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Final Report
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January 1990 to
July 1990

Solid Propellant Temperature Profiles Obtained Through Embedded Microthermocouples

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FOREWORD

This final report was submitted on completion of JON; 573000Q3 with the Astronautics Laboratory (AFSC), Edwards AFB CA. AL Project Manager Dr Tim Edwards.

This report has been reviewed and is approved for release and distribution in accordance with the distribution statement on the cover and on the DD Form 1473.



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REPORT DOCUMENTATION PAGE

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INTRODUCTION

The purpose of this technical report is the documentation of experimental results obtained from a solid propellant combustion study conducted January-July 1990. Experimentation was carried out in the Combustion Research Laboratory of the Astronautics Laboratory, Edwards Air Force Base, CA. The experiments involved embedded thermocouples measuring temperature profiles through the combustion wave of solid propellants. Ammonium Nitrate (AN) was the primary propellant studied. However, three other types of propellants were also studied: double base, HMX1 (energetic binder), and HMX2 (inert binder).

This project stems from another in the Combustion Research Laboratory in which Laser Induced Fluorescence (LIF) is used to obtain temperature profiles of various propellants. LIF runs into trouble when propellants blow off large quantities of soot as they burn. These "sooty" burns make it impossible to receive a useful return signal. In these instances another method is needed to acquire data. The method of embedding thermocouples into solid propellants is a proven reliable method as it dates back to the 1950's. This report deals solely with the embedded thermocouple method. No data using other methods is presented.

EQUIPMENT AND PROCEDURES

Thermocouples

Type-S thermocouples (platinum-platinum/10% rhodium) were used exclusively in this experiment. They were fabricated here in the lab from 25 and 75 micron leads which were welded together. The two leads were cut from their respective spools. For 75 micron wire the leads were cut at 2.5"-3.0", whereas the 25 micron leads were cut at approximately 5.0". Originally shorter leads were cut for the 25 micron wires, however many broke due to handling. Therefore, longer leads allowed rougher handling without the threat of breakage. The leads were placed between two magnets with their ends butted together. The junction was then placed under a 30-power microscope. A miniature hydrogen/oxygen torch, made by Smith Equipment, was then ignited and brought towards the junction. This torch allowed for very small flames which made it easy to apply pin-point heating to the junction. The wires melt, bead up, and recede as shown in Figure 1.

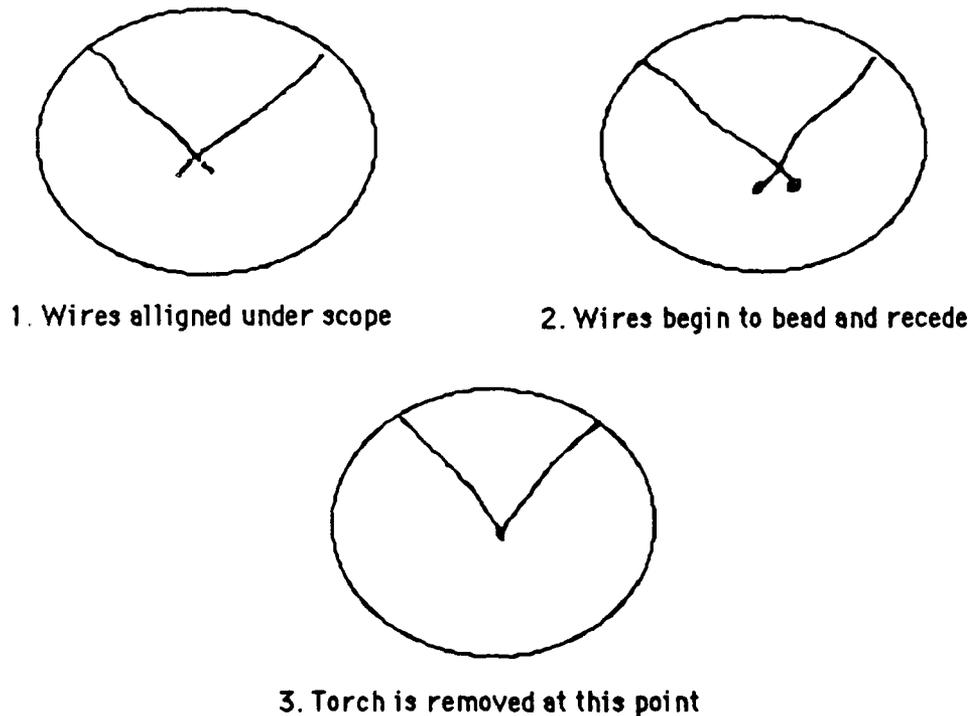


Figure 1. Demonstration of Thermocouple Fabrication

Once the two beads joined, the torch was removed and the weld was complete. The bead size was restricted to 2 to 3 times the diameter of the wire to insure sufficient response time from the thermocouple.

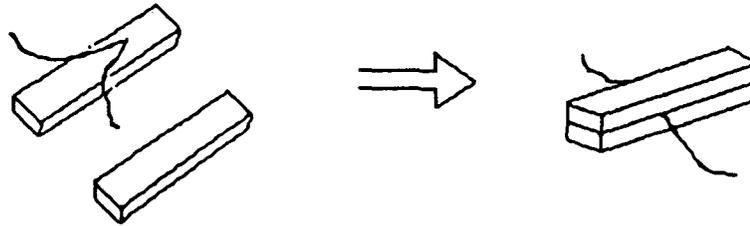
Once a thermocouple was made, it was tested with an Omega 660 thermocouple thermometer. If it responded well to heat fluctuations, it was then ready for embedding into the solid propellant.

Embedment

There were different methods used in the embedment process of the thermocouples. The methods varied in their use of different adhesives, thermocouple lead angles, and in the manner in which the propellant was cut.

There were two ways in which the propellant was cut. One method was the "sandwich method." The other was the "pie-piece method." These are both demonstrated in Figure 2.

Sandwich Method



Pie Piece Method

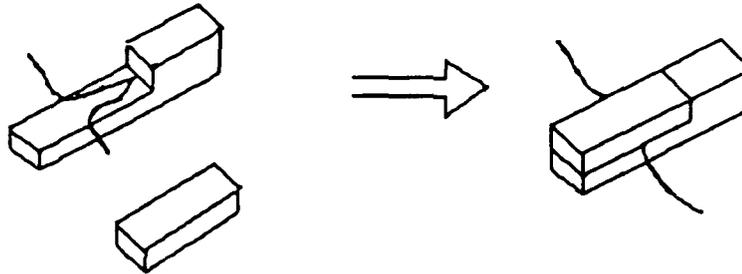


Figure 2. Embedment Methods

The pie-piece method was an idea obtain from an Army report on solid propellant combustion studies¹. As can be seen from the figure, the pie-piece method offers some possible advantages over the sandwich method. One being that there is no epoxy involved in the propellant combustion before the thermocouple emerges from the surface. Testing that compared the two methods was also performed.

The Army report contained research material pertaining to various lead angles for the embedded thermocouples, and presented the results for angles of 30, 90, and 150 degrees. Data taken from AN burns using 30, 90, and 150 degree angles are illustrated later in this report.

Once the propellant was cut and the lead angle set, the sample was adhered together. When AN or HMX propellants were bonded, an epoxy (DEVCON 5 minute

epoxy) was used. However, acetone was utilized to bond the double base propellant back together. The propellants were then held in a vise until they had completely bonded. The double base propellant took approximately 3 days to bond and the epoxy-bonded propellants took approximately 1 day.

Combustor

Once embedment is complete the propellants are ready to be burned. Just prior to burning, the propellant's sides are coated with a lubricant (TRIPOLUBE-16 by Aerospace Lubricants). This procedure insures that the flame does not burn down the sides, thus an even burn from top to bottom is achieved. The strands were burned in a nitrogen-purged, high pressure combustor². The combustor is capable of pressures up to 1000 psig.

Detectors and Electronics

The thermocouples are fed through the combustor by use of a Conax gland fitting. The signal is carried to the data collection device where a program³ using an Apple Macintosh IIx with a MacADIOS A/D board collects the data at 210 hertz.

The collected data is then run through a program⁴ written to convert millivolt signals into useful data for temperature profiles, correcting for any effects due to heat transfer.

Surface Position Determination

The surface temperature of these propellants was determined by utilizing a method described in a 1965 paper⁵.

It begins by stating that the one-dimensional flow of heat in a medium with heat generation q_x is given by:

$$\frac{\partial}{\partial x} \left(-\lambda \frac{\partial T}{\partial x} + m C_p (T - T_0) \right) = q_x$$

It goes on to state that if specific heat C_p and thermal conductivity λ are assumed constant the following holds:

$$\frac{\partial \ln(T-T_0)}{\partial x} = \frac{\dot{m}C_p}{\lambda} - \frac{1}{(T-T_0)\lambda} \int_{-}^{+} q_x \partial x$$

With the burning propellant slabs, there is no heat generation until the surface area is reached. Thus, in the non-reactive region of the solid:

$$\frac{\partial \ln(T-T_0)}{\partial x} = \frac{\dot{m}C_p}{\lambda}$$

The report goes on to state that $\ln(T-T_0)$ vs. x is a straight line for such a medium. Deviations from this straight line would indicate the onset of a reactive region - or, the surface of the burning propellant.

The method of how this linearity factor is implemented with temperature profiles is shown in the 1965 report and is implemented in this report. The use of the linearity factor in this report is demonstrated in the first four AN log plots. Although not presented here, this is the method used for all other T_s measurements acquired throughout the remainder of this report.

The T_s is measured to ± 25 K. This holds for T_s measurements of all propellant types in this report. While the T_m presented is the maximum measured temperature of the flame, it should not be viewed as the absolute maximum flame temperature for any of the propellants used in this experiment. Too many variables may be encountered during combustion which may alter the maximum reading the thermocouple receives. For example, the thermocouple may get blown around in the flame and thus an absolute maximum flame temperature becomes unattainable.

Data Smoothing

Some of the temperature profiles in this report were smoothed. Smoothing is done as a five point running average. For example: data point #10 = $(\#8 + \#9 + \#10 + \#11 + \#12)/5$, etc. This allowed for many of the ambiguities of T_s determination of unsmoothed curves to be eliminated. It thus hopefully yielded a more accurate determination of the surface temperature. Smoothing was only done when deemed necessary. Examples of unsmoothed curves vs. smoothed curves are shown in Appendix A-D.

PROPELLANT DATA

Propellant Formulations

Most propellant used in this experiment was produced at the Astronautics Laboratory. Their formulations are presented below, and are left general to clear up any classification problems.

Table 1. Propellant Formulations

Ingredient, wt % (approx)	AN	DB	HMX1	HMX2
HMX (200/20 μm)	0	0	73	80
PGA binder	0	0	10	20
PEG binder	0	29	0	0
TMETN	12	0	17	0
NG	0	71	0	0
AN	67	0	0	0
GAP binder	21	0	0	0

HMX = cyclotetramethylene tetranitramine $\text{C}_4\text{H}_8\text{N}_8\text{O}_8$, TMETN = trimethylolethanetrinitrate $\text{C}_5\text{H}_9\text{O}_9\text{N}_3$, NG = nitroglycerin $\text{C}_3\text{H}_5\text{N}_3\text{O}_9$, AN = ammonium nitrate NH_4NO_3 , GAP = glycidyl azide polymer, PEG = polyethylene glycol-based polymer $\text{C}_{4.5}\text{H}_9.1\text{O}_{2.3}$, PGA = polydiethylene glycol adipate $\text{C}_{4.58}\text{H}_{7.50}\text{O}_{2.34}$

Burn Rate Data

Burn rates were achieved from two sources. The first is a report by Tim Edwards⁶. Not all burn rate data necessary was available, so the other burn rates were determined experimentally.

Several known-length strands of propellant were burned at certain pressures. A stopwatch was used to time the burns from ignition to completion. Knowing the slab

length and the burn time yielded a burn rate. The trials of each propellant at each pressure were then averaged to acquire the final rates presented below.

Table 2. Propellant Burn Rate Data (in mm/sec)

Pressure (psi)	AN	DB	HMX1	HMX2
100	1.57	2.7	.84	No burn
150	--	2.9	1.06	0.65
250	3.2	3.3	1.5	1.0
450	--	4.1	2.2	1.4
500	5.2	--	--	--

DISCUSSION OF EXPERIMENT

As previously stated, the main objective of this report was to present temperature profiles valuable to the chemical and the kinetic modeling of solid propellant combustion. Also, it was imperative to investigate certain variables to set some test parameters for the experiment. Different lead angles and embedment methods were studied. Different size thermocouples were also used.

Seventy-five (75) micron wire used to establish a consistent manner in the fabrication of microthermocouples. These 75 micron thermocouples were also used to reproduce some previous data and thus insure all instruments were working as planned. Smaller wires have faster response times as reflected in the AN temperature profiles in Appendix A. The 25 micron curves are, in general, a bit smoother and less ambiguous than the 75 micron curves.

Three different lead angles of 30, 90, and 150 degrees were studied. Previous experimentation had been done with these angles⁷. Theoretically, the smallest lead angle,

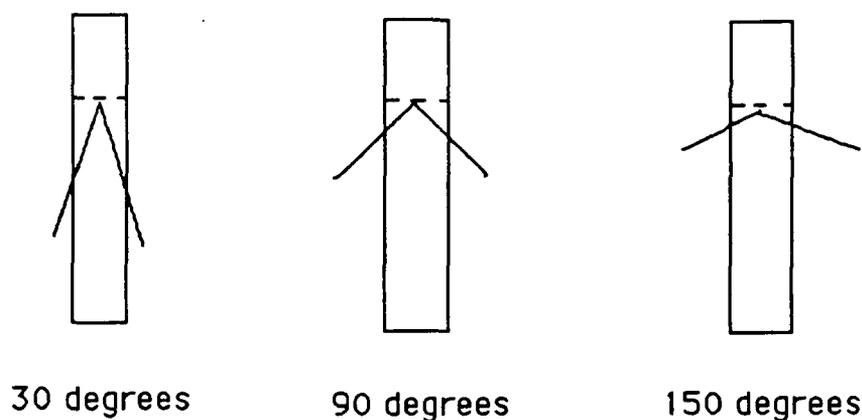


Figure 3. Various Embedment Angles

as defined in Figure 3, will perturb the temperature field the most by conducting heat away from the junction site. Fig. 4 illustrates this effect using AN data at 100 psi.

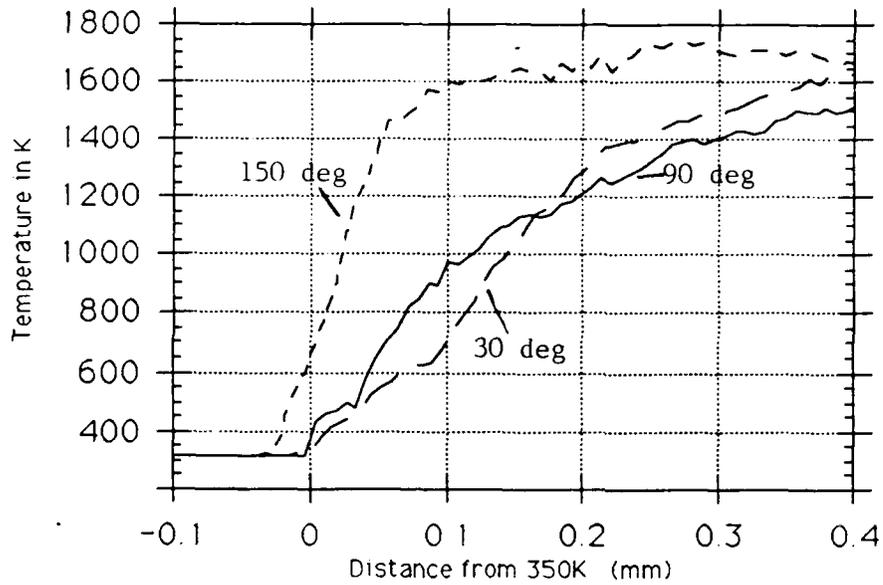


Figure 4. Lead Angle Comparison

In reference to the embedment process, the "pie-piece" method became the preferred method for the combustion experiments. This preference resulted from the observation that the epoxy was not burning with the HMX2 propellants at 250 psi, as shown in Figure 5. Although the other propellants tested did not exhibit the same "lack of epoxy burn" characteristics to the degree of HMX2, it seemed only logical to use as little epoxy as necessary to achieve a truer propellant temperature profile.

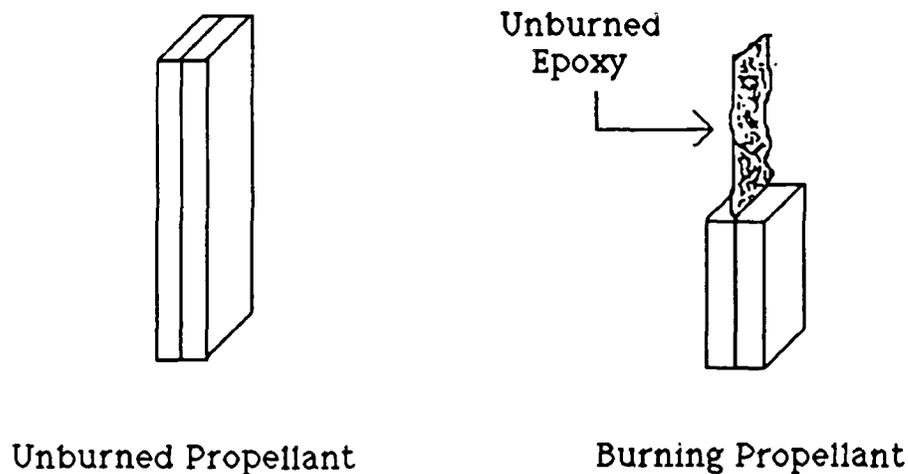


Figure 5. Schematic of Typical HMX2 Burn

Ammonium Nitrate

Ammonium nitrate (AN) was studied back in the 1950's as a solid propellant. Ammonium perchlorate (AP) turned out to be much more energetic than AN. So AN was put aside and not much has been done with it since. However, AP based solid propellant (such as that used in the space shuttle boosters) produces large amounts of HCl as an exhaust product and also a lot of smoke. HCl is harmful to the environment and a smoky exhaust makes a missile very visible to the enemy. Therefore, the search for a "cleaner" propellant is on. AN, a non-HCl producing, smokeless propellant base, is coming under investigation again. Its low energetic capacity and poor burning qualities can hopefully be overcome to the point where it can be used as a clean, useful fuel. AN's burning characteristics are not well known as of yet and this portion of the report is meant to bring more insight into its characteristics. Temperature profiles have been made as well as physical burn characteristic observations. These physical observations are discussed later.

AN was burned at 100, 250, and 500 psi. The actual temperature profiles are shown in Appendix A. The following is a tabular summary of those profiles.

Table 3. AN Temperature Data

Size μ	Pressure psi	Angle degrees	T _m (K)	T _s (K)
75	250	---	1700	525
75	250	---	1900	510
75	250	---	1900	520
25	100	30	1700	600
25	100	90	1700	580
25	100	150	1400	450
25	100	150	1750	450
25	100	150	1700	440
25	250	30	1850	550
25	250	30	1850	600
25	250	30	1800	600
25	250	30	1900	525
25	250	30	---	600
25	250	30	1875	550
25	250	30	1900	550
25	500	90	1900	550
25	500	90	1900	550
25	500	150	1900	600

The surface temperature results compare well with the data compilation of Beckstead⁸. He found the surface temperature of AN to be in the range of 500-600 K.

Double Base

Double base propellant was also studied in this report using only 75 μ thermocouples. Surface temperatures were obtained using the same methods previously

tested with other double base propellants⁹. The schematic below shows from where the surface temperature was determined.

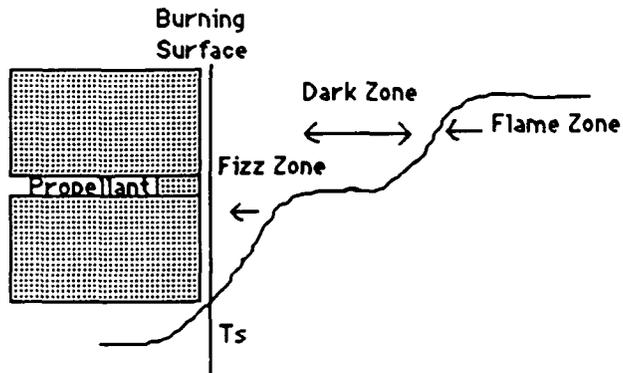


Figure 6. Description of DB Combustion Wave

Double base data obtained in this experiment is illustrated in the table below.

Table 4. Double Base Temperature Data

Pressure psi	Size μ	Angle degrees	T _m K	T _s K
250	75	90	1900+	510
250	75	90	1375+	510
250	75	90	1500+	550
250	75	90	1650+	550
250	75	90	1350+	550

This data is comparable with that obtained by Sabadell¹⁰ in Table 5:

Table 5. DB Comparison Data

Pressure psi	T _s , range of values	T _s , ave K
50	523-623	605
100	573-623	606
150	523-553	541

This validity comparison of data shows that viable test methods were used and lends credence to the accuracy of other reported data. The true importance of good double base comparisons comes from the use of acetone (instead of epoxy) in the embedment process. A good comparison using acetone and a good comparison when using the epoxy (as seen later with HMX2) dispels some belief that the epoxy may be adversely affecting the temperature profiles. The actual temperature profiles for the double base propellant can be seen in Appendix B.

HMX1

HMX1 burns at a much higher temperature than any of the other propellants described in this report. It was investigated at 150, 250, and 450 psi. Through the combustion of HMX1, the thermocouples were expected to melt and then break. This was to have shown the thermocouples response to rapid heating. The thermocouples were all set at lead angles of 90 degrees in the HMX1. The temperature profiles are presented in Appendix C. Data is also presented in Table 6.

Table 6. HMX 1 Data

Pressure psi	Size μ	T _m (K)	T _s (K)
250	75	1850	700
250	75	1980	700
250	75	1970	600
250	75	2000	600
250	75	1980	630
250	25	1900	610
250	25	1900	700
250	25	1900	670
450	25	1875	680
150	25	1900	640

HMX2

HMX2 is virtually the same as HMX1 without the energetic plasticizer (TMETN) in its formulation. This difference does however lead to a much less energetic burn than that of HMX1. HMX2 data was primarily taken for comparison purposes. The data obtained is compared below to that of Alspach¹¹ (who used 75 μ thermocouples) and Kubota.¹²

Table 7. HMX2 Data

Size μ	Pressure (psi)	Angle degrees	T _m K	T _s K
25	150	90	1100	670
25	150	90	1400	700
75	250	30	1900	700
75	250	30	1450	720
75	250	30	1500	730
75	250	30	1500	700
25	250	90	1400	700
25	250	90	----	700
25	250	90	1400	700
25	450	90	1900	600
25	450	90	1850	680
25	450	90	1850	680

Table 8. Comparison of Data

	Pressure (psi)	T _m K	T _s K
Kubota's Data	150	975	673
	450	1873	673
Alspach Data	150	1050	700
	450	1900	700

As can be seen above, the reported HMX2 data compares well with that of these two other researchers. This further establishes the accuracy of the method used in this report. The actual temperature profiles for HMX2 are shown in Appendix D.

Comparison of 75 μ v. 25 μ Temperature Profiles

A comparison between 75 μ and 25 μ thermocouple temperature profiles is shown below. Several useful observations can be made from this data. The 75 μ thermocouples

start indicating a heat flux sooner than the 25 μ . Also, the indicated temperature increase for the 75 μ is more gradual than the 25 μ profiles. However, surface temperatures and "maximum" temperatures values for the two different thermocouple diameters correspond almost identically. This is an important fact because it shows that 75 μ thermocouples measurements can be used if T_s is the property being investigated, while 25 μ thermocouples should be used if spatial resolution is of interest. Figures 7-9 are temperature profiles at 250 psi for thermocouples having the same lead angles and embedment methods for the three different propellants. These figures show that this trend is not dependent on a particular propellant type, lead angle, or embedment method.

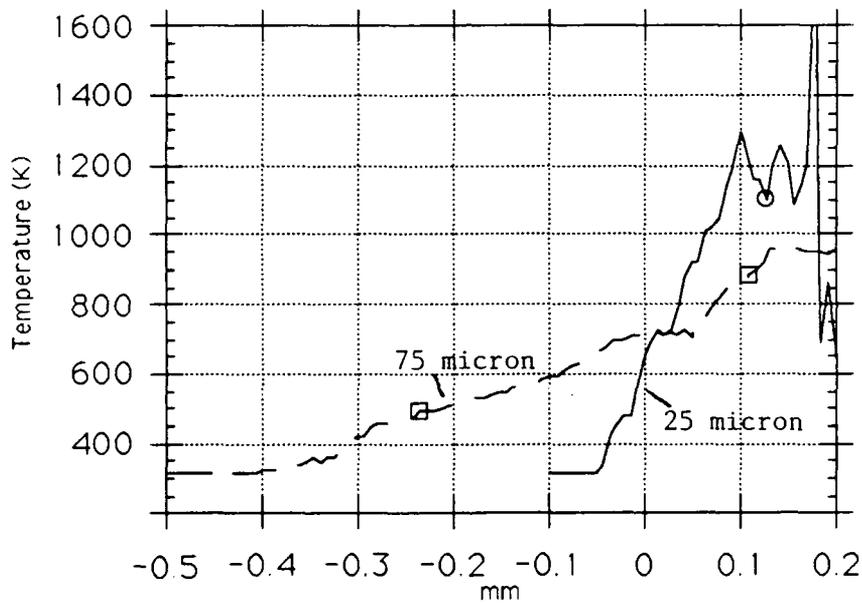


Figure 7. 75 v. 25 Micron Thermocouples - HMX1

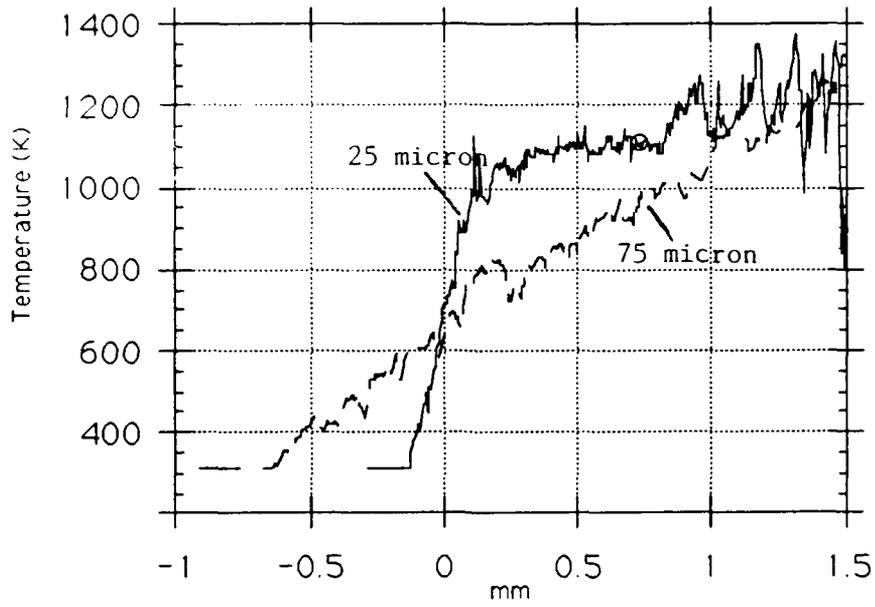


Figure 8. 75 v. 25 Micron Thermocouples - HMX2

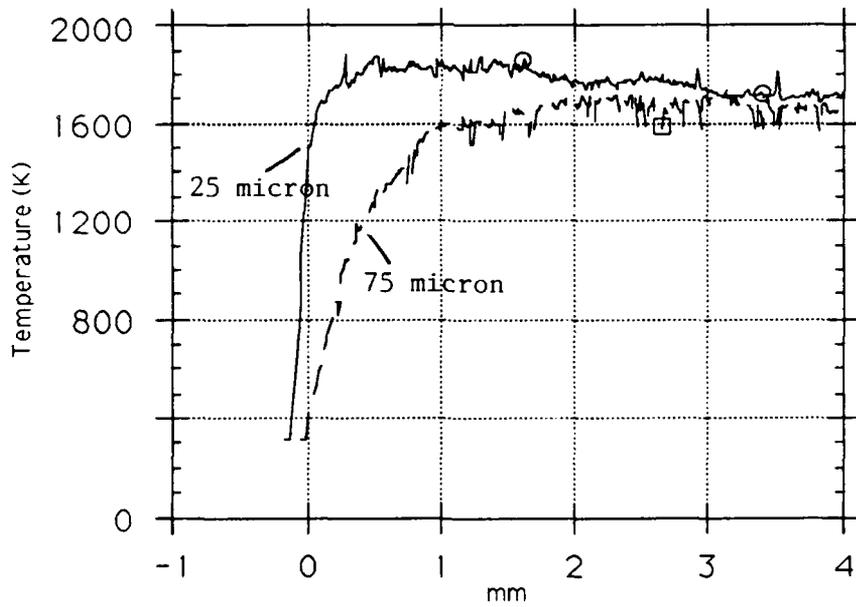


Figure 9. 75 v. 25 micron Therocouples - AN

OBSERVATIONS

It has been shown through this experiment and previous work that ammonium nitrate has a low burning temperature. While observing AN combustion in the high-pressure bomb and the remnants of the fuel after the burn, an interesting characteristic was noted. It was evident that not only is AN a "sooty" burning propellant, but the soot becomes harder at higher pressures. For instance, at 100 psi the "soot" has the composition of ash and it is blown apart by the evolving gas and coats the inside of the bomb. At 250 psi, the "soot" does become more rigid and remains attached to the propellant strand mounting post. The soot maintains its shape but is very porous and will collapse into ash upon handling. At 500 psi the "soot" remaining after combustion maintains the same volume as the original propellant slab prior to combustion, although much more porous. It is also much harder and more brittle than the "soot" produced at lower pressures. Although it is not the objective of this report to explain this phenomenon, it is speculated that the binder may not be burning with the propellant at these pressures. If this is actually the case, the possibility remains that another binder may be found which enhances the burning properties of AN.

One problem that occurred during experimentation and is worth noting to benefit other researchers involved the use of an AC power source as an ignitor. When the original DC source for the ignitor posts broke down, it was temporarily replaced with an AC source. Problems arose from the interference of the AC power source with the thermocouple electronics. An AC current produces a magnetic field around the wire carrying the current. In our experiment this produced a magnetic field around the ignitor posts. Unfortunately, the thermocouple wires were close enough to the ignitor posts so that the true signal was interfered with by this field. Thus, the data from the thermocouples came out as a sine wave. The problem was remedied by insuring that the AC power source was turned off as soon as the propellant ignited.

SUMMARY

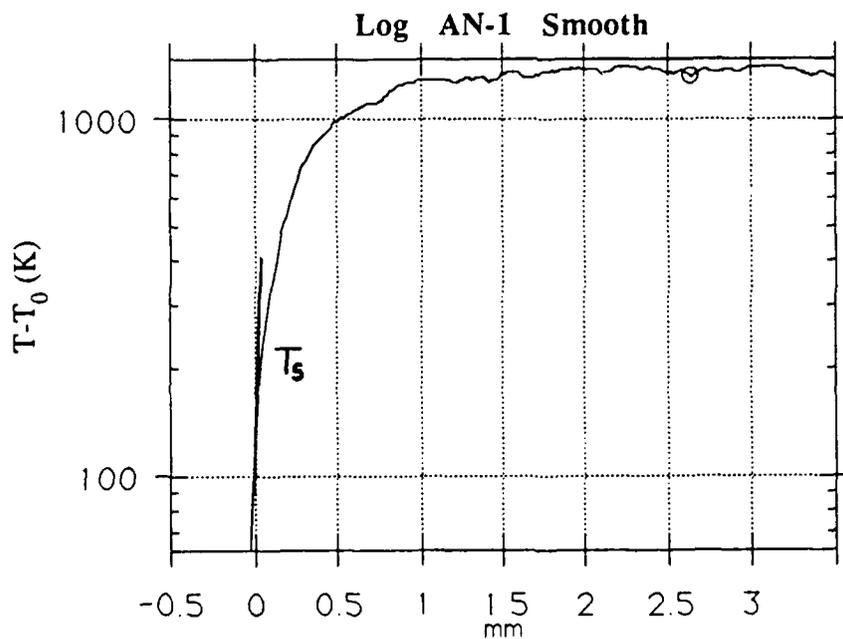
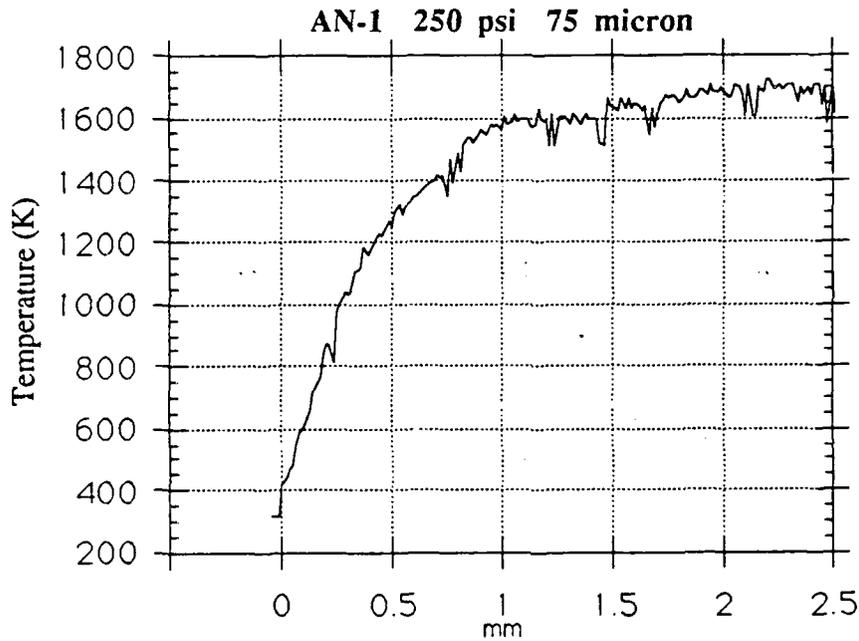
This experiment achieved several pre-determined goals. First, a viable method of producing useful temperature profiles through the flame of burning solid propellants was established. Also, temperature profile data for AN propellant was produced. Data was analyzed and AN was found to have a burning surface temperature of 500-600 K. The troubles with AN as well as some possible ways to better its poor burning characteristics were studied. Three other propellants were also studied to compliment the AN work. A double base propellant was found to have a surface temperature of 510-550 K. HMX1 surface temperatures were found to be 600-700 K, while HMX2 surface temperatures were measured at 600-730 K.

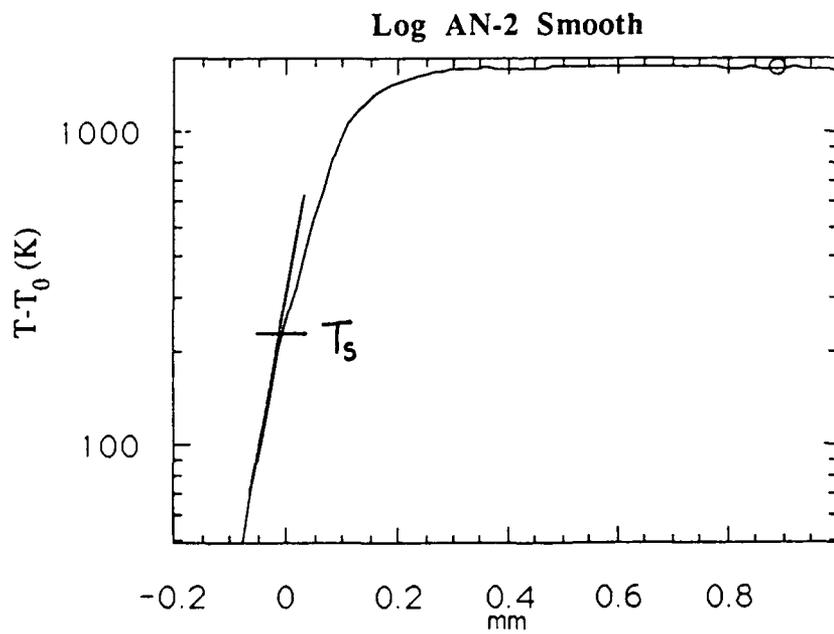
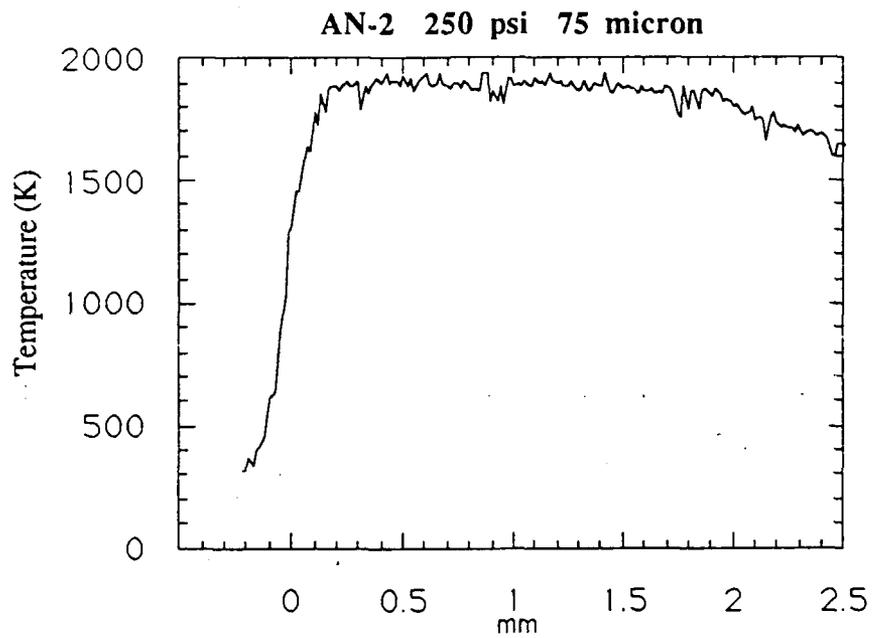
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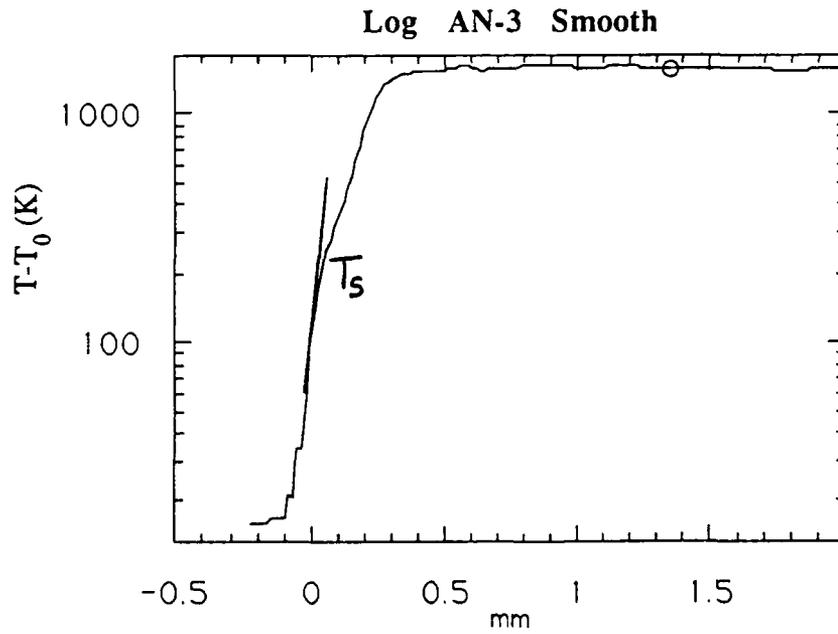
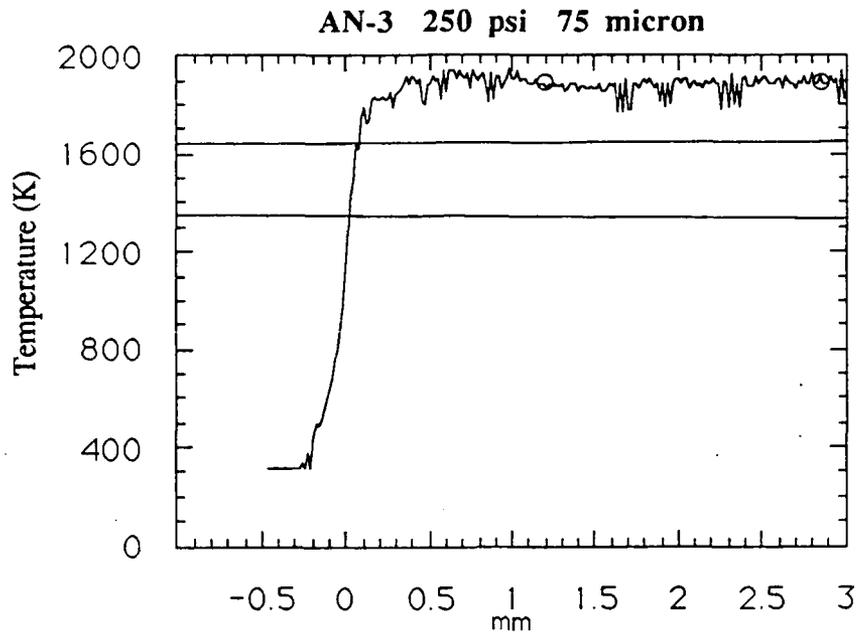
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APPENDIX A. TEMPERATURE PROFILES FOR AMMONIUM NITRATE

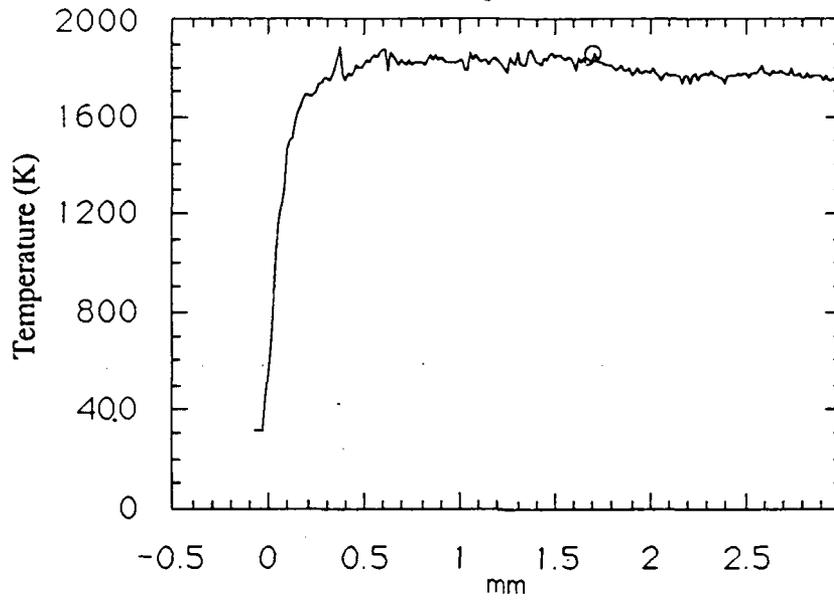
This Appendix is a compilation of all of the ammonium nitrate temperature profiles which were collected. The presentation of these profiles illustrates exactly how each T_s and T_m measurement was acquired. Some of the curves were smoothed to reduce some of the ambiguity of finding the T_s . Those that were smoothed are so indicated.



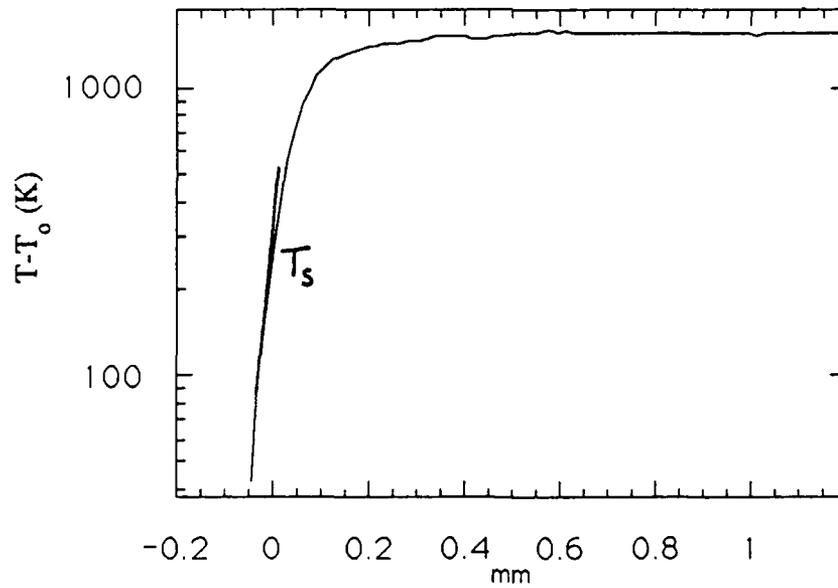


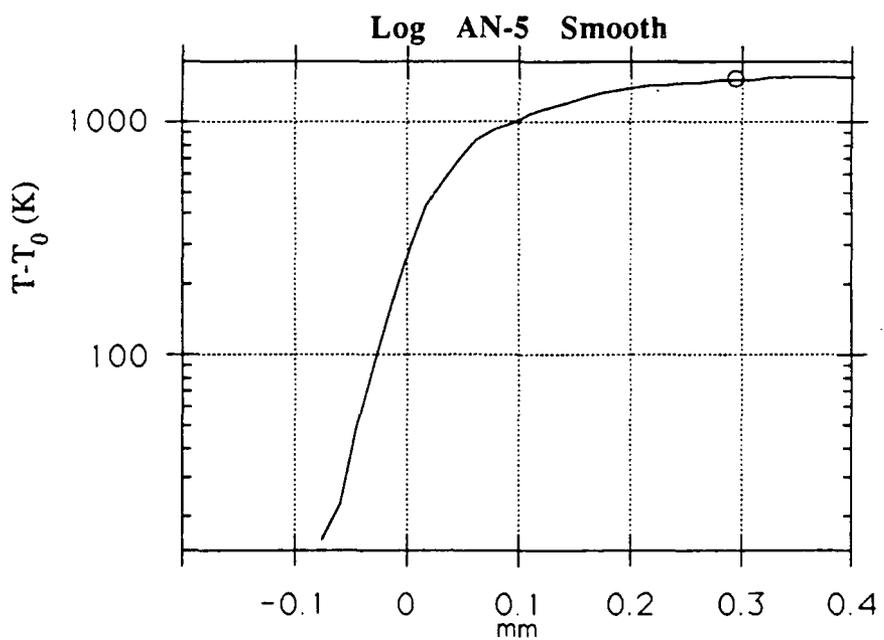
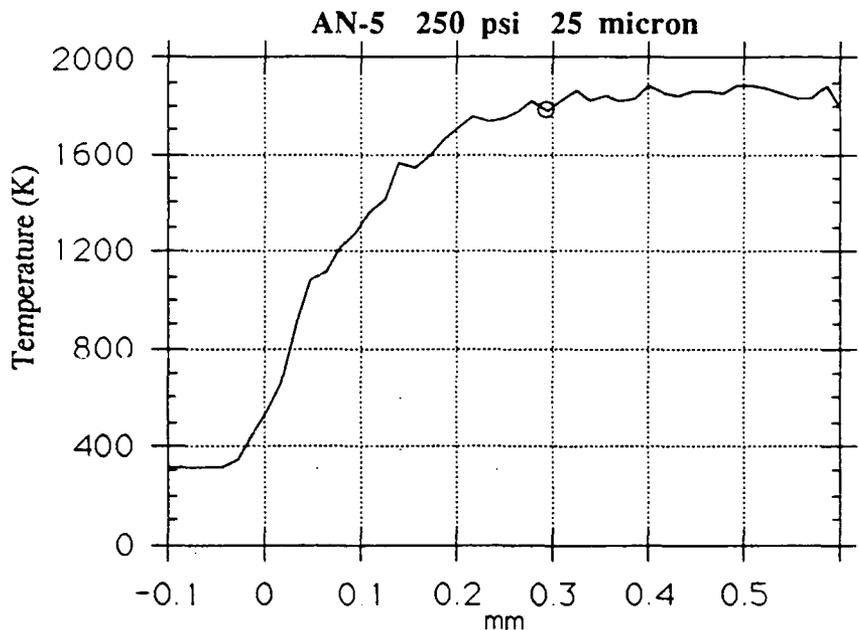


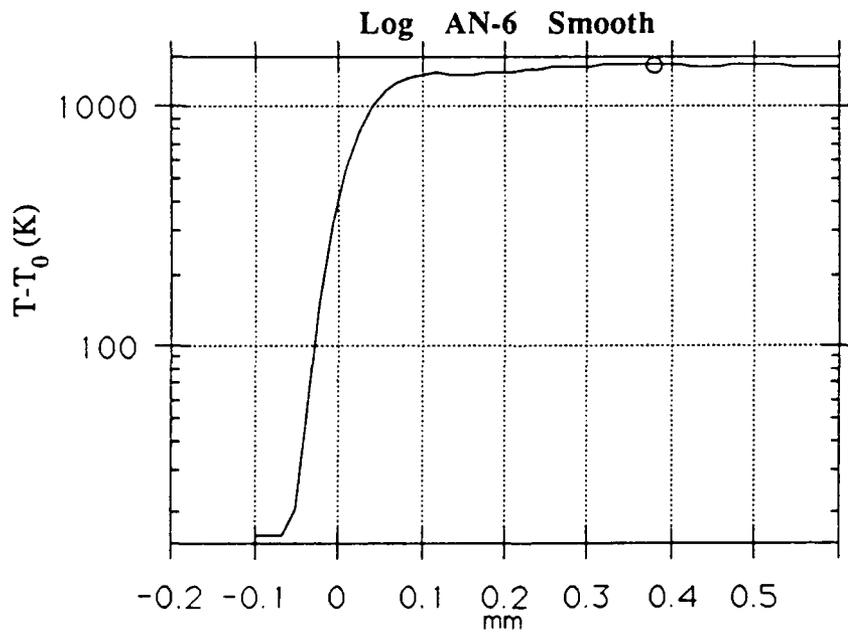
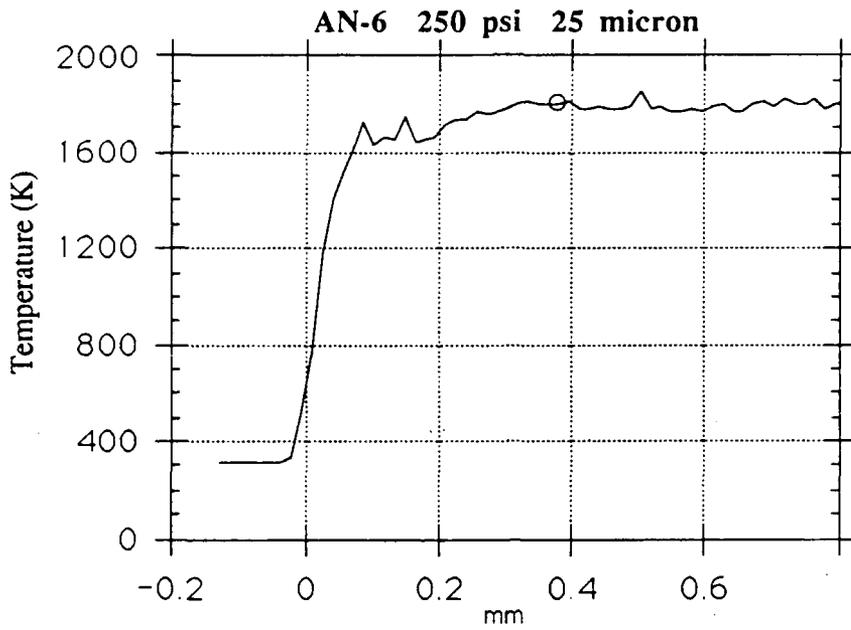
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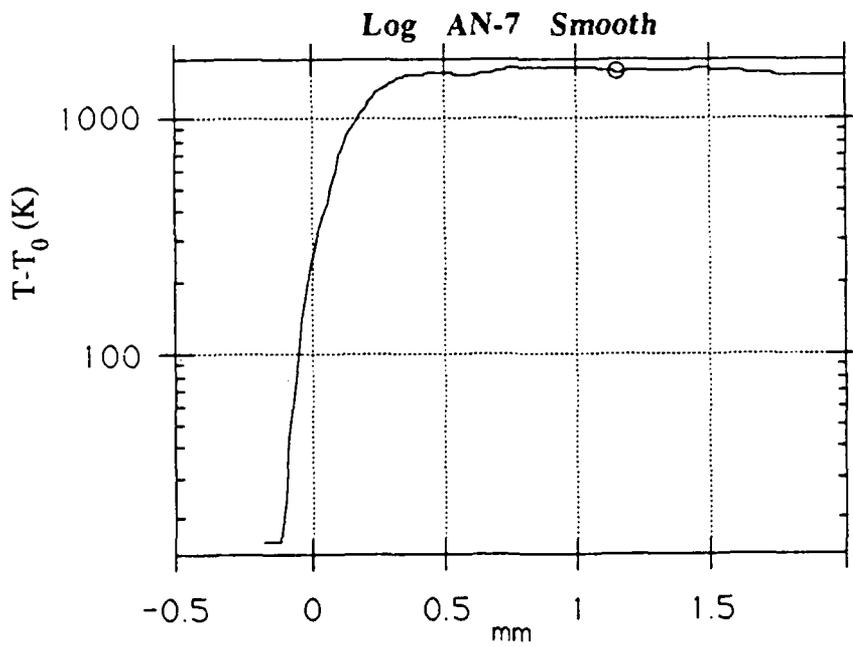
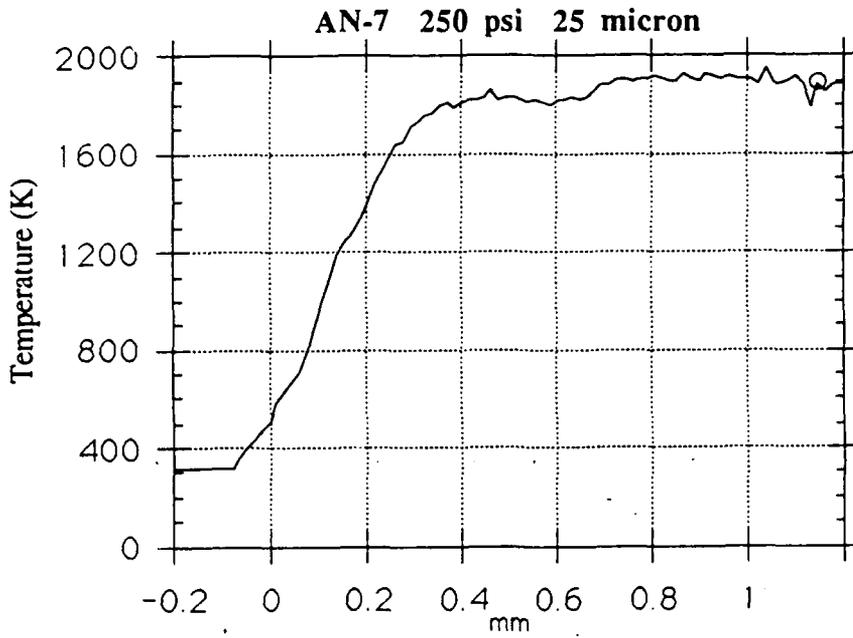


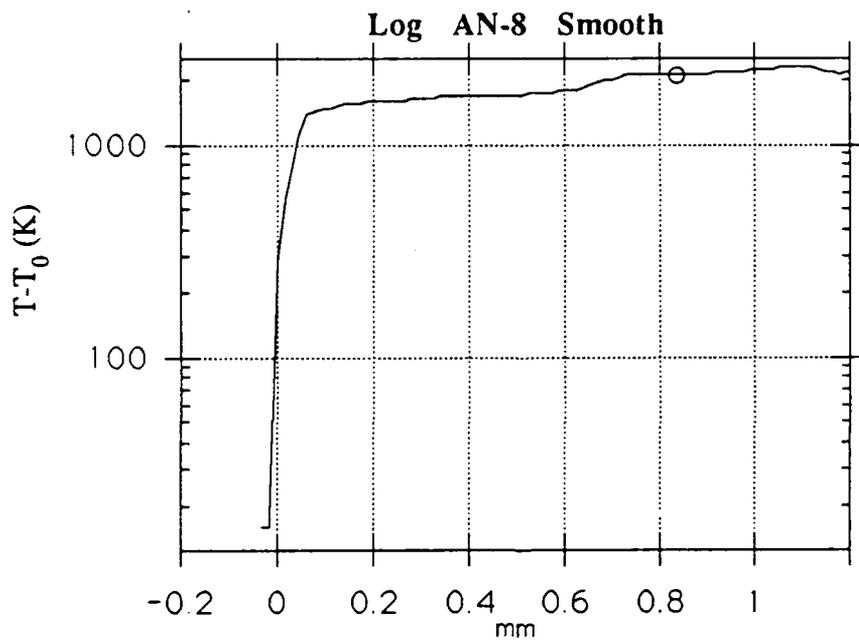
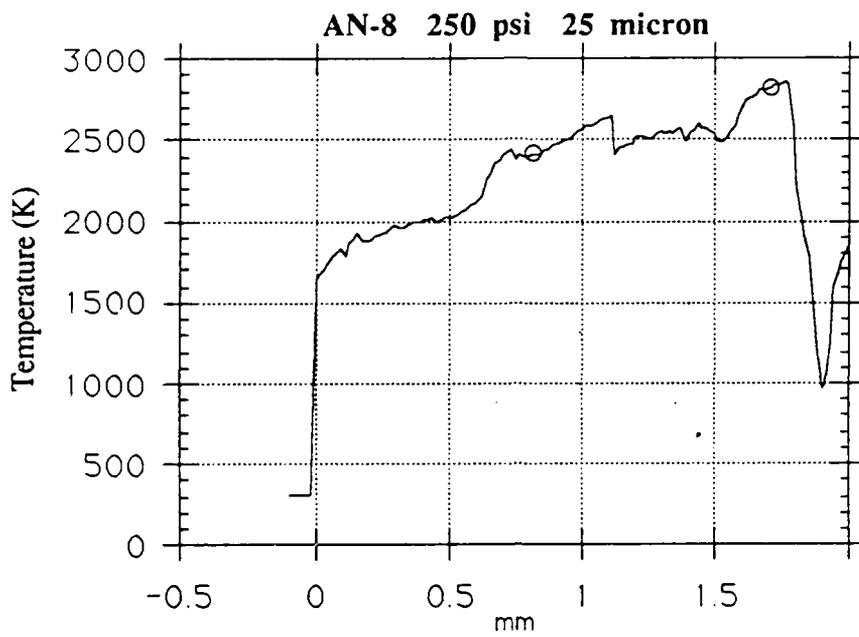
Log AN-4 Smooth

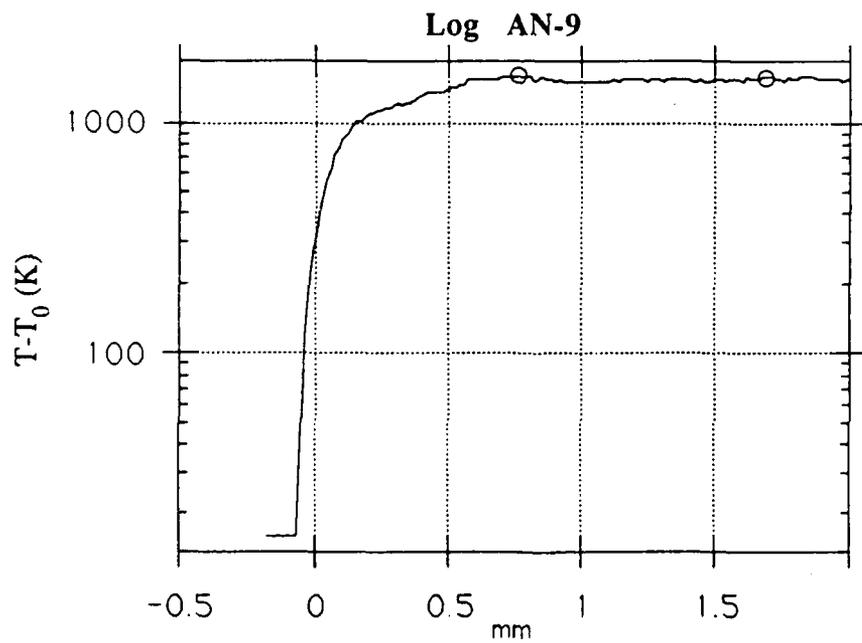
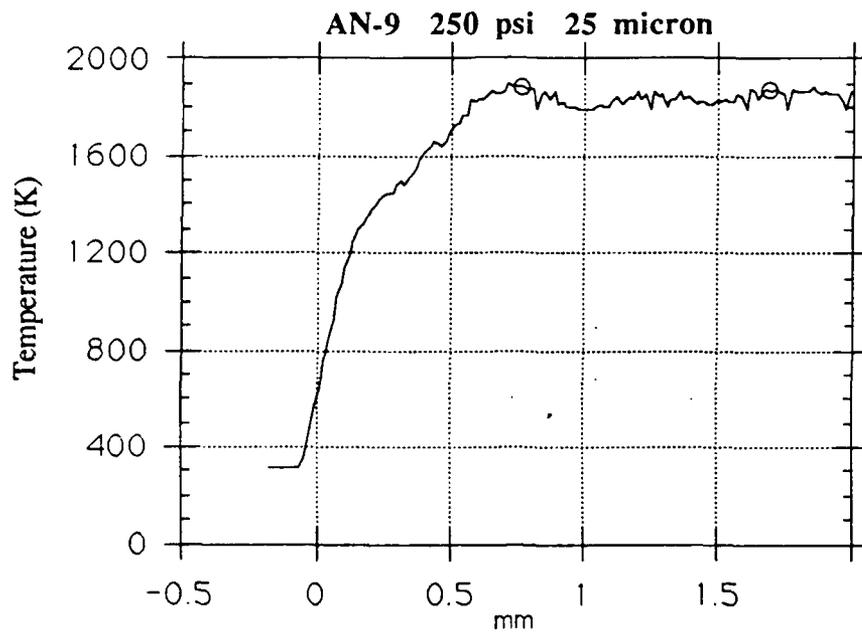


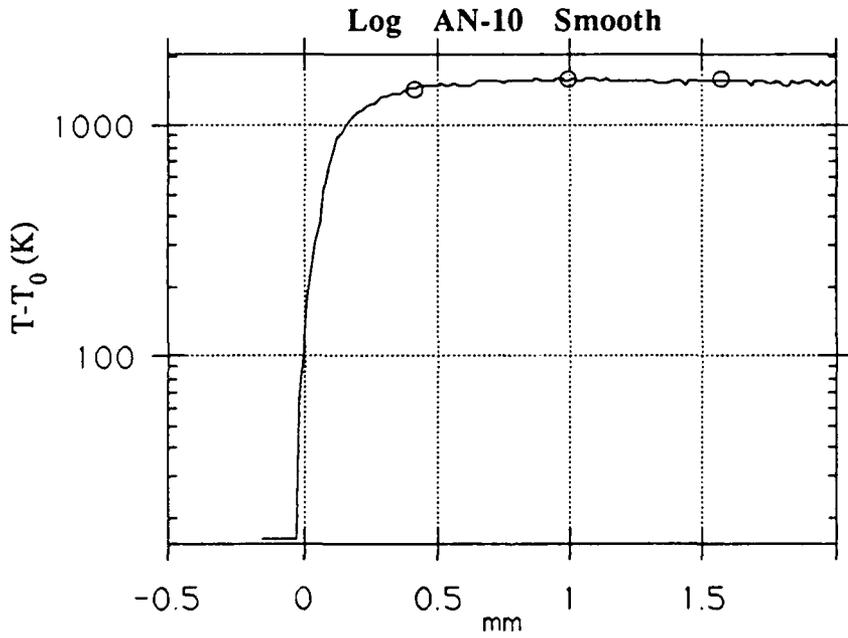
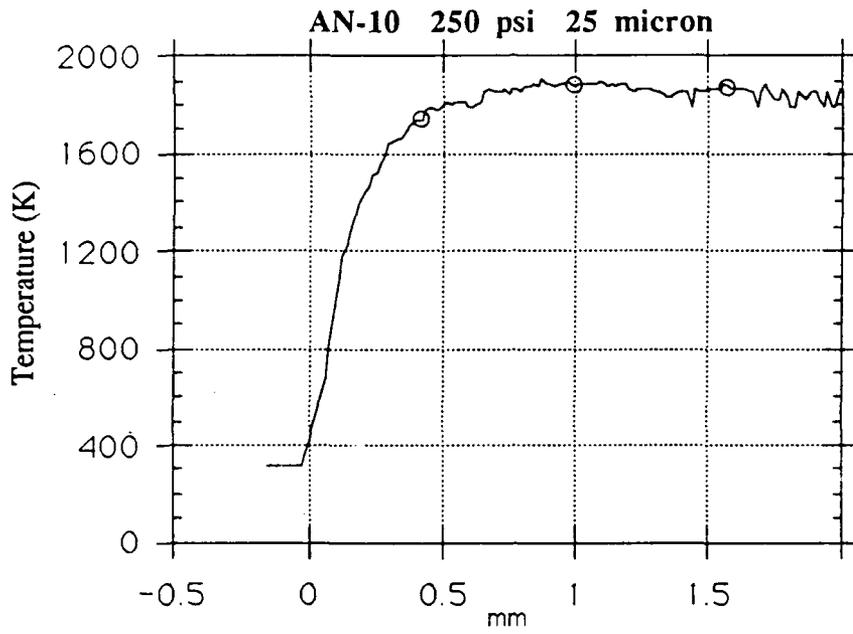


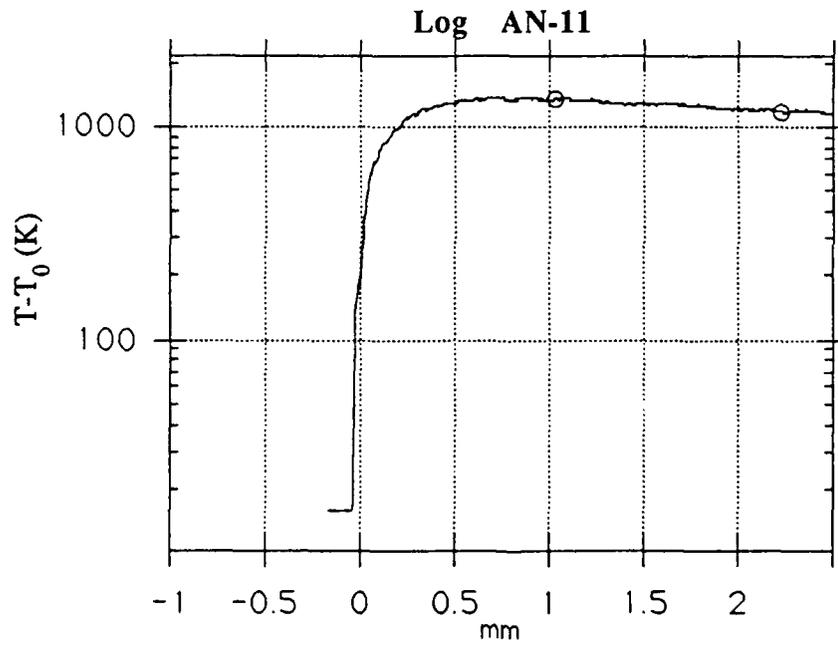
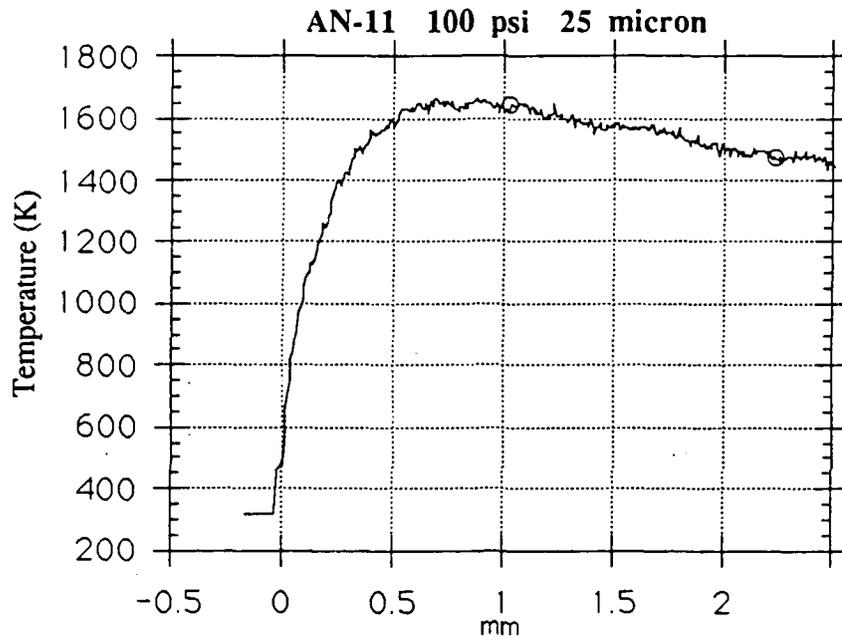


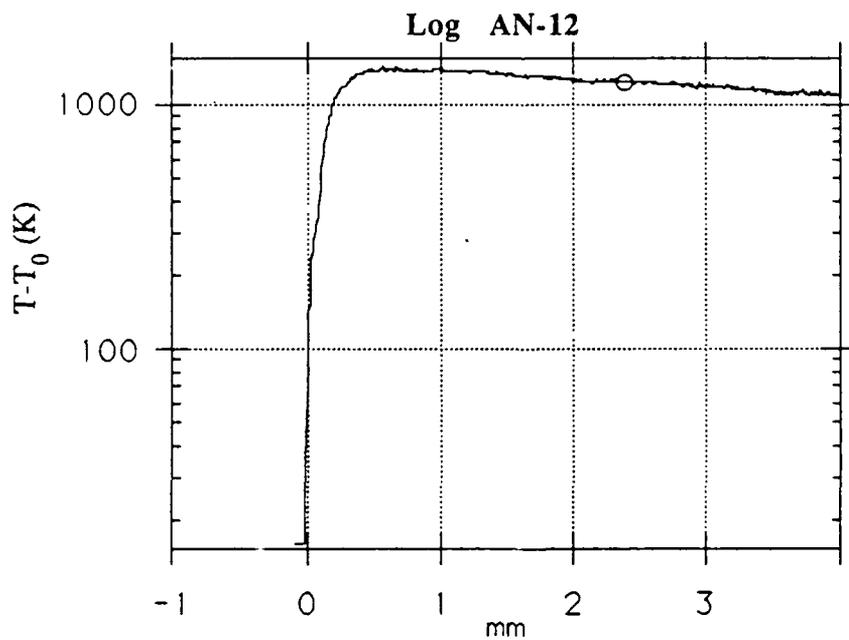
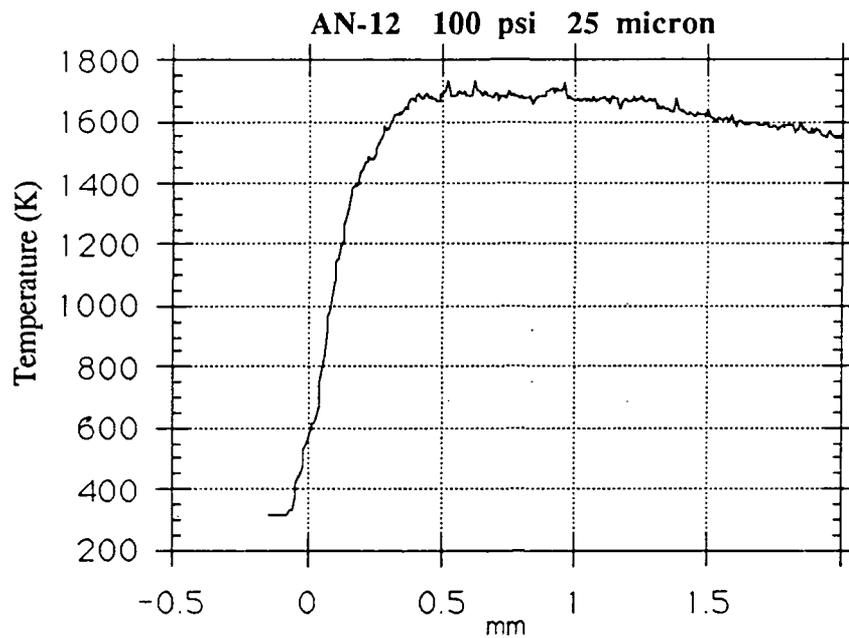


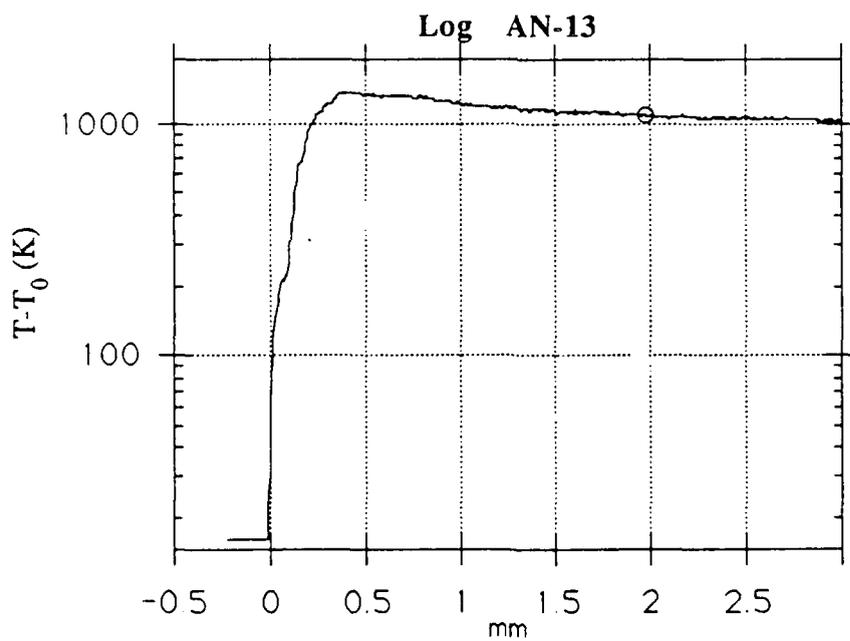
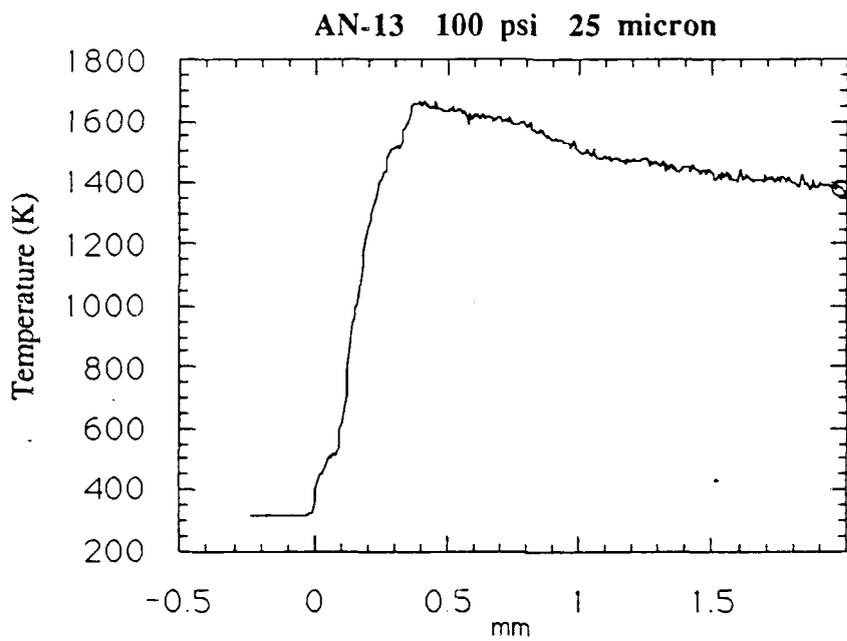


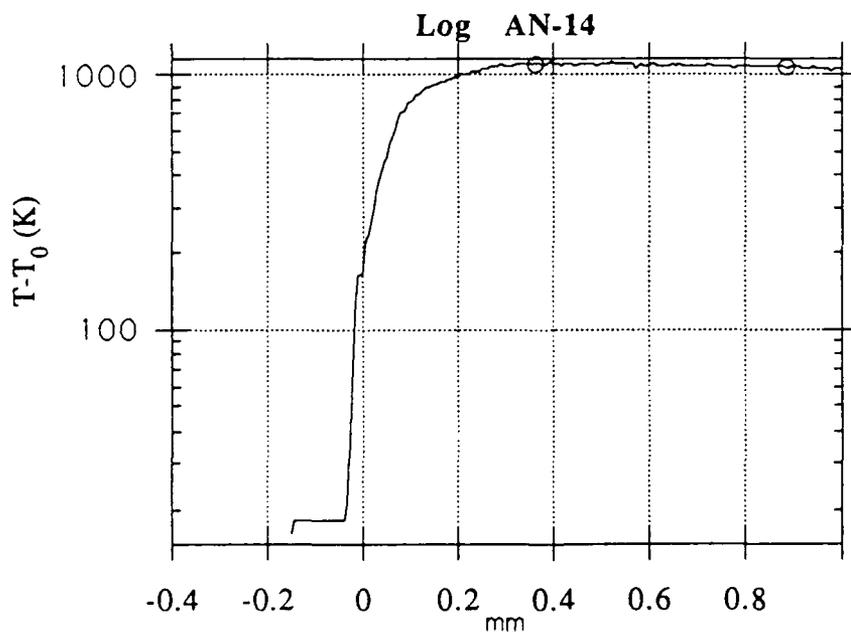
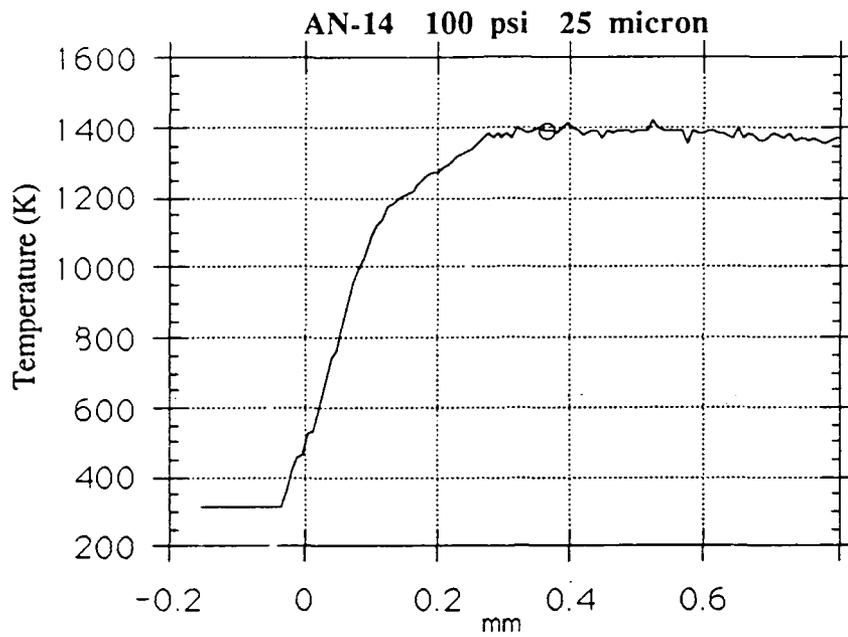


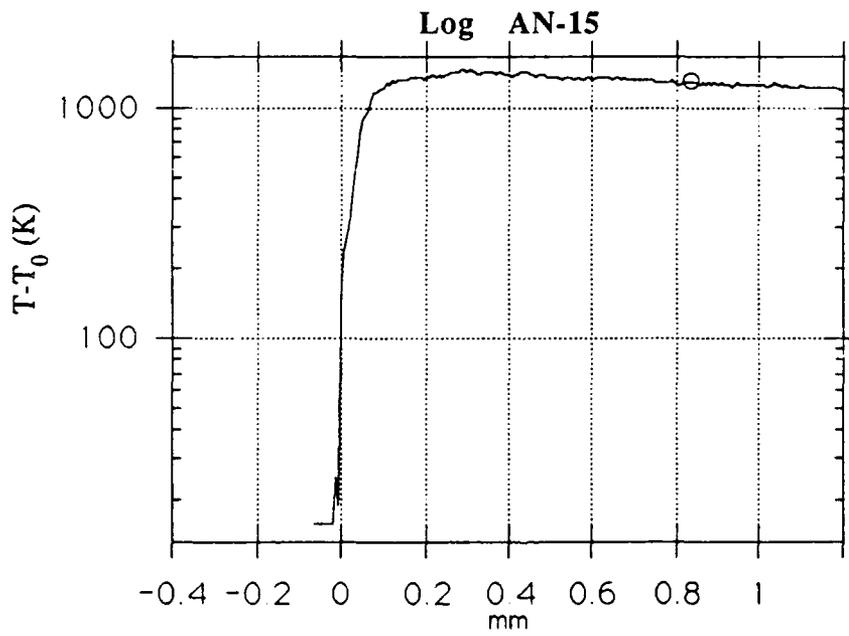
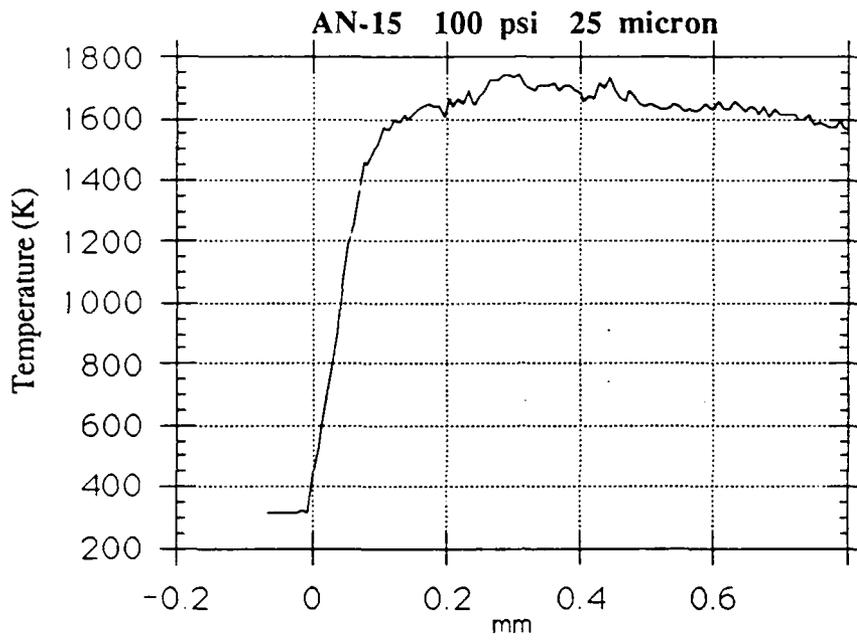


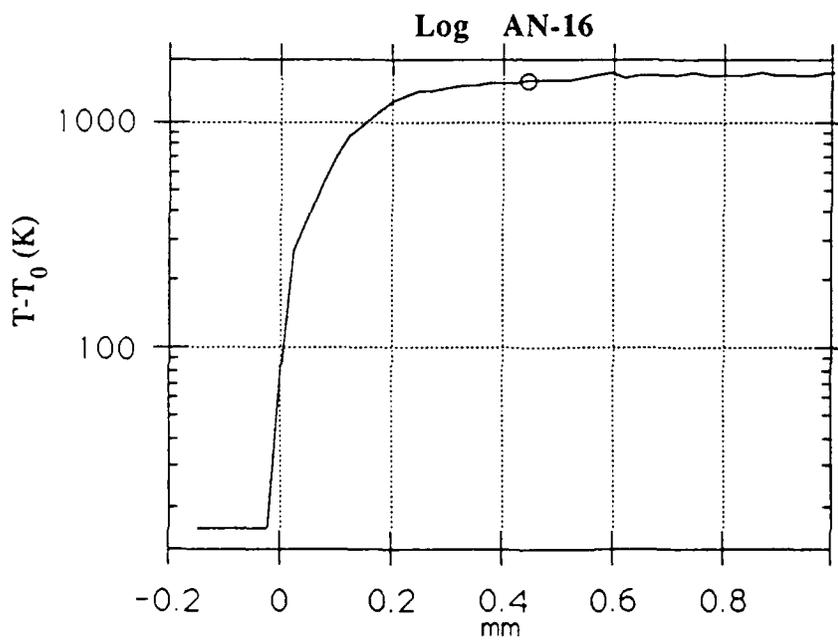
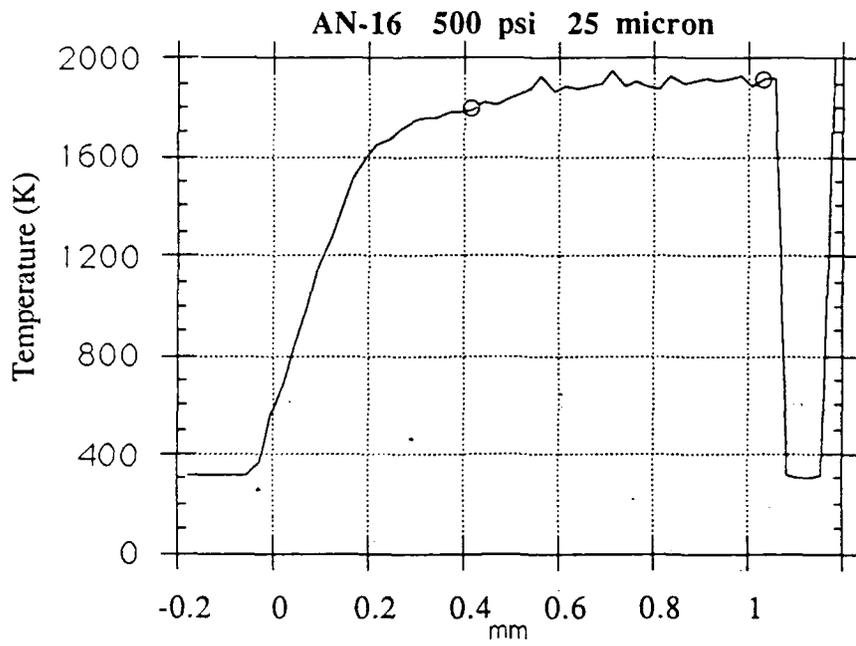


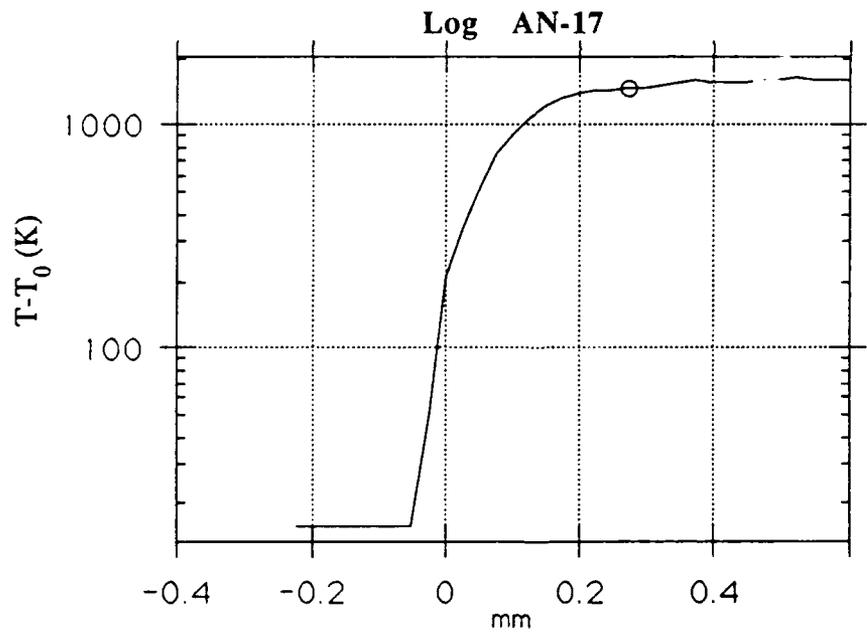
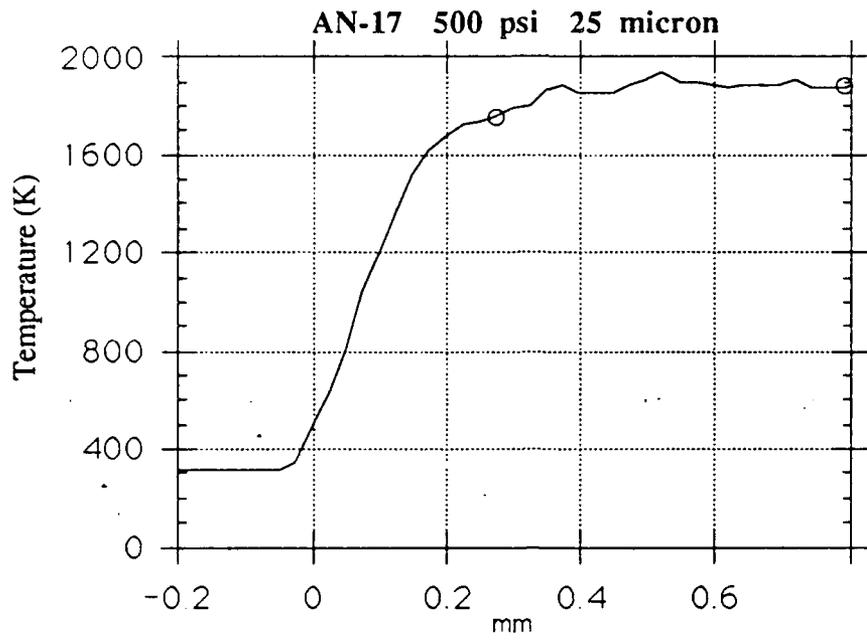


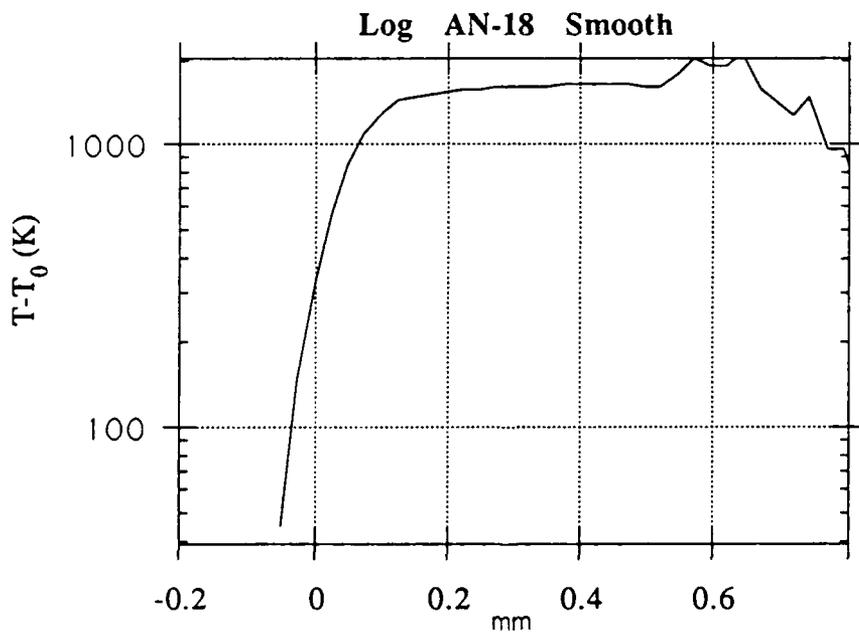
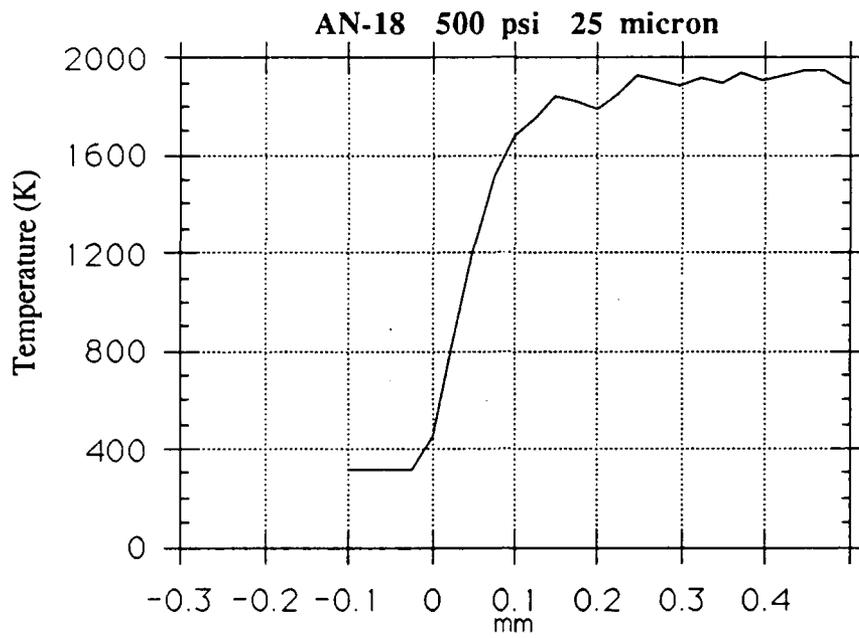






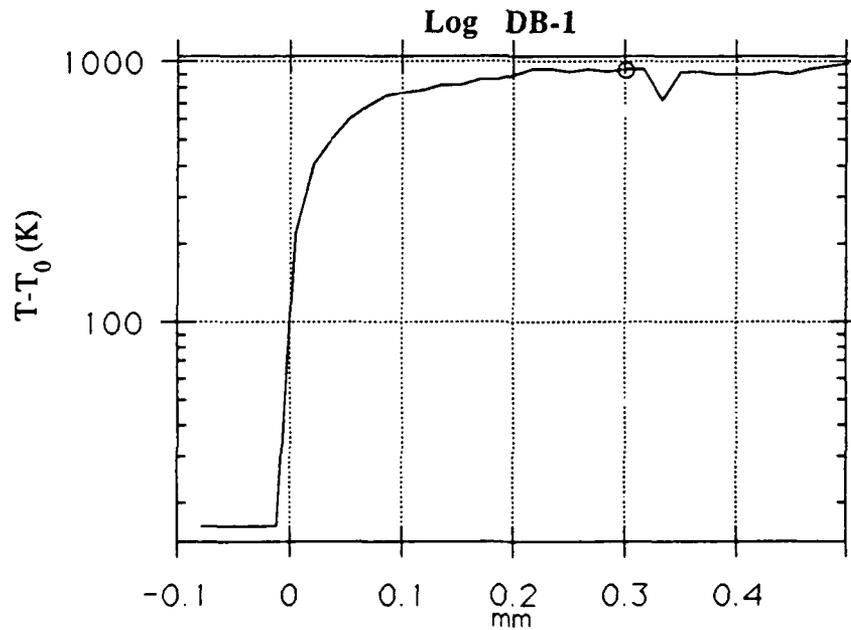
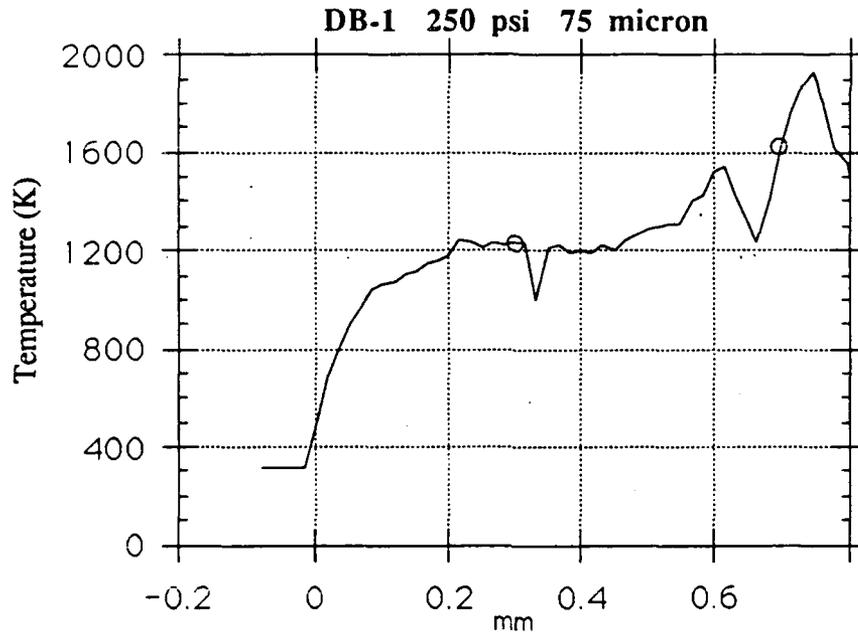


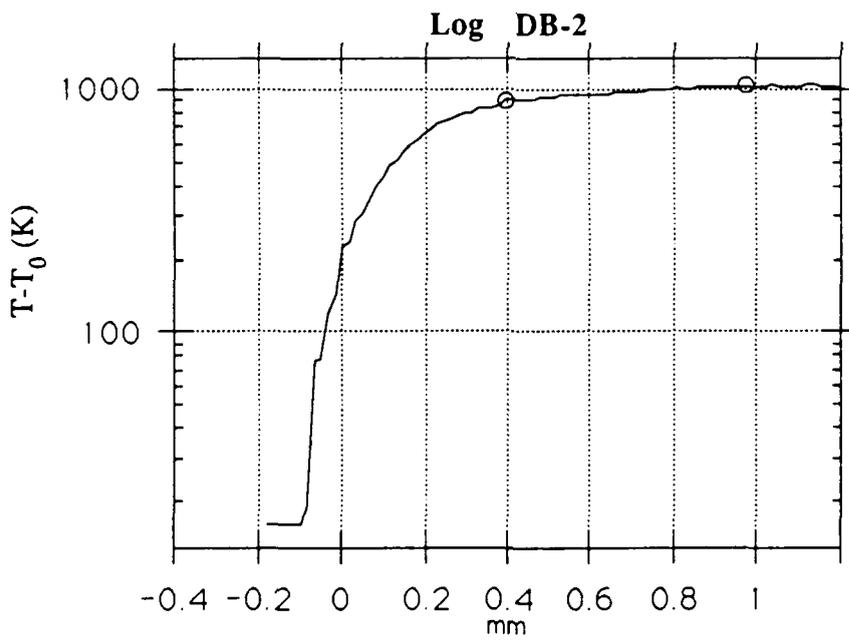
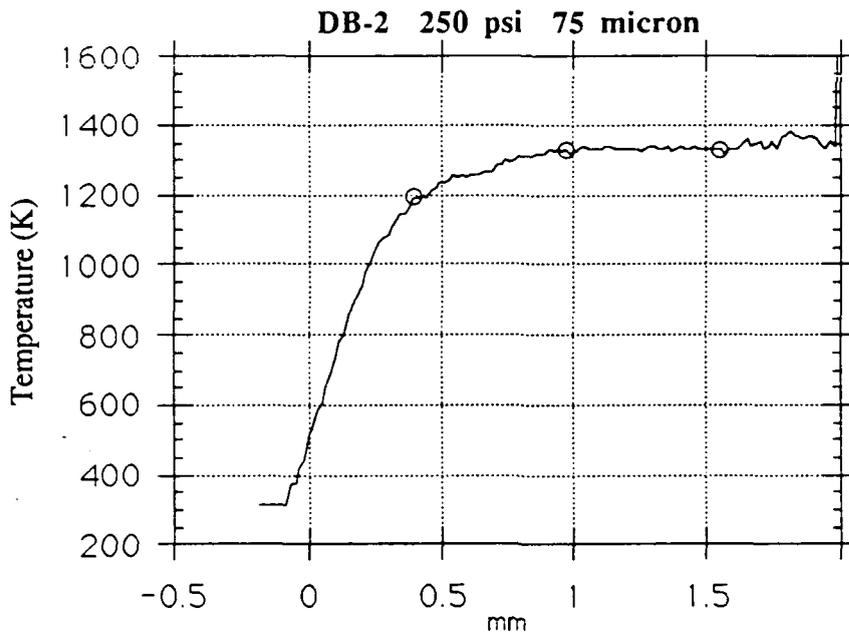


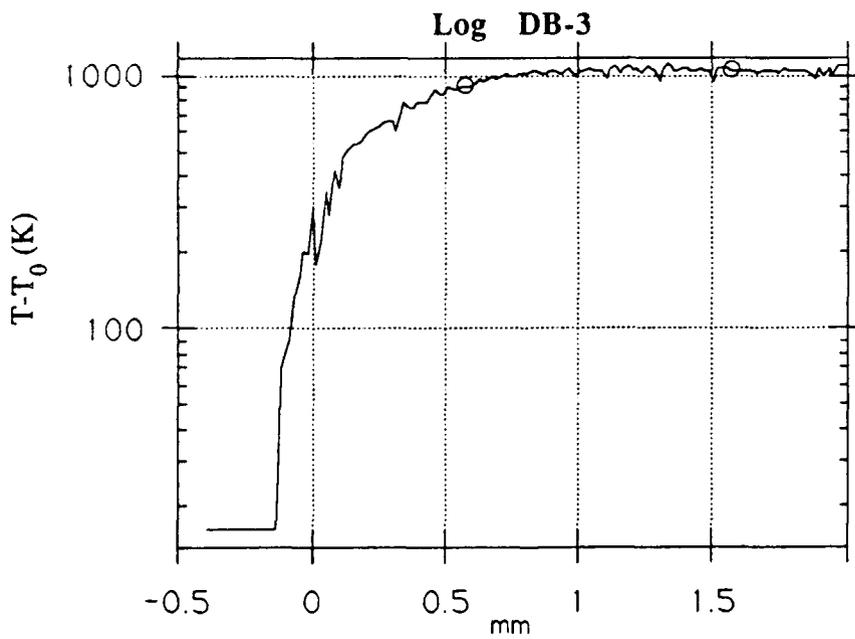
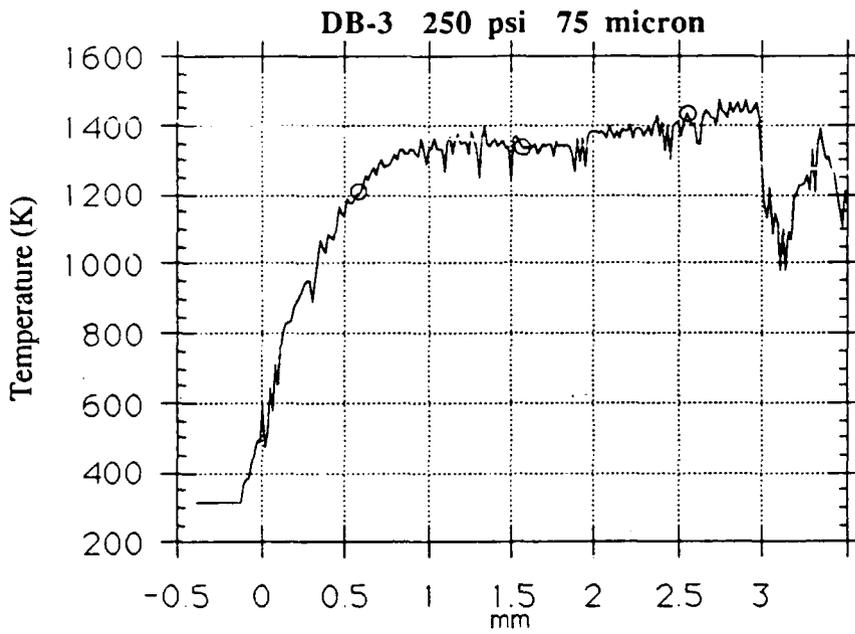


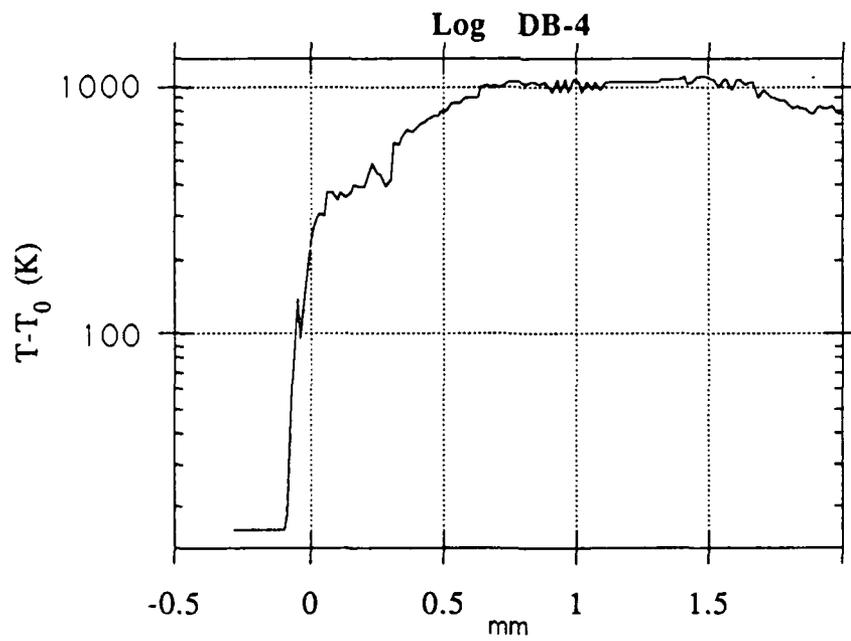
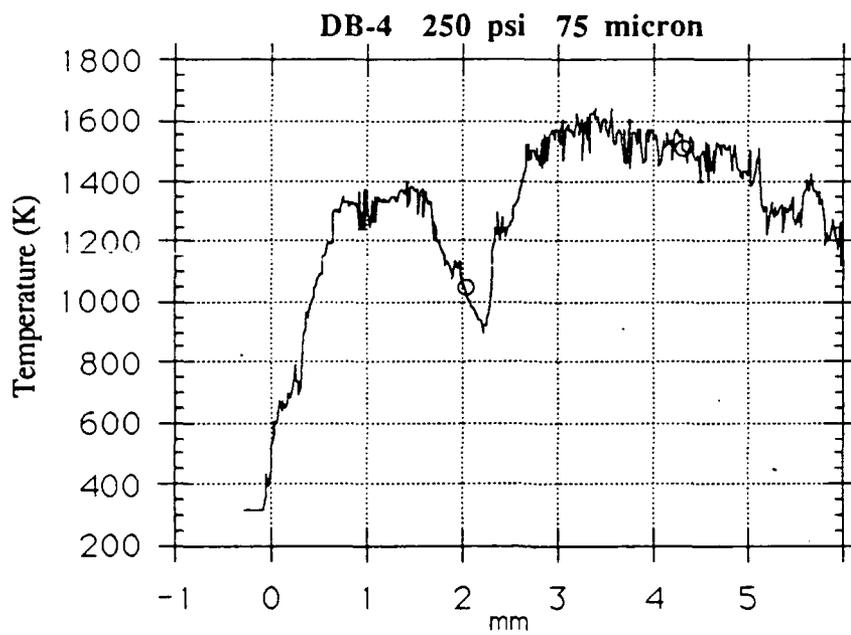
APPENDIX B. TEMPERATURE PROFILES FOR DOUBLE BASE

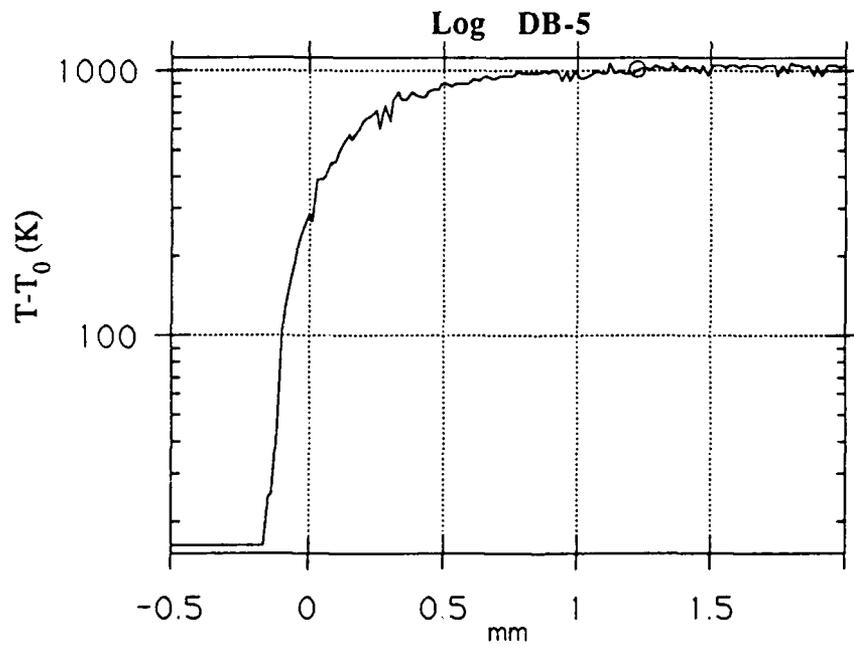
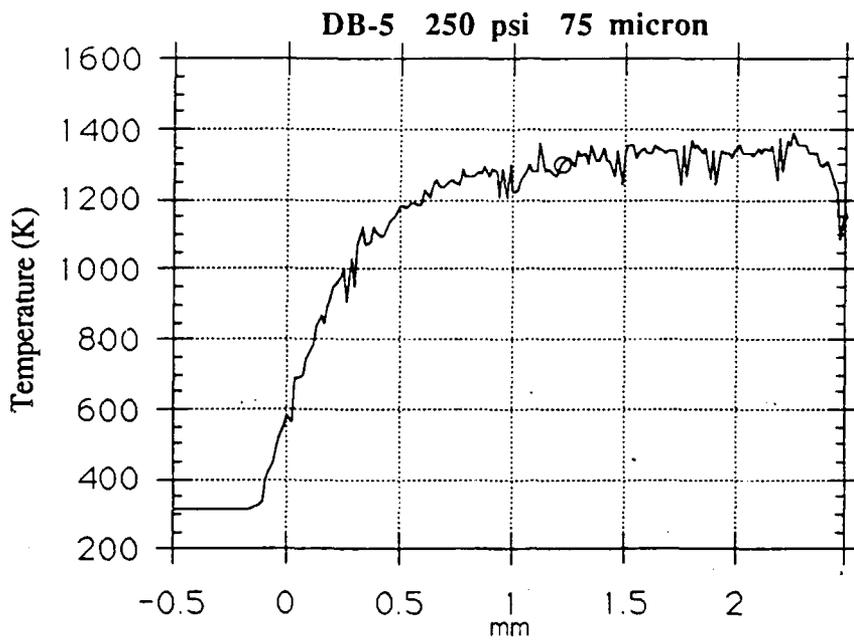
This Appendix is a compilation of all of the double base temperature profiles which were collected. One interesting aspect of these profiles is the point at which the thermocouple seems to have broken. It appears that the thermocouple often broke just as its bead was leaving the dark zone and entering the flame zone.





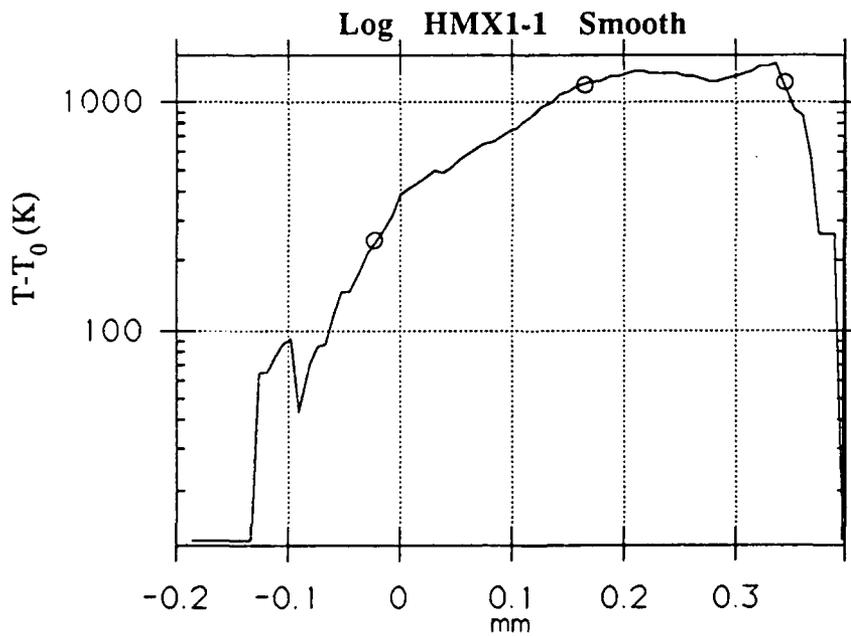
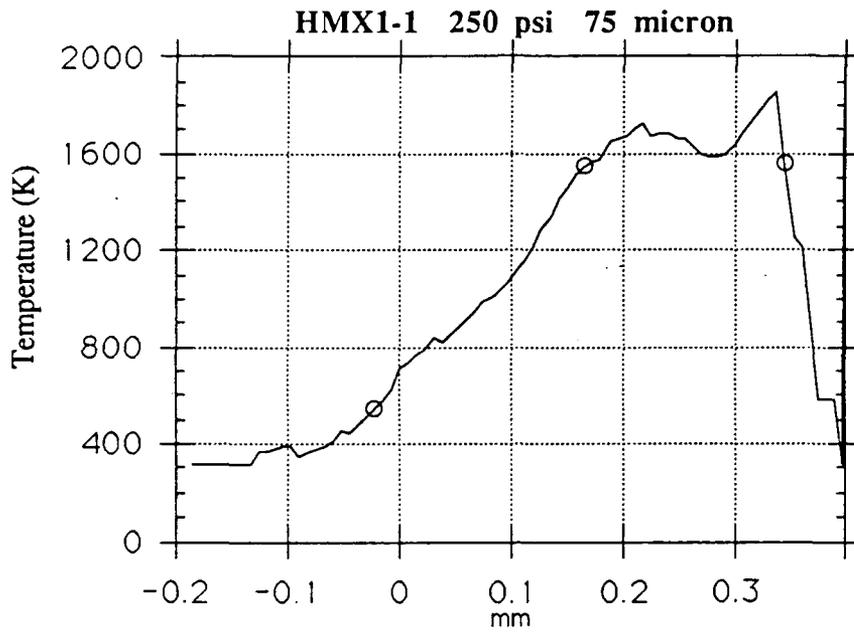


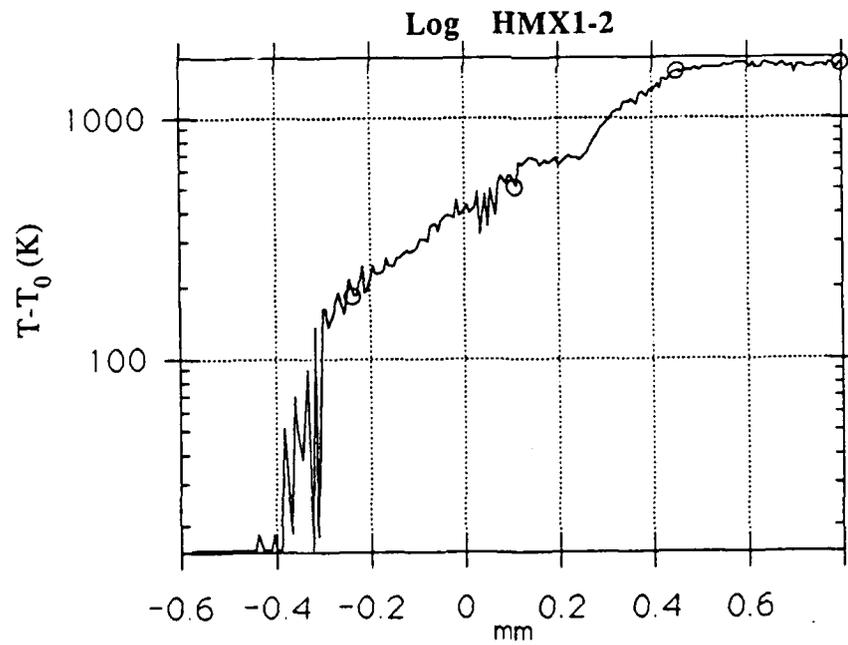
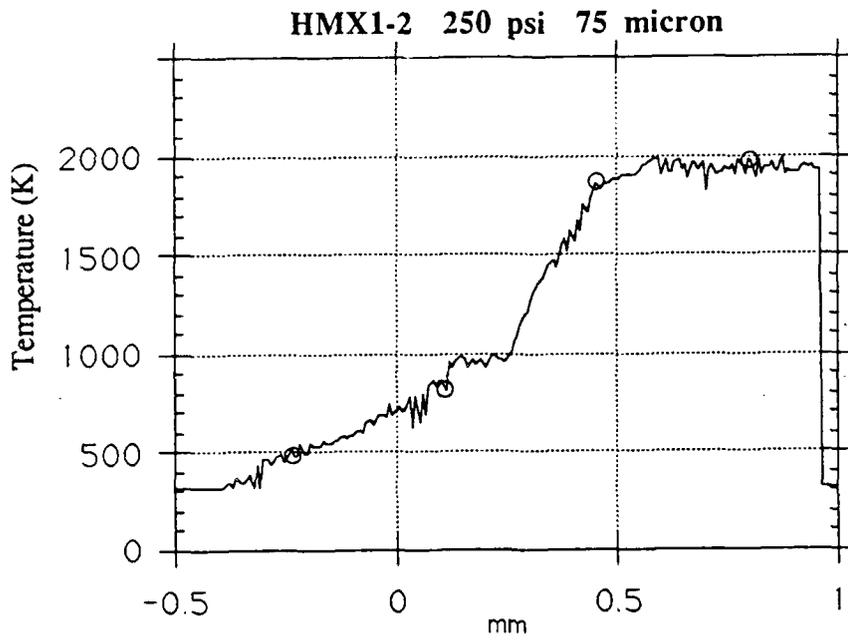


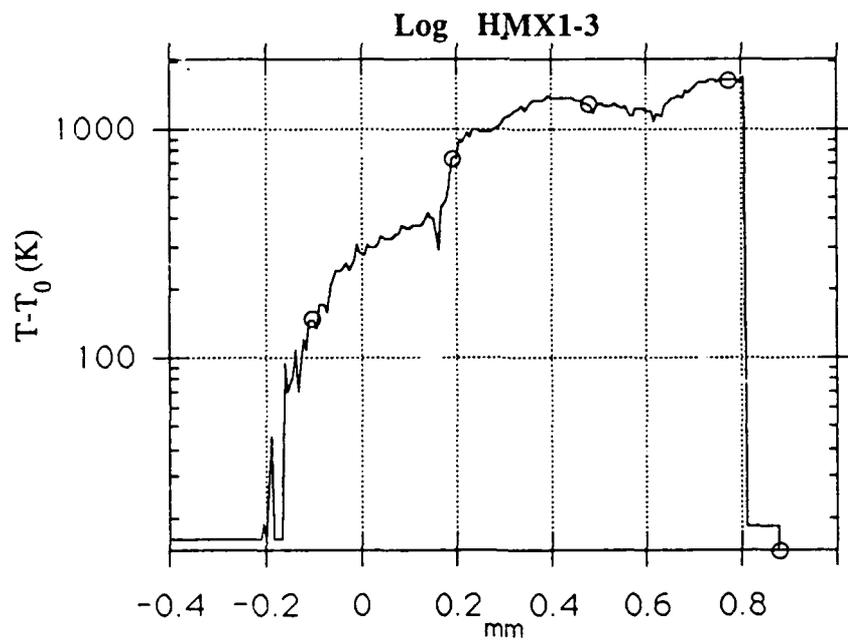
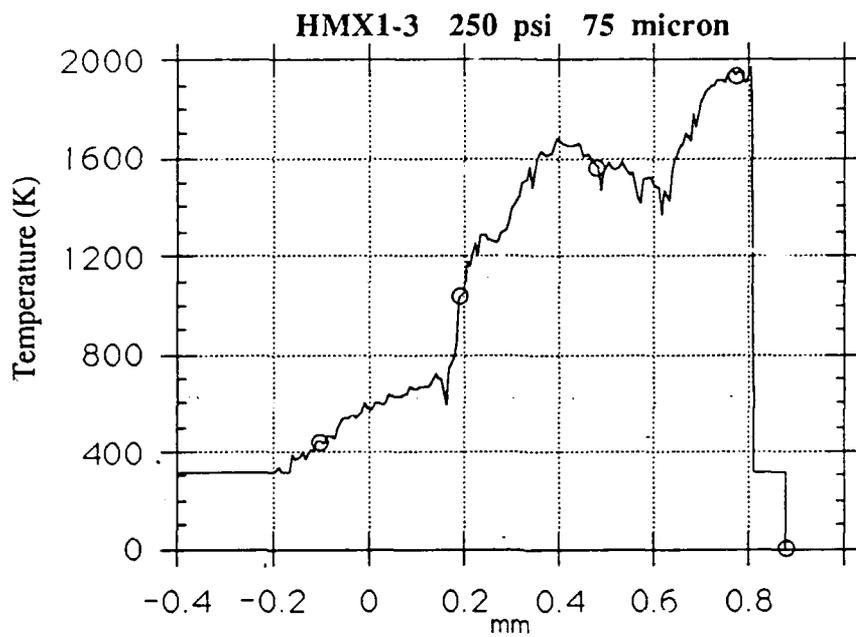


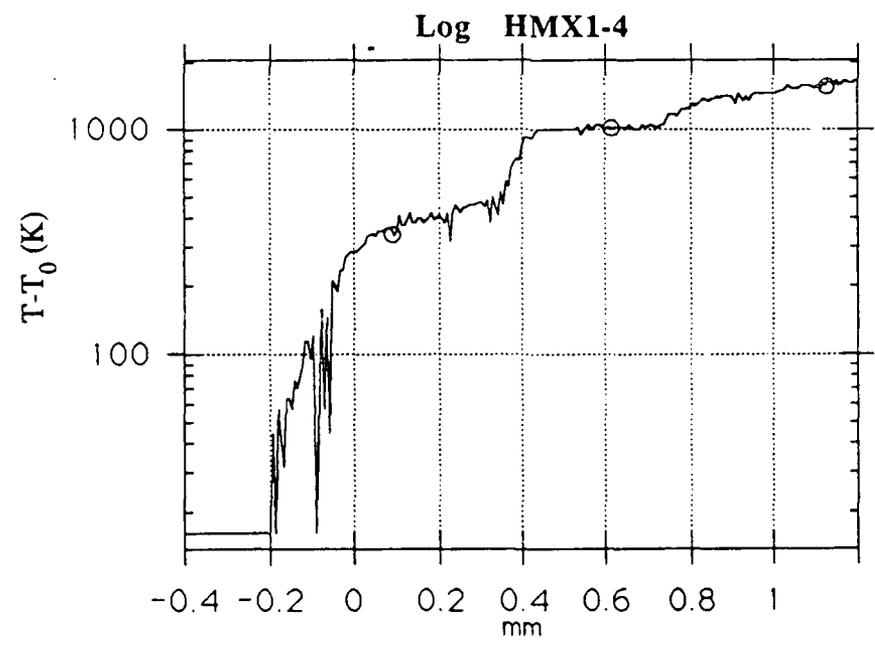
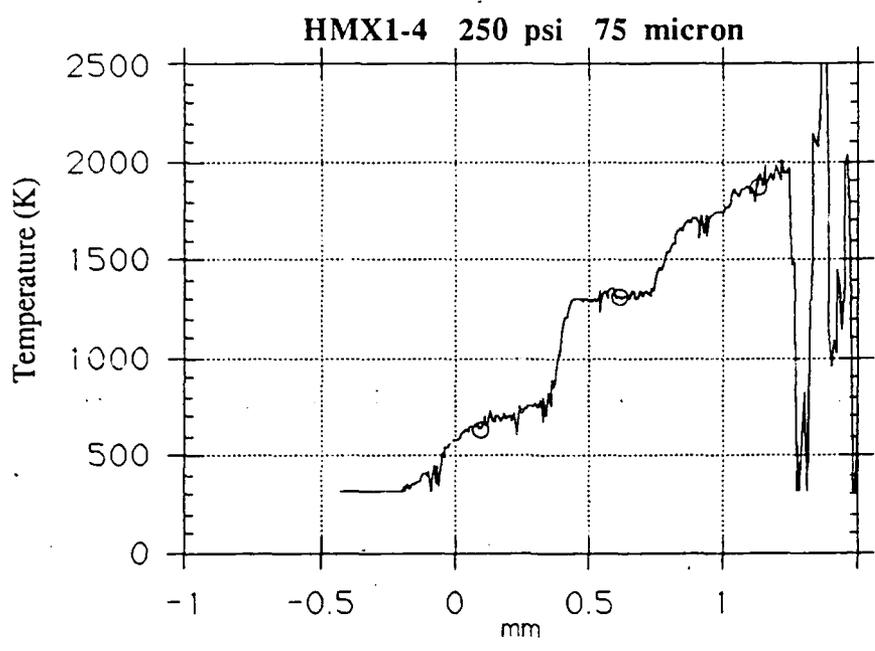
APPENDIX C. TEMPERATURE PROFILES FOR HMX1

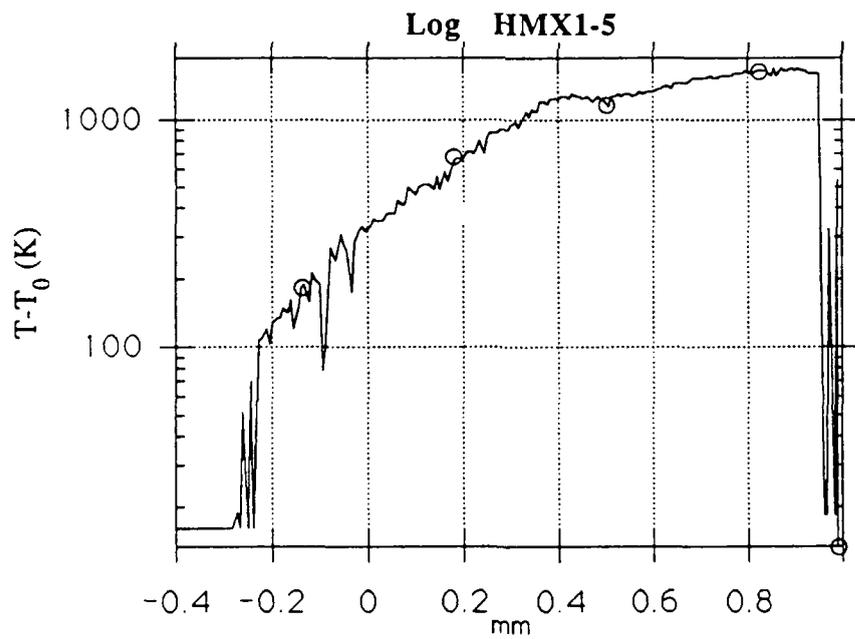
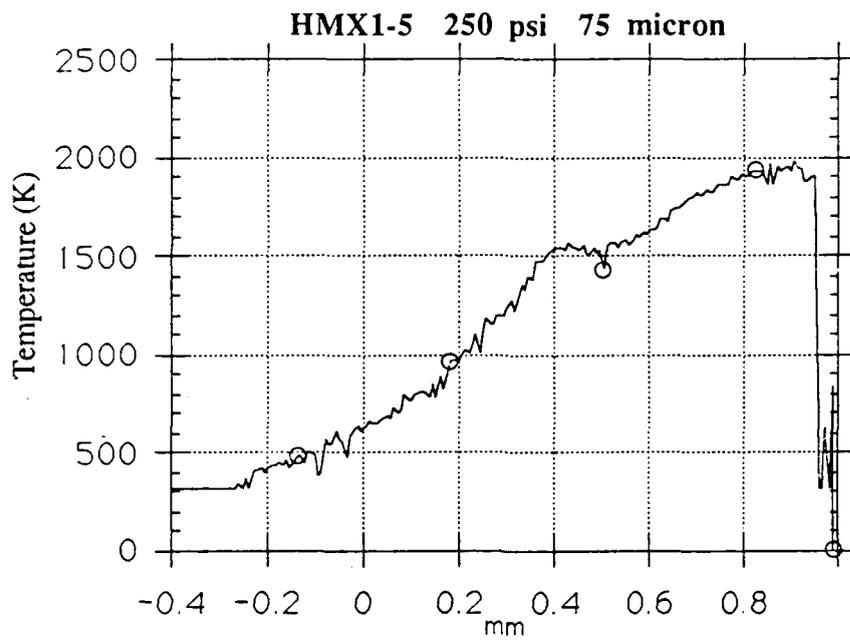
This Appendix is a compilation of all of the HMX1 temperature profiles which were collected.

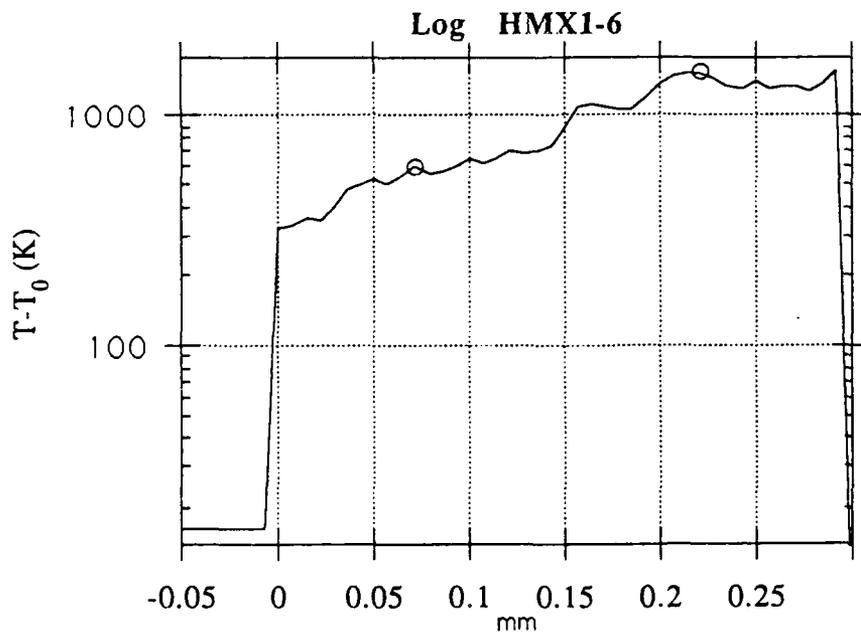
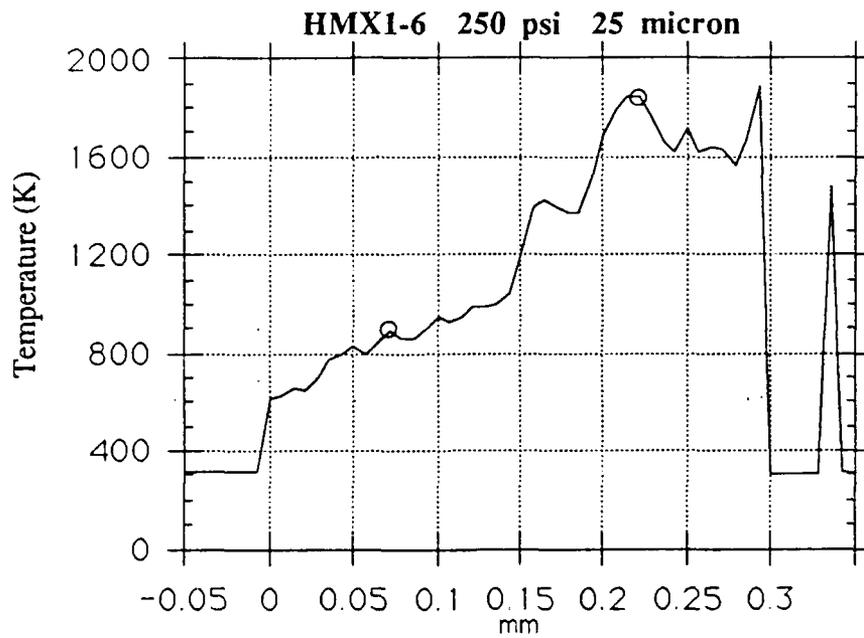


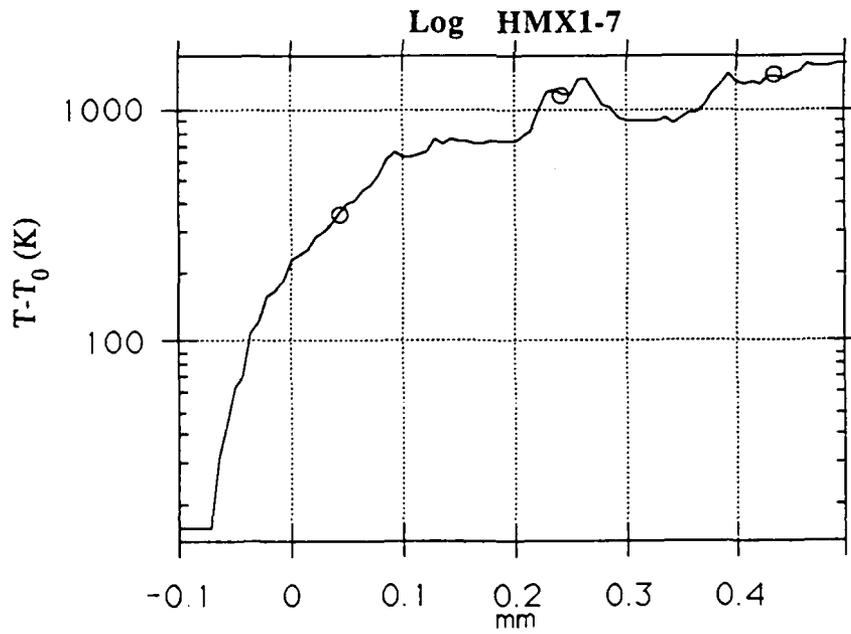
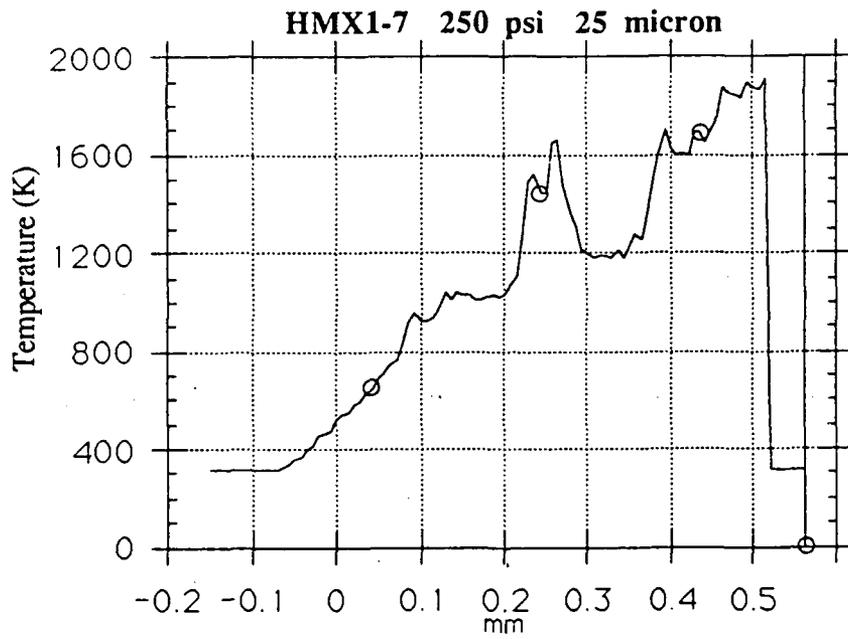


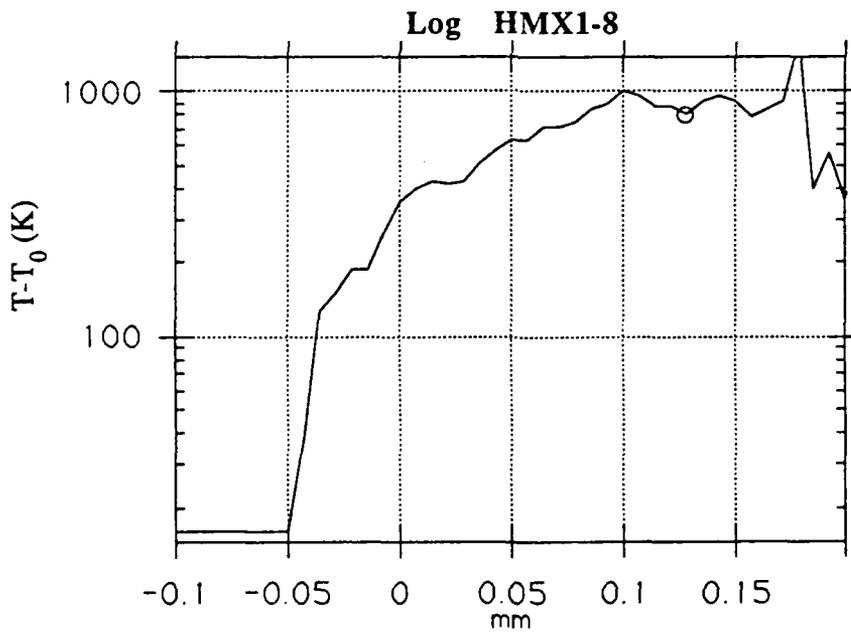
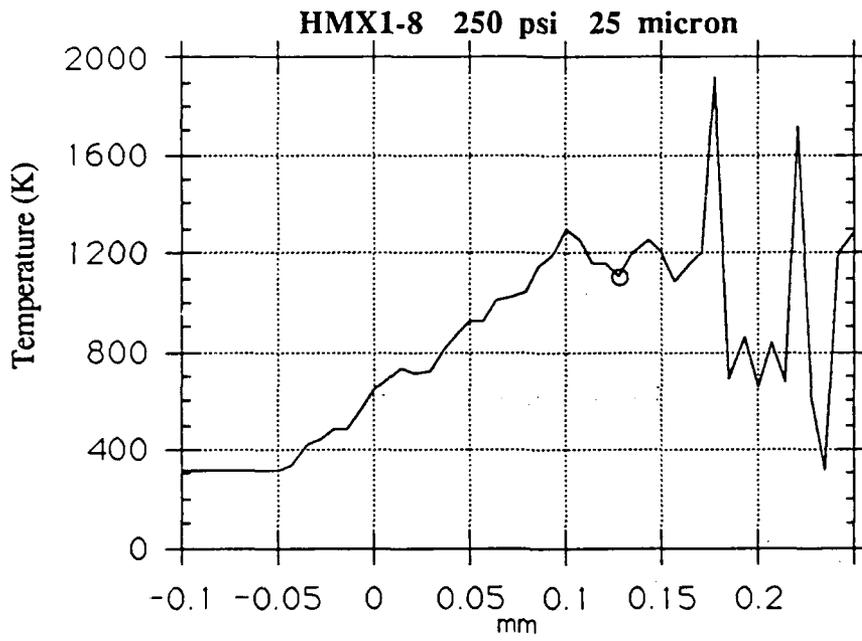


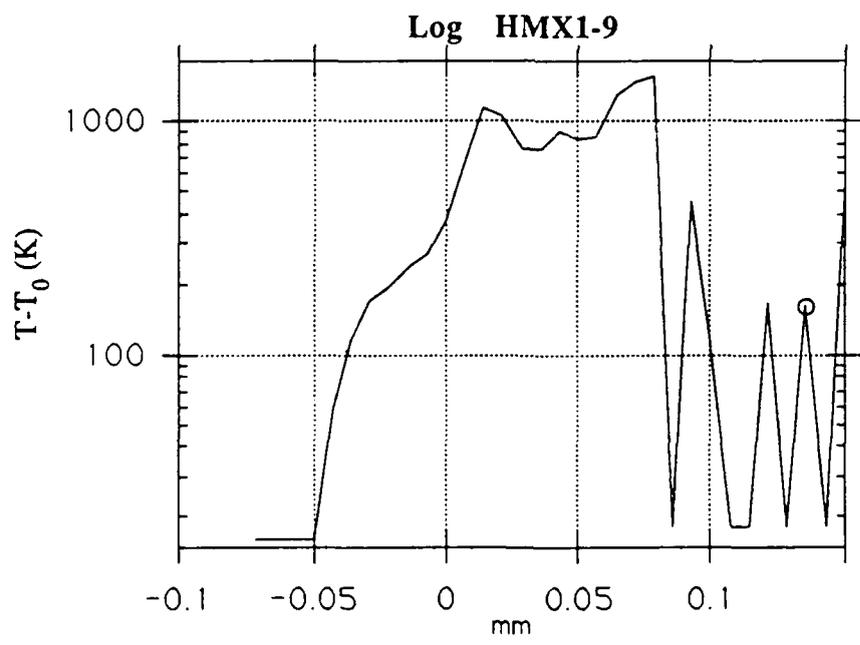
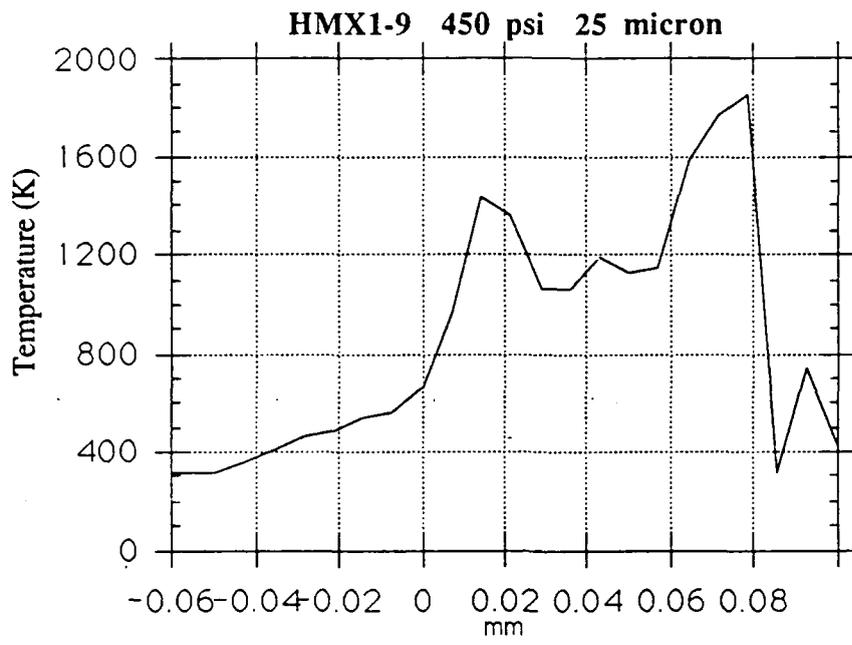


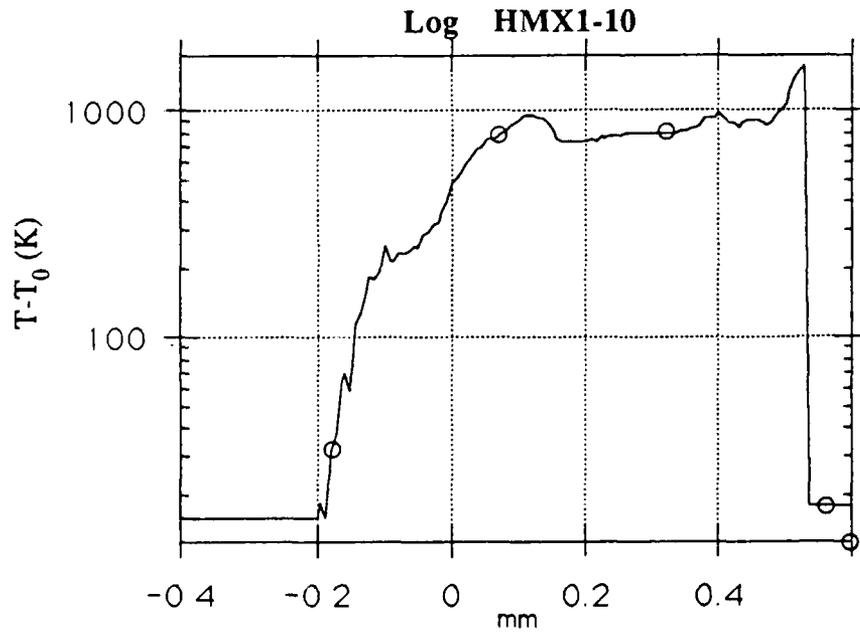
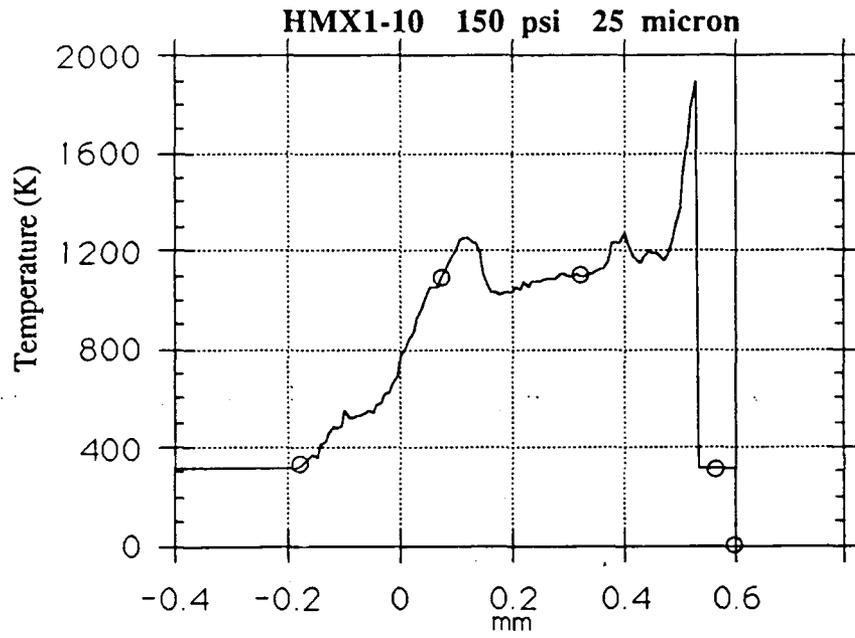












APPENDIX D. TEMPERATURE PROFILES FOR HMX2

This Appendix is a compilation of all of the HMX2 temperature profiles which were collected.

