Unknown Material Identification through Energy Dispersive X-ray Spectroscopy (EDS)

Unknown material identification is often challenging, particularly when there are limited amounts of the unknown material. Energy Dispersive X-ray Spectroscopy (EDS) is a quantitative x-ray technique that can identify and measure elemental compositions at the microscale when it is carried out inside a scanning electron microscope (SEM). During EDS analysis, the SEM focuses a high energy electron beam on the sample surface. The energetic primary electrons knock inner-shell electrons out of atoms on the sample surface. Electrons from an outer shell of the atoms fill the lower energy level vacancies, and the difference in energy between the two states of the electrons is emitted as a characteristic x-ray. Figure 1 shows a schematic diagram of the characteristic x-ray generation process during EDS. Each element emits a unique set of characteristic x-rays which are dependent on the element's atomic number (Z) and the orbital transitions involved. Obtaining the spectrum of characteristic x-rays allows for elemental composition analysis of the material surface.

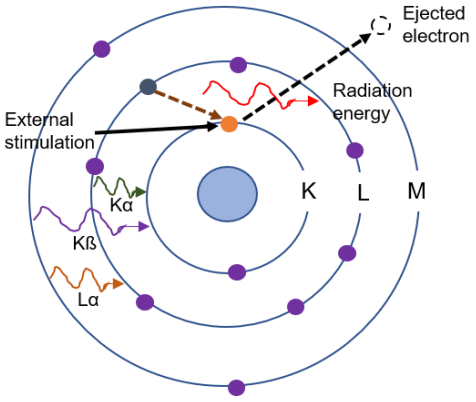

Figure 1. Schematic of characteristic x-ray generation using an electron beam in SEM.

In this application note, an automotive hood ornament of unknown composition was analyzed via SEM and EDS. The chemical composition was analyzed by EDS following ASTM E1508 using an accelerating voltage of 15 kV. The analysis was performed using a standardless method with P/B-ZAF matrix correction. Figure 2 shows a SEM image of the fractured surface in the core area of the hood ornament and the corresponding EDS spectrum. In addition, four other randomly selected areas were also analyzed using EDS at 200X magnification.

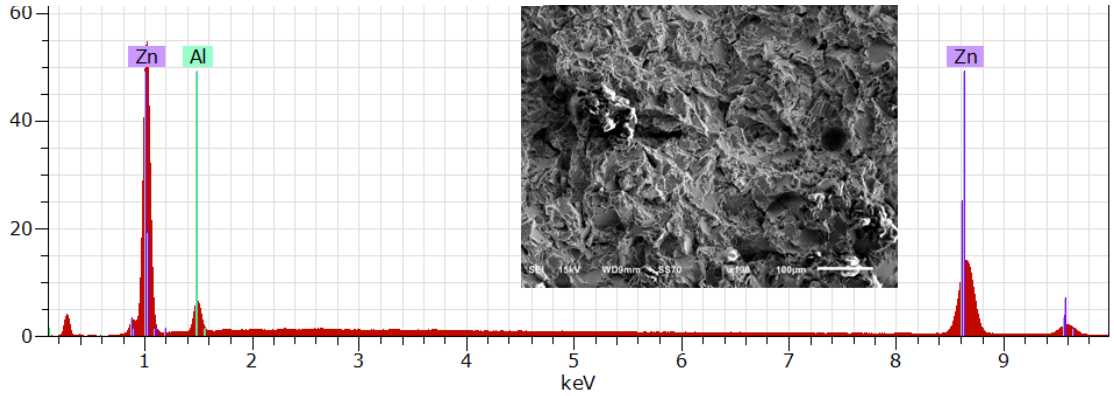

Figure 2. SEM image (inset) and corresponding EDS spectrum obtained from the core area of the hood ornament.

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The EDS spectra indicate that the core of the hood ornament is composed of zinc and aluminum. Table 1 lists the EDS elemental composition results obtained from these five measurement areas. From Table 1, it can be seen that the average concentrations of Zn and Al are 93.8 wt% and 6.2 wt%, respectively. This composition is common for zinc die casting applications. Zinc alloys have excellent finishing characteristics for plating and chromate treatments. They are low cost, have excellent thin wall capabilities, and possess high strength and hardness. These are probably the reasons zinc alloy was used for this hood ornament application.

Table 1 Elemental Composition of an Automotive Hood Ornament Measured via EDS Analysis

Element	Area 1	Area 2	Area 3	Area 4	Area 5	Average
Zn (wt %)	92.0	94.8	94.2	94.0	94.1	93.8
Al (wt %)	8.0	5.2	5.8	6.0	5.9	6.2

EDS is not only capable of performing spectroscopic analysis, but can also generate high resolution maps of the elemental distribution. In addition to the elemental composition measurement of the core area, an EDS map was taken on the cross section at the location near the outer surface of the hood ornament. From the EDS elemental map shown in Figure 3, it is obvious that surface coatings have been applied to the Zn alloy core of the hood ornament. The coatings consist of three layers: a Cu layer in direct contact with the Zn alloy core, a middle Ni layer, and a thin Cr outer layer. These coatings were likely prepared through electroplating processes where the Cu layer was used to facilitate Ni coating. The thicknesses of the Cu, Ni and Cr coatings were estimated from the EDS mapping to be approximately 15, 20, and 2 μm, respectively.

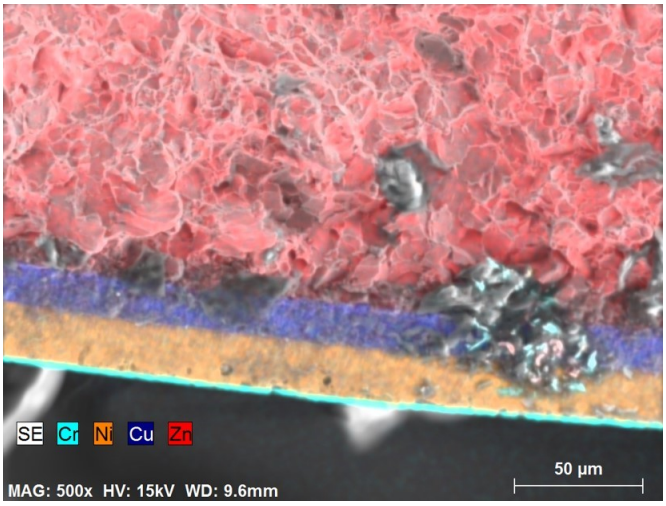

Figure 3. EDS elemental map of the cross section at the location near the outer surface of the hood ornament.