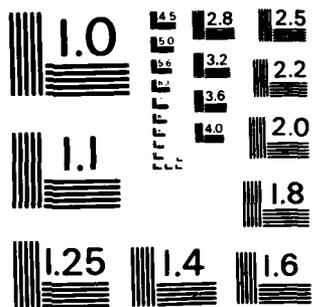


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THERMOPHYSICAL PROPERTIES RESEARCH LABORATORY

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THERMOPHYSICAL PROPERTY DETERMINATIONS
USING TRANSIENT TECHNIQUES

Annual Report to AFOSR for the Period
15-Feb-82 to 15-Feb-83

by

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

April 1983

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JUL 26 1983

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Obtained using two techniques: (1) short range results from 477-486°K just after the phase transition ($\beta \rightarrow \delta$), and (2) extended range results from 415-485°K. The second technique was possible due to hysteresis in the conversion of δ -HMX back to β -HMX following cooling from above the phase transition temperature. The δ phase results for a powdered blend of HMX were obtained and yielded good agreement of the single crystals. Also, the specific heat of HMX inter-mixed with decomposition products was found to be slightly larger than for pure HMX.

Studies to determine the thermal diffusivity of HMX have begun. The initial effort has been to determine the best sample configuration for the diffusivity measurement. A sample holder and mounting technique has been found that will allow the diffusivity measurements to proceed without much difficulty. The first attempt to measure the thermal diffusivity of HMX has been completed. As a result of this measurement, it appears that the reaction rate constant and the heat of activation for HMX decomposition can be obtained from the thermal diffusivity.

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THERMOPHYSICAL PROPERTY DETERMINATIONS USING TRANSIENT TECHNIQUES

RESEARCH OBJECTIVES

The primary purpose of this research is to determine the thermophysical properties of HMX, RDX and AP. The following is a list of the initial goals for this effort:

- The modification of present instrumentation to allow the measurement of the specific heat and the thermal diffusivity of energetic materials.
- The examination of the optical properties of the energetic materials in the infrared region, to determine if an IR detector can see into the material in its different forms; e.g. single crystal and pressed powder.
- The investigation of each material's decomposition temperatures and rates.
- The determination of each material's specific heat in its different phases.
- The examination of the specific heat measurement's heating rate sensitivity for the various materials.
- An examination of how sensitive the specific heat is to decomposition products within the sample.
- The examination of the sensitivity of the thermal diffusivity to decomposition products and sample changes as a result of decomposition.

STATUS OF RESEARCH EFFORT

Several of the goals have been completed. The instruments used for the specific heat measurements (Perkin Elmer DSC II) and the thermal diffusivity have been modified to allow the safe handling of these materials as well as to insure accurate measurements. The major modification to the DSC was to add a flow-through chamber. The DSC had been computerized and it was necessary to change the data collection and data analysis procedures to correct for the effects of decomposition product evolution at the end of the run. The measurement of the energetic material in the flash diffusivity apparatus required the development of a new type of sample holder. The sample is heated by a short IR laser pulse about 800 microseconds long and the transient temperature of the back face of the sample is measured with an IR detector. It was necessary to cover the front face of the sample with another material to avoid direct exposure of the energetic material to the laser pulse. This could

cause localized decomposition or possibly combustion of the sample. Since a pressed powder was used, the sample had to be pressed into a cup and because of the sample's fragility, it was left in the cup for the diffusivity measurement. This required a cup design that would not allow heat flow into the sample except from the front face. The sample's rear face temperature is monitored with an IR detector so it was necessary to determine the material's IR transmission properties. If the material was partially transparent to the IR then the transient temperature being measured would be that of the interior of the sample and not that of the rear face. This would violate the assumptions used in the diffusivity measurement and the result in erroneous results. In the case of pressed powders of HMX and RDX it was found that there was no significant IR transmission as far as the IR detector was concerned. However, the transmission of single crystal HMX, RDX and AP has yet to be tested.

The temperature of the onset of the beta to delta phase change for HMX has been measured as well as the specific heat of both phases. The beta to delta phase transition began at 453°K and decomposition began to become significant above 490°K. The specific heat results for HMX are presented in Figure 1. The heating rate dependency of the specific heat measurement was investigated for both powder and crystalline samples. The rates tested were 2.5 K/minute, 5 K/minute and 10 K/minute. As long as the rate was kept at or below 10 K/minute the results of the measurement were within the experimental error for the instrument. The same was found to be true for RDX. The specific heat for RDX in powder form for temperatures well below its melting point are presented in Figure 2. Above 455°K the RDX undergoes significant decomposition. Single crystal RDX samples have not yet been obtained so that phase of the work has not started.

The thermal diffusivity of HMX has been measured over a short temperature range so far. These results are in Figure 3. The sample used was a powder pressed into a cup made of stainless steel and teflon. These results give only a small part of the picture since the diffusivity will vary with density and sample composition. A preliminary attempt was made to determine the rate of decomposition for a pressed powder sample held at 460°K using the change of thermal diffusivity with time as an indicator. The results are presently being

analyzed. The preliminary analysis suggests that it may be possible to determine the reaction-rate constant for HMX decomposition at various temperatures from the thermal diffusivity. Using those constants, it may be possible to determine the heat of activation.

PUBLICATIONS

"Thermophysical Properties of Fine-Weave Carbon-Carbon Composites," Spacecraft radiative Transfer and Temperature, 83, 96-108, AIAA, Inc., 1982, T.E. Horton, Ed., R.E. Taylor, H. Groot and R.L. Shoemaker.

"Specific Heat of Octahydro - 1,3,5,7 - Tetranitro - 1,3,5,7 - Tetrazocine (HMX)," submitted to AIAA Journal, February 1983, L.G. Koshigoe, R.L. Shoemaker and R.E. Taylor.

PERSONNEL

Dr. R.E. Taylor	Director
Dr. R.L. Shoemaker	Associate Senior Researcher
Mrs. Leslie G. Koshigoe	Associate Researcher
Mr. John L. Stark	Master's Candidate

INTERACTIONS

Several contacts have been made with R. Glick (Purdue) concerning Propellant research and possible decomposition studies. An in-state source of AP mixed with binder and aluminum was located. This would be important if we should incorporate research on mixtures.

While carbon/carbon research was not extended into the present contract period, we remain interested in the diffusion of heat through fiber-reinforced composites. This interest is expressed in personal discussions with Dr. Balageas, Office National d'Etudes et de Recherches Aerospatiales (ONERA), Chatillon-sous-Bagneux, France, at the 8th European Conference on Thermophysical Properties (September 1982, Baden-Baden, Germany), Dr. Roy Taylor of the University of Manchester Research Institute (October 1982, Manchester, England) and Mr. Scott Theibert of Wright-Patterson Air Force Base (several telephone conversations). Dr. R.E. Taylor will be presenting a paper on this subject

at the Carbon Conference to be held in July 1983, at San Diego.

OTHER STATEMENTS

Some single crystals of AP were obtained from Dr. T. Boggs of NWC. Several pressed HMX samples were obtained from Dave Flannigan of Thiokol. Samples of RDX at Thiokol have yet to be shipped as they are waiting on clearance. Sample procurement remains a bottleneck.

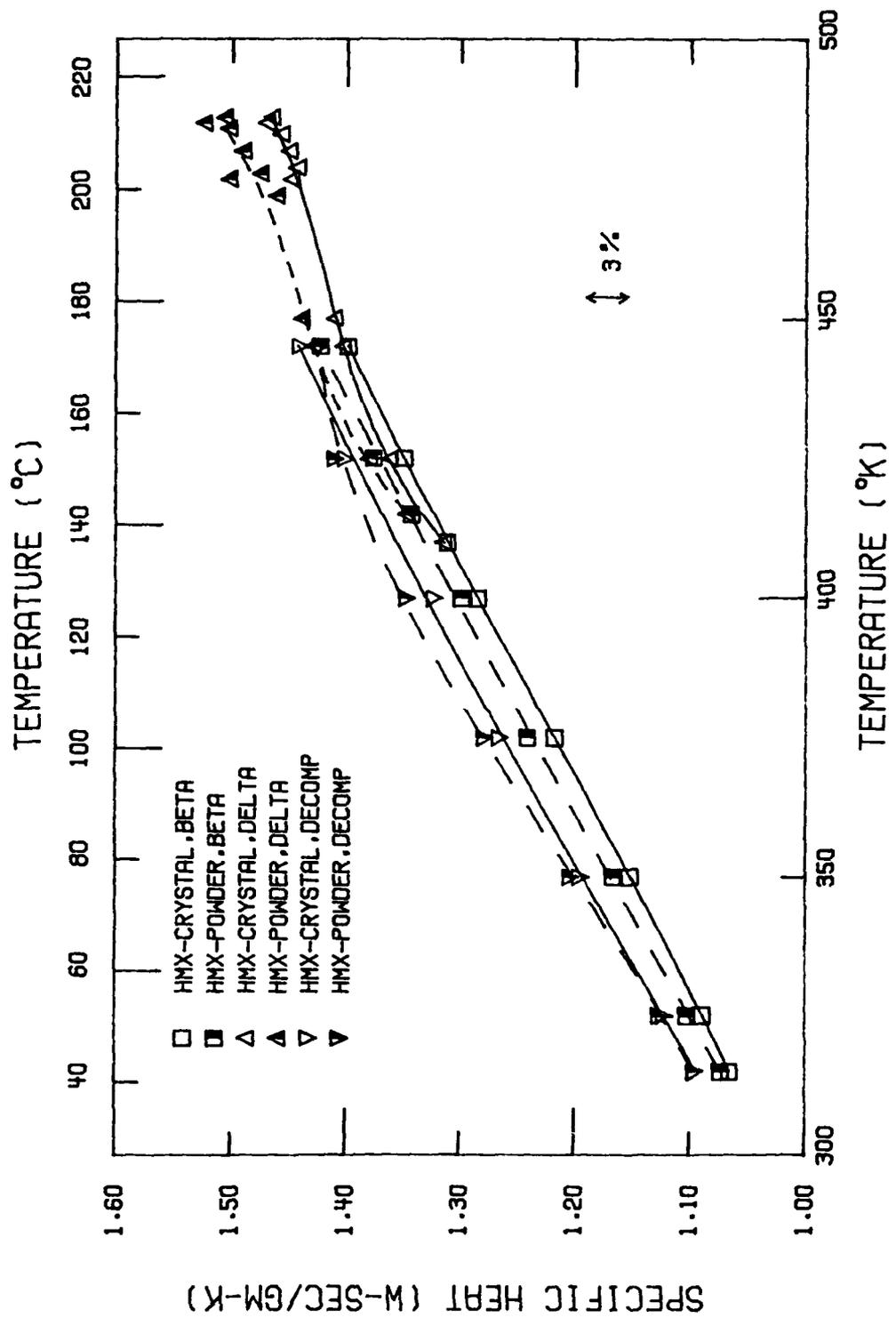


Figure 1. Specific Heat Results (HMX)

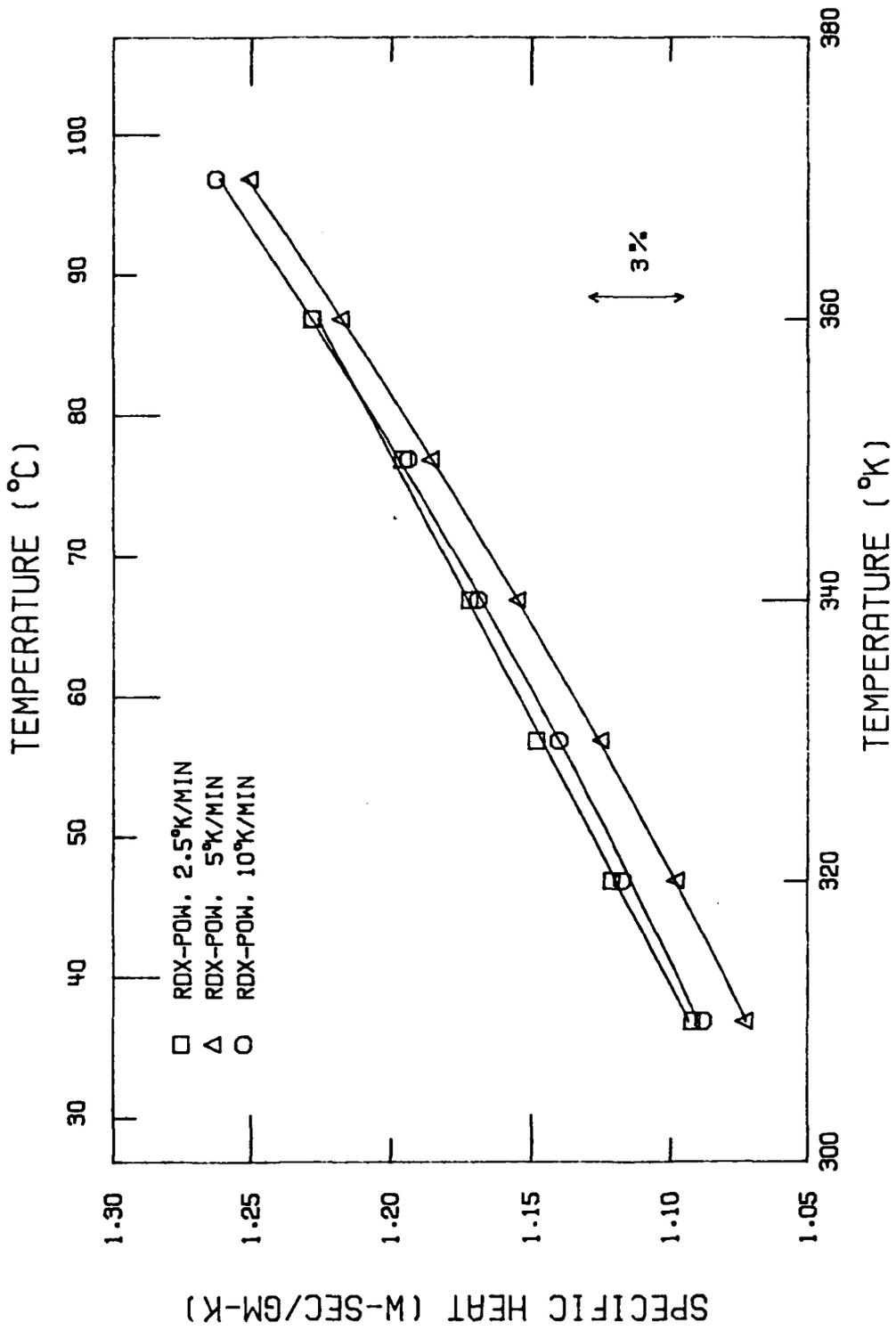


Figure 2. Specific Heat Results (RDX)

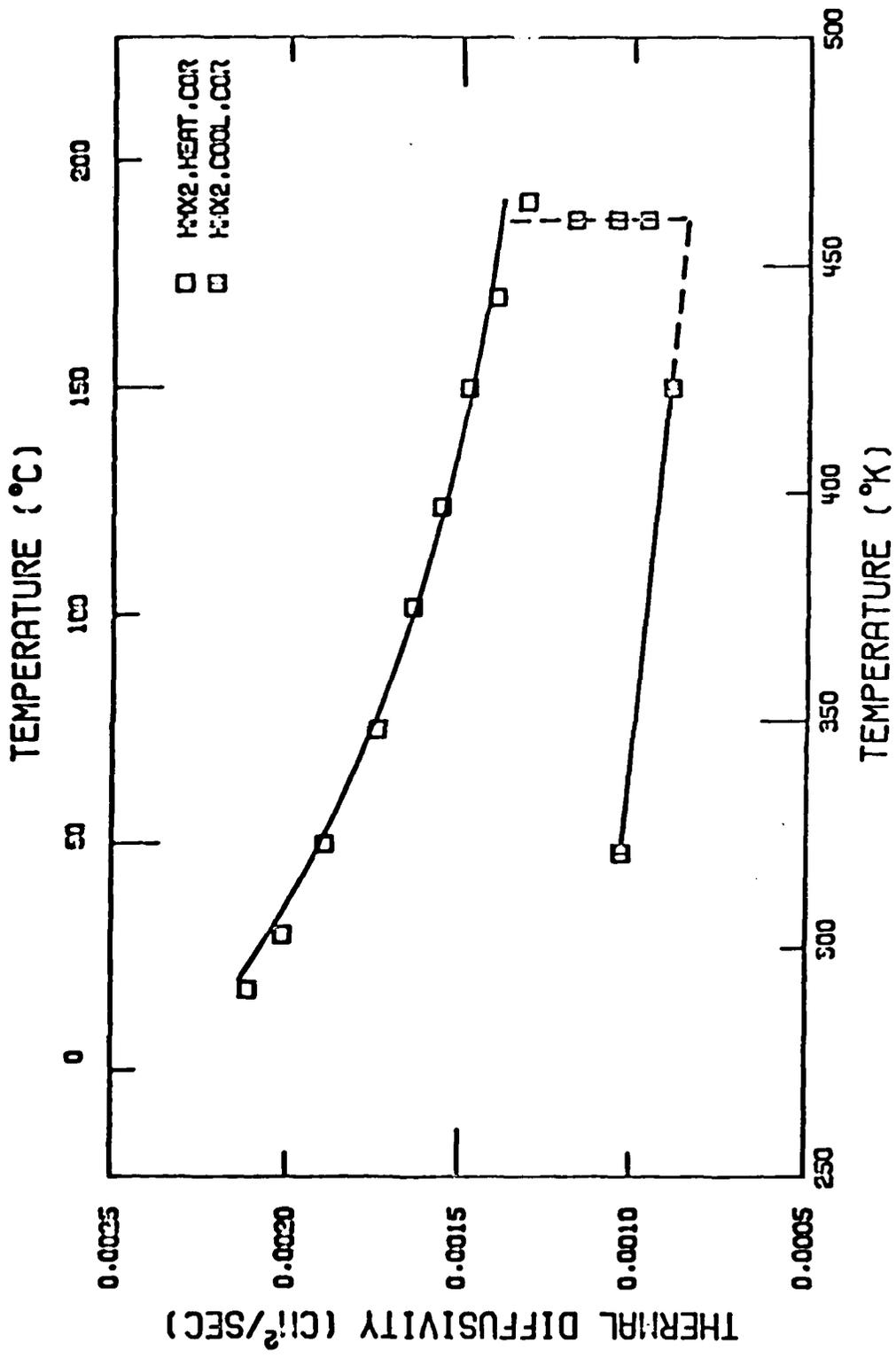


Figure 3: Thermal Diffusivity Results (HMX)

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