An Improved, Compact High Temperature Sample Furnace for X-ray Powder Diffraction

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Abstract

A compact sample furnace was designed and tested at the X-ray Powder Diffraction (XPD) beamline at NSLS-II to heat samples to 2000 - 2300°C for diffraction data collection. Since these temperatures weren't reached, engineering studies as described herein were undertaken to improve performance.

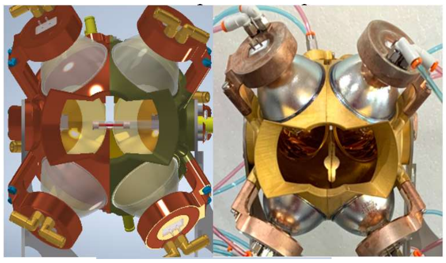

Figure 1: XPD Infrared Sample Furnace looking upstream (a) rendering (b) photo

Introduction

The XPD Sample Furnace uses infrared lamps with ellipsoidal reflectors to focus heat rays at samples. Seven ways to improve furnace performance were considered:

a. Use IR lenses to focus forward-directed IR rays otherwise lost as halo.
b. Improve sample tube holder design
c. Use high-temperature coatings/films to insulate and improve heat transfer.
d. Design a secondary reflector to capture forward-directed IR rays.
e. Use a CO_2 laser for supplementary (or primary) heating
f. Use an inert gas to prevent oxidation.
g. Optimize heat lamp selection/position.

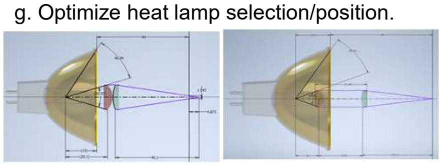

Figure 2: IR lenses with focus (a) behind sample and (b) at sample center

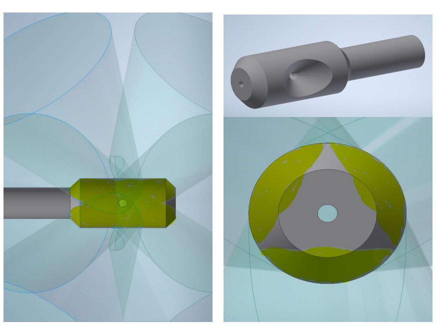

Figure 3: Redesigned sample tube holder (a) looking downstream, (b) end view with heat flux pattern in yellow, and (c) view looking upstream with ±45° exit opening to collect diffraction data

Methodology

Seven possible solutions noted in the introduction were explored theoretically for viability. Based on these results, Lab tests were then undertaken to determine the best combination of solutions to improve furnace performance.

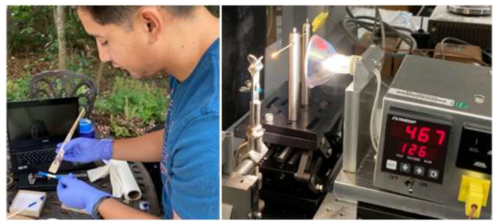

Figure 4: (a) Applying ceramic coatings and (b) testing ceramic materials and IR lamps

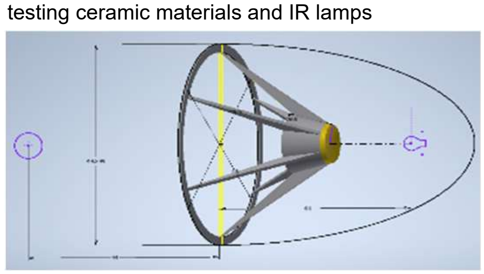

Figure 5: Design of a secondary spherical reflector to capture forward-directed IR rays

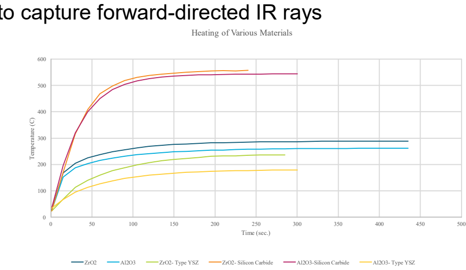

Figure 6: Heating Curves for Various Materials and Coatings

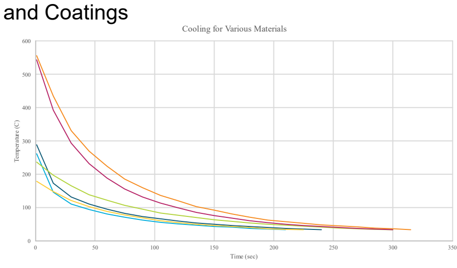

Figure 7: Cooling Curves for Various Materials and Coatings

Gas	Specific Heat Capacity (KJ/kg K)	Max Temp Achieved (˚C)
Helium	5.1926	185
Argon	0.5203	258
Air	1.039	242
Nitrogen	1.005	243

Table 1: Specific Heat and Maximum temperature achieved for various gases

Conclusions

Material research in air >1750°C requires careful sample holder material selection and an understanding material properties at high temperatures. Refractory metals undergo oxidation, high-temperature ceramics are difficult to machine, and thermal absorptivity and emissivity are wavelength dependent when using infrared heat sources. Some materials (e.g. sapphire) are mostly transparent to near infrared rays, and many ceramics are poor heat conductors. Cost is also a consideration. The conclusions obtained are:

- the authors were not able to improve furnace performance with infrared lenses
- a redesigned sample holder improves heat absorption, provides a short heat conduction path to samples, and reduces convective heat transfer losses
- high-temperature coatings worked well, but coating adherence and maximum coating temperature need further improvement
- secondary reflectors diminish lamp life with insufficient thermal gain
- CO_2 lasers (~10µm wavelength) can be used as primary or secondary heat sources, but tests were not yet undertaken
- inert gases helps prevent high-temperature oxidation; specifically, Ar gas has much promise
- infrared lamp position adjustments showed temperature improvements

Solutions chosen for implementation include items b, c, and g (and maybe f).

Acknowledgements

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