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THERMOPHYSICAL PROPERTY DETERMINATIONS USING TRANSIENT TECHNIQU--ETC(U)
MAR 82 R E TAYLOR, R L SHOEMAKER, L KOSHIGOE F49620-81-K-0011
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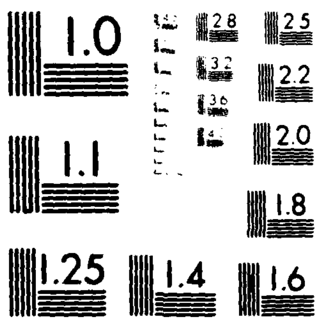

MICROCOPY RESOLUTION TEST CHART
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This program has as its objectives the measurement of the thermophysical properties of energetic materials (AP, HMX, and RDX) and of carbon/carbon materials. The specific heat of ammonium perchlorate up to the beginning of decomposition has been measured with a differential scanning calorimeter. The theory for the ramp heating of a sample with a heat pulse input for determining diffusivity and the heat conduction theory for strip heating have been investigated. Software has been written to accomodate ramp heating at rates up to 30°C per second using the pulse heating method for measuring diffusivity.

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Thermal diffusivity and specific heat measurements have been made on coarse weave carbon/carbon materials. The heat flow through both the individual fibers and the matrix material have been investigated using the flash diffusivity technique, for the purpose of verifying the applicability of the concept of diffusivity to this type of composite material. This work has indicated that an off-axis measurement procedure could be developed which uniquely defines the heat conduction in the various fiber directions for fiber composite material.

TPRL

THERMOPHYSICAL PROPERTIES RESEARCH LABORATORY

AFOSR-TR- 82-0446 TPRL 280

THERMOPHYSICAL PROPERTY DETERMINATIONS
USING TRANSIENT TECHNIQUES

(Annual Report to AFOSR Contract F49620-81-K-0011
Covering Period 15 Feb. 1981 through 15 Feb. 1982)

by

R. E. Taylor, R. L. Shoemaker and L. Koshigoe

March 31, 1982

School of Mechanical Engineering

Purdue University, West Lafayette, Indiana

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RESEARCH OBJECTIVES

A. RESEARCH OBJECTIVES OF ENERGETIC MATERIALS

I. Develop a ramp heating technique using the flash diffusivity apparatus.

   A. Solve the heat conduction equation with a sample undergoing ramp heating with puse heating for thermal diffusivity.
   B. Write the data collection and analysis software for ramp heating.
   C. Build the ramp heating equipment.
   D. Test the ramp heating system using a well known material such as stainless steel 304.
   E. Develop a method for holding the energetic material for the ramp heating using laser pulse heating.
   F. Investigate the best way to measure the temperature transient of the sample.
   G. Measure the thermal diffusivity of the energetic materials (AP, HMX, RDX) to as high a temperature as possible.

II. Develop a high speed strip heating technique for measuring the thermal diffusivity and specific heat of the energetic materials.

   A. Solve the heat conduction equation for the high spped strip heating of a thin layer of material for thermal diffusivity and specific heat.
   B. Write the data collection and analysis software for strip heating.
   C. Build the hardware for the strip heating apparatus.
   D. Test the technique with a well known material such as Lucite.
   E. Measure the thermal diffusivity of AP, HMX, and RDX.

III. Measure the specific heat of the energetic materials.

   A. Purchase and install a flow through cover for the DSC.
   B. Modify our present software to allow corrections for decomposition of the sample.
   C. Measure the specific heat of AP, HMX, and RDX very accurately up to decomposition.
   D. Use the strip heating method to measure the specific heat past the decomposition temperature.

B. RESEARCH OBJECTIVES OF CARBON/CARBON MATERIALS

The objective of the research on carbon/carbons is to investigate the thermal response of fiber-reinforced materials to large transient heat fluxes. In particular, the validity of thermal diffusivity measurements and even the usefulness of the concept of thermal diffusivity in such situations can be questioned legitimately. Important technical applications include rocket nozzles and re-entry nose tips. This investigation is to be concentrated on two areas:

A. Determination of the thermal diffusivity of a carbon/carbon composite material by measuring the heat conduction of in-situ fibers and of matrix material separately.

B. Measurements of transient temperature response in the orthogonal and off-axis directions in selected carbon/carbons.

STATUS OF THE RESEARCH EFFORT

The first step in measuring the thermal properties of AP, HMX and RDX has been to develop a method to measure the thermal diffusivity accurately as a function of temperature. In order to minimize the decomposition of the energetic materials during the diffusivity measurements, procedures to collect the diffusivity data rapidly were investigated. One approach involves heating the sample at a constant rate while collecting diffusivity data by superimposing heat pulses on the sample's front surface. The heat pulses are generated with a laser. The rear face temperature rise curve would be an addition of the ramp temperature and the temperature rise from the heat pulse. It was therefore necessary to remove the effects of the ramp heating from the temperature rise due to the pulses.

Computer programs have been written to collect and analyze data during ramp heating. The separation of the ramp heating curve from the temperature rise data is greatly simplified if the ramp heating is linear. Therefore, a temperature control device has been developed to heat a stainless steel tube furnace at a very constant rate. This device is in the final stages of testing.

It has been necessary to investigate the optical properties of the energetic materials to determine the best way to pulse heat the samples and to measure the temperature response. An infrared laser is normally used for pulse heating the sample and an infrared detector is used for tranaient temperature measurements. The materials are transparent to certain infrared frequencies requiring the use of a special sample holder for measuring the diffusivity. Sample holders have been developed and the energetic material is presently being pressed into these holders.

An alternate technique for measuring the thermal diffusivity for these materials has been under investigation. This method uses much higher heating and data collection rates that the ramp heating. It is anticipated that the higher heating rates will allow the sample to go to higher temperatures before decomposition begins. The investigation of the heat conduction equation for a thin layer of sample on a strip heater is underway. A metallic strip can be heated very rapidly by using a large current surge. It is hoped that the heating rates will approach 10^5 K°/sec.

The differential scanning calorimeter has been modified to allow the decomposition products from the energetic material to escape from the sample holder area. The modifications to the DSC software have been made and the specific heat of AP has been measured from room temperature to 500 °K. Several different sample masses, ranging from 5 mg to 25 mg, were used, as well as different heating rates, from 2.5°K/minute to 10°K/minute. The different heating rates were used to determine how well the AP absorbed heat from the sample holder. It was found that there was no significant difference in heat transfer to the AP at heating rates at least up to 10°K/minute. Figure 1 is a plot of the specific heat results, the measurements made by Westrum and the JANNAF Table Data. The agreement was within 1% up to 500°K where AP undergoes a phase change. The specific heat of the second phase and the heat of transition have not yet been determined.

The current status of our knowledge of thermal diffusivity as applied to carbon/carbon materials is:

I. The important parameters governing the diffusivity are:

(1) Magnitude of diffusivity of fibers and of matrix,

(2) fiber fraction ratio in direction of heat flow,

(3) thickness of sample in relation to fiber bundle spacing,

(4) rear face temperature sampling area and location.

What has been learned:

(1) It is possible to measure diffusivity values which correspond to thermal conductivity values by using a sufficiently thick sample and large viewing area. The sample thickness required becomes less as the temperature is increased.

(2) It is possible to measure in-situ diffusivity values of fibers and of matrix by using point source temperature sensors and thin samples.

(3) It is possible to determine the effects of imperfections such as delaminations on the thermal diffusivity. These effects are manifest by matrix values lower than those for regions where the defects are not present (point source detector methods) and by an increase in diffusivity values for artifacts measured in gaseous environments as opposed to vacuum.

(4) The nature of off-axis heat flow is beginning to be understood. The preliminary results are encouraging but are not completely internally consistent. The use of off-axis testing may be the best method for fully-characterizing heat flow in 3-D material.

(5) The tools to determine the skin depth in artifacts subjected to rapid heating have been developed. Beyond this surface layer the material may be treated as homogeneous for purposes of transient temperature response. For continuous heating this surface layer thickness is of the order of a yarn bundle spacing.

(6) Within the surface layer there are substantial gradients in planes perpendicular to the heat flow (parallel to the surface). These may be of importance in ablation but are not necessarily important in bulk heat conduction.

(7) The temperature of the fiber bundle near the surface is less than that of the surrounding matrix. At greater depths the difference goes through zero and then the bundles become hotter than the surrounding matrix.

CHRONOLOGICAL LIST OF PUBLICATIONS AND REPORTS

Note that carbon/carbon technology is a sensitive area (carbon/carbon materials are on the munitions list) and thus the opportunity to publish results are at least delayed. Thus we have included technical reports and Current Awareness Bulletin articles as these are major sources of controlled dissemination of information in this area.

1. Publications in Open Literature

a. "Variations in the Measurement of Thermal Diffusivity on Coarse-Weave Carbon/Carbon Composites in Terms of Fiber Fraction Involvement," M.S. Deshpande, R.H. Bogaard and R.E. Taylor, International Journal of Thermophysics, 2(4), pp. 357-70, 1981.

b. "Thermophysical Properties of Fine-Weave Carbon/Carbon Composites," R.E. Taylor, H. Groot and R.L. Shoemaker, AIAA Progress in Astronautics and Aeronautics, in publication, 1982.

2. Publications in Current Awareness Bulletin of the Metals and Ceramics Information Center, Battelle Memorial Institute.

a. "Thermophysical Properties of a Coarse Woven Carbon/Carbon Rocket Nozzle Material (TPRL 206R) Issue No. 8, 1980.

b. "Coarse Weave C-C Composites: A Viewing Spot Size Problem Occuring for Thermal Diffusivity Specimen," M.S. Deshpande, R.H. Bogaard and R.E. Taylor, Issue No. 11, 1981.

c. "Analysis of Transient Temperature Response in a Carbon-Carbon Composite During Flash-Method Thermal Diffusivity Tests," J. Jortner, Issue No. 12, 1981.

d. "Analysis of Transient Temperature Response of a Carbon-Carbon Composite During Continuous Heating at One Surface," J. Jortner, submitted.

3. Technical Reports

a. "Thermophysical Properties of a Carbon-Carbon Composite," PRL 162, August 1980.

b. "The Thermophysical Properties of a Coarse Woven Carbon-Carbon Rocket Nozzle Material," TPRL 206, February 1980.

c. "Thermophysical Properties of Carbon-Carbon Composites," AFOSR-81-0716 (TPRL 235), October 1980.

d. "Thermophysical Properties of a Carbon/Carbon Composite," AFOSR-(TPRL 244), May 1981.

e. "Thermal Diffusivity in Carbon/Carbon Composites," AFOSR-(TPRL 256), March 1982.

PROFESSIONAL PERSONNEL

Dr. R. E. Taylor, Co-Principal Investigator

Dr. R. L. Shoemaker, Co-Principal Investigator

Mrs. L. Koshigoe, start May 1981, Professional Staff

Mr. J. Stark, start May 1982, Graduate Student

H. Groot, Professional Staff

INTERACTIONS

1. Presentations

a. "On the Measurement of Thermal Diffusivity on Coarse-Weave C-C Composites and the Dependence Upon Fiber Fraction," M.S. Deshpande, R.H. Bogaard and R.E. Taylor, AIAA 16th Thermophysics Conference, June 1981, Palo Alto, California.

b. "Thermophysical Properties of Fine Weave Carbon/Carbon Composites," R.E. Taylor, H. Groot and R.L. Shoemaker, AIAA 16th Thermophysics Conference, June 1981, Palo Alto, California.

c. "Thermal Diffusivity Measurements of HEPN," R.E. Taylor and H. Groot, Third Carbon-Carbon Rocket Nozzle Technology Meeting, October 1981, Hampton, Virginia.

d. "Off-Axis Thermal Diffusivity Testing of 3D Carbon-Carbon Composites," J. Jortner and R.E. Taylor, submitted to Fourth Rocket Nozzle Technology Meeting, October 1981, Monterey, California.

CONSULTING AND ADVISORY FUNCTIONS

A. Letter concerning round-robin measurements on Carbon-Carbon materials to Les Tepe, AFRPL, March 9, 1982.

B. Consultation with W. Hoffman, APFPL, concerning properties of carbon-carbon materials, March 10, 1982.

C. Numerous contacts with L. S. Theibert, AFML, concerning carbon-carbon materials.

D. Work with McDonnell Douglas personnel on exit cone material (A. B. Guvescjuck), TPRL 268, December 1981.

E. Ongoing work with Bendix personnel (Dr. E. Mays) on C-C brake linings plus upgrading their flash diffusivity apparatus.

F. Ongoing interactions with C-C data bank (R. Bogaard and M. Deshpande of CINDAS) concerning validity of data and test methods used for C-C research.

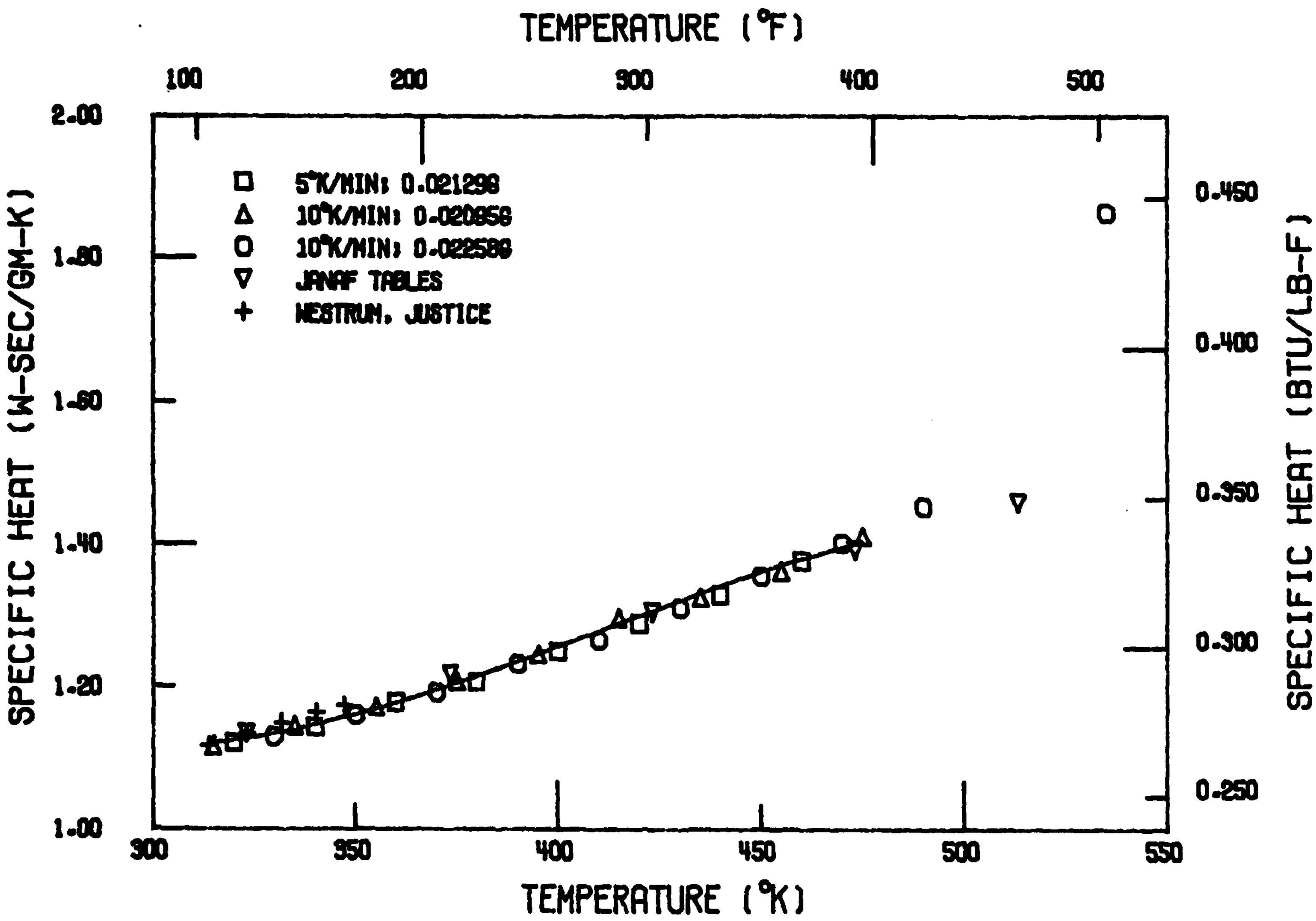

FIGURE 1. Specific heat of ammonium perchlorate.