GATAN

Experiment Brief

In-Situ EELS Spectrum Imaging at Elevated Temperature

Title

Observation of bimetallic nanoparticle oxidation via *in-situ* EELS.

Gatan instrument used

Gatan imaging filter with a DENSsolutions Wildfire heating holder.

Background

Synthesis of hollow shell bimetallic oxide particles is of great interest for multiple technological applications, including electrocatalysis, lithium batteries, and supercapacitors. However, the oxidation mechanisms involved are not yet well-understood. *In-situ* transmission electron microscopy (TEM) enables direct observation of bimetallic nanoparticles during the oxidation process and provides insight into these mechanisms. Combining electron energy loss spectroscopy (EELS) spectrum imaging with scanning TEM (STEM) provides detailed information on the compositional heterogeneity of the resulting structures.

Materials and Methods

A DENSsolutions Wildfire heating holder was used within an environmental TEM (ETEM) to achieve an elevated temperature in a gas environment of 0.2 Torr pure O_2. STEM-EELS spectrum imaging was performed using a GIF Tridiem® system to map the O k, Co $L_{2,3}$, and Ni $L_{2,3}$ edges over multiple particles, as seen in Figure 1.

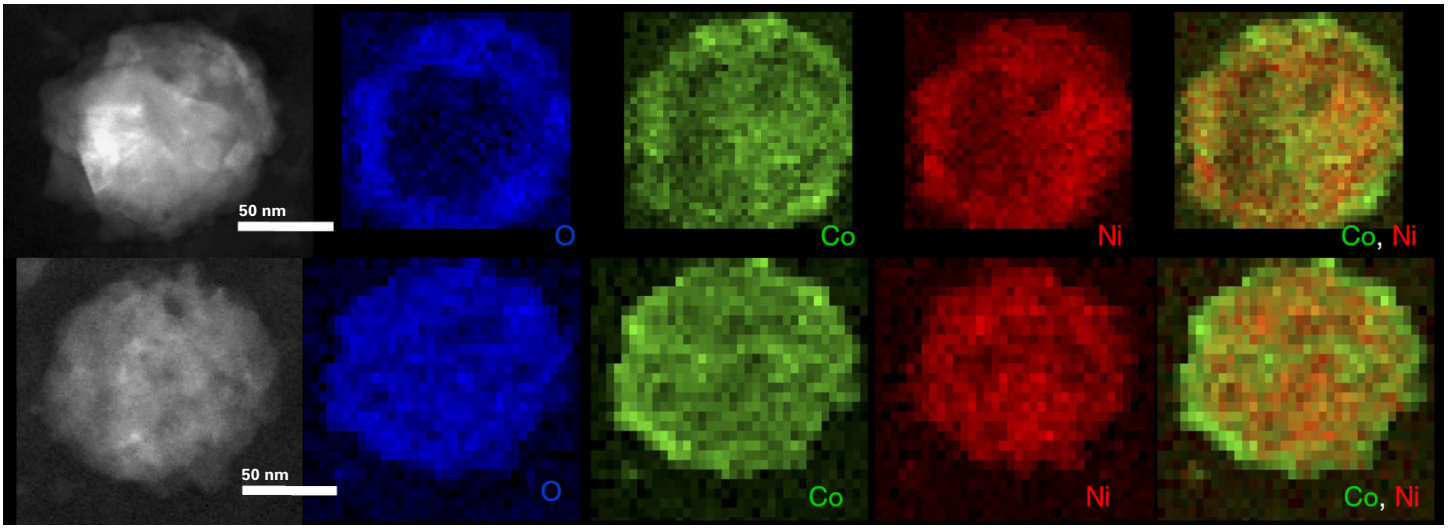

Figure 1. Series of *in-situ* EELS spectrum images showing particle oxidation. The top row shows a particle after initial oxidation at 450 °C, where a clear oxide shell is observed. The bottom row shows a different particle after further oxidation at 550 °C, where the entire particle was finally oxidized.
Han, L.; Meng, Q.; Wang, D.; Zhu, Y.; Wang, J.; Du, X.; Stach, E. A.; Xin, H. L., Interrogation of bimetallic particle oxidation in three dimensions at the nanoscale. Nat. Commun. 7 (2016) 13335. doi:10.1038/ncomms13335.

Summary

In-situ EELS spectrum imaging was used to map the composition of multiple bimetallic nanoparticles as they were oxidized. The study indicated that oxide shells form initially, followed by diffusion of metal ions up to the surface where they are oxidized, forming voids in the structure. If the oxide cracks, allowing oxygen to penetrate through the outer shell, oxidation can additionally proceed in the central region of the particle. The internal and external surfaces of the oxidized nanoparticles were found to be Co-rich.

Credit(s)

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