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Activation of Group III and IV Metals for Tritium Absorption

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Motivation

Rare-earth and transition metals are often used in hydrogen storage applications. However, these metals tend to passivate when exposed to air, which reduces their ability to sequester hydrogen during processing. In industry, high temperature is used prior to loading to breakdown the native oxide layer and therefore thermally "activate" the metal. The aim of this study is to determine the approximate minimum temperature by which a given metal can be considered thermally activated. *In-situ* XPS of Group III and IV metals during thermal annealing was performed to determine the phase content of the sample surface as a function of temperature.

Experimental Methods

- Samples were synthesized via electron beam evaporation under UHV conditions and from high purity precursor metals
- Samples were exposed to lab air for a period of several weeks to allow for native oxide layer formation
- Samples were then heated in increments of 100 °C (up to 900 °C), with a one hour wait period between spectrum collections
- CasaXPS software was used to analyze the resulting spectra

XPS Fitting Methods

- Metal peaks were fitted to an asymmetrical peak shape of the form LA(1.1,5,7)
- FWHM values between doublet peaks were constrained to be equal for Zr and Y
- FWHM values in Sc and Ti were only constrained with maximum values due to Coster-Kronig broadening
- Peak positions were constrained to within one standard deviation of the average value based on the NIST XPS database, then constrained to +/- 0.05eV when propagating the resulting fit

Conclusions

- A transition occurs as the sample is activated from an oxide surface to a metallic surface
- Oxygen diffuses into the film, producing an oxygen rich metal in the interior and a cleaner metal surface.
- Various oxide phases appear during the transition.
- The carbon phases are a complicated determination and are less certain at this point.
- Metals from the same group have similar activation temperatures and properties.
- Activation temperature roughly follows predominant oxide heat of formation
- Activation proceeds in these steps:
  1. Removal of hydroxides
  2. Diffusion of oxygen into film
  3. Potential appearances and removal of carbides
  4. Clean metal surface to react with hydrogen.

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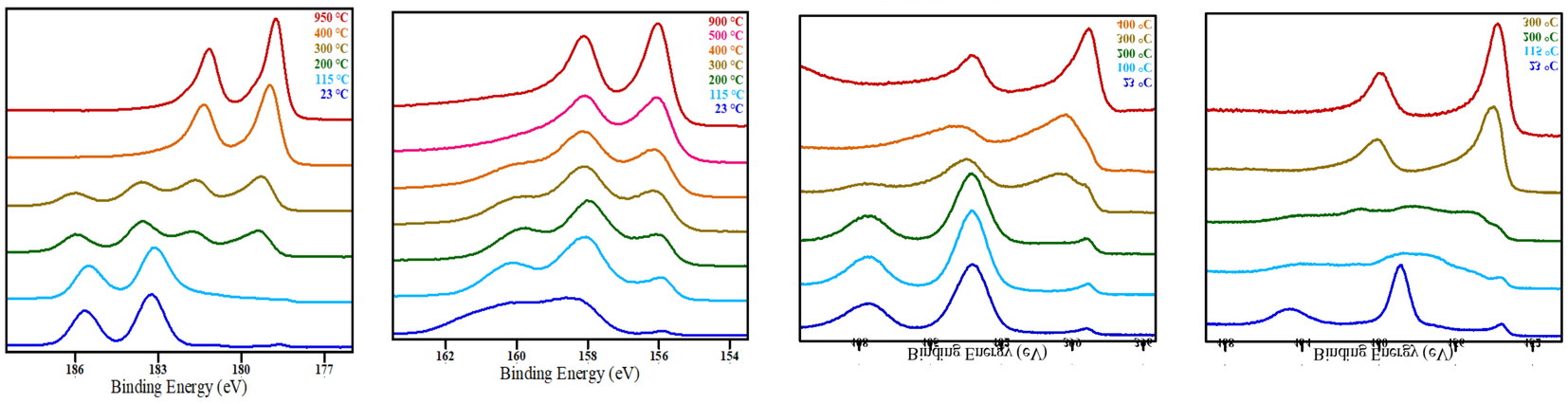
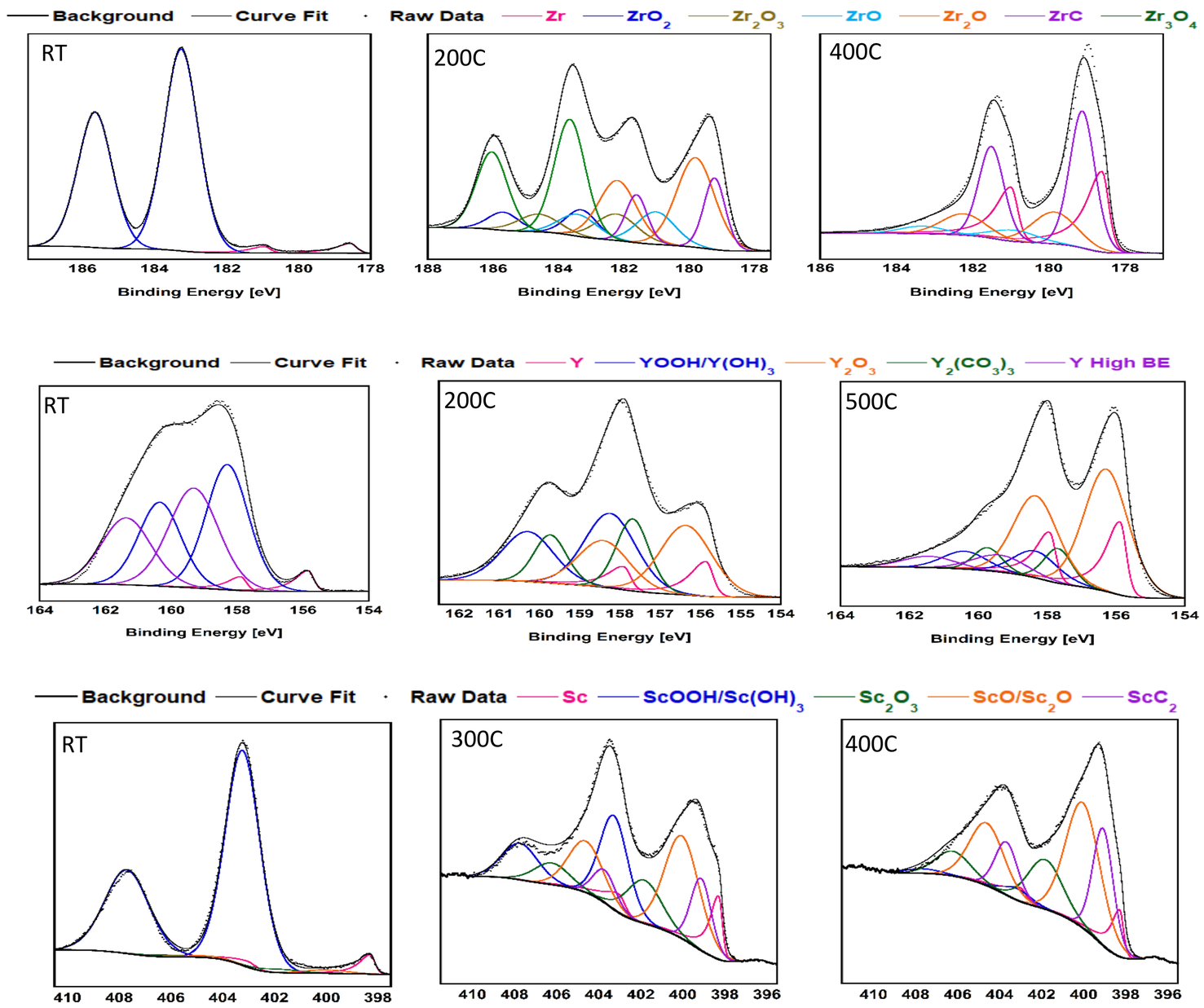
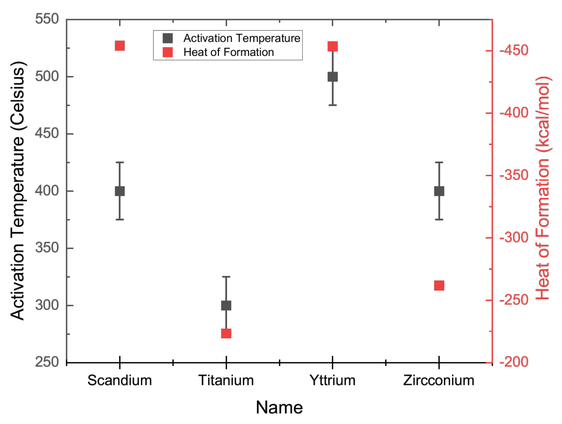
Temperature Dependent Metal Scans



Metal Peak Component Analysis





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