EXPERIMENTAL TECHNIQUES TO STUDY STRUCTURE AND THERMODYNAMICS AT ULTRA-HIGH TEMPERATURES

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The data on high temperature structure and thermodynamics of refractory oxides, carbides, nitrides, and borides are required for developing and validation of novel thermal protection systems and nuclear fuels. Traditional differential thermal analysis (DTA) is limited by interaction with container and to ~2500 °C by WRe thermocouples. Aerodynamic levitation and laser heating allow structural and thermodynamic measurements on not electrically conductive solid and liquid oxides at temperatures limited only by sample evaporation. Drop calorimetry using aerodynamic levitator with splittable nozzle (Figure 1) was used to obtain first data on fusion enthalpies for ZrO_2, HfO_2, Lu_2O_3, and Yb_2O_3 [1]. The existing aerodynamic levitators in user programs at synchrotron and neutron diffraction facilities in the US and Japan, originally developed for study of structure of oxide melts, can also be used to obtain thermal expansion and structural data on solids to the melting temperatures (Figure 2). Electromagnetic levitation (EML) has been employed for more than 50 years for measurements of high temperature thermodynamic and thermophysical properties of metals [2-4]. The bulk Joule heating, absence of cooling by levitation gas, and sample access, provides inherent advantages for EML application to measurements on electrically conductive carbides (Figure 3), nitrides, and borides; however it was not employed until now for these key components of ultra-high temperature ceramics.

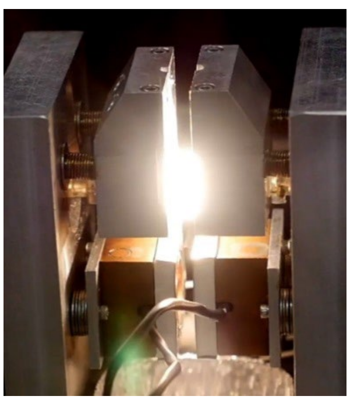

Figure 1. Drop calorimetry experiment on Er_2O_3 sample (~2.5 mm in diameter)

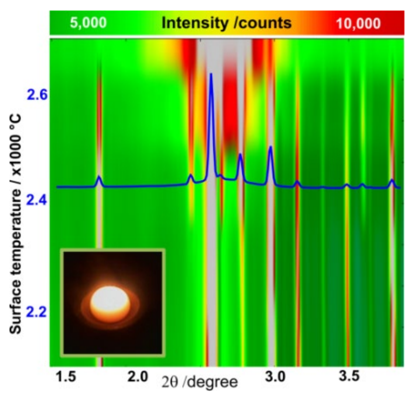

Figure 2. Cubic to hexagonal (C-H) phase transformation in Er_2O_3 above 2000 °C from synchrotron diffraction on levitated laser heated samples.

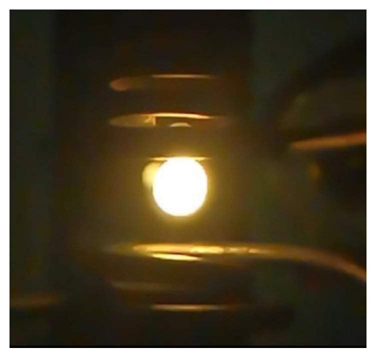

Figure 3. Electromagnetically heated and levitated solid ZrC pellet (~5mm in diameter).

References

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