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Phase transformations in oxides above 2000°C: Experimental technique development

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Phase transformations in oxides above 2000 °C: experimental techniques development

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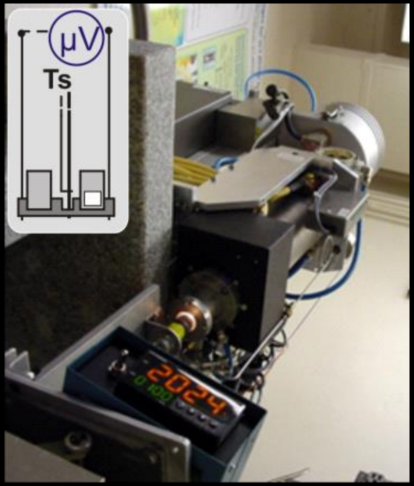
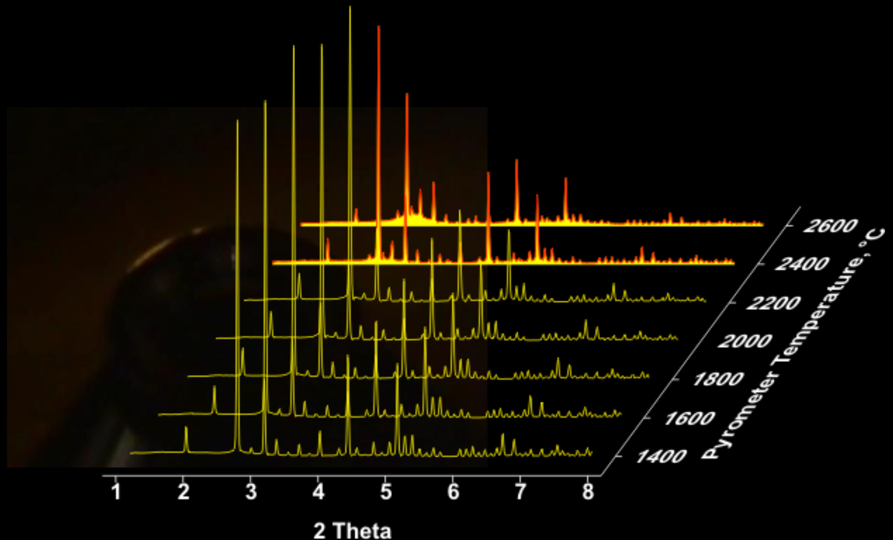
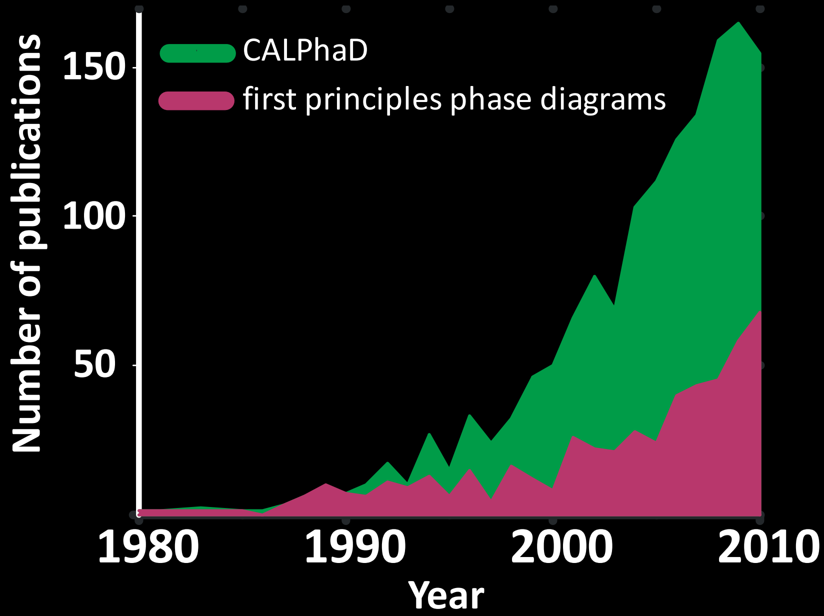
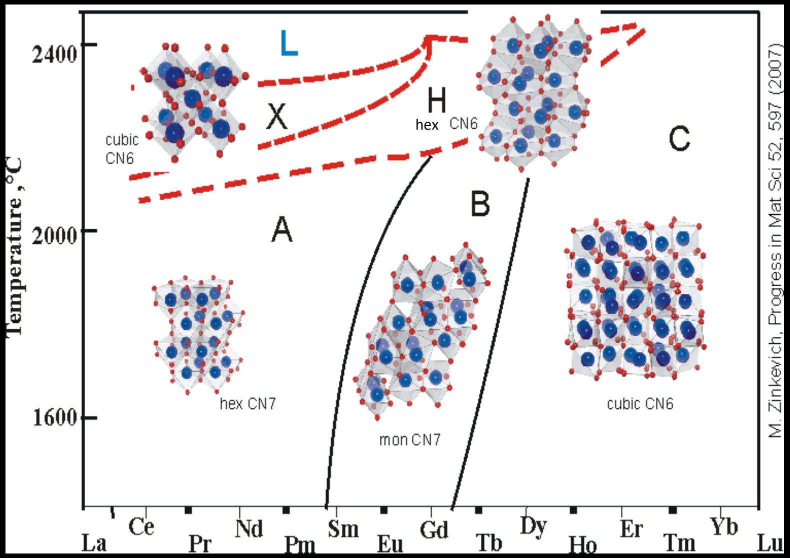
UC Davis Peter A. Rock Thermochemistry Laboratory, Davis, CA

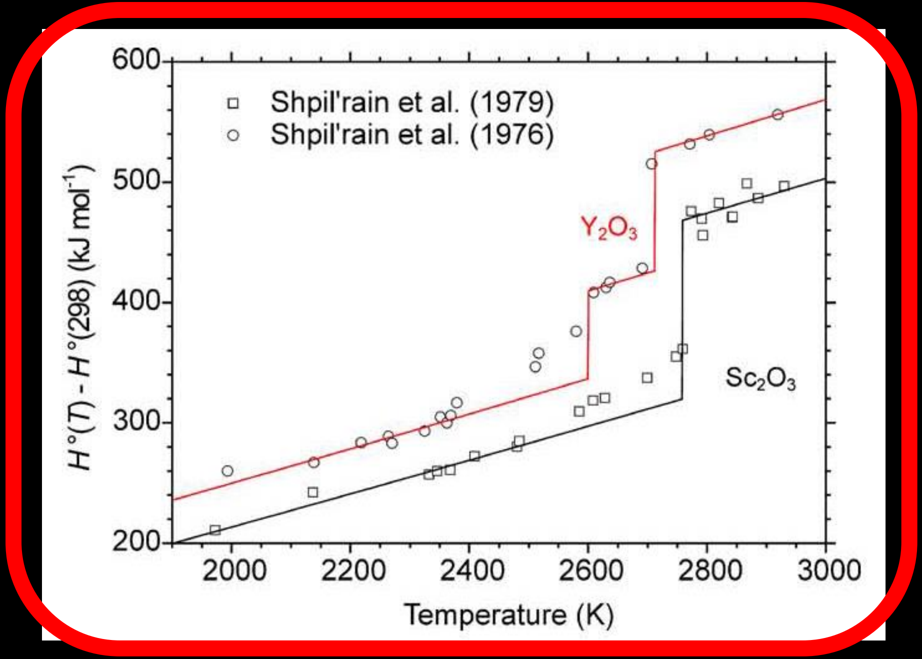
1. Differential Thermal Analysis
2. Drop calorimetry
3. X-ray and neutron diffraction

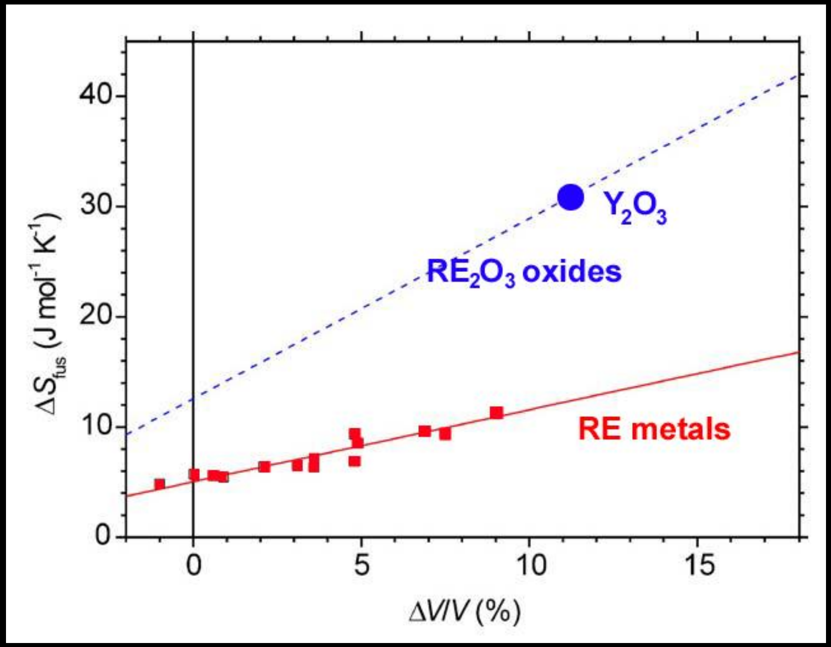












M. Zinkevich, Progress in Materials Science 52, 597 (2007) www.nist.gov/publications/open-calphad-free-thermodynamic-software 80% opencalpahd

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PUBLICATIONS

Open Calphad - a free thermodynamic software

Published: January 17, 2015

Author(s)

Bo Sundman, **Ursula R. Kattner**, Mauro Palumbo, Suzana G. Fries

Abstract

The use of thermodynamics in many applications in material science like simulation of phase transformation suffers from the lack of high quality open source software for calculations of multicomponent systems. The goal of the Open Calphad (OC) software is to bridge this gap. The OC software has a GNU license which makes it possible for the interested scientist to implement and test new ideas for models and algorithms. Currently such development can be done only using proprietary software. The assessment of model parameters using both experimental data and data from theoretical calculations like DFT will have increased efficiency using software that can be directly integrated in various applications.

Citation: Integrating Materials and Manufacturing Innovation

Volume: 4

Pub Type: Journals

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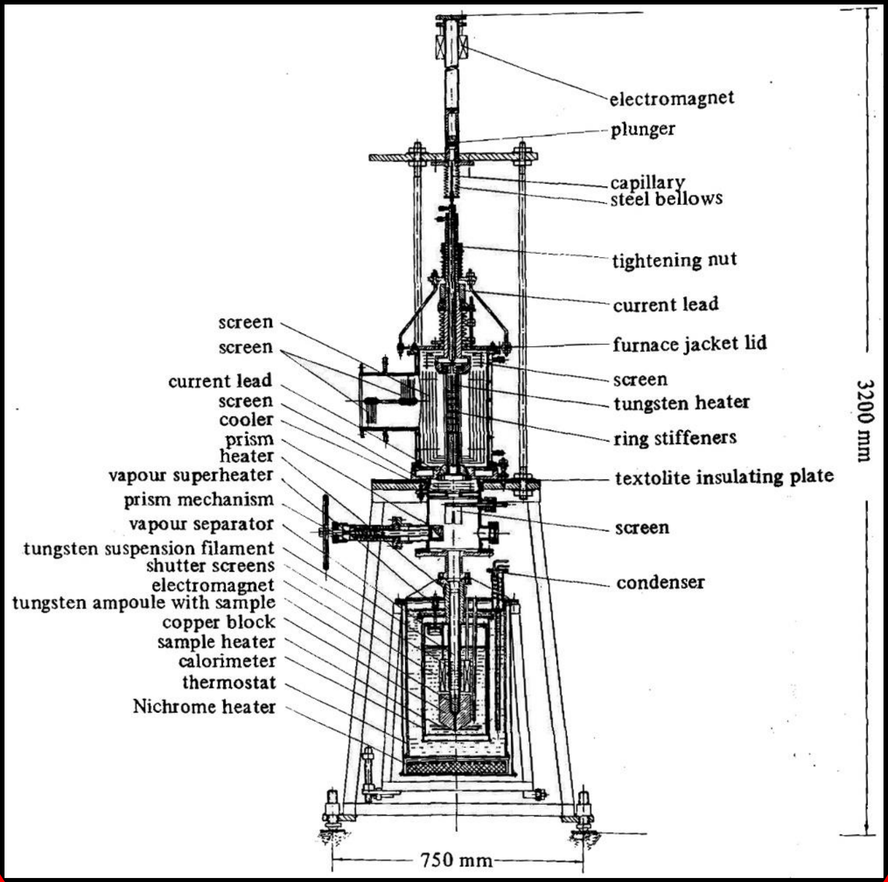
Shpil'rain 1972
DROP calorimeter



1960s - Drop calorimetry from above 2000 °C

1962-1963 Kantor, Sheindlin et al. Al_2O_3 heat of fusion

1963 Pears et al. report on thermal properties of 26 solids to 5000 °F

1968 Hein et al. heat of fusion of UO_2

Cooling traces from solar furnace

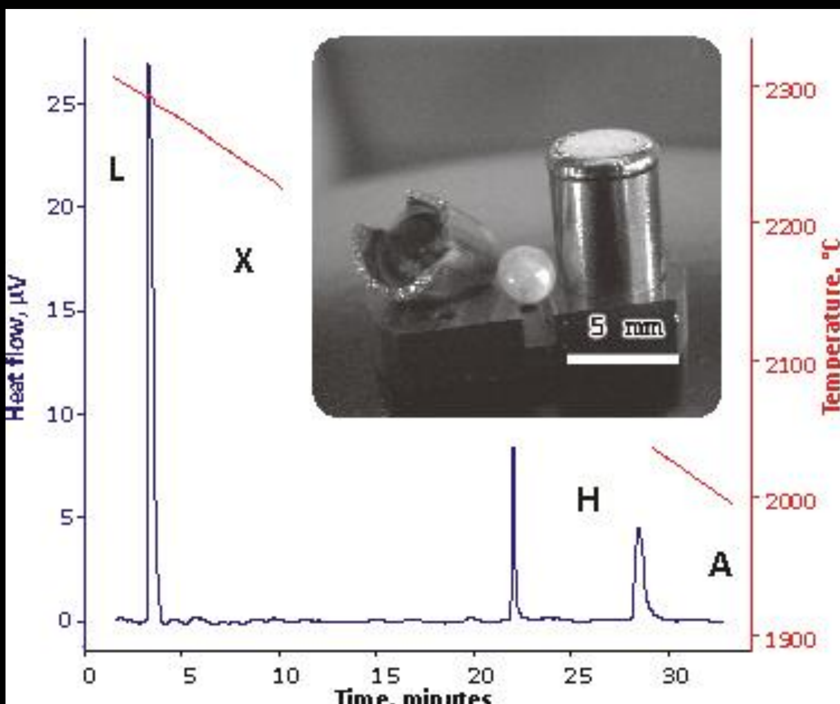
1966 Foex and Traverse

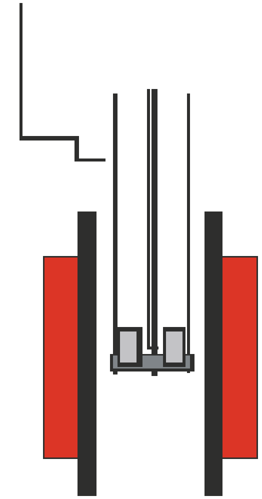
1976 Coutures, Rounet et. al

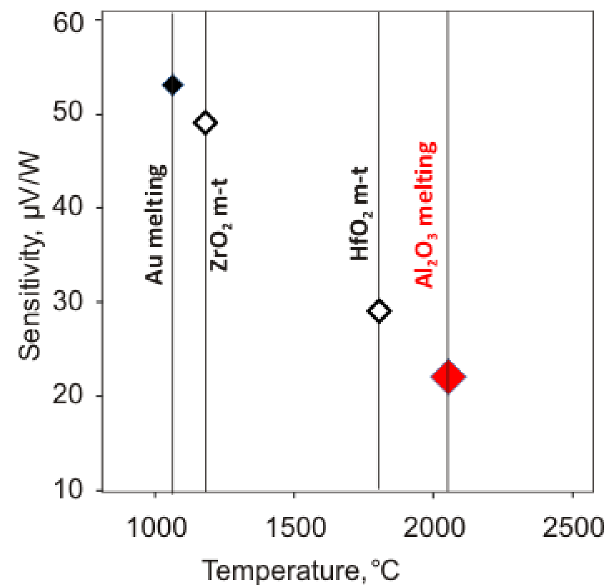
1980s – DTA to 2600 °C

1981 Shevchenko and Lopato

Setaram Setsys TG/DTA 2400

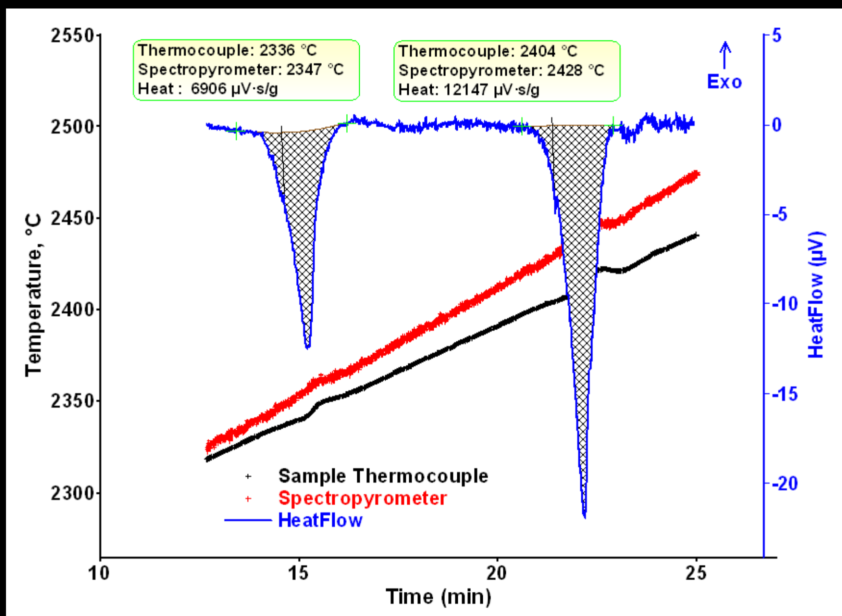




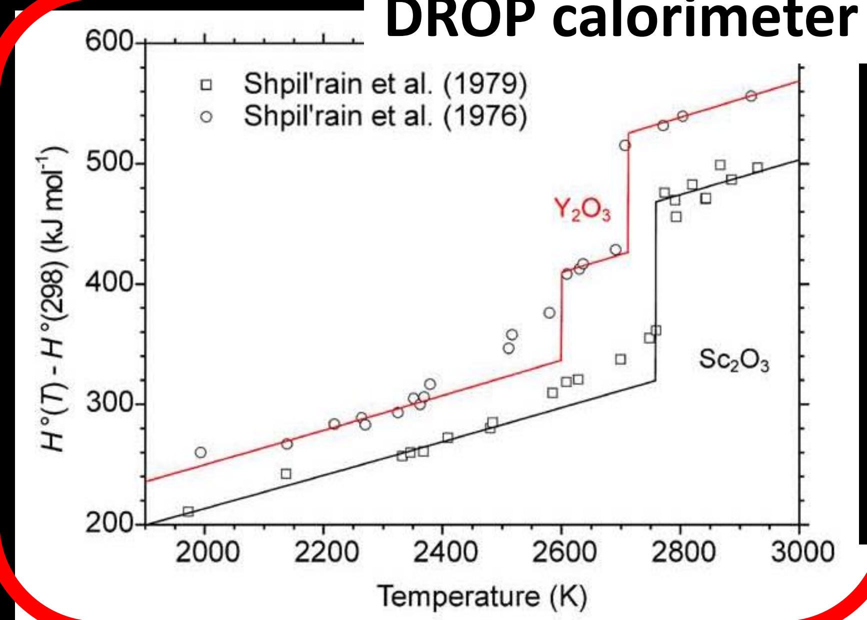


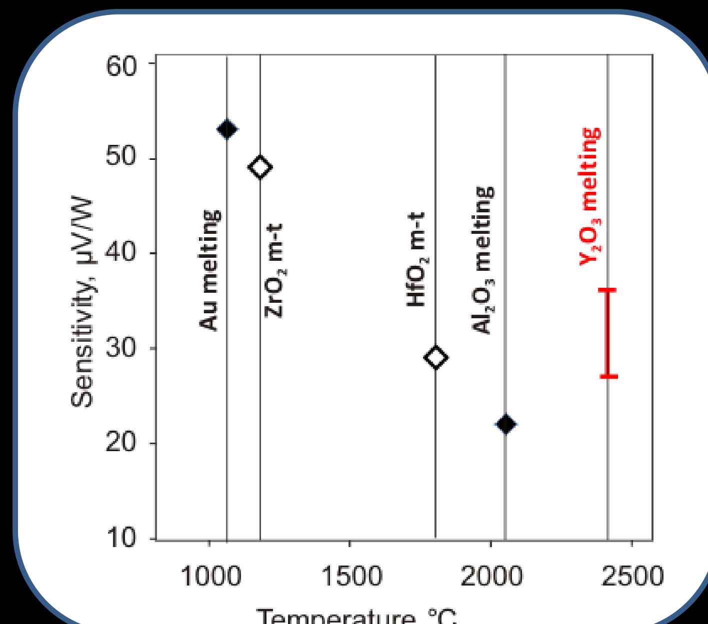
SV Ushakov, A Navrotsky, JMR, 26(7), 845 (2011)

Setaram Setsys TG/DTA 2500



DROP calorimeter





SV Ushakov, A Navrotsky
Journal of the American Ceramic Society 97 (5), 1589-1594

Levitation calorimetry

M.G. Frohberg / Thermochimica Acta 337 (1999) 7–17

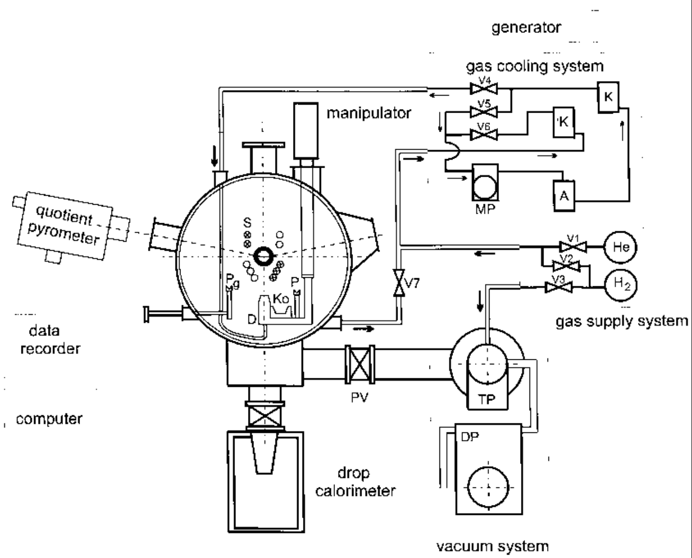


Fig. 1. Levitation drop calorimeter.

Table 1
Overview to drop calorimetric investigations until 1975

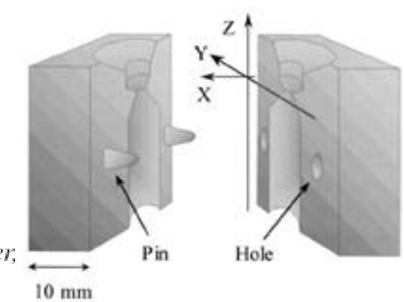
Year	Authors	Element	Temperature range (K)
1970	Chaudhuri et al. [25]	Cu	1386–1887 (l)
1970	Chaudhuri et al. [25]	Pt	2202–2631 (l)
1970	Treverton and Margrave [26]	Mo	2692–3112 (l)
1971	Berezin et al. [27]	Mo	2890–2925 (l)
1971	Treverton and Margrave [28]	Fe	1804–2142 (l)
1971	Treverton and Margrave [28]	Ti	1969–2313 (l)
1971	Treverton and Margrave [28]	V	2205–2638 (l)
1971	Treverton and Margrave [29]	Co	1774–2345 (l)
1971	Treverton and Margrave [29]	Pd	1846–2334 (l)
1972	Sheindlin et al. [30]	Nb	1650–2707 (s)
1972	Berezin et al. [31]	V	2084–2325 (l)
1974	Stretz and Bautista [32]	Y	1799–2360 (l)
1974	Stephens [33]	Cu	1428–2007 (l)
1974	Stephens [33]	U	1428–2398 (l)
1975	Stretz and Bautista [34]	Nd	1446–2246 (l)
1975	Stretz and Bautista [35]	La	1250–2420 (l)

M.G. Frohberg – "Thirty years of levitation calorimetry – a balance" (1999)

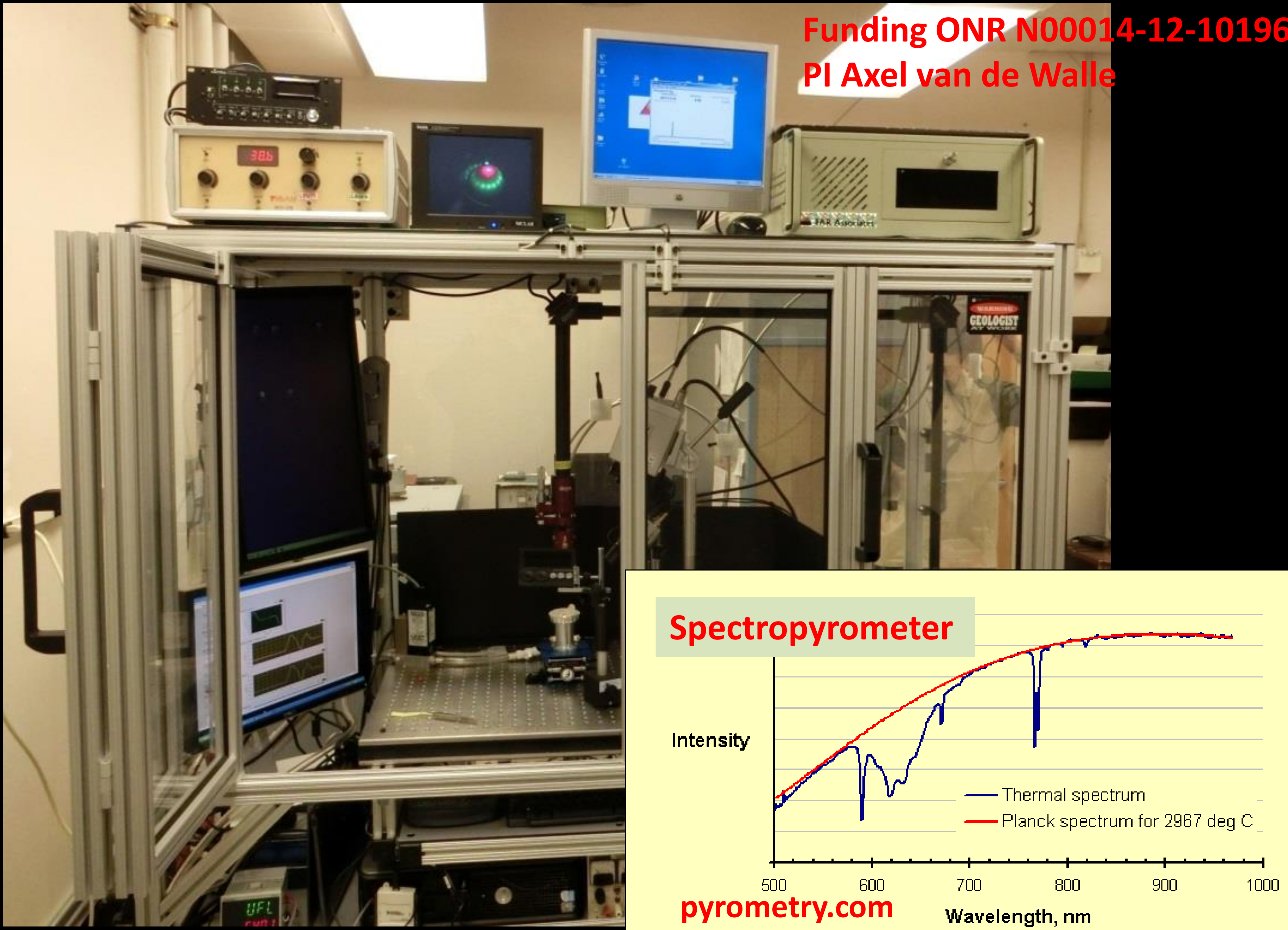
REVIEW OF SCIENTIFIC INSTRUMENTS VOLUME 74, NUMBER 2 FEBRUARY 2003

An aerodynamic levitation system for drop tube and quenching experiments

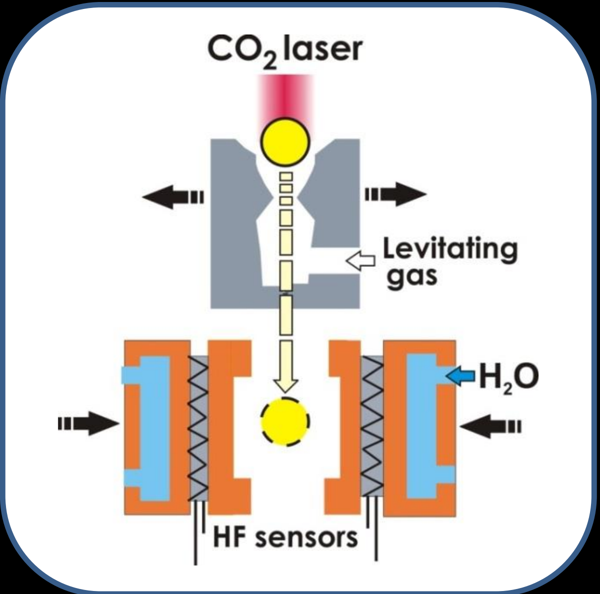
Yasutomo Arai,[a)] Paul-François Paradis, Tomotsugu Aoyama, Takehiko Ishikawa, and Shinichi Yoda
National Space Development Agency of Japan, Space Utilization Research Program, Tsukuba Space Center, 2-1-1 Sengen, Tsukuba City, Ibaraki 305-8505, Japan

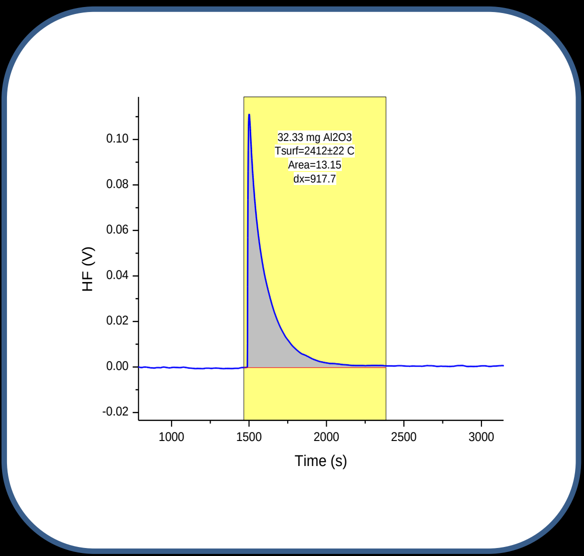


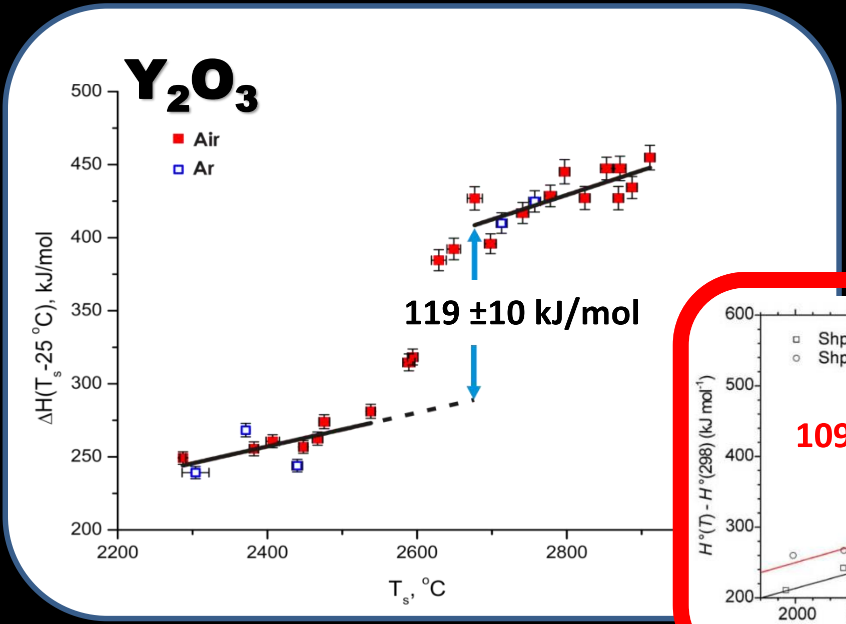
Funding ONR N00014-12-10196
PI Axel van de Walle

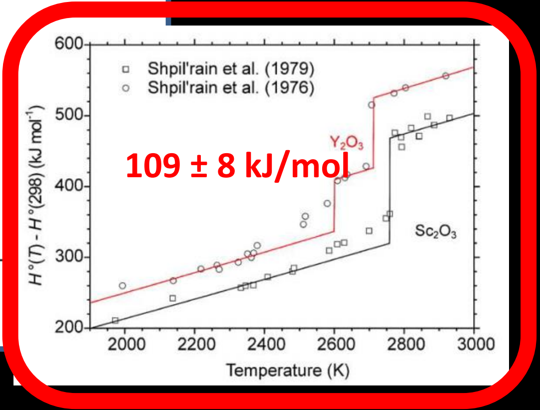


Drop-n-Catch calorimeter









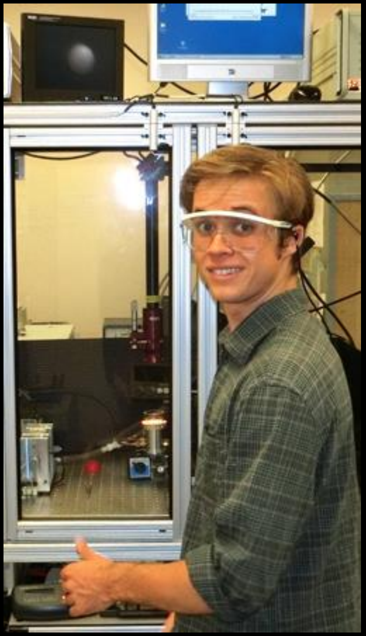
SV Ushakov, A Shvarev, T Alexeev, D Kapush, A Navrotsky, J. Amer. Cer. Soc. 100 (2), 754-760 (2017)

D Kapush, SV Ushakov, A Navrotsky, QJ Hong, H Liu, A van de Walle Acta Materialia 124, 204-209 (2017)

DTA and DnC above 2000 °C: summary

Experimental capabilities for drop calorimetry from above 2000 °C were re-established

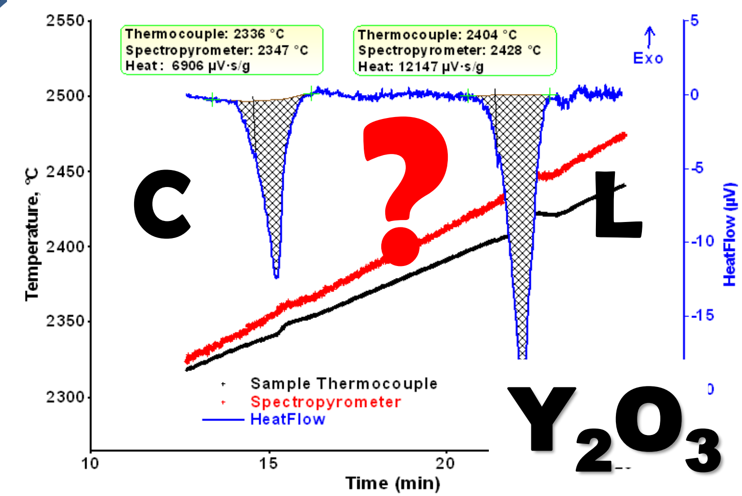
Quantitative DTA is possible to 2400 °C with commercially available instruments

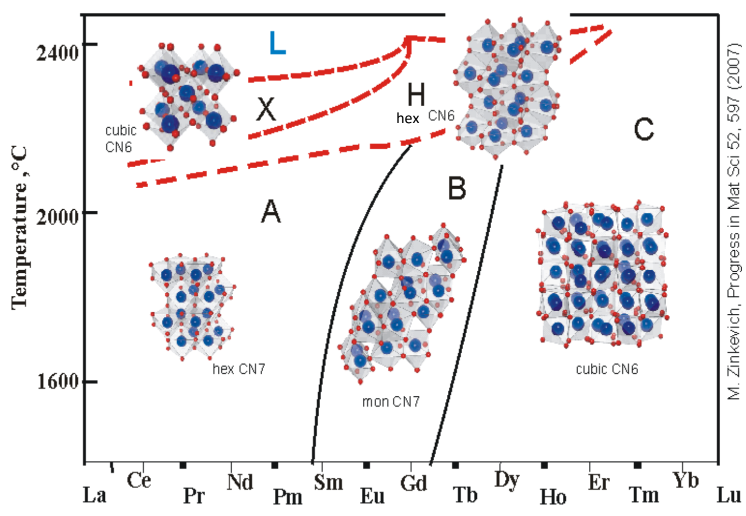


Matthew Fyhrie

Thermodynamic data from high-T diffraction

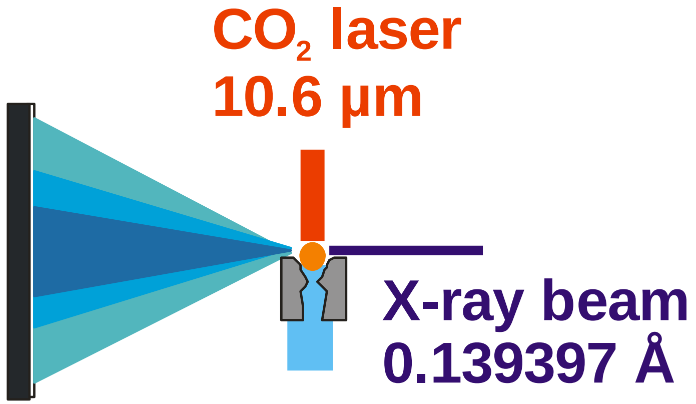
- Molar volumes as a function of temperature
- ΔV for solid state phase transitions
- ΔS_c for order - disorder transitions
- *In situ* phase diagram determination



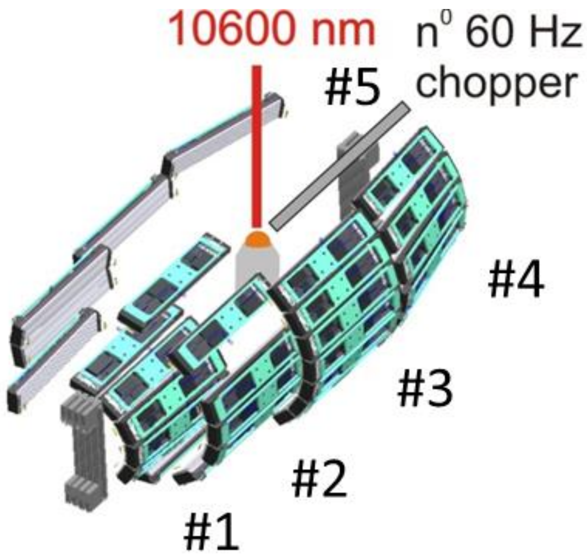


X-ray and neutron diffraction above 2000 C

APS 6-ID-D

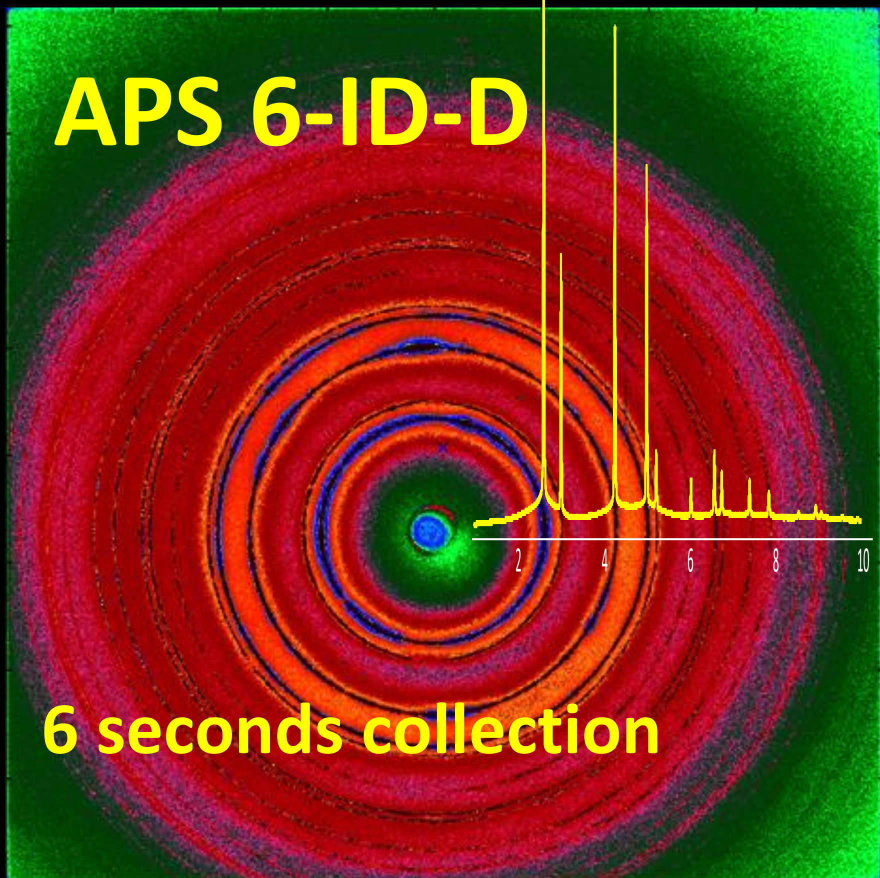


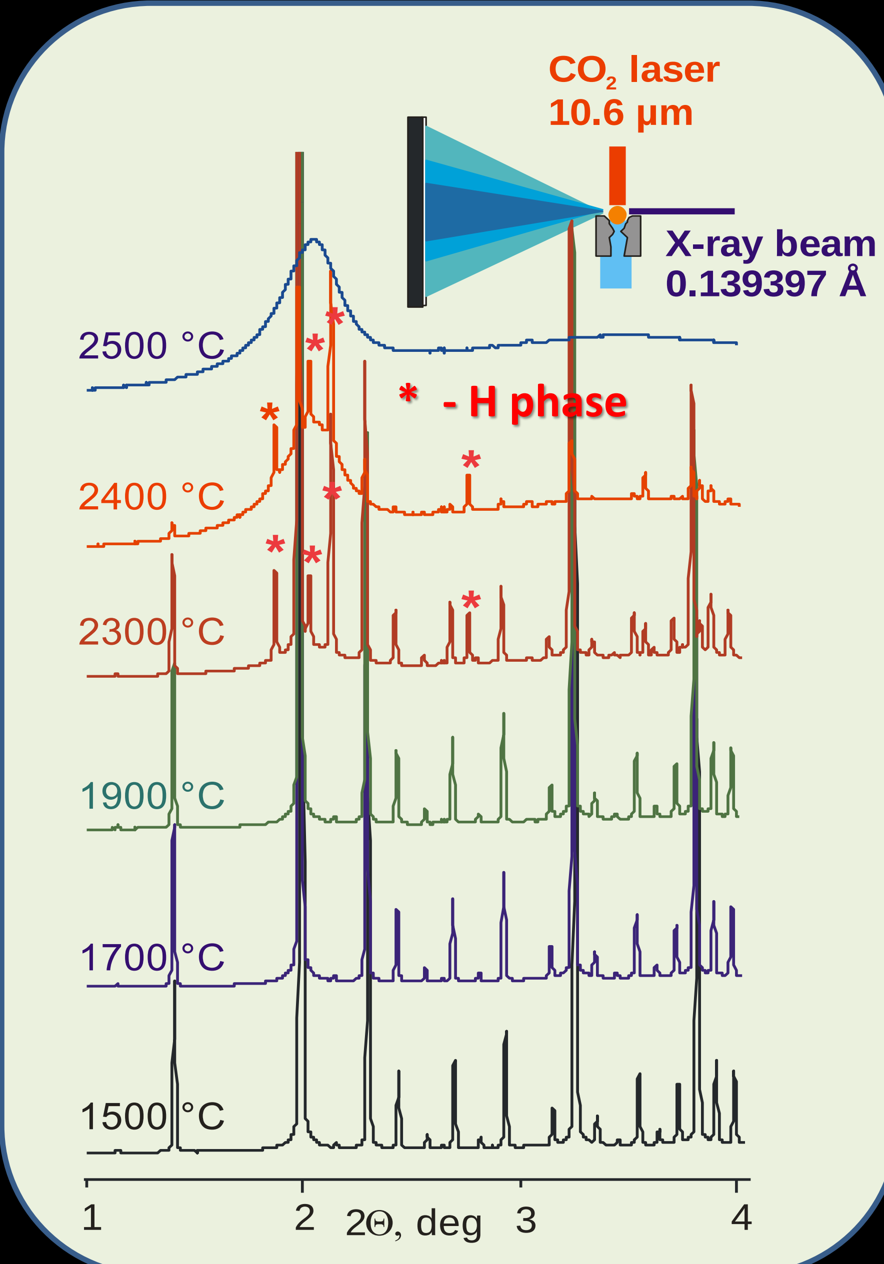
SNS NOMAD BL1B



Chris Benmore Richard Weber Joerg Neuefeind

HT XRD Y_2O_3





Ushakov S.V. & Navrotsky A. (2012) *J. Am. Ceram. Soc.* 95, 1463

Results from 3 days of diffraction experiments on levitator at APS (June 15-17, 2011)

- **Y_2O_3 transforms in H-type before melting in Ar and O_2. Volume reduction on C-H phase transition was refined.**

- **Pyrochlore structure of $La_2Zr_2O_7$ persists to the melting temperature. Thermal expansion was used to validate high temperature *ab initio* calculations.**

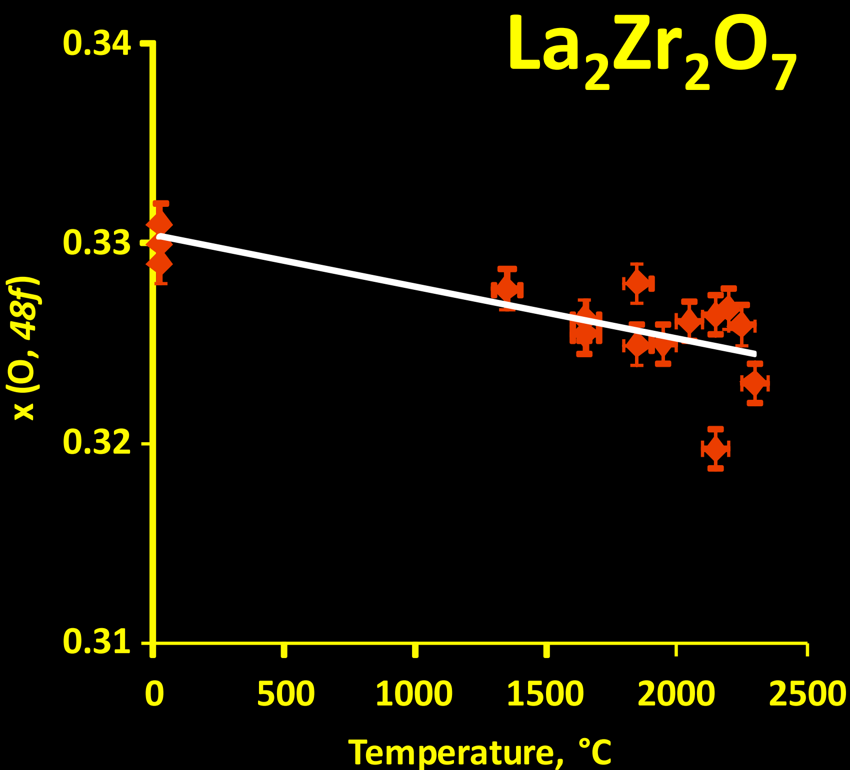
- **Thermal expansion of Eu_2O_3-ZrO_2 DF solid solutions and anti-site occupancies on pyrochlore-DF phase transition in $Eu_2Zr_2O_7$ were refined.**

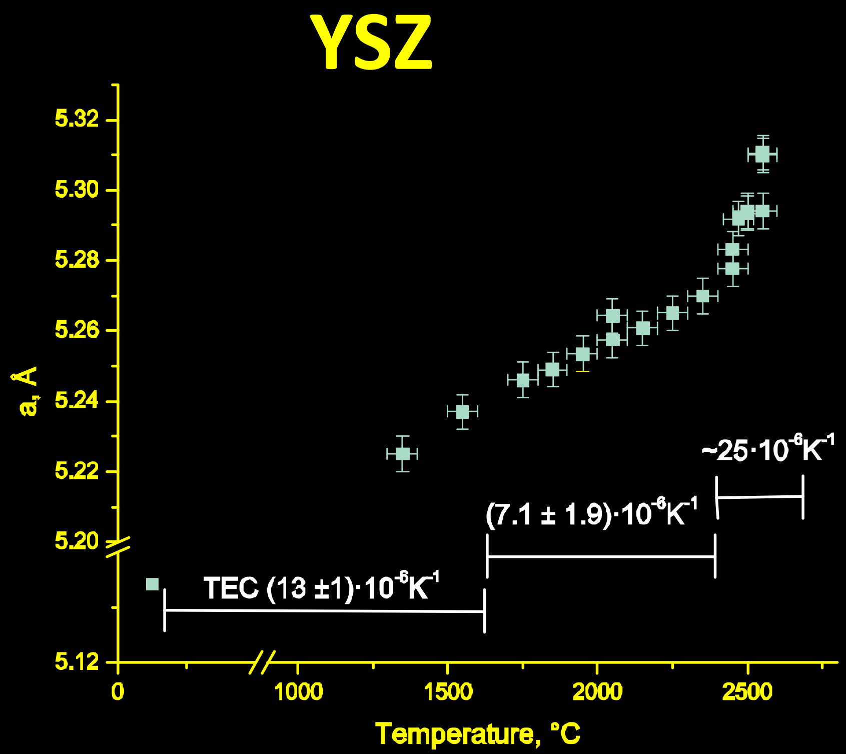
Maram PS, Ushakov SV, Weber RJK, Benmore CJ, & Navrotsky A (2015) *J. Am. Ceram. Soc.* 98 (4), 1292

Hong Q-J, Ushakov SV, Navrotsky A, & van de Walle A (2015) *Acta Mater.* 84, 275

Ushakov SV & Navrotsky A (2012) *J. Am. Ceram. Soc.* 95, 1463

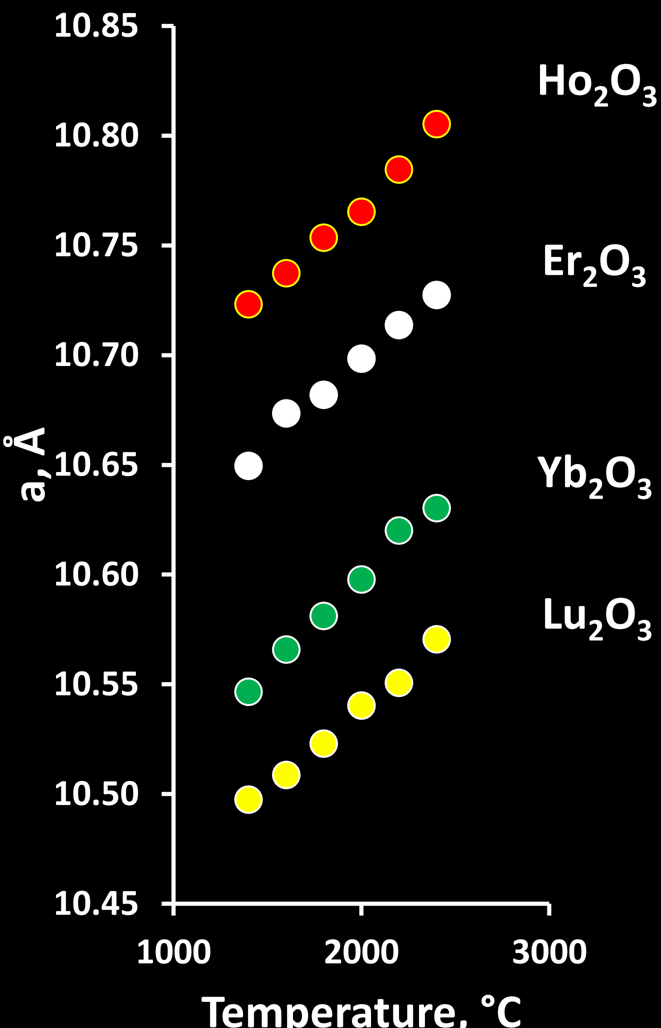
Bredig transition in YSZ from neutron diffraction at SNS, NOMAD (Aug 23-29, 2014)



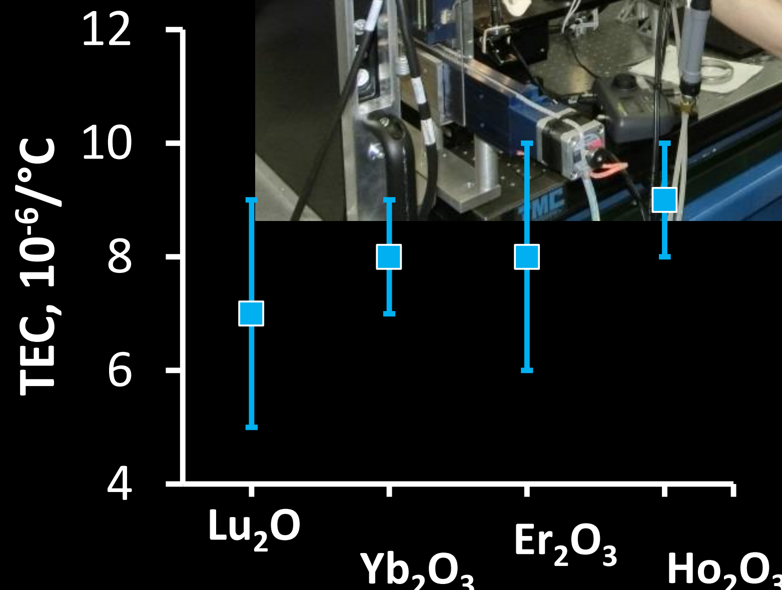


Ushakov, S. V., Navrotsky, A., Weber, R. J. K. and Neuefeind, J. C. J. Am. Ceram. Soc., 98: 3381 (2015),

Thermal expansion of C-type RE_2O_3 in oxygen flow

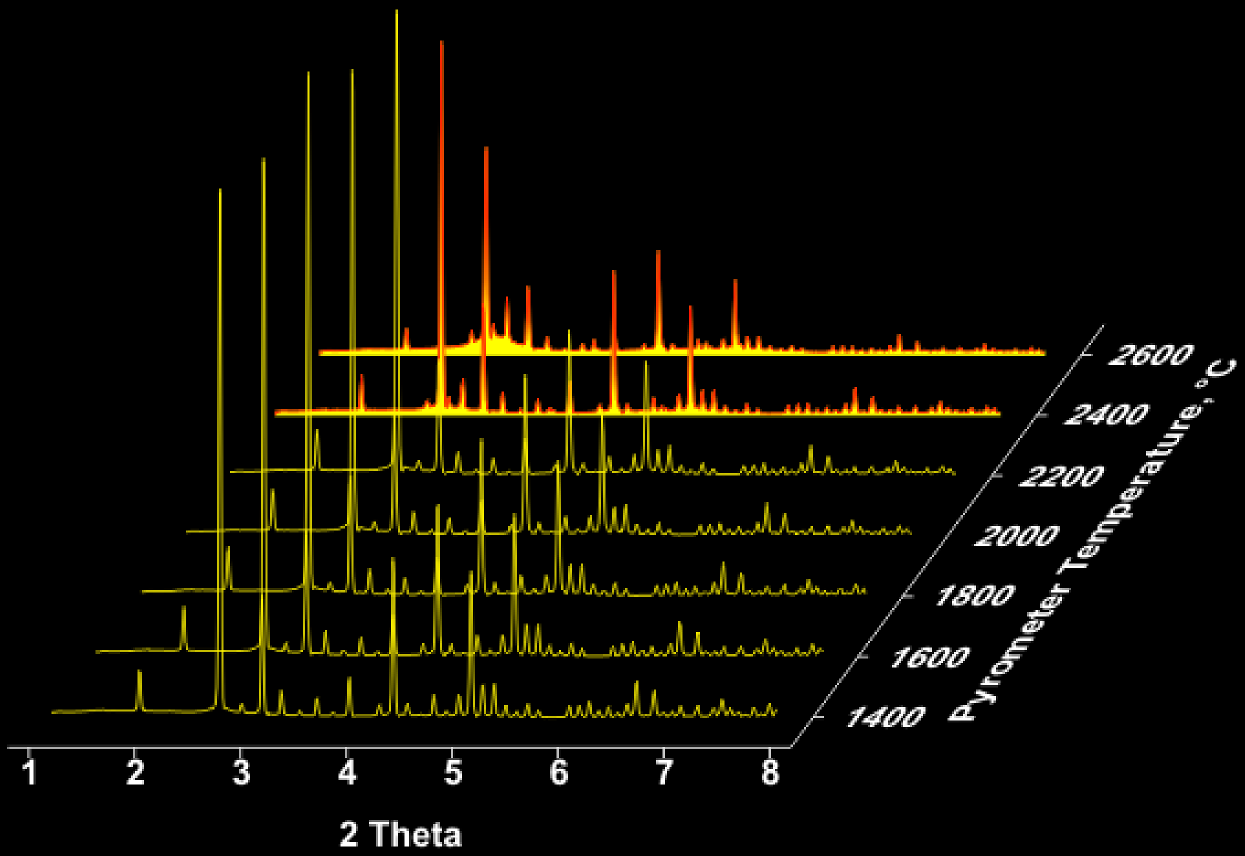


Alfred Pavlik



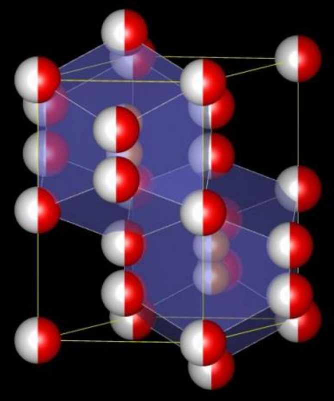
A Pavlik, SV Ushakov, A Navrotsky, CJ Benmore, RJK Weber, Journal of Nuclear Materials, 495 (2017) 385-91

C-H pre-melting phase transitions RE_2O_3



Pre-melting C-H phase transition in Er_2O_3 in Ar

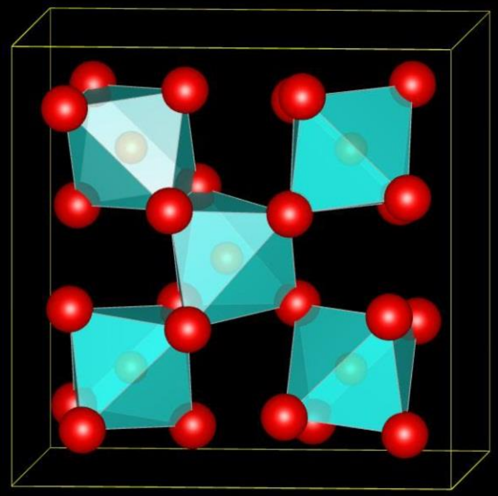
H-type Z=1



Y_2O_3 ΔV -3.1%

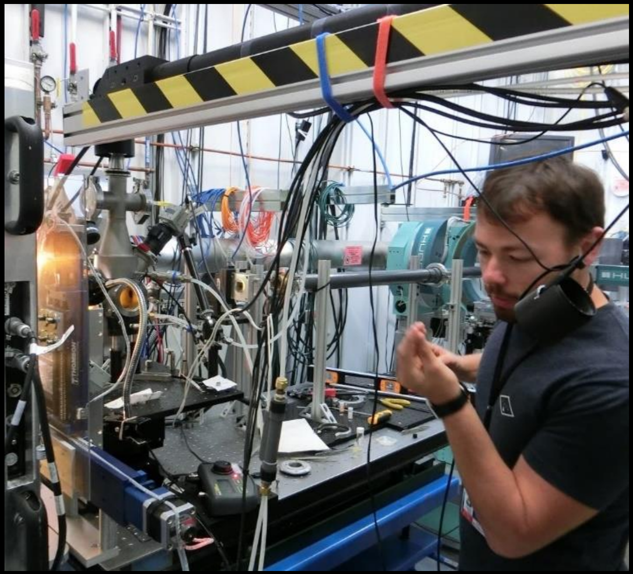
Er_2O_3 ΔV -3.4%

Ho_2O_3 ΔV -3.9%



C-type Z=16

Summary: Structure refinement above 2000 °C is possible using aerodynamic levitators available at APS 6-ID-D and SNS NOMAD



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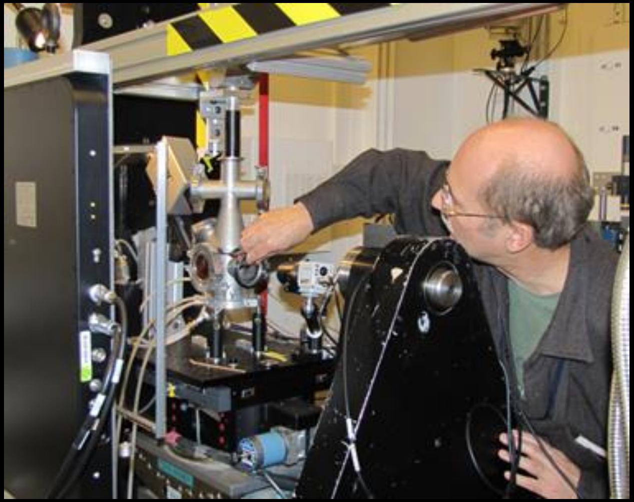

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Qi-Jun Hong, Brown U
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