Auger Electron Spectroscopy

Physics:

Auger Electron Spectroscopy (AES) is an analytical technique to study the surfaces of materials. The auger effect is the analysis of the surface of materials based on the emission of energetic electrons. The electrons are emitted from an atom that is excited by the means of a photon or an electron beam. An electron is emitted from the core of the atom leaving a hole in the atoms inner orbital. The hole will be filled by an electron from the outer shell. The electron will then move to a lower energy level emitting the excess energy that is in the magnitude of the difference in orbital energies which is characterized by the orbital binding energy. Orbital energies are characteristic and unique to atoms of specific element or compounds, the energy emitted can be used to characterize the material of a surface. [1-3]

Figure 1.0 is the AES process. Where Figure 1.0 (a) is the illustration of the atom being excited by a photon or an electron beam releasing an electron from the inner orbital. Figure 1.0 (b) demonstrates how an electron from a higher energy orbital drops into the inner orbital to fill the hole left from the electron on part (a). The last part of the figure shows the electron moving orbitals emitting an auger electron energy that will characterize the element on the surface.

Figure 1.0 Auger Electron Emission [1]
Auger electron spectroscopy study of MgB$_2$ surface

Experimental:

The highly dense samples of MgB$_2$ were synthesized by using (99%) Furuuchi Chemical commercial MgB$_2$ powder. The samples were placed in a BN crucible at a temperature of 1273 K and pressure of 3.5 GPa for 2 hours. The bulk samples attained a density of approximately 2.63 g/cm$^3$. The samples were tested using XRD to determine the phases within the sample. The major phase had been characterized with the major peaks at MgB$_2$ and minor peaks of MgO. MgO is considered an impurity. The sample was also tested for Magnetization that gave a superconducting transition with the onset and midpoint temperature at 38.3 and 37.8 K, respectively. The sample preparation included a mechanical surface polishing with alumina powder of particle size of 0.05 µm and absolute ethanol. Once the surface reached the desired reflection (shininess) it was cleaned with acetone. The sample was polished to obtain a clean and smooth surface. The final step of the sample preparation was to glue the sample with an adhesive conductive tape on an AES sample holder and introduce into an AES ultra-high vacuum (UVH) chamber.

Once the sample was prepared the AES experiments were performed using a standard Scanning Auger Microprobe SAM PHI-680 from (Physics Electronics, USA). The experiment was conducted in UHV with a base pressure of 5 x 10$^{-8}$ Pa. The AES spectrum was acquired using a cylindrical mirror analyzer (CMA) and recorded in the pulse counting mode. The primary electron beam was set to 10 keV with 10 NA and incident to the surface normal. Depth analysis was conducted by the AES with an Ar$^+$ ion beam with 1.0 keV energy to sputter the surface with an incident angle of 30$^\circ$ with respect to the sample surface. The Ar$^+$ ion beams were focused to 1mm x 1mm area and the sputtering rate was about 12.4 nm/min. The morphology of the MgB$_2$ surface was observed by the use of secondary electron images (SEM) and the compositional distribution on the surface was revealed by Auger compositional images.

Results and Discussion:

The SEM micrograph shows the as-polished the MgB$_2$ surface’s crystallites and grain boundaries, and the AES spectra with the measurements taken at specific locations marked on the SEM image as A (top of the crystallite) and B (near the grain boundary). The AES spectra revealed four Auger peaks with Kinetic Energies 175.2, 266.5, 510.0, and 11848.8 eV that correspond to the B KLL, CKLL, O KLL, and Mg KLL, respectively. The atomic concentrations at the locations A and B were calculated using sensitive factors and found to be B (25%), C (42%), O (21%), and Mg (17%), respectively. The composition distribution was analyzed by mapping the surface and resulted in a spatially-resolved correlation of surface composition with surface morphology. There is a significant amount of C that exists on the surface, O is accumulated near the grain boundaries, and B is concentrated on top of the crystallites. The ratio of Mg:B of the as-polished surface is measured to be 1:2.1 which is close to the theoretical data of 1:2.0. Near the grain boundaries the Mg:B ratio exhibits 1:1.6 thus showing that this surface region is Mg enriched. The enrichment of Mg and O at the grain boundaries indicates the presence of Mg oxidation.

Auger sputter depth was also measured in another area marked by the white rectangle in Figure 3 (a). and demonstrated the compositional changes of the MgB$_2$ surface. The concentration of B increases for the first 3 minutes of sputtering then continues to increase slowly. The Mg concentration increases rapidly to
a definite value of 0.5 minutes of sputtering, then remains at the same value. The concentration of O decreases slowly during the Ar\(^+\) ion sputtering, and the concentration of C decreases rapidly to a minimum value. The mean atomic concentration also changes in this region from B (7%), C (42%), O (38%), and Mg (15%) after 10 minutes of sputtering. Resulting in a mean ratio of Mg: B of 1:0.5 before sputtering and 1:3.4 after sputtering. The mean ratio of Mg:B decreases with increasing sputtering time indicating that the Ar\(^+\) ion beam removes the oxidized surface and preferentially Mg from the MgB\(_2\) surface. Auger spectra was also taken from Figure 4 (a) where A is marked, on top of the crystallite, in comparison to the as-polished surface of MgB\(_2\), it can be seen from the sputtered surface a small Ar LMM (215.7 eV) signal is present due to the Ar\(^+\) ion sputtering, both C KLL and O KLL signals are reduced, and the B KLL signal is increased. The atomic concentration for this section is calculated to be B (75%), C (6%), O (7%), Mg (6%), and Ar (6%), implying that the ratio of Mg:B is 1:12.5 indicating that the regions on top of the crystallite are highly Mg depleted after the 10 minute sputtering. Auger images were also mapped out for this region and found that small amounts of O and C are accumulated along the grain boundaries, and B is concentrated at the top of the crystallites after sputtering. The white regions indicate the presence of MgO found in Figure 5.

**Conclusions:**

The surface of a high dense sintered MgB\(_2\) was characterized by AES, SEM, and sputter depth profiling. The as-polished surface was found to be Mg-rich and oxidized; this was due to the higher reactivity of Mg relative to B, especially near the grain boundaries. Care environment conditions must be present in the formation of MgB\(_2\). The sputtered surface was found to be Mg depleted, especially on top of the crystallites. The high indication of Ar\(^+\) indicates that it is not ideal for sputtering and affects the surface-sensitive studies of MgB\(_2\).

**Articles References:**


Our References:


