Auger Electron Spectroscopy Analysis of Fresh and Aged Alumina-Supported Silver Catalysts

Overview

Auger electron spectroscopy (AES) is an effective method for characterizing surface features with very high lateral resolution. The primary electron probe beam can be focused to spot sizes less than 10 nm in diameter, making it an ideal choice for analysis of small features such as corrosion pits and catalyst materials. Traditionally, AES analysis of insulating materials has been difficult due to sample charging related to electron bombardment, which can result in spectral peak shifts, appearance of multiple peaks, or data spikes. However, proper sample mounting techniques and acquisition conditions can alleviate the effects of sample charging and extend the analytical capabilities of AES to insulator-supported catalysts.

Sample Measurements

In this work, fresh and aged catalyst particles (Scientific Design Company, Inc., Little Ferry, NJ) were analyzed in a Physical Electronics *710* Scanning Auger Nanoprobe to understand what happens to the constituent materials during the aging process, which may then be correlated to catalyst performance. The cesium-promoted silver catalysts were contained in alumina supports.

Comparison of Fresh and Aged Alumina-Supported Silver Catalysts

Secondary electron (SEM) images were collected from fresh (Figure 1A) and aged (Figure 1E) catalysts at 10 µm fields of view (FOV). Large nodules were observed in both the fresh and aged samples, and additional small particles were dispersed across the fresh sample. AES mapping reveals the large nodules as the alumina supports, and they did not appreciably change in size during the aging process (Figure 1C, 1G). Silver catalyst

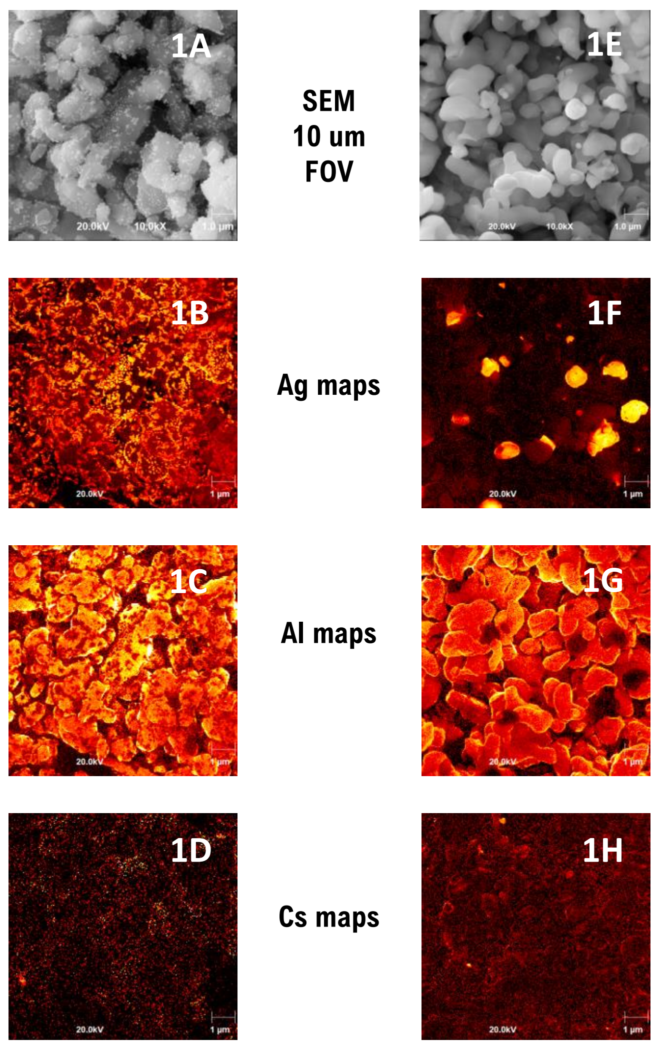

Figure 1. SEM images and associated Auger maps for Ag, Al, and Cs on fresh (left column) and aged (right column) catalyst samples at 10 µm FOV.

particles were well-dispersed in the fresh sample, with particle size ~0.05 – 0.1 µm (Figure 1B). However, in the aged sample, silver showed dramatic segregation and agglomeration into particles up to approximately 1 µm in size (Figure 1F). Cesium was well-dispersed across the alumina in both samples, and initial studies suggest that the overall surface concentration increased slightly after aging.

Analysis of Aged Catalyst

Figure 2 shows a 20 µm FOV SEM image and associated Auger spectrum collected from the entire image area. The alumina support was observed as well as small amounts of Ag and Cs catalyst material. A map of Cs reveals several locations with high intensity, and the survey spectrum from one of these regions confirms a higher Cs concentration compared to the surrounding area (Figure 4). Re was also observed, even though it was not detected in the large area spectrum. Figure 5 shows a 2 µm FOV SEM image of this region, and maps of Cs and Re indicate that these elements are in the same location.

Conclusions

We have shown the ability to observe the distribution of major and minor catalyst constituents on an insulating catalyst support material and observe changes in morphology of the catalyst constituents between fresh and aged catalysts. We also show the ability to locate and characterize sub-micron localized features with the use of Auger imaging and spectroscopy.

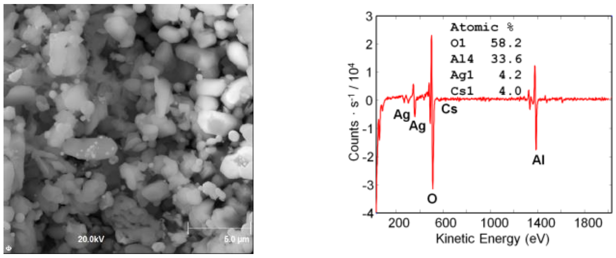

Figure 2. SEM image of aged catalyst with 20 µm FOV and average Auger spectrum from the entire field of view.

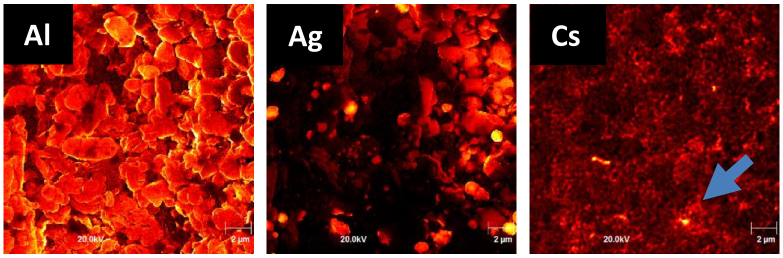

Figure 3. Auger maps for Al, Ag, and Cs from the 20 µm FOV SEM image shown in figure 2.

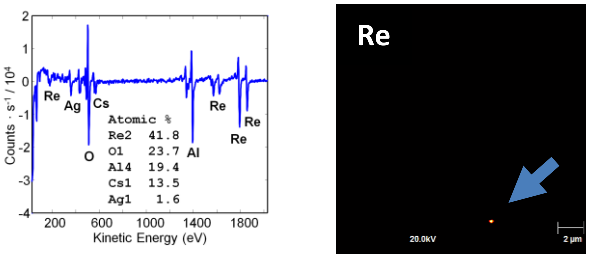

Figure 4. Auger spectrum and Re map from the high Cs area marked in Figure 3.

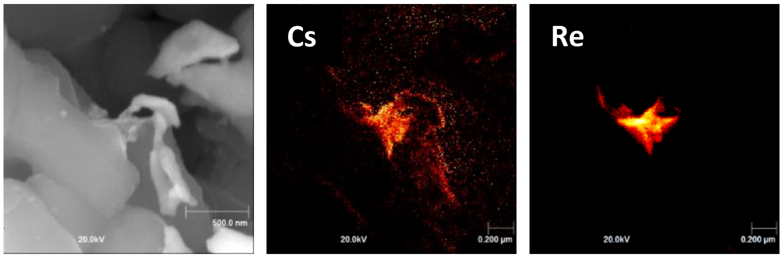

Figure 5. 2 µm FOV SEM image taken in the high Cs region and Auger maps for Cs and Re.