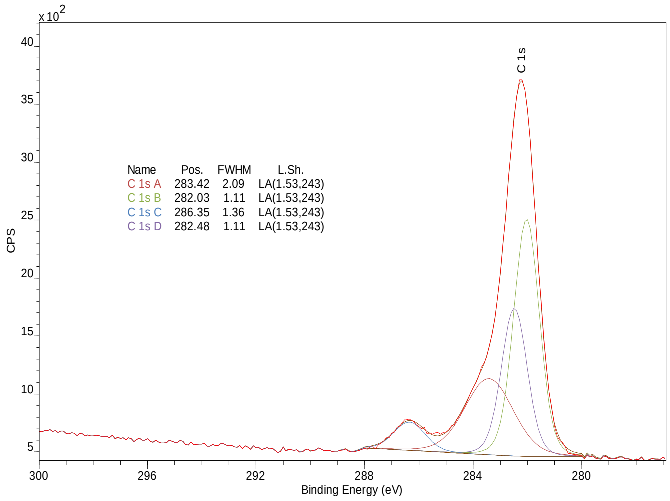

Fig -13: Peak fitting of carbon in XPS using Casa XPS Software

XPS also confirms the presence of copper sulphide(CuS), iron sulphide(FeS) and lead sulphide (PbS). This copper sulphide and lead sulphide further reacts with oxygen and water to form sulphuric acid. Sulphur on reacting with oxygen formed sulphates(SO_4) which were also found during XPS scanning. Thus, causing the failure of the device. Some other elements may also be present which are responsible for failure of these data storage devices.

4. CONCLUSIONS

Environmental Scanning Electron Microscopy is a high-resolution imaging technique which is used to study the composition, topography and surface structure of the sample. PCB used here has amorphous structure. With the help of ESEM, the spectrum for EDS were obtained which depicts the amounts of elements present in the USB devices.

XPS gives the quantitative information for the elements with chemical composition. From XPS, it was confirmed that the low pH and high metal content compounds like copper sulphide, iron sulphide, lead sulphide & sulphates were solely responsible for the failure of the two USB devices.

From the EDS data, the presence of various types of compounds was found which reacted with oxygen and carbon to form sulphides and other toxic compounds. Different sulphide and ferrite was confirmed by the scans of XPS. These compounds were also responsible for the acid mine drainage.

Therefore, it can be concluded that more rugged and reliable devices should be used which should not react in highly acidic regions for storing data in a copper mine. Rugged devices are capable for the efficient working in the acidic environment. Hence, this will help us prevent the loss of data and will prevent companies from financial losses.

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BIOGRAPHIES

Patience, attitude and discipline are the traits that completely define. Hardwork and enthusiasm to work is my nature. Exploring and working on new things and gifting them to the world in the form of writing is my passion.