Application Note

Using AES, EDS, and FIB to Detect, Identify, and Image Buried Metallic Particles

Overview

Chemical information about buried particles can be very useful in helping to determine important parameters such as the origin of defects, corrosion mechanisms, coating problems, etc. Auger Electron Spectroscopy (AES) is a powerful analytical tool that provides quantitative elemental information from surfaces of solid materials, and when combined with other bulk analysis techniques can greatly assist in identifying buried particles. This type of analysis would be challenging, if not impossible, with any one technique alone. Specifically, AES in conjunction with a focused ion beam (FIB) can produce site specific imaging of microscale features beneath a sample surface. The combination of the two techniques allows for high spatial resolution analysis of buried particles and defects. Energy dispersive x-ray spectroscopy (EDS) is a complementary technique to Auger analysis as it provides information from much deeper below the sample surface (tenths of a µm to a few µm). With the combination of AES, FIB, and EDS, buried metal particles can first be located and characterized with EDS, followed by the subsequent FIB milling and high spatial resolution Auger spectroscopy and imaging of the particles.

Sample Measurements

Defect sites present on a Ti passivated Al sheet sample were identified and analyzed using a PHI *710* Scanning Auger Nanoprobe equipped with a 25kV Schottky field emission electron gun and a coaxial cylindrical mirror analyzer (CMA). The Auger instrument was equipped with a PHI FIB ion gun and a windowless EDS detector. Secondary electron images, Auger spectra and Auger maps were acquired with a 20 kV electron beam that provided a 10 nA incident beam current and a probe diameter of 10 nm.

Results

A defect region was observed in the SEM image and localized Fe and Si were detected in the EDS maps of the particle (Figure 1). Based on the EDS maps indicating the location of the FeSi particle, Auger spectroscopy was used to obtain a more surface sensitive spectrum of the particle region. Contrary to the EDS data, no Fe or Si were detected by AES on the particle surface (Figure 2), indicating that the FeSi particle is buried below the surface and deeper than the Auger depth of analysis (5-10 nm). A FIB cut was then made to investigate the chemistry of the buried particle in cross section using AES (Figure 3). The AES Fe map of the exposed FIB face shows an elevated Fe signal within the particle and shows that the particle is buried well beneath the sample coating and within the Al substrate.

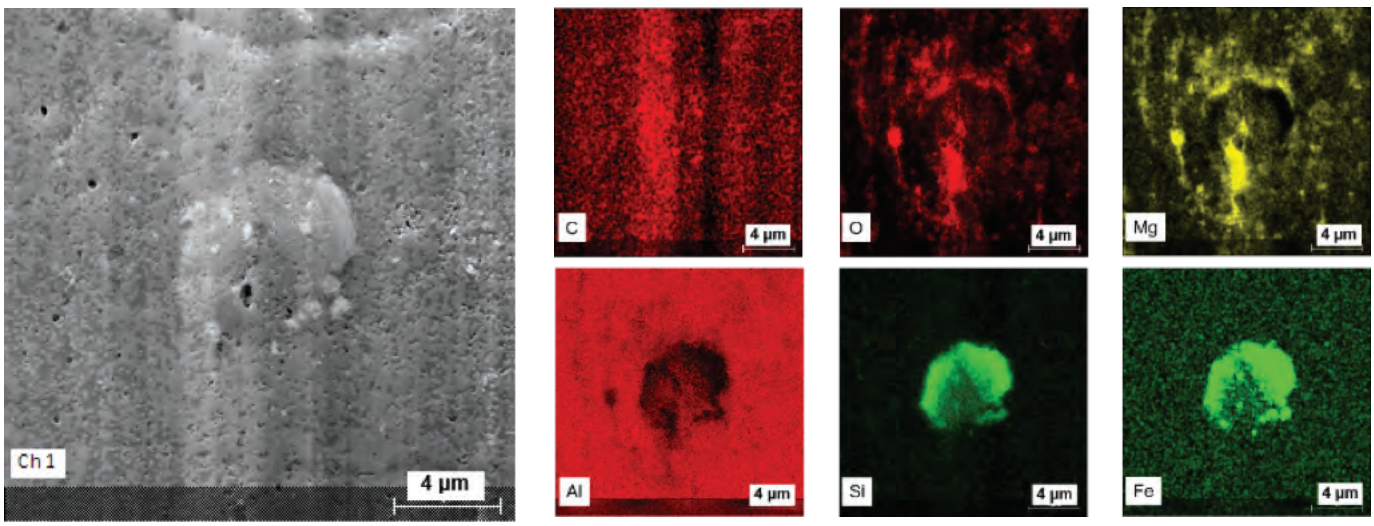

Figure 1. SEM image (left) and corresponding EDS maps of the defect region (right). The field of view for all images is 20 microns.

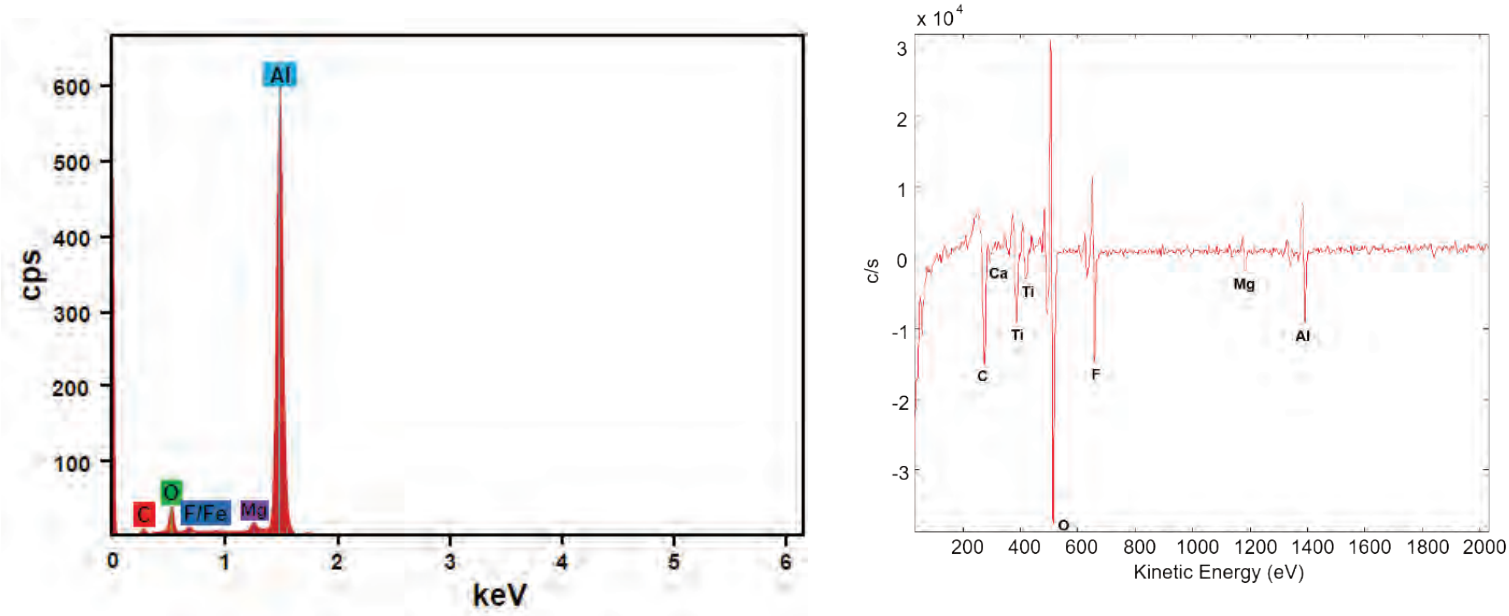

Figure 2. EDS spectrum (left) and AES survey spectrum (right) of the defect region.

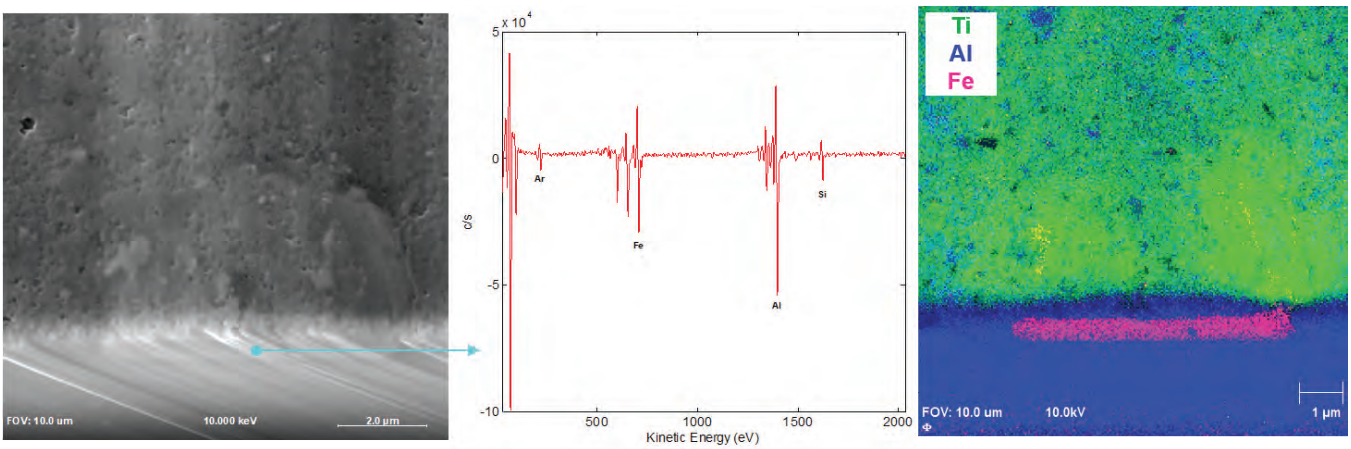

Figure 3. SEM image of the particle after a FIB cut (left) with a point spectrum acquired from the FIB face (center) and AES color overlay (right) of the FIB face with Ti coating in green, Al substrate in blue, and Fe particle in magenta.

Conclusions

Auger electron spectroscopy was used in conjunction with EDS and FIB to detect and characterize a buried metallic particle. The deep depth of analysis of EDS allowed a subsurface FeSi particle to be quickly located while the FIB provided an in-situ cross-section of the particle. Auger's surface sensitivity and superior spatial resolution were then used to characterize the true composition of the freshly exposed particle.