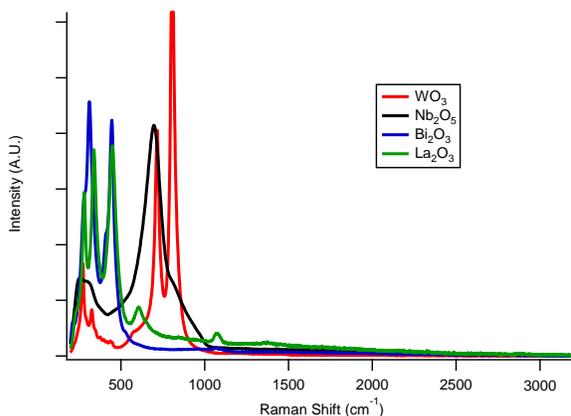


# In Situ Raman Spectroscopic Measurements of Metal Oxides under High Temperature

## High Temperature Raman Fiber Probe Designed for Harsh Environment

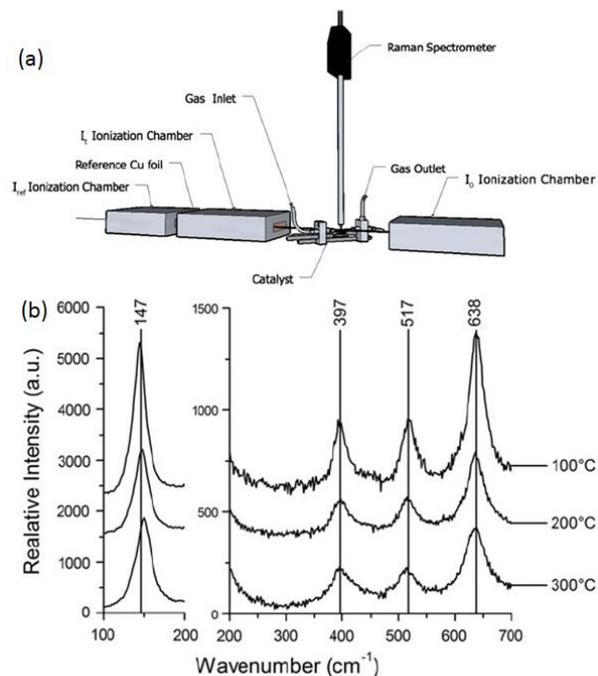
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Transition metal oxides are compounds composed of oxygen bound to transition metals and they are versatile materials commonly used in various applications such as heterogeneous catalysts. To study the mechanism of the catalytic process as well as monitor and optimize the reaction conditions, multiple analytical methods such as XAS, NMR, and Raman spectroscopy have been investigated. As an in situ, non-invasive, and sensitive technology to probe and analyze chemical compositions and structures with high specificity (shown in **Figure 1**) and without sample preparation, Raman spectroscopy has been widely utilized in *in-situ* catalytic process monitoring with metal oxides, even achieved multi-technique characterization in *operando* by combining with other instrumentations.



**Figure 1:** Raman spectra of four typical metal oxides.

BaySpec's *PeakFinder*<sup>TM</sup> high temperature fiber optic Raman probes feature optical filtering of  $10^6$  for efficient attenuation of the Rayleigh line for background-free spectra. The pressure-tight stainless-steel extension tube is equipped with a compression-welded sapphire window at the tip to ensure outstanding optical performance under harsh environment of -50 to 500 °C. Additionally, optional video camera can be integrated with the standard fiber optical probe in order to show the video image of the excitation laser spot.



**Figure 2:** Schematic of the setup for combined, operando XAS/Raman experiment (a). Raman spectra of pure  $TiO_2$  catalyst collected during the reaction cycle (b).<sup>1</sup>

As shown in **Figure 2**, Prof. Frenkel's group combined BaySpec's 532nm Raman spectrometer and high temperature Raman probe with XANES and EXAFS to monitor the catalytic process of CO oxidation in operando.<sup>1</sup> Results of all complementary measurements suggest that both phases of copper oxide (CuO and Cu<sub>2</sub>O) must be present in the active state of the catalyst at 200 °C. Besides, Raman studies highlighted the role that the support material (TiO<sub>2</sub>) plays in this reaction.

### References

- 1) Patlolla, A., et al., *Top. Catal.*, **2013**, 56, 896-904.