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Distribution of this document is not restricted.

13. SUPPLEMENTARY NOTES

14. ABSTRACT
An ultrahigh temperature laser flash thermal properties analyzer was acquired by the Missouri University of Science and Technology (formerly the University of Missouri-Rolla). The analyzer consists of a remote controlled Class I neodymium glass laser; furnace modules that are complete with their own power supplies, temperature controllers, and temperature measurement cells, for several temperature ranges; sample holders; auxiliary modules capable of vacuum, high vacuum, and gas purging; a data acquisition system; and software. The analyzer is capable of determining thermal diffusivity, heat capacity, and thermal conductivity at temperatures from cryogenic (-180°C) to ultrahigh temperature (2800°C) in inert or reducing atmospheres. The system is located in a laboratory that is available to users from the university or from companies, research laboratories, or other universities.

15. SUBJECT TERMS
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Objectives

The objective of this project was to acquire a state-of-the-art thermal properties analyzer capable of determining thermal diffusivity, heat capacity, and thermal conductivity at temperatures from cryogenic (-180°C) to ultrahigh temperature (2800°C) in inert or reducing atmospheres.

Status of the Effort

This is the final project report for the effort. At the close of the project, a commercial laser flash thermal properties analyzer has been acquired and installed. The analyzer system includes a computer controlled laser pulse system; furnace modules complete with their own power supply, temperature controller, and temperature measurement cell; a sample holder; a vacuum pump/gage module; a data acquisition system; and software. The system is capable of measuring thermal diffusivity to ±3% or better accuracy from 10 to 0.001 cm^2/s and heat capacity to ±5% accuracy, allowing the determination of thermal conductivity from 0.1 to 2000 W/m-K. The system has been located in a laboratory facility at the Missouri University of Science and Technology (Missouri S&T; formerly known as the University of Missouri-Rolla) where it has been made available to other researchers at Missouri S&T, other universities, and companies through the Missouri S&T Graduate Center for Materials Research (MRC).

Accomplishments

The primary objective of this effort has been accomplished. The laser flash thermal properties analyzer has been installed and all of the components are working. The system has been used to collect thermal property test data for a variety of material systems. The sections that follow describe the overall laser flash analyzer, with separate descriptions of the laser pulse system, the furnace modules, and the data acquisition system. Finally, there is also a section that includes some of the results that have been collected using the analyzer.

A. Laser Flash Thermal Properties Analyzer

A laser flash thermal properties analyzer (Anter Corp., FLASHLINE™ 5000) was purchased and installed. The analyzer consists of a computer controlled laser pulse system; furnace modules complete with their own power supply, temperature controller, and temperature measurement cell; a sample holder; a vacuum pump/gage module; a data acquisition system; and software (Figure 1). The system meets all specifications for ASTM Standard E1461. Since its installation, the system has been used for testing the thermal diffusivity of ceramic-based samples from room temperature to 2000°C.

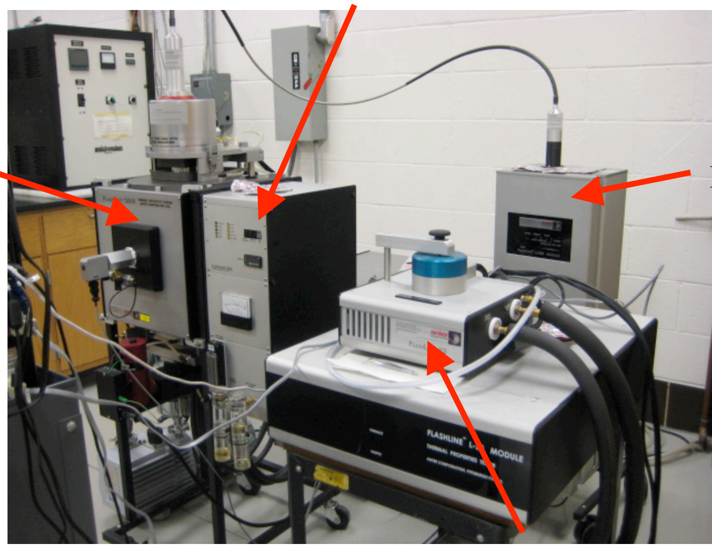

Figure 1. The Anter FLASHLINE™ 5000 laser flash thermal properties analyzer that was purchased with DURIP funds showing the laser pulse system, furnace modules, and data acquisition system.

B. Laser Pulse System

The FLASHLINE™ 5000 uses a laser to irradiate a small, disc-shaped specimen over its front face with a very short pulse of energy. A fast response optical pyrometer or thermocouple, depending on the furnace module being used, records the temperature of the rear face of the specimen as a function of time just after the laser pulse is applied. Thermal diffusivity is determined from the rise time of the thermal pulse at the specimen's rear face to reach half of its ultimate temperature rise. The laser pulse system consists of remote-controlled Class I Nd: glass laser (~35 joules maximum power), a fiber optic laser power delivery wand with safety interlocks to attach it to the separate furnace modules, a fiber optic link that can be used to shape the laser pulse, a pulse shape mapping circuit, a built-in laser alignment device, and a liquid nitrogen cooled InSb detector. The laser system stands ~1.5 m tall and has a footprint of nominally 0.7 m by 0.7 m (Figure 2).

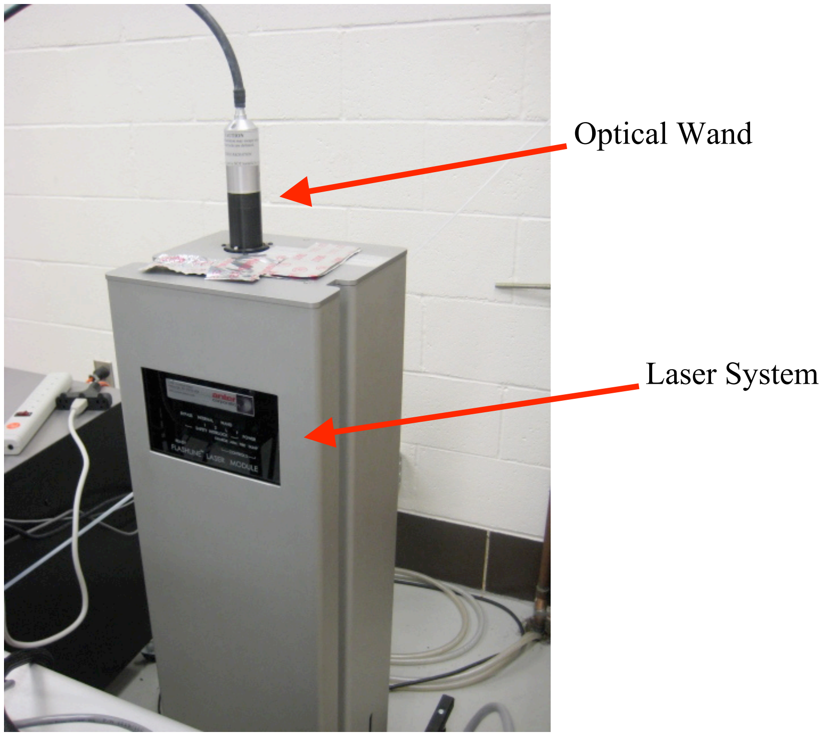

Figure 2.   The Anter FLASHLINE™ 5000 laser pulse system.

C. Furnace Modules

Purchased with the system were two furnace modules with operating ranges from -150°C to 200°C, and RT to 2800°C. Each furnace module is self-contained to include its own power supply, temperature control module, over temperature controls, safety interlocks, etc. Also included with each furnace module is a thermocouple or optical pyrometer for solid-state temperature readings. Details regarding the furnace modules and their temperature ranges are listed below:

- *Cryogenic Module* (-120°C to 200°C) – single specimen operation, uses a built-in contact type thermocouple for temperature sensing, dry inert gas or dry air purge required, and liquid nitrogen cooling (Figure 3).
- *Ultra High Temperature Graphite Furnace* (RT to 2800°C) – six specimen carousel, either inert gas purge or vacuum (10^{-3} torr) atmosphere, a proprietary optical pyrometer monitors the furnace temperature (Figure 4).

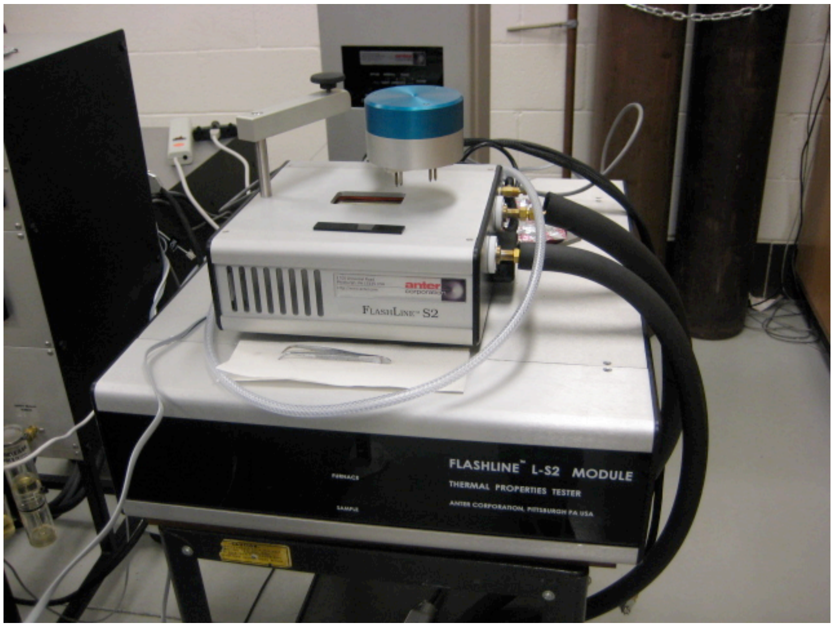

Figure 3. The Anter FLASHLINE™ 5000 cryogenic module.

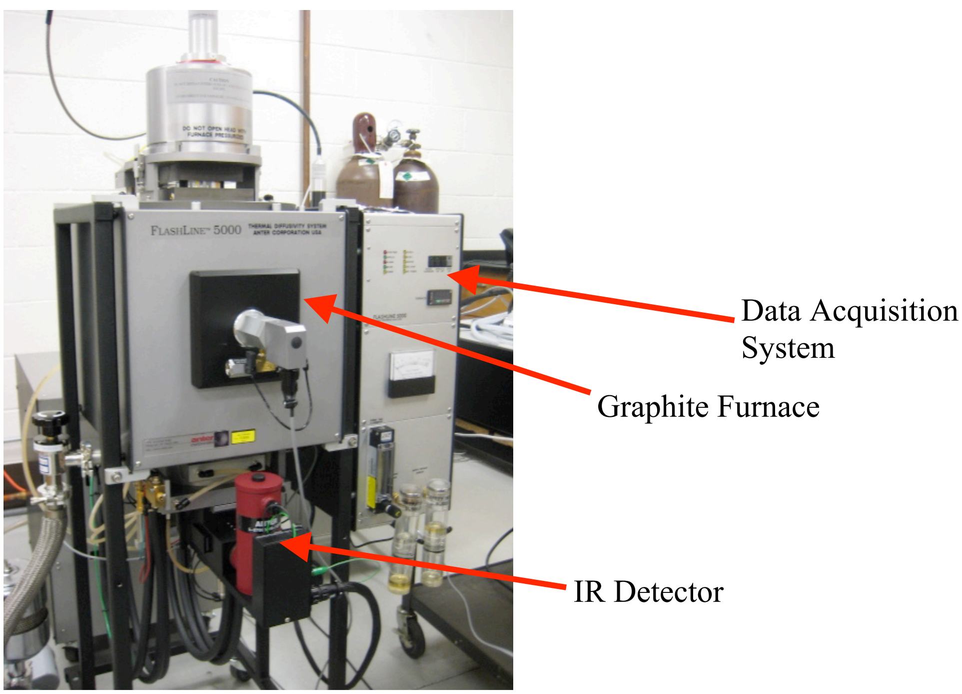

Figure 4. The Anter FLASHLINE™ 5000 ultrahigh temperature furnace module.

D. Data Acquisition System

The data acquisition system that was supplied with the thermal diffusivity unit can acquire > 15,000 data points per second. This capability allows for thermal diffusivity to be calculated with accuracy to 10^{-3} cm^2/sec. The software uses several common models to calculate thermal diffusivity including Clark and Taylor, Degiovanni, Cowan, etc. Heat capacity can also be measured based on temperature rise compared to that of a known standard, graphite for this instrument. Using the measured thermal diffusivity and heat capacity the system calculates thermal conductivity (accurate to 0.1 W/m•K).

E. Examples of Test Data

The new thermal property measurement instrument is currently available to users from Missouri S&T. The Graduate Center for Materials Research at Missouri S&T has staff that coordinate the administrative (sign-up, billing, etc.) and logistic (routine maintenance, repair, upgrades, etc.) aspects of not only the laser flash thermal property instrument, but a wide range of materials characterization equipment including mechanical testing equipment, x-ray diffractometers, electron microscopes, and surface characterization equipment. The system has been used for several types of projects at Missouri S&T. Some of the projects that have made use of the equipment are summarized in Table 1. Specific properties that have studied involve thermal conductivity measurements to evaluate solid solution effects and thermal diffusivity changes as a result of diffusional mechanisms. Examples of the types of test data that have been collected and analyzed are shown in Figures 5-7.

Table 1. Summary of thermal property testing accomplished using Anter FL5000 system.

Project	Max Temperature	Funding Agency
ZrB_2	2000°C	National Science Foundation
ZrB_2	2000°C	Air Force Office of Scientific Research
$MgAl_2O_4$	1100°C	Department of Energy

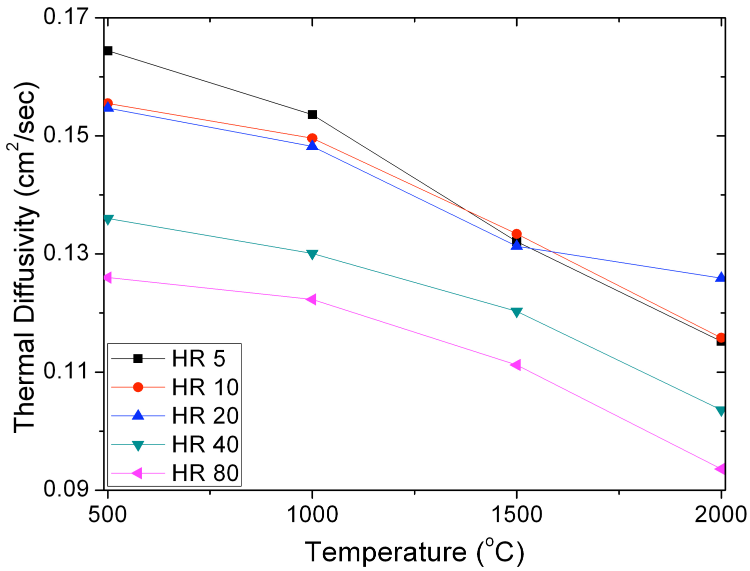

Figure 5. Thermal diffusivity measured as a function of temperature for specimens that were hot pressed using heating rates ranging from 5°C/min to 80°C/min.

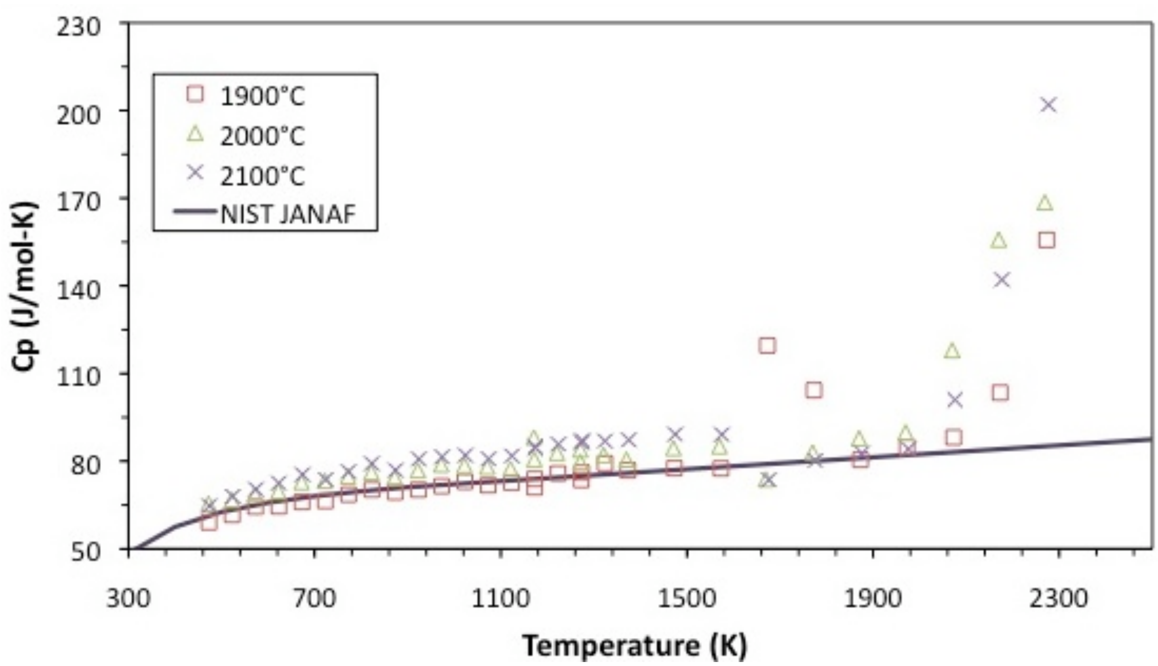

Figure 6. Heat capacity measured as a function of temperature showing a similar trend with varying hot pressing temperature. However, a small increase with higher hot pressing temperatures has been observed.

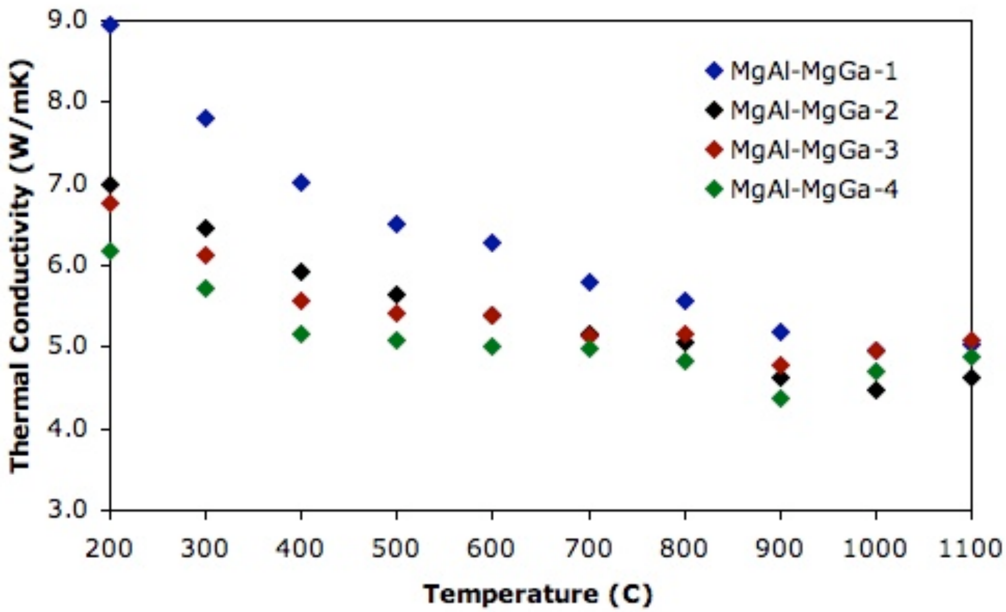

Figure 7. Thermal conductivity calculated from measured thermal diffusivity and heat capacity as a function of temperature showing that with increased solid solution content (5% to 50%), thermal conductivity decreased.

Personnel Supported

No program funds were expended for salary or travel.

Publications

At this point, this project has not lead to work that has been published.

Interactions/Transitions

A. Conferences and meetings

Results obtained with the new test system have been presented at several technical meetings. A list of presentations to date that have used data collected with the newly acquired thermal diffusivity test system is provided below.

1. K. O'Hara, J. D. Smith, J. Hemrick, "Solid Solution Effects on the MgAl2O4 System," UNITCER, October 2009, Salvador Bahia, Brazil.
2. M.J. Thompson, W.G. Fahrenholtz, and G.E. Hilmas, "Effects of Heating Rate and Carbon Content on Mechanical and Thermal Properties of Zirconium Diboride" an oral presentation at the 34th International Conference and Exhibition on Advanced Ceramics and Composites, January 28, 2010, Daytona Beach, FL.

3. W.G. Fahrenholtz and G.E. Hilmas, "Fundamental Thermal and Mechanical Properties of Boride Ceramics," AFOSR High Temperature Aerospace Materials Contractors Review, February 1-5, 2010, Arlington, VA.

B. Consultative and advisory

None to report.

C. Transitions

None to report.

New Discoveries, Inventions, Patent Disclosures

None to report.

Honors/Awards

None to report.