In-situ High Temperature X-ray Diffraction up to 1500°C in Air: Lab Source XRD and Quadrupole Lamp Furnace

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Introduction

Instrumentation has been developed for high temperature x-ray diffraction (HTXRD) in reflection geometry on powder samples in air up to 1500°C using a laboratory source diffractometer. The samples are heated using a quadrupole lamp furnace (QLF), which was mounted on a Bruker D8 GADDS diffractometer equipped with a 2D VÅNTEC-500 detector. The QLF uses four 150 W Xenon lamps with ellipsoidal gold-plated reflectors to create a real image of the lamp filaments at the furnace center, where the sample is placed. A refractory crucible made of alumina was used as the specimen holder. A Pt-Rh Type B thermocouple embedded inside the powder sample was used to determine sample temperature. Using Cu Kα x-rays HTXRD data can be acquired with high S/N ratio over a 2θ range of 10-80°. The performance of the apparatus was verified by measurement of the thermal expansion properties of standard materials and comparison with synchrotron data collected at NSLS-II, BNL.

Objective:
To develop instrumentation for in-house high temperature x-ray diffraction to study the thermo-physical properties of materials.

In-Situ Phase Transformations

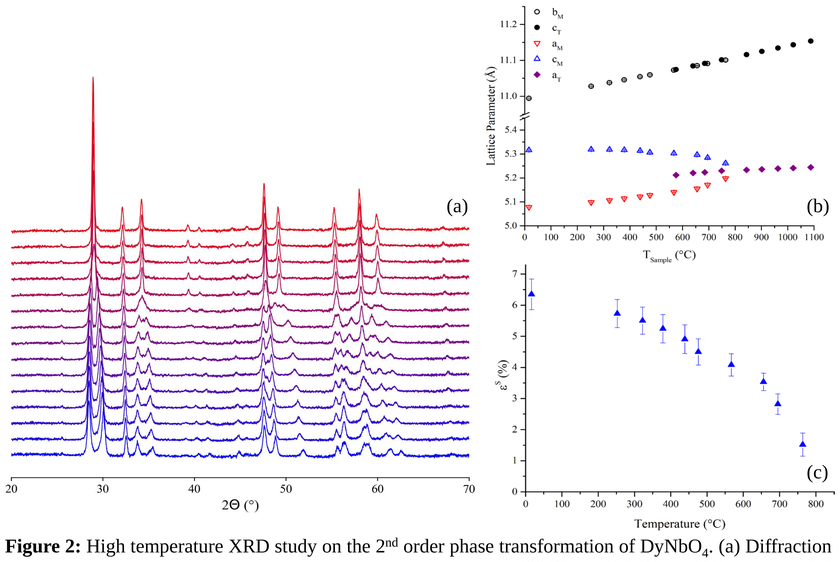

Figure 2: High temperature XRD study on the 2nd order phase transformation of $DyNbO_4$. (a) Diffraction patterns were collected in air at room temperature and between 200°C and 850°C at 50°C intervals. (b) Crystal structure lattice parameters were refined using the Rietveld method and (c) From HTXRD datasets it is possible to determine the second order phase transformation temperature (T_{Tr}) using Landau theory and the spontaneous strain parameter.
Note: Methods for accurate sample temperature determination are still being optimized. Sample temperature measured using the thermocouple is sensitive to the location of the thermocouple with respect to the furnace hot spot. For the above data the sample temperature was determined by fitting the monoclinic *b* parameter to data collected at XPD, NSLS-II, Brookhaven National Laboratory.

Conclusions

- A Quadrupole Lamp Furnace has been adapted for use with a laboratory Bruker D8 XRD for high temperature experiments.
- The capability to study 2nd order phase transformations in situ has been demonstrated.
- The ability to study the complete thermal expansion tensor using powder diffraction has been demonstrated

❖ Procedures for accurate determination of the sample temperature need to be optimized.

Acknowledgments

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QLF on Bruker D8 Discover X-ray Diffractometer for HTXRD

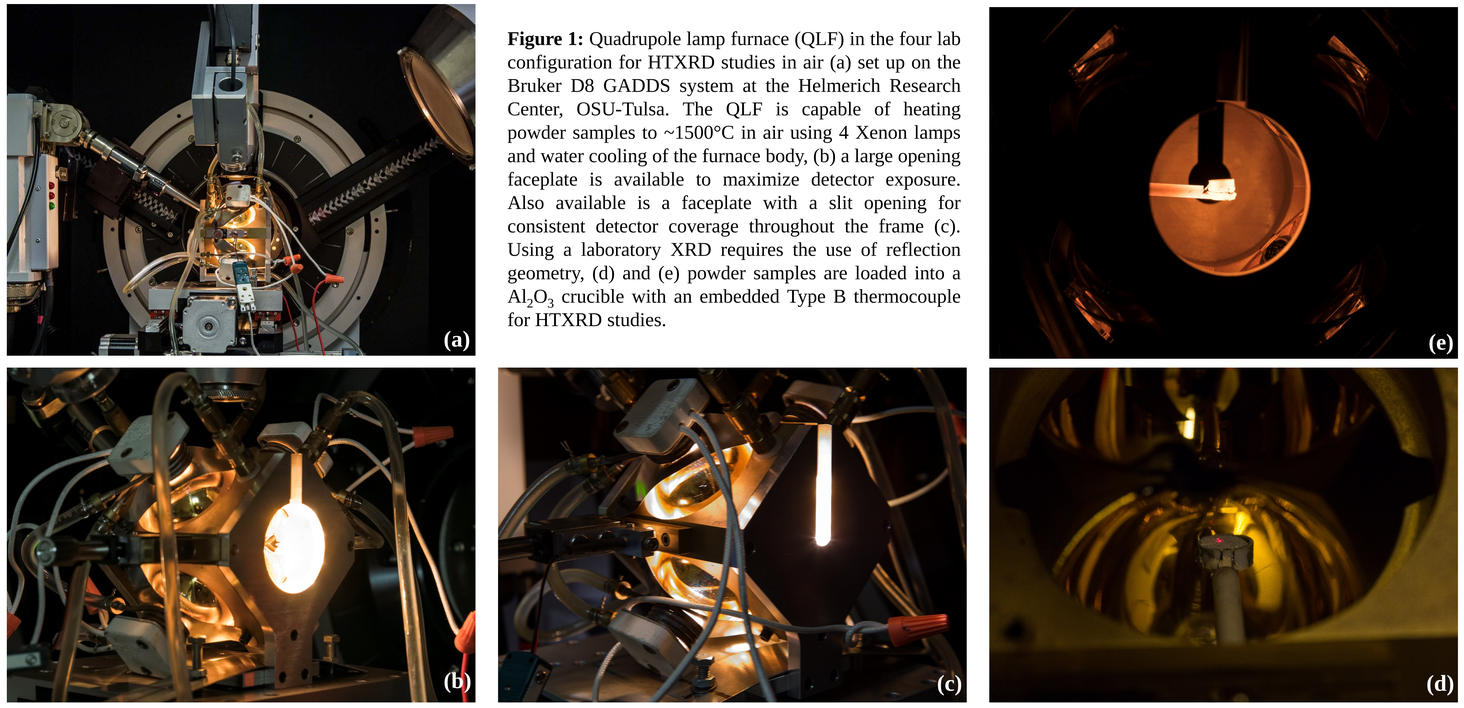

Figure 1: Quadrupole lamp furnace (QLF) in the four lab configuration for HTXRD studies in air (a) set up on the Bruker D8 GADDS system at the Helmerich Research Center, OSU-Tulsa. The QLF is capable of heating powder samples to ~1500°C in air using 4 Xenon lamps and water cooling of the furnace body, (b) a large opening faceplate is available to maximize detector exposure. Also available is a faceplate with a slit opening for consistent detector coverage throughout the frame (c). Using a laboratory XRD requires the use of reflection geometry, (d) and (e) powder samples are loaded into a Al_2O_3 crucible with an embedded Type B thermocouple for HTXRD studies.

Thermal Expansion Analysis

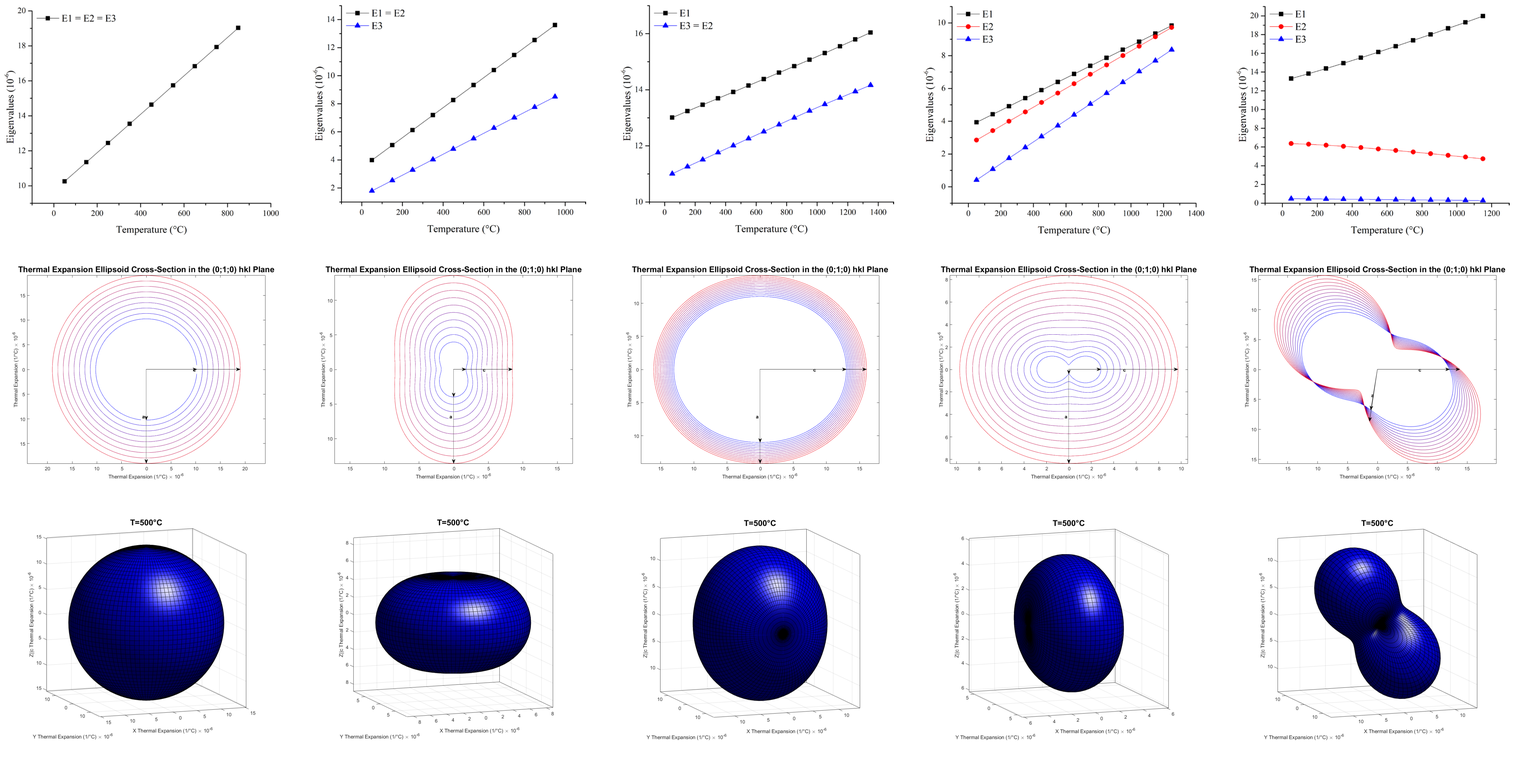

Figure 3: Thermal expansion analysis of selected material systems representative of a range of crystallographic symmetry. HTXRD datasets from powder samples were used to refine the crystal structure of the studied materials using the Rietveld refinement method. The Coefficient of Thermal Expansion Analysis Suite (CTEAS)[1] software was used to determine the CTE tensor elements. Presented are the thermal expansion eigenvalues which represent the maximum and minimum magnitudes of thermal expansion, a 2D representation of the thermal expansion ellipsoid as a function of temperature (ac-plane), and a 3D representation of the thermal expansion ellipsoid, at 500°C.

[1]"CTEAS: A GUI Based Program to Determine Thermal Expansion from HTXRD," Z.A. Jones, P. Sarin, R.P. Haggerty, and W.M. Kriven, *Journal of Applied Crystallography*, Vol. 46, 550 – 553 (2013).