

Particle Elemental Identification in the ORION® PLUS Spectra

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Application

We discuss in this application note the use of helium ion scattering spectroscopy (ISS) in identifying small particles on surfaces. This capability is often useful in failure analysis and material study applications, where a sample may have present small amounts of material of unknown origin.

ORION® PLUS Spectra Capabilities

High resolution secondary electron imaging on surfaces can identify topological features very sensitively. Backscattered ion imaging can augment this with atomic number contrast that can flag the presence of particles. A novel ion spectroscopy method is provided that takes advantage of the extreme surface sensitivity in helium ion scattering. Energy proportional backscattered particle detection makes it possible to obtain spectra in the ORION® tool. Simulation software allows fitting the data to model to aid in particle identification.

Background

The identification of particles on surfaces is often valuable in failure analysis or in materials studies. This task becomes increasingly difficult, however, when the particle size is on the nano-scale. This is especially true in circumstances when the particle is in a field of other materials and not isolated. In such a case it is desirable that the spectroscopic signal comes from the very small spatial region containing the particle, and that there is also a way to differentiate the analytical signal of the particle from that of the substrate upon which it lies.

Challenge

Techniques such as SEM-EDS, while easy to apply, do not have the necessary spatial resolution for chemical information necessary to identify a particle separately from its environment. The probed volume in EDS is much larger than the SEM imaging resolution, since the interaction volume in which x-rays are produced is on the order of a micron. Improving the spatial resolution through the use of S/TEM requires a thinned sample to be made, increasing the preparation burden. A surface sensitive technique such as Auger Electron Spectroscopy requires ultrahigh vacuum conditions because it is defeated by even small amounts of contamination or oxidation. ISS is typically done with a broad beam and offers no imaging capability.

ORION® PLUS Spectra Solution

A combination of very high spatial resolution, extreme surface sensitivity, and ISS capabilities means

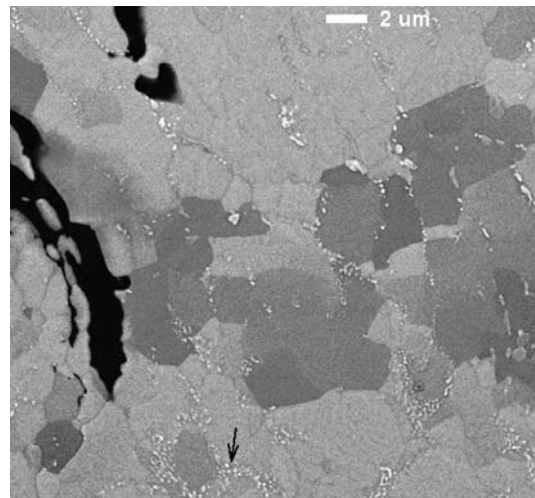


Figure 1: Area of tin solder ball, 25 µm field of view. The arrow points to the area where one of the particles was investigated by ISS.



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that the ORION® PLUS Spectra can address this void in the analytical applications space. Spectroscopic capability is enabled by a specially modified silicon drift detector which is provided in a sample-facing port that allows the acquisition of spectra either in a spot mode or while imaging. The spectrum collected is a total of an entire scanned area, or a spot mode spectrum can be obtained by parking the beam at one spot. The user chooses the region of interest via the image. If a particle is to be imaged, the field of view is set to enclose just that particle. The minimum analyzable volume depends on the sample material and geometry and is not yet fully characterized for this novel technology. In the example shown in this note, a 100 nm particle is studied. We will explain the analysis technique by looking next at this particular example.

The sample of interest in this case was a nickel-tin-copper solder joint (sample courtesy of Fraunhofer Institute Halle, Germany). It was being imaged for its metallurgical properties. The first step was apply Rutherford Backscatter Imaging (RBI) to the sample. In this mode, the backscattered helium ions from the sample are collected to form the image, rather than secondary electrons. RBI mode offers strong material contrast as well as grain orientation contrast. This can be useful for imaging polycrystalline metal samples such as solder. Figure 1 shows an image of a 25 μm area of the tin solder ball. The grain orientation contrast provided by RBI imaging reveals the polycrystalline structure of this material. We can also see that there is a network of particles, approximately 100 nm in size, which are resident at the grain boundaries. Since RBI mode brings out material contrast as opposed to topological contrast, it is evident that this material is not tin. Thus an investigation with the Spectra option of the ORION® tool was carried out.

As a part of the investigation, ISS was carried out on areas of the pure metals in the sample: nickel, tin, and copper. The primary beam energy was 34 keV. With a helium ion beam current of 8.5 pA, spectra are captured in about 1 minute each with a 12.2 eV channel width. The analyzed area in each case was 1 μm \times 1 μm . Since the resolution of the detector is about 4 keV for helium particles, the data

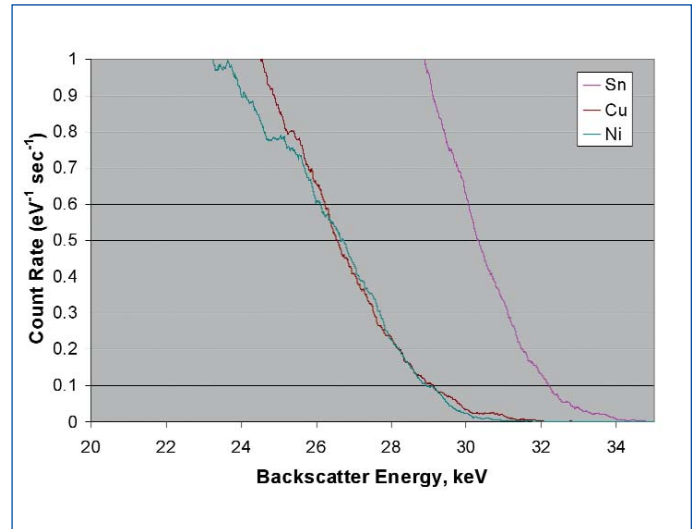


Figure 2:
ISS from nickel, copper, and tin areas on the solder joint.

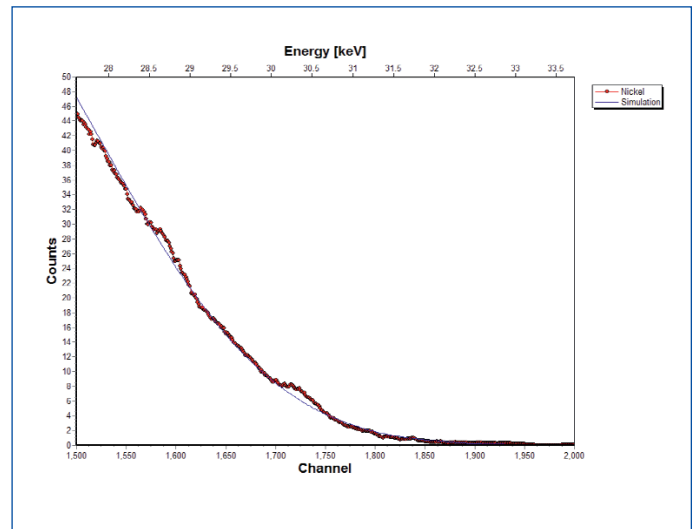


Figure 3:
SIMNRA fitting of peak for nickel.

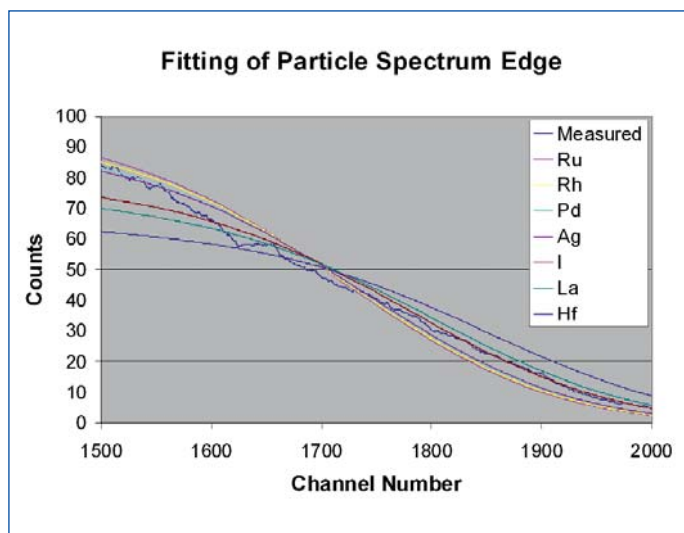


Figure 4:
ISS edge measured from small particle, along with fits from several candidates for its identity.

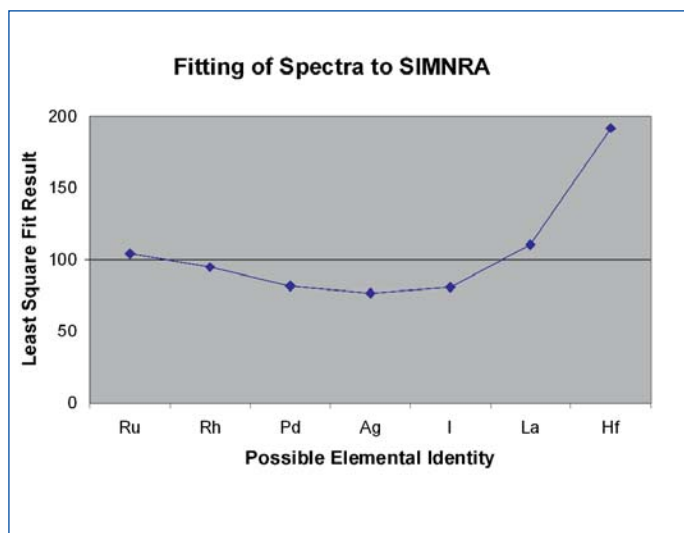


Figure 5:
Result of least squares fit of simulated spectra to measured data.

is filtered by means of a running average in a 300 eV wide window. This manages noise without sacrificing data integrity. For bulk materials, ISS produces a spectral shape in the form of an edge. There is a maximum backscattered energy corresponding to an elastic recoil at the top monolayer of the material, while ions that scatter deeper in the sample experience inelastic losses that cause them to be detected at lower energies – theoretically all the way to zero energy. We concentrate here only on the part of the spectra near the edge. Figure 2 shows the spectral edges from the three materials. We notice immediately that the highest energy of each edge changes with atomic mass of the material being analyzed. Nickel, the lightest element, appears at lowest energy while tin appears at the highest, as it is heaviest. The shape of the nickel and copper peaks is sufficiently different to distinguish them, even though they are neighbors on the periodic table. The lighter element begins to rise at lower energy, then it rises above the copper peak below about 28 keV. This shape may be characteristic and is seen also in the particle analysis below. The shapes of the edges deviate from one another at even lower energies, as can be seen again in the nickel and copper spectra. This may be due to crystallographic effects. It is certainly known that channeling effects will reduce peak intensity for some grain orientations, complicating interpretation. We concentrate on just the high energy portion of the edges for the data interpretation, however.

To provide a baseline for the analysis we need to correlate the results on these known materials to models. This is accomplished in the ORION® PLUS Spectra through the use of the simulation and data fitting software SIMNRA (developed by Matej Mayer at the Max Planck Institute for Plasma Physics). The program, originally intended for RBS and NRA measurements, can be extended to lower energy, with the assumption that its models remain valid. The sample information, geometries, and detector response function are input into the model. The program can then import experimental spectra and perform a fit. An example of the fit is shown in Figure 3 for the nickel edge. (Note that SIMNRA works in energy

units of channels.) The fitting, done over a range from about 28 to 34 keV, was used to calibrate the energy scale offset. The copper and tin edges then were in agreement with the models.

At this point we can turn our attention to the particle analysis. A region in the tin was chosen near where it is indicated by an arrow in Figure 1. A particle, approximately 100 nm in size, was analyzed by making the ion scan area equal to the particle size. The edge spectrum was obtained and calibrated according to the parameters determined for the bulk spectra. At this point the analysis needed to determine which element would best fit the observed spectral edge. The model in SIMNRA here consisted of two layers: a tin substrate and a top layer approximately 100 nm thick with a roughness of 100 nm as well. This approximated a particle for the sake of the fitting. It was noted that the position of the edge was close to that of the tin ($Z=50$), so a range of elements from $Z=44$ to $Z=72$ were modeled. The simulated spectra are compared in Figure 4. We note that the lighter elements modeled (Ru, for example) have lower signal than the measured data at higher energies but is stronger below about channel 1700. This, like the nickel and copper comparison above, is indicating that this element is lighter than the actual particle material. On the high mass extreme (Hf), the edge is higher at higher energies and lower at lower energies compared to the real particle data. To identify the element that fit best, a least squares difference measure was extracted for each candidate relative to the actual collected data. The result of this is shown in Figure 5. The best fit is obtained for silver ($Z=47$). The minimum of the fit curve is shallow, so the uncertainty in the elemental identification is probably only accurate to within 5-10 amu. This assignment is indeed correct, though, as the tin solder used in microelectronics is alloyed with 3-5 % silver. The imaging revealed silver that segregated into the grain boundaries and agglomerated into nanoparticles.

In conclusion, the ORION® PLUS Spectra is capable of distinguishing elements and has the spatial resolution and surface sensitivity to analyze small particles.

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