

# Multiple Station Thermal Diffusivity Instrument

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## **ABSTRACT**

A multiple furnace laser flash thermal diffusivity system has been developed. The system is equipped with a movable Nd:Glass laser unit, two IR detectors and four furnaces for precise measurements of thermal diffusivity over the temperature range from  $-150^{\circ}\text{C}$  to  $2500^{\circ}\text{C}$ . All furnaces can operate in vacuum and inert gas; the environmental effects furnace also supports oxidizing and reducing environments. To increase testing speed the graphite and aluminum furnaces are both equipped with six-sample carousels. Thermal diffusivity measurements of three standard reference materials show excellent results over the entire temperature range.

## **I. INTRODUCTION**

Since its introduction in 1961[1], the laser flash technique has become a standard testing method for thermal diffusivity measurements of solids [2,3]. Traditionally, a single furnace is used for a certain temperature range and a few classes of materials.

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The rapid development of new materials often requires a laser flash system to cover a wider temperature range and to operate with different environments. This is particularly true for materials research and development. However, it is virtually impossible to find a single furnace type that can service the entire temperature domain of practical use, and to accommodate every material to be tested. Throughput in testing is a paramount factor even for fully automated systems.

The system which is the subject of this article is based on a traversing laser, multiple furnaces and two photovoltaic IR detectors. To suit a variety of testing requirements, the system is equipped with an ultra-high temperature graphite furnace (500°C to 2500°C), a high temperature Kanthal Super™ furnace (RT to 1700°C), a low temperature aluminum Monoblock™ furnace (-150°C to 500°C), and a high speed quench furnace (RT to 1200°C). It also provides the ability to add to and/or reconfigure an existing system as needs change. High throughput leads to lower unit cost per test and higher level of utilization. Although the laser flash technique is much faster than static methods, a single sample test over a moderate temperature range could still take a whole day in traditional systems. Concurrent testing of multiple samples was made available on this system. The graphite and aluminum furnaces are both equipped with six-sample carousels. They provide an opportunity to study five or six specimens concurrently under completely identical conditions.

The purpose of this paper is to summarize the system capabilities and report results for tests on standard reference specimens. Armco iron, stainless steel and AXM-5Q graphite were tested and compared with data in the literature. The tests showed excellent results over the entire temperature range.

## **II. Laser Flash Thermal Diffusivity System**

### **Ultra High Temperature Subsystem**

A schematic of the system is shown in Fig.1. The ultra-high temperature graphite furnace is designed to operate from 500°C to 2500°C in inert gas environment and up to 2000°C in vacuum. It employs a water cooled stainless steel outer shell, molded fibrous carbon insulation, and pyrolytic graphite reflector inside. The graphite heating element is free standing with both power connectors on the bottom. The sample support structure, also of graphite, is an integral part of the furnace. A precision slide mechanism allows the sample holder to be lifted out, co-

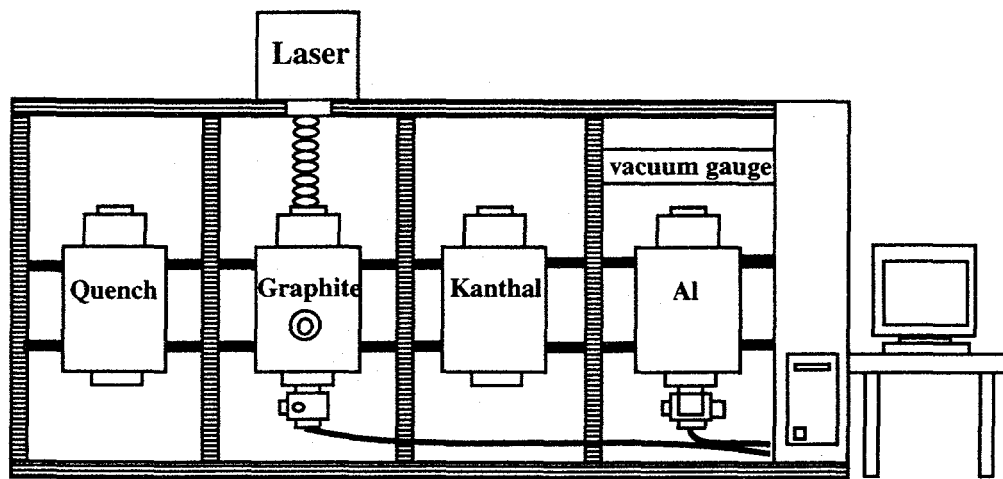


Figure 1. The thermal diffusivity testing system.

linearly with the optical axis. Proper closure is ensured with a sliding seal that engages without a jolt to the sample. A graphite multi-sample carousel allows up to six samples to be loaded and tested during one run. The sample holder is located in the uniform temperature zone of the furnace, and the temperature of the sample holder is monitored by a one-color optical pyrometer.

### High Temperature Subsystem

The high temperature Kanthal Super™ furnace is designed to operate between room temperature and 1700°C. The furnace cavity has a 4 inch long hot zone ensuring excellent uniformity and stability in the sample region. The high purity alumina muffle and sample support components allow operation in high vacuum, ( $10^{-6}$  torr), oxidizing and reducing atmospheres. The alumina muffle also prevents the Kanthal elements from direct exposure to the operating gas. The shell is air-cooled and is no more than warm to the touch at maximum operating temperature. Temperature control and sensing is via Type S thermocouples, one close to the heaters and another adjacent to the sample. To operate with hydrogen atmosphere, special valves and a built-in hydrogen sensor were incorporated into the gas controls to monitor the muffle and the furnace cavity in case the muffle tube cracks. The alumina sample holder is configured for a single sample. With judicious use of

opaque shields in the sample holder, the shine-by of the laser beam due to transparency of alumina is kept at an acceptable level.

### **Moderate Temperature Subsystem**

The operating range of the low temperature aluminum Monoblock™ furnace is from -150°C to 500°C. The design allows the furnace to run in high vacuum, ( $10^{-6}$  torr) and inert gas environment. Measurement below room temperature is made possible by introducing liquid nitrogen to cooling channels within the furnace block. The sample holder is a six-sample carousel for multiple testing. It is made out of aluminum and stainless steel and has similar design to the graphite furnace carousel.

### **Quench Furnace**

The fourth furnace is a high speed quench furnace. The radiantly heated sample holder structure will be operable between ambient and 1200°C. Cooling is provided by a blast of helium once the heat source is turned off. Through various timed and tracked signals, the lower quenched temperature can be programmed. The furnace is designed to measure the thermal diffusivity of a sample at a certain high temperature and then quench the sample at about 200°C per second to a secondary temperature to test it again without first having to go ambient. This furnace will be installed and tested during the first quarter of 1996.

### **Optical Subsystems**

Laser Unit: The laser unit has a Nd:Glass laser with pulse energy up to 35 Joule. The laser head can be moved along a rigid rail above each of the four furnaces. The front face of the laser is equipped with a full complement of key-locked manual controls for diagnostic testing. An interlocked flexible shield connects the laser and the furnace. Full locking is checked by actuated safety switches before firing may occur. Once the beam paths are shielded and interlocked, Class 1 operation is ensured and thus there is no need for safety goggles in the vicinity. The laser power can be varied by changing either the charging voltage (1200 to 2800V) or the number of high voltage capacitors in parallel connection (1 to 5). The pulse-width may be adjusted by varying the number of parallel capacitors. In addition, the pulse-width may be stretched by the addition of inductors in the discharge circuit. Several optical components were used to aid the alignment of the laser. A helium/neon alignment laser is located on the laser rail in the same housing. It is used for the

alignment of the primary laser, and during thermal diffusivity tests to confirm the presence of a sample in the sample holder.

Detection System: Two different photovoltaic detectors are incorporated into the system for versatility. They are mounted underneath the furnaces and monitor the temperature change of the rear surface of the specimen. A high temperature silicon photodiode detector with adjacent preamplifier is shared by the graphite and Kanthal furnaces. Its properties are well suited for use with wavelengths common in high temperature testing. A fiber optic link connects the furnace port and the detector, located 6 feet away. A cryogenically cooled InSb detector with built-in preamplifier is designed for the low temperature aluminum furnace.

A secondary detector subsystem is incorporated into the design to determine the exact shape of the laser pulse. The system maps every laser shot to obtain the precise pulse shape and apply the various pulse width corrections.

### **Other Subsystems**

The vacuum system consists of a mechanical roughing pump and a turbo molecular pump for high vacuum. All the furnaces share the same vacuum system. In practical use, only one furnace at a time is under vacuum.

Purging gas may be directed to an individual furnace during testing. Two-stage bubblers are used for the exhaust of all the furnaces. The bubbler has an oil reservoir located at the bottom of the assembly and a second reservoir located directly above the first. The design has a ball seal and isolation valve to prevent any air from being pulled into the system during vacuum operation or rapid cool down.

The power supply for the system consists of a water cooled step-down transformer controlled by a low noise controller. Current limiting, water flow sensing, etc., are fully implemented.

### **Samples**

The system is designed to adapt to a variety of samples. Typical samples are disk-shaped or square-shaped, 6 to 12.5mm in diameter and 1 to 6mm thick. In addition to bulk specimens, powder and molten metal samples can also be tested. Special containment capsules of quartz or sapphire, are used to test materials through the melt.

### III. OPERATING SOFTWARE AND DATA ANALYSIS

The operation of the system is fully computer controlled and automatic. The following functions are provided by the application software:

- Sample identification, test parameters and test setup information.
- Choice of filters and their effect in reducing noise.
- Detailed tabulated corrections by the various methods for each test shot at each temperature.
- Rear temperature vs. time plot for each shot as it occurs. Full display of laser pulse shape, and rear surface temperature excursion.
- Tabulated summary of diffusivity averaged over the multiple measurements at each temperature.

The data analysis methods of the laser flash technique have been well documented [2,3]. The first analysis method was developed by Parker, et. al. [1]. Thermal diffusivity,  $\alpha$ , was calculated from the thickness,  $D$ , and half rise time,  $t_{0.5}$ , of the rear surface temperature:

$$\alpha = CD^2/t_{0.5} \quad (1)$$

where  $C$  is a dimensionless parameter. In Parker's method,  $C=0.1388$  under adiabatic conditions. Cowan [4] later developed a mathematical model to include radiation heat losses. Temperature changes at  $5t_{0.5}$  or  $10t_{0.5}$  are compared with the temperature change at  $t_{0.5}$  to determine the parameter  $C$ . Clark and Taylor [5] proposed a new algorithm for analyzing the radiation heat losses from the temperature vs. time plot before the maximum temperature had been reached. Koski [6] and Heckman [7] incorporated a parameter,  $L$ , for heat loss from the front to the rear of the sample for each condition of Clark and Taylor as well as Cowan's method.

The system software incorporates all of the data analysis techniques mentioned. One technique can be chosen before the test as a primary one. After the test, thermal diffusivity values obtained from the various methods are given in a table.

Two layer and three layer materials can also be tested in the system. The analysis is based on the technique developed by Lee and Taylor [8]. Specific heat, density, thickness of each layer and thermal diffusivity of the substrate material are

required for the calculation. The application software can also calculate the contact resistance between two layers with known diffusivity[8].

#### IV. STANDARD MATERIALS TESTING

Several standard materials, SRM 8425 graphite, SRM 1462 stainless steel and ARMCO iron, were used to test the performance of the system. All the samples were disks 12.5mm in diameter. Thermal diffusivity of each material was measured as a function of temperature and compared with reference values. The tests were repeated three times to check the reproducibility of the system. Thermal diffusivity data of standard materials are available in the TPRC Data Series from Purdue University [9] and NIST Research Material data sheet.

SRM 8425 graphite samples were tested in the aluminum furnace from 100°C to 500°C and continued in the graphite furnace from 700°C up to 1900°C. Reference data were calculated from the thermal conductivity values provided on the NIST Research Material data sheet, specific heat values existing in the literature [10] and a density for graphite of 1.73 g/cm<sup>3</sup>. The testing results of SRM 8425 graphite

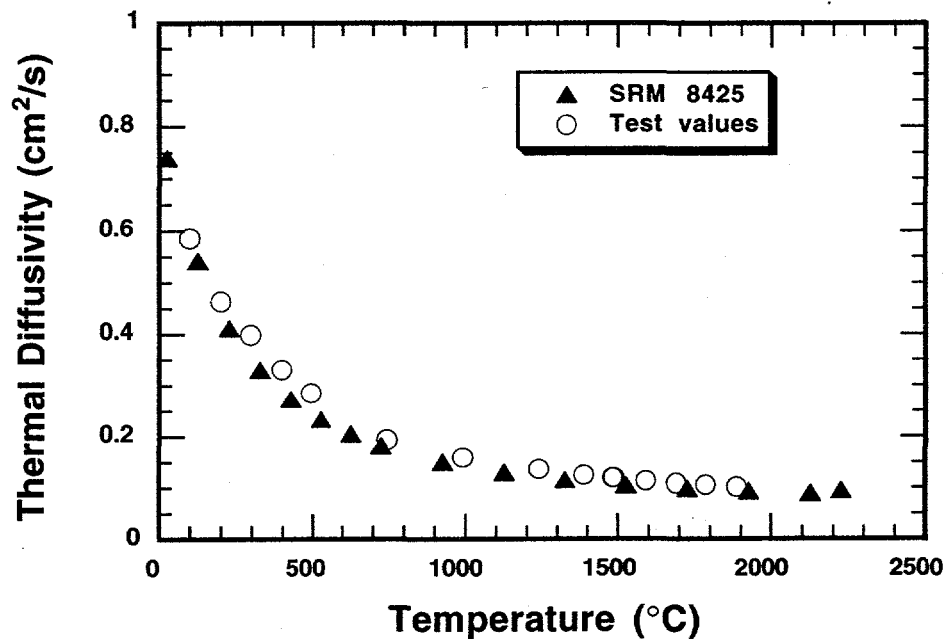


Figure 2. Thermal diffusivity test on SRM 8425 graphite standard sample.



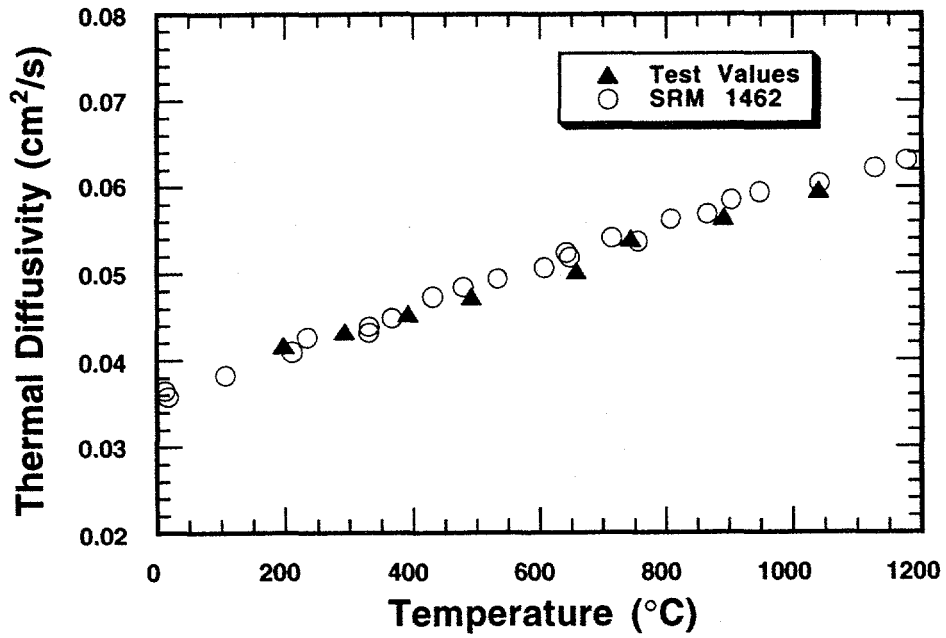


Figure 3. Thermal diffusivity measurement on SRM 1462 stainless steel sample.

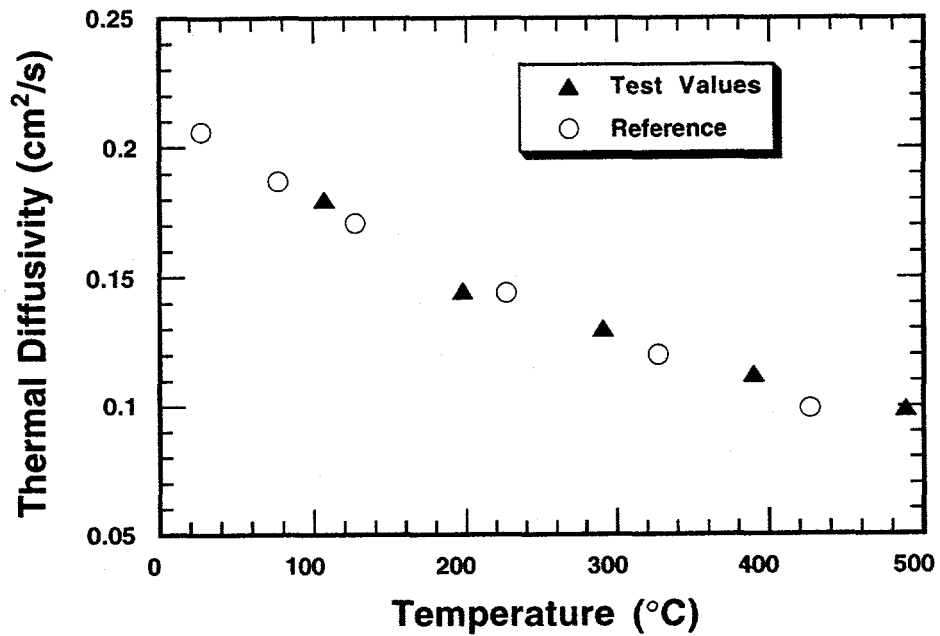


Figure 4. Thermal diffusivity measurement of ARMCO iron standard.

obtained from the system agreed within  $\pm 5\%$  with the reference curve as shown in Fig. 2. The standard deviation of a single test temperature determined by multiple measurements (multiple laser shots) during each test and among the three repeated tests are within  $\pm 3\%$  of the experimental values shown in Figure 2.

Stainless steel, SRM 1462, was tested from 100°C to 1050°C in both furnaces and compared with reference data [9,11]. The results shown in Figure 3 exhibit less than 5% deviation from the average reference values. An ARMCO iron sample was tested in the low temperature furnace under vacuum condition. The results from 100 to 500°C are shown in Figure 4. They showed very good agreement with Taylor and Clark's data [12] and are within  $\pm 5\%$  of the recommended reference curve [9]. For all the three reference materials, the estimated uncertainties of the reference data values are  $\pm 5\%$  for test temperatures above 300K.

## V. SUMMARY

A system has been developed for thermal diffusivity measurements over a wide temperature range and in different environments. The multiple station and concurrent testing concepts have been proven successful for materials characterization. Testing of standard reference materials also showed exceptionally good agreement with literature values; in all cases the experimental values were essentially equal to the reference values. Further tests on layered samples, melts and the quench furnace are planned.

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