(NASA-TM-82500) EXOTHERMIC FURNACE MODULE DEVELOPMENT (NASA) 14 p HC A02/MF A01
CSCL 22A
N83-10093
Unclas
G3/12 38308

NASA TECHNICAL MEMORANDUM

NASA-82500

EXOTHERMIC FURNACE MODULE DEVELOPMENT

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Materials Processing In Space Projects Office

September 1982

NASA

George C. Marshall Space Flight Center
Marshall Space Flight Center, Alabama

MSFC - Form 3190 (Rev June 1971)

TECHNICAL REPORT STANDARD TITLE PAGE

1. REPORT NO.
NASA TM -82500

2. GOVERNMENT ACCESSION NO.

3. RECIPIENT'S CATALOG NO.

4. TITLE AND SUBTITLE

Exothermic Furnace Module Development

5. REPORT DATE
September 1982

6. PERFORMING ORGANIZATION CODE

7. AUTHOR(S)
Roy R. Darnell and Richard M. Poorman

8. PERFORMING ORGANIZATION REPORT #

9. PERFORMING ORGANIZATION NAME AND ADDRESS

George C. Marshall Space Flight Center
Marshall Space Flgith Center, Alabama 35812

10. WORK UNIT NO.

11. CONTRACT OR GRANT NO.

13. TYPE OF REPORT & PERIOD COVERED
Technical Memorandum

12. SPONSORING AGENCY NAME AND ADDRESS

National Aeronautics and Space Administration
Washington, D. C. 20546

14. SPONSORING AGENCY CODE

15. SUPPLEMENTARY NOTES

Prepared by Materials Processing In Space Projects Office

16. ABSTRACT

An Exothermic Furnace Module (EFM) has been developed to rapidly heat and cool a 0.820-in. (2.1 cm) diameter by 2.75-in. (7.0 cm) long TZM Molybdenum alloy crucible. The crucible contains copper, oxygen, and carbon for processing in a low-g environment. Peak temperatures of 1270°C were obtainable 3.5 min after start of ignition, with cooling below 950°C some 4.5 min later. These time-temperature relationships were conditioned for a Foam-Copper Experiment, Space Processing Applications Rocket (SPAR) Experiment 77-9, in a sounding rocket having a low-g period of 5 min.

17. KEY WORDS
Exothermic
Furnace
Foam-Copper
Low gravity
Space Processing Applications Rocket (SPAR)
Materials Processing in Space

18. DISTRIBUTION STATEMENT

Unclassified — Unlimited

19. SECURITY CLASSIF. (of this report)	20. SECURITY CLASSIF. (of this page)	21. NO. OF PAGES	22. PRICE
Unclassified	Unclassified	14	NTIS

For sale by National Technical Information Service, Springfield, Virginia 22151

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TECHNICAL MEMORANDUM

EXOTHERMIC FURNACE MODULE DEVELOPMENT

I. SUMMARY

An Exothermic Furnace Module (EFM) has been developed to rapidly heat and cool a 0.820-in. (2.1-cm) diameter by 2.75-in. (7.0-cm) long TZM Molybdenum alloy crucible. The crucible contains copper, oxygen, and carbon for processing in a low-g environment. In the EFM peak temperatures of 1270°C were obtainable 3.5 min after start of ignition, with cooling below 950°C some 4.5 min later. These time-temperature relationships were conditioned for a Foam-Copper Experiment, Space Processing Applications Rocket (SPAR) Experiment 77-9, in a sounding rocket having a low-g period of 5 min. The overall furnace enclosure is 8-in. (20-cm) diameter by 9-in. (23-cm) long, weighing 30 lb (14 kg) when mounted in an 11-in. (28-cm) SPAR cannister. Ignition of the exothermic material requires 22 A at 34 V for a period of 1.5 min.

II. BACKGROUND

SPAR VII was scheduled to process a Foam-Copper Experiment (SPAR Experiment 77-9) per MSFC-SPEC-937. Dr. R. B. Pond, Sr., and J. M. Winter, Jr., are the Principal Investigators (PIs). The experiment was originally intended for processing in the General Purpose Rocket Furnace (GPRF). Evaluation of the flight model showed that this furnace could not reliably meet the experiment temperature requirements.

Independent of the Foam-Copper Experiment, and prior to the GPRF functional evaluation, some feasibility tests (3.0) had been made using exothermic materials. This work was primarily related to a cast iron solidification experiment that was under consideration. These tests did define a reliable electrical ignition system and the potential of very rapid heating to high temperature, 1400°C.

With this background, an effort was initiated in February 1981 to develop (4.0) an EFM for processing SPAR Experiment 77-9.

III. FEASIBILITY TESTS

Exothermic materials have been used for many years as high energy heat sources. Literature shows several companies marketing exothermic material for various heat treating processes. Exomet, Inc., being one, was contacted and they recommended two of their commercial products. First, their Exomold LDK has a moderate burn rate and internal temperatures of 1500°C. Second, Exomold LDKK has a higher burn rate and internal temperatures of 1700°C. Work was initiated with the latter.

The first step was to develop an electrical ignition or start of the exothermic reaction. A small sample of the Exomold LDKK was compacted around a 0.028-in. (0.7-mm) diameter Kanthal A-1 resistance wire. The wire had been formed into a 0.19-in. (4.8-mm) diameter helix. Tests showed good positive ignition when 10 to 12 A were passed through the wire.

Next step was to compact Exomold LDKK into a 4-in. (10-cm) diameter by 9-in. (23-cm) long cylinder around a stainless steel rod. The rod, which was 0.75-in. (1.9 cm) in diameter and approximately 7-in. (18-cm) long, was placed so the rod and cylinder were coaxial. The end of the rod was flush with the end of the cylinder of the exothermic material. The assembly was placed with the flush end on a fire brick for the evaluation.

The electrical ignition system was embedded at the bottom and energized. A burn rate of 2 to 3 in. (6.3 cm) per minute was observed. The steel rod in the center did not melt. However, some superficial surface melting was indicated. A few additional tests confirmed the ignition system and the rapid heating to high temperatures and slow cooling rates for a thermally insulated package. Cooling rates of 20°C/min were noted.

IV. DEVELOPMENT TESTS

In February 1981, the need for a furnace to process the Foam Copper Experiment in SPAR became apparent and a development effort was started. A basic approach was defined:

1) An ignition system, essentially as had been tested, would be baselined

2) Use multiple segments of Exomold LDKK

3) Sample would be centered inside a cylinder of Exomold LDKK

4) Gas cooling system - carbon dioxide

5) Two ignition points for a shorter period of burn to completion.

All of these are now shown in Figures 1, 2, and 3.

First tests were made with Exomold LDKK segments 3.5-in. (9-cm) diameter, a 0.88-in. (2-cm) hole, and 0.63-in. (1.6-cm) long. Basically the segment was a "donut" configuration. Some nine segments were stacked. A stainless steel crucible was supported in the center of the stack. The stack was insulated with 2.5-in. (6.3-cm) thick high temperature wool, basically quartz. The crucible was sized to simulate the experiment sample, both in outside dimensions and specific heat. Thermocouples were mounted on each end of the stainless steel crucible.

Upon ignition, the lead wires to both thermocouples melted where they were exposed to the hot exotherm. The stainless steel crucible melted and ran freely into the alumina support system. It was concluded that the peak temperatures were in excess of the melting point of stainless, 1400°C.

Subsequent runs were made with 2.5-in. (6.3-cm) diameter exothermic segments and improved thermal insulation on the thermocouples. Temperatures measured were well in excess of 1200°C. Next, 2-in. (5-cm) diameter segments were used. They provided temperatures between 1100 and 1200°C and have been used in all subsequent work.

Based on tests to this point, a more flight-type configuration was made. A ground test TZM crucible-copper sample supplied by the PI was substituted for the stainless steel crucible. The configuration is shown in Figure 1, 2, and 3. Figure 1 shows the basic furnace, which is 8 in. (20 cm) in diameter and 9-in. (23 cm) long, with cooling gas bottle and nonpropulsive vent all mounted in an 11-in. (28-cm) long SPAR cannister, 17.5-in. (44.5-cm) diameter. Figure 2 shows two coils of ignition wire "B." Each has 31 in. (79 cm) of 0.0285-in. (0724-mm) diameter Kanthal A-1 wire. The helix is 0.153-in. (3.89-mm) outside diameter. The ignition coils are paralleled on 34 Vdc and together draw a total of 22 A. Nine segments of Exomold LDKK were used in this configuration and Inconel 718 and Type 303 stainless made up the support members. Cooling gas was stored in a 4 × 8 in. (10 × 20 cm) high pressure gas storage bottle. A solenoid valve, when activated, allowed gas flow through an orifice at the base of the exothermic support structure. Gas flowed through ducts directly to one end of the TZM crucible.

Various insulations and sleeves were tested around the sample and exotherm stack until the desired time-temperature relationships were obtained: 1) A peak temperature between 1130 and 1200°C, 2) a temperature in excess of 1130°C for more than 1 min, and 3) the time from start of copper melt until the copper had resolidified shall be less than 5 min, the low gravity period for SPAR. However, a study of the temperature data in these tests gave indications that the temperature in the center of the TZM crucible was significantly hotter than the ends, where the thermocouples were mounted.

This flight configuration with carbon dixode cooling gas met the Foam Copper Experiment conditions. A flight package, less electronics for signal conditioning of the thermocouple outputs, was vibration tested to the SPAR prototype criteria (reference Test Laboratory, ET19, vibration report "SPAR Exothermic Furnace Module Vibration, B-D-563"). There were no anomalies. The solenoid valve and fill valve were observed by Stress Analysis EP46. They suggested the addition of mounting supports for this plumbing to improve its long term reliability.

V. ADDITIONAL DEVELOPMENT EFFORTS

For several reasons, the EFM was not flown on SPAR as planned. In May 1982, there appeared to be another sounding rocket launch that might accommodate the Foam Copper Experiment. This rocket, however, would not have the capability of in-flight power, temperature recording system, or timing signals for proper initiation of the cooling gas. Thus additional development was required.

A close study of data at this point revealed that it might be possible to refine the thermal insulation, use a more precise amount of exotherm, and meet the time-temperature requirements without the use of cooling gas. Since the original experiment did not have in-flight temperature records, work was directed to provide a furnace operating with constraints of the May 1982 sounding rocket.

The following tests were made with a thermocouple in the center of the TZM crucible and one on the bottom. As suspected, the center was found to be hotter than the end, 150°C hotter. Hence, a new wrinkle was tested, namely, an inert segment (firebrick) was placed in the middle of the stack. This provided a desired reduction of the center temperature. The temperature difference from center to end of the TZM crucible was measured as low as 50°C. Further refinements could probably reduce this temperature difference even more.

Additional balancing of the exotherm, insulation, and brick segments give satisfactory temperatures. However, the time from melt to resolidification was 5 min. In that the cooling rate very likely would be less in flight than on the ground, further refinement was desirable.

The next series of tests used a heavy walled aluminum sleeve inside the furnace housing and around the exotherm stack. In principle, the aluminum sleeve reduced the quartz wool insulation thickness. Also, the quartz wool was now packed to a greater density and the venting of the spent exotherm gases more critical. However, this approach did reduce the time from melt to resolidification to about 4 min and provided a satisfactory time-temperature profile. With essentially the same configuration, three consecutive runs were made. The time from ignition power on until resolidification of all the copper was 7 min. There was a total variance of 15 sec within these three runs. The peak temperatures had a variance of 16 and 4°C for the crucible center and bottom, respectively (Figs. 4, 5, and 6). Thus, the EFM had proven its reproducibility.

VI. GASES OF THE BURNING EXOTHERMIC MATERIAL

The exothermic material has a phenolic based binder. During the reaction of Exomold LDKK, a significant volume of gas is released. It is estimated at 4 standard cubic feet (110 liters) for the EFM. A gas analysis was made of some gases collected during the reaction. It is shown below.

H_2	51 percent
N_2	36 percent
O_2	01 percent
CO_2	12 percent

One might expect this composition to change during the reaction. No attempt was made to define the change, if any exists.

VII. POTENTIAL APPLICATIONS

The EFM has four very different characteristics than furnaces in common use today.

1) When it is started, it goes to completion in a pattern determined almost entirely by its design.

2) It carries, in the exotherm, the energy for the designed temperature profile.

3) It appears to be highly reliable with no element burn-outs. In all of the tests of this effort, perhaps 20, the exotherm performed in a consistent manner. No anomalies were noted related to the burn.

4) A significant volume of gas is released during the exothermic reaction.

These characteristics have advantages in MPS applications requiring rapid heating, high energies, and high temperatures. Sounding rockets and free fliers are likely to be vehicles for this combination of furnace and experiment. Shuttle applications may be constrained by the off-gases of the exothermic reactions.

A wide range of exothermic materials are marketed today. They can be sized for the desired temperature conditions. The upper limit for temperature is probably 1500 to 2000°C when using commonly known materials.

VIII. RECOMMENDATIONS

It is recommended that the EFM be utilized to process the Foam Copper Experiment on either SPAR X or any other suitable sounding rocket. The EFM is extremely simple in operation and should prove highly reliable. Preintegration testing should be initiated for SPAR X when concurrence is received from the PIs and the SPAR Configuration Control Board.

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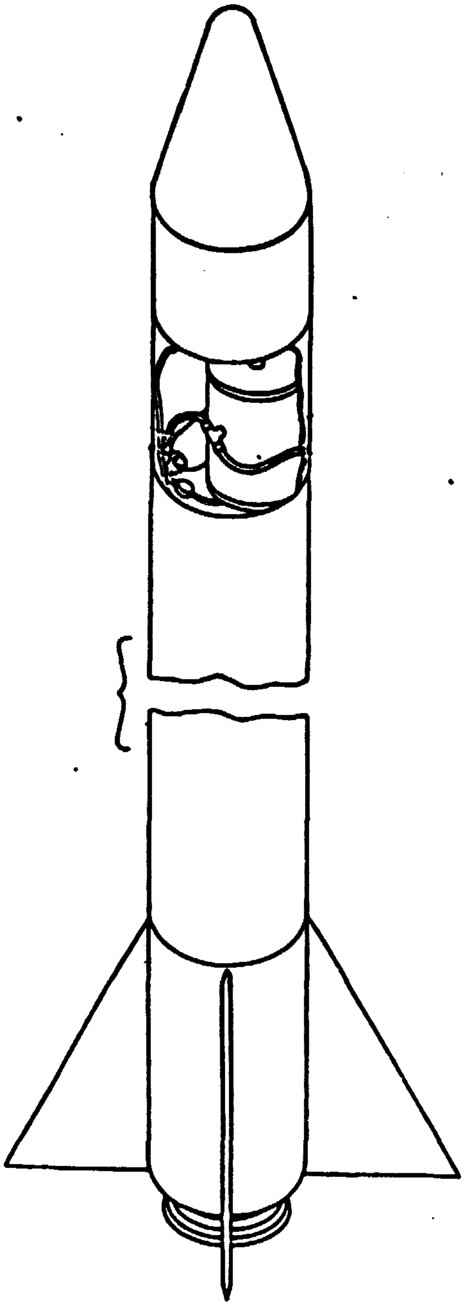

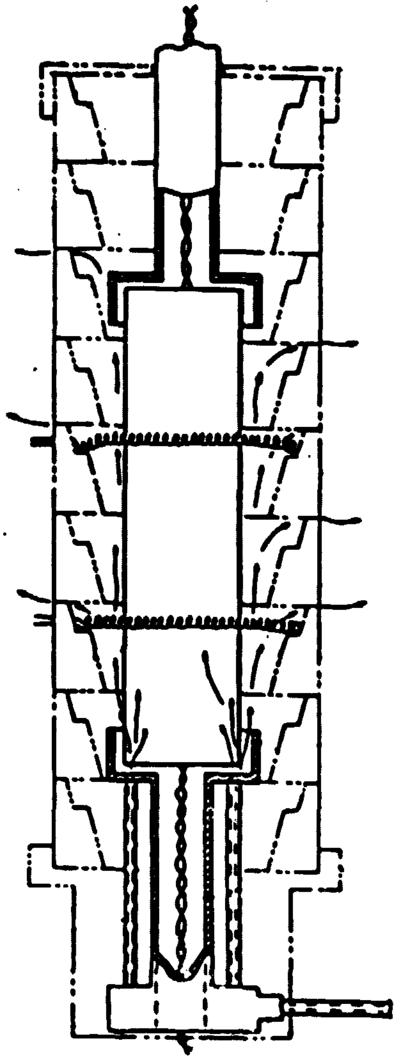

Figure 1. Basic furnace installation.

Figure 2. Basic furnace configuration.

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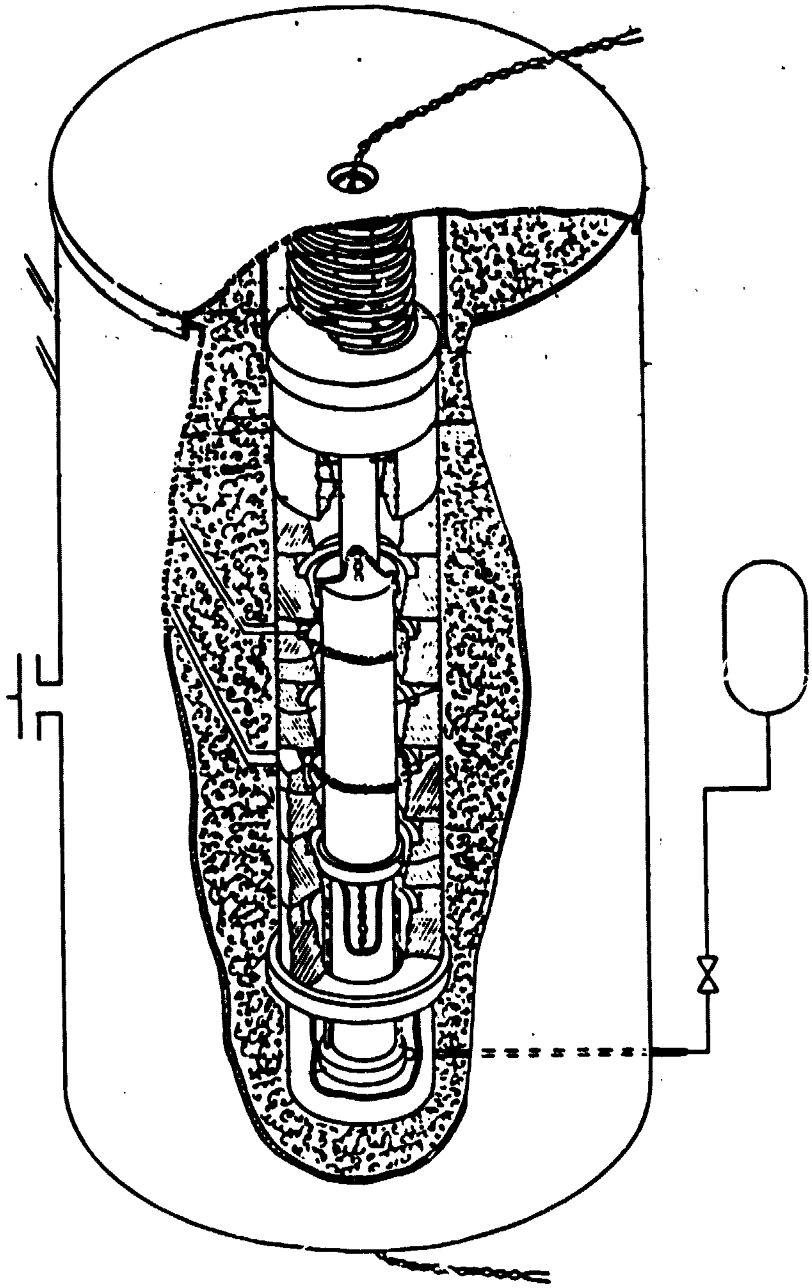

Figure 3. Furnace assembly.

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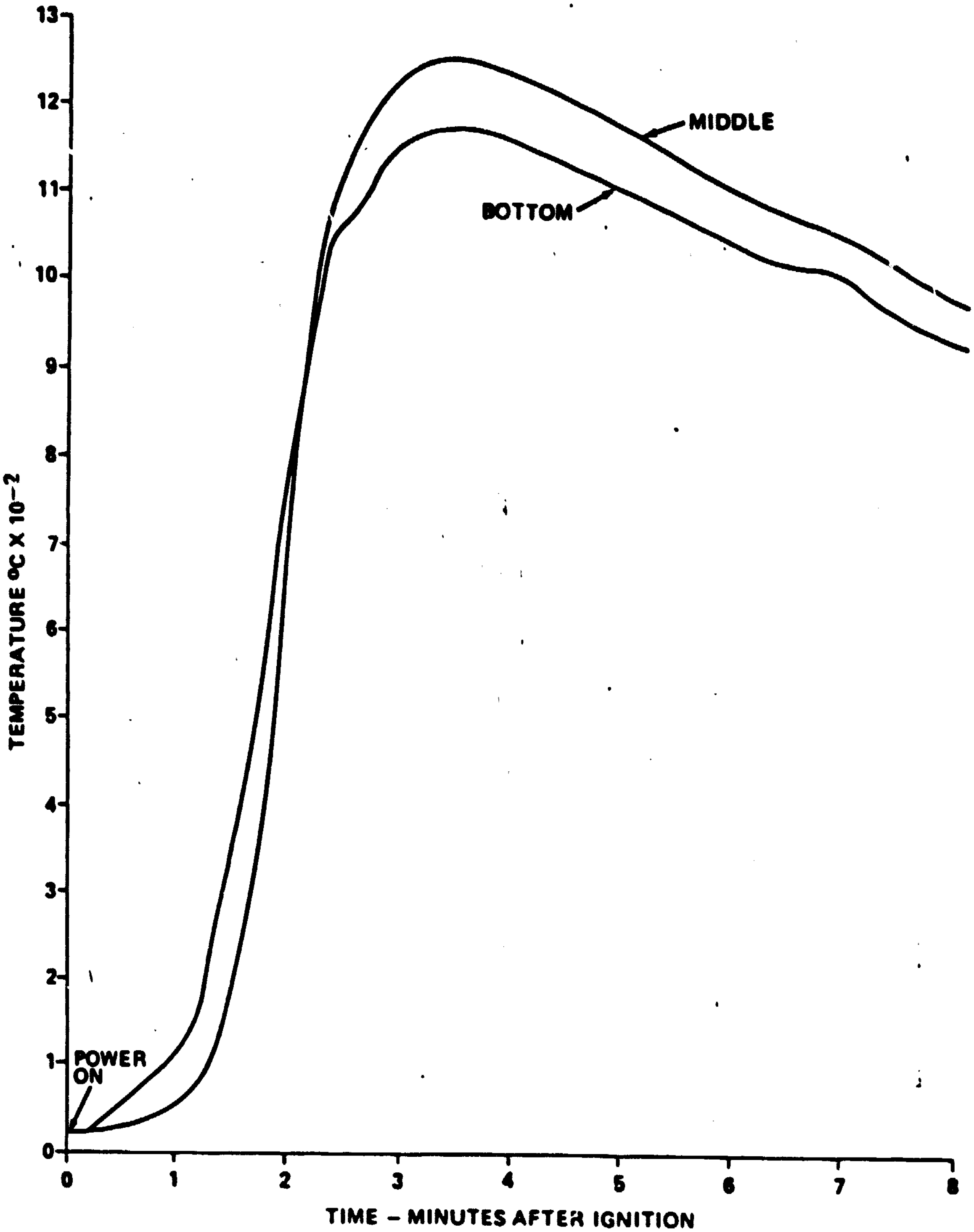

Figure 4. Exothermic furnace module, Run No. 9, May 21, 1982.

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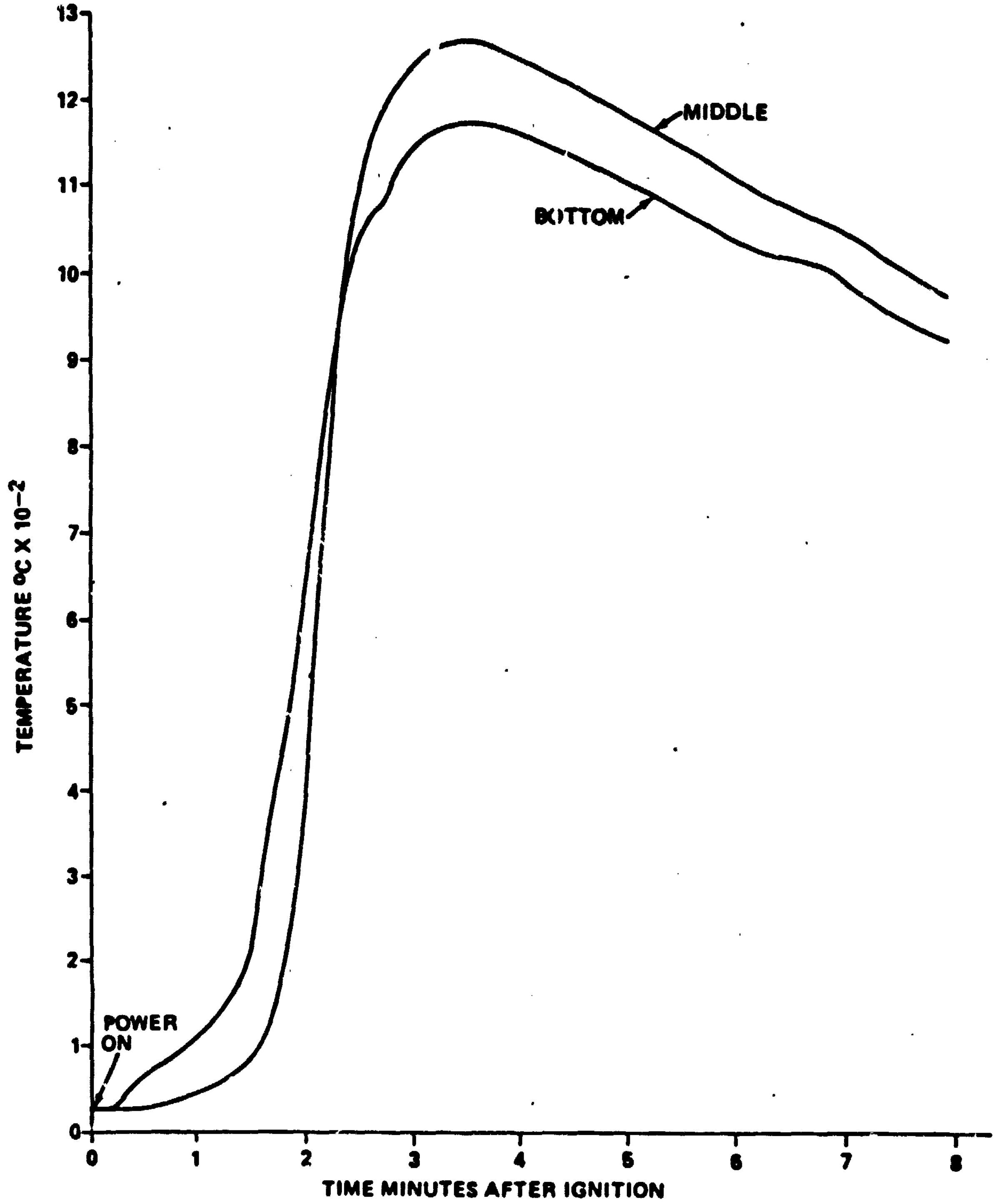

Figure 5. Exothermic furnace module, Run No. 10, June 4, 1982.

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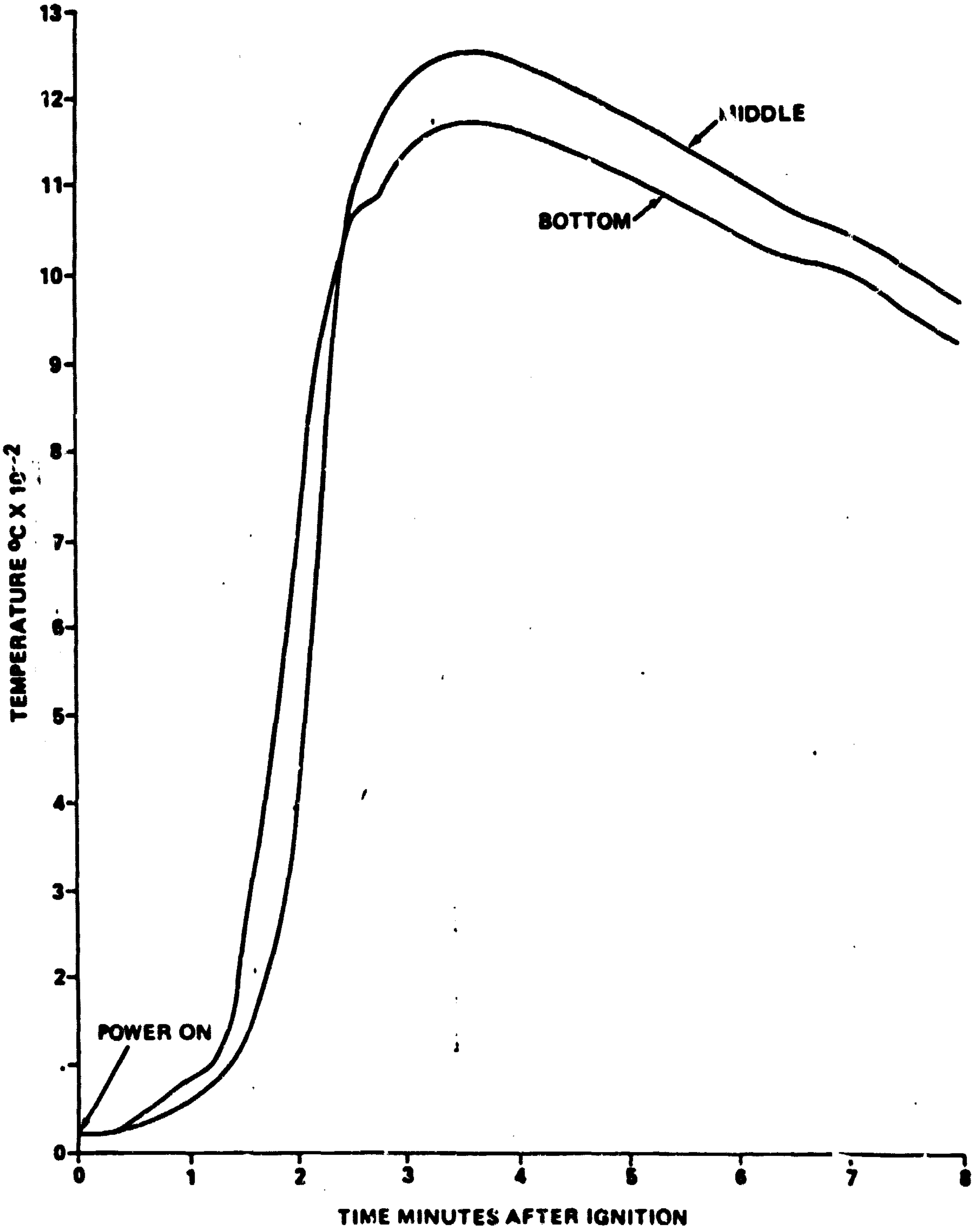

Figure 6. Exothermic furnace module, Run No. 11, June 10, 1982.

APPROVAL

EXOTHERMIC FURNACE MODULE DEVELOPMENT

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The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

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