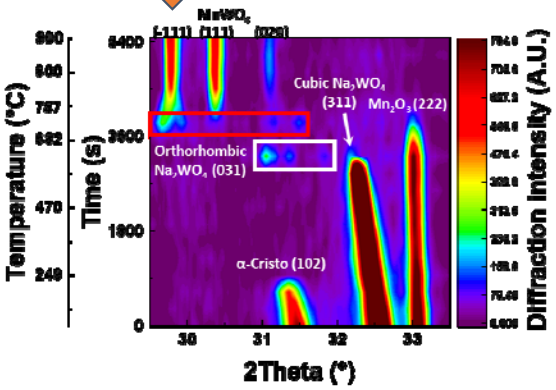
Experimental

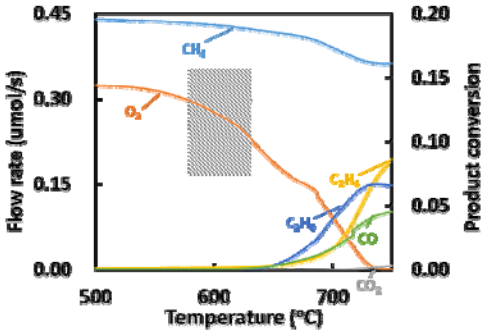
In situ XRD-MS

In situ Bulk structure



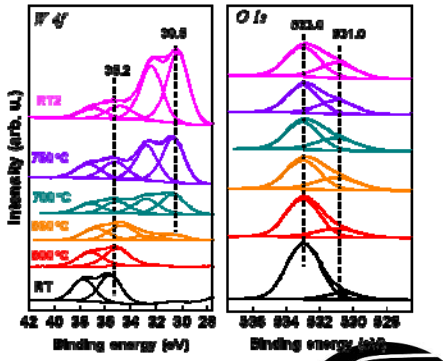
Online MS

Activity



In situ XPS

Surface electronic structure after in situ reaction

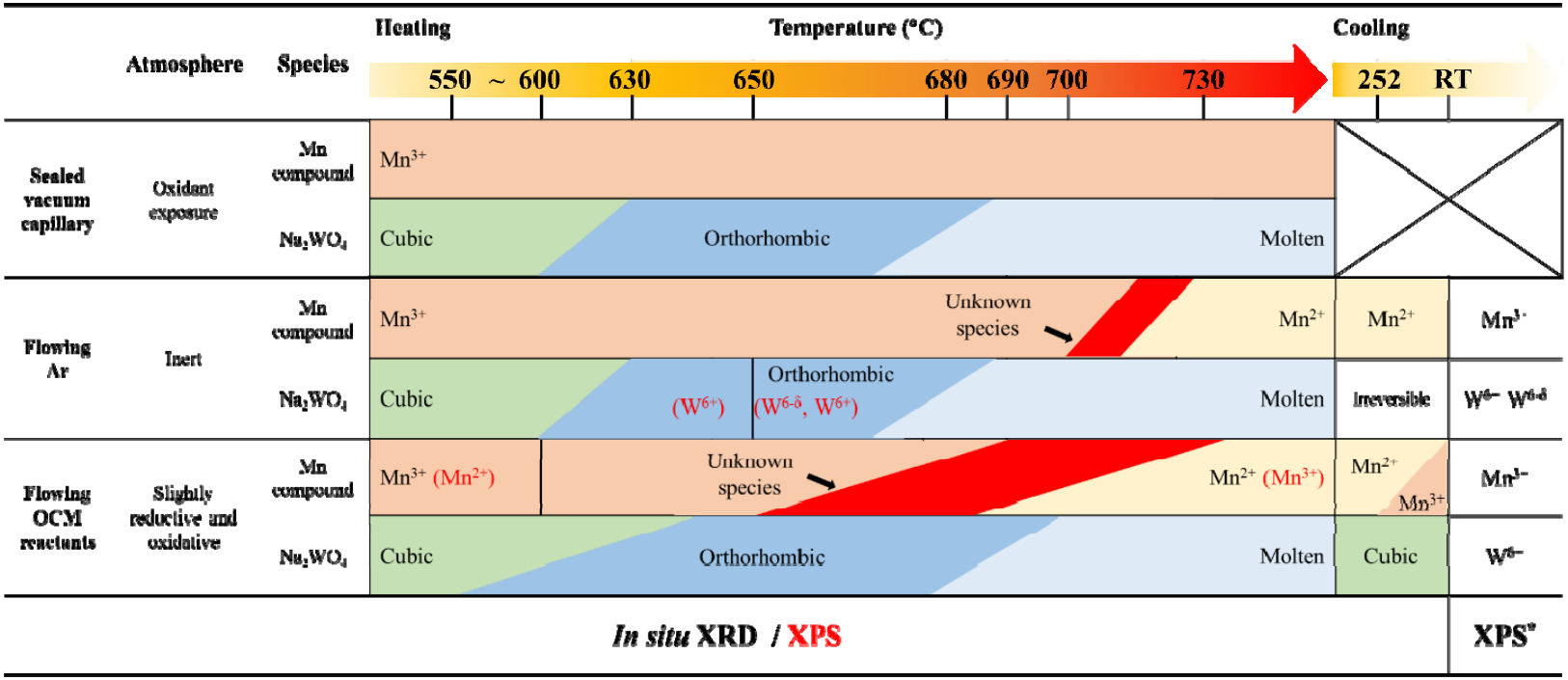


Yang, Y.; Zebang, L.; Li, J.P.H.; Evgeny, V. Reaction control and mass spectrometry workstation for coupling an X-ray spectroscopic characterization instrument with an in-situ reaction cell. 2022.

Discussion Ti-NaWMnSi

XRD and XPS results of Mn and W at different temperatures and different atmosphere conditions

*** *In situ* treatment in HPGC, the spectrum is collected after cooling down to RT**



- The reduction from Mn^{3+} to Mn^{2+} is favored by inert or slightly reductive gas exposure while a high temperature (> 650 °C) is always required.
- It also shows that to inhibit Mn^{3+} reduction to Mn^{2+} at the same high temperature, or to reverse the reduced Mn^{2+} to Mn^{3+}, slightly oxidative exposure other than inert gas must be provided.
- The "unidentified species" should be related with the complex catalyst components and is a transition state which can only be observed at high temperature.
- W self reduction observed after melting under inert condition.

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