

# Diffraction Facilities at ORNL

## *In-situ x-ray, Synchrotron and Neutron Diffraction*

### Abstract

The Diffraction User Center (DUC) assists academic, industrial, and U.S. Department of Energy users in the characterization of materials and systems using X-ray, synchrotron, and neutron diffraction.

DUC specializes in real-time in-situ diffraction studies where diffraction data are collected at temperatures from 10 to 2400 K under various controlled environments and pressures. Such in-situ diffraction methods are used to characterize the crystal chemistry and stability of ceramics, alloys, catalysts, and other industrially relevant materials. Traditional applications such as phase identification, quantitative phase analysis, unit cell refinement, and texture studies are also performed.

The data are used to relate materials processing and performance with phase transformations, reactions (solid-solid, liquid-solid, and gas-solid), lattice expansion, atomic structure, crystallization from the melt, and phase stability. In addition to supporting users' diffraction needs, the diffraction facilities are extensively used in a wide variety of ceramic and alloy research and development efforts sponsored by DOE and other agencies.

### Scintag PAD-X In-Situ X-Ray Diffractometer



- ❖ Buehler HDK 2.3 furnace
  - Temperatures up to 2200°C
  - Operates in air (up to 1500°C), vacuum, inert, oxidizing, or reducing gases
  - O<sub>2</sub> partial pressure control and monitoring
- ❖ Solid-state detector for high precision measurements
- ❖ Position sensitive detector for high speed measurements

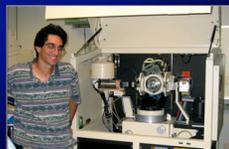
### Scintag XDS2000 Cryogenic X-Ray Diffractometer



- ❖ Displex closed-cycle He cryostat
  - Temperatures from 10 to 300 K
  - Cold sample loading
  - Controlled gas environment
- ❖ Solid-state detector

### Scintag PTS 4-Circle Goniometer

- ❖ 4-axis goniometer
- ❖ Measure texture or preferred orientation



### Scintag PAD-V X-Ray Diffractometer

- ❖ Vertical  $\theta:2\theta$  goniometer
- ❖ Peltier-cooled Si(Li) energy dispersive detector
- ❖ Multiple stage sample holder with spinner

### PANalytical X'Pert Pro Multipurpose Diffractometer

- ❖ Optics
  - Programmable slits
  - Monochromator
  - Pseudo-parallel optics
    - Multilayer mirror
    - Polycapillary lens
- ❖ Detectors
  - High count rate proportional detector
  - X'Celerator real-time multiple strip detector for rapid data collection
- ❖ Anton Paar XRK900 furnace
  - Volumetric heating up to 900 °C
  - Controlled atmosphere
  - Sample spinner
- ❖ Anton Paar HTK16 furnace
  - Temperatures up to 1500 °C
  - Controlled atmosphere
- ❖ Multipurpose sample stage
  - Mount and accurately position large and irregularly shaped samples



Anton Paar XRK900 Furnace



Interchangeable Optics

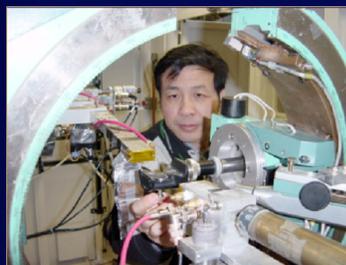


Anton Paar HTK16 Furnace



Multipurpose sample stage

### X14A Synchrotron Beamline at NSLS



- ❖ High flux, parallel beam
  - X-ray energies from 3 to 26 keV
- ❖ 6-circle Huber goniometer
- ❖ Low temperature displx cryostat
  - Down to 10 K
- ❖ Position-sensitive curved detector
  - Measure 45 °2 $\theta$  in ~1 sec



- ❖ High temperature capillary furnace
  - To 1500 °C in controlled gas environment
  - Spinning capillary sample

### Neutron Powder Diffraction at HFIR



- ❖ High resolution neutron diffractometer
- ❖ Furnace operates up to 1600 °C
- ❖ Cryostat operates down to 10 K
- ❖ Pressure cell operates at temperatures of 10 to 300 K and pressures of 0 to 350 bar



The HFIR Center for Neutron Scattering is a national user facility operated by ORNL for the U.S. Dept. of Energy.

### Access HTML User Program facilities via our website: [www.html.ornl.gov](http://www.html.ornl.gov)

#### Acknowledgements

Research sponsored by the Assistant Secretary for Energy Efficiency and Renewable Energy, Office of FreedomCAR and Vehicle Technologies, as part of the High Temperature Materials Laboratory User Program, Oak Ridge National Laboratory, managed by UT-Battelle, LLC, for the U.S. Department of Energy under contract number DB-AC05-00OR22725