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Report Number 1558

PERFORMANCE EVALUATION
OF "THERMO-LAG" MATERIAL FOR
ENTRY HEAT PROTECTION OF
ADVANCED MANNED SPACECRAFT

Material Development Report
1 October 1962 to 1 April 1963

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HOUSTON, TEXAS

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SUMMARY

Primary effort during the first six months of Contract NAS 9-877 was directed toward the planning and conducting of the basic specific task accomplishments in all phases of the program. Emphasis was on obtaining test data for solid-state thermal, mechanical, and environmental properties, and parameter evaluation data of "THERMO-LAG" T-500 materials. This was for the purpose of obtaining a thorough understanding of the modes of "THERMO-LAG" T-500 material in order to perform an accurate evaluation of its performance within the region of interest specified for afterbody conditions during the final reporting period.

PHASE I.

Solid-state thermal properties that would characterize "THERMO-LAG" T-500 materials for the modes of heat transfer and low-vacuum space environment were conducted. All test data is reported in tables and is illustrated in figures in order to more concisely indicate characteristics and trends of the thermal properties. Inspection of Figure 7-2 in Section VII indicates that the only thermal property tests not completed are measurements of the emissivity of virgin and charred "THERMO-LAG" T-500 materials and the gas chromatograph study.

PHASE II.

This section contains a record of the mechanical properties of "THERMO-LAG" T-500 materials which were tested during the reporting period. All material test data for both elevated and cryogenic temperature conditions is reported in tables and is illustrated in figures. Examination of Figure 7-4 in Section VII indicates that only the flexural stiffness and acoustical environmental test are not completed.

PHASE III.

Results of flight simulation tests performed during this reporting period are recorded in tables. Results of the reduced test data indicating the thermal performance of "THERMO-LAG" T-500 materials are illustrated. This section contains results of the low shear, oxidation, and low incident radiation simulation tests which are indicated in Figure 7-3 in Section VII.

PHASE IV.

Specie selection studies were performed to determine the primary and secondary bonding

techniques most suitable over the environmental temperature range from -150°F to +300°F. Results of bond shear and tensile tests performed on "THERMO-LAG" T-500-4 for five of the promising bonding techniques are recorded in tables. As indicated in the figures, further selection studies are required. Studies of secondary bonding materials indicate that further screening efforts are necessary before conducting bond shear and tensile tests.

PHASE V.

Three artificial curing techniques considered to have utility under field conditions were investigated for utilization of "THERMO-LAG" T-500 in the roles of joint, gap, and edge fillers. Investigation indicated that the chemical composition of "THERMO-LAG" T-500 prevented sufficient cure by the conductive heat and chemical accelerator techniques. Further investigation of infrared heat and polymer effects on artificial curing techniques for "THERMO-LAG" T-500 putty will be continued in the next reporting period.



SECTION I

INTRODUCTION

This report is intended to convey, clearly and concisely, the results of all tests, experiments, developments, studies, and other efforts which were either conducted or planned for use in Emerson Electric's performance evaluation of "THERMO-LAG" T-500 during the first six months of Contract NAS 9-877.

The format of this report distributes the contents as they are grouped in the Program Plan. Sections II and III present work accomplishments of the solid phase and thermal and mechanical properties, respectively, for environmental aspects. Section IV presents the test matrices and results and data reduced from the conducted flight simulation tests. These tests and results were placed in the section covering thermal performance because of the relationship of its parameter evaluation to the numerical ablation program. Sections V and VI present work accomplishments for the attachment techniques for "THERMO-LAG" T-500 materials and structural schemes, respectively. These six sections present the details of work accomplished with tables, curves, and sketches as required to convey proper meaning as specified in Schedule, Part VII, Materials Report. Section VII reflects present program planning through schedules and relates work accomplishments to overall objectives.



SECTION II

THERMAL PROPERTY AND PARAMETER EVALUATION

SOLID-PHASE THERMAL PROPERTIES (VIRGIN AND CHARRED).

THERMAL CONDUCTIVITY. Species of virgin and charred "THERMO-LAG" T-500 materials were tested for thermal conductivity values by the guarded hot-plate method described in ASTM C177-45. The test method is designed to determine the thermal conductivity of the material at various temperatures above, at, and below a standard laboratory atmosphere.

The material test specimens were circular disks 2.5 inches in diameter and 0.125 inch thick. (See Figure A-1, Appendix A.) A series of approximately thirteen successive observations were made during each test. The observations were made at intervals not exceeding one hour. For the virgin "THERMO-LAG" materials, seven tests were performed on T-500-4 and ten tests were performed on T-500-6. The results of the tests are recorded in Table 2-1 and illustrated in Figure 2-1. The data indicates that the thermal conductivity, k , of "THERMO-LAG" T-500-4 is approximately linear over the temperature range of -150°F to the sublimation temperature, and can be reasonably fitted to the empirical expression

$$k = k_o \left(\frac{T}{T_o} \right)^{0.147}$$

where the subscript (o) denotes known values. Indications are that the empirical expression may also apply to the thermal conductivity above room temperature for the virgin T-500-6 material.

The plotted data for the T-500-6 material at the temperature range below about 0°F indicates the increasing influence the glass mesh material has upon the over-all thermal conductivity of "THERMO-LAG" T-500-6. The thermal conductivity of the glass mesh material increases with decreasing temperatures below 0°F .

For the charred "THERMO-LAG" materials, nine tests were performed on T-500-4 and ten tests were performed on T-500-6. The results of the tests are recorded in Table 2-1 and illustrated in Figure 2-2. The data indicates that over the test temperature range the variation in thermal conductivity is about 12 percent. A reasonable value of the thermal conductivity would be for

"THERMO-LAG" T-500-4	0.036 BTU/HR FT $^{\circ}\text{F}$
"THERMO-LAG" T-500-6	0.0379 BTU/HR FT $^{\circ}\text{F}$

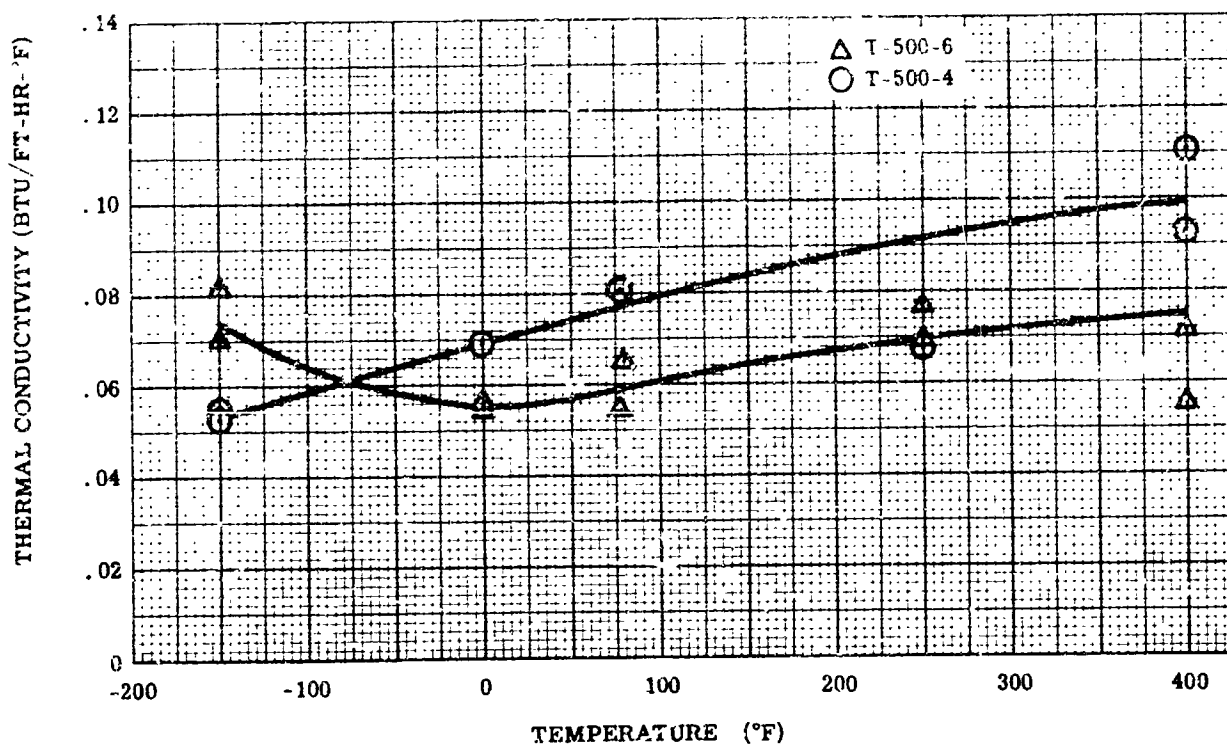


Figure 2-1. Thermal Conductivity of Virgin "THERMO-LAG" T-500-4 and T-500-6

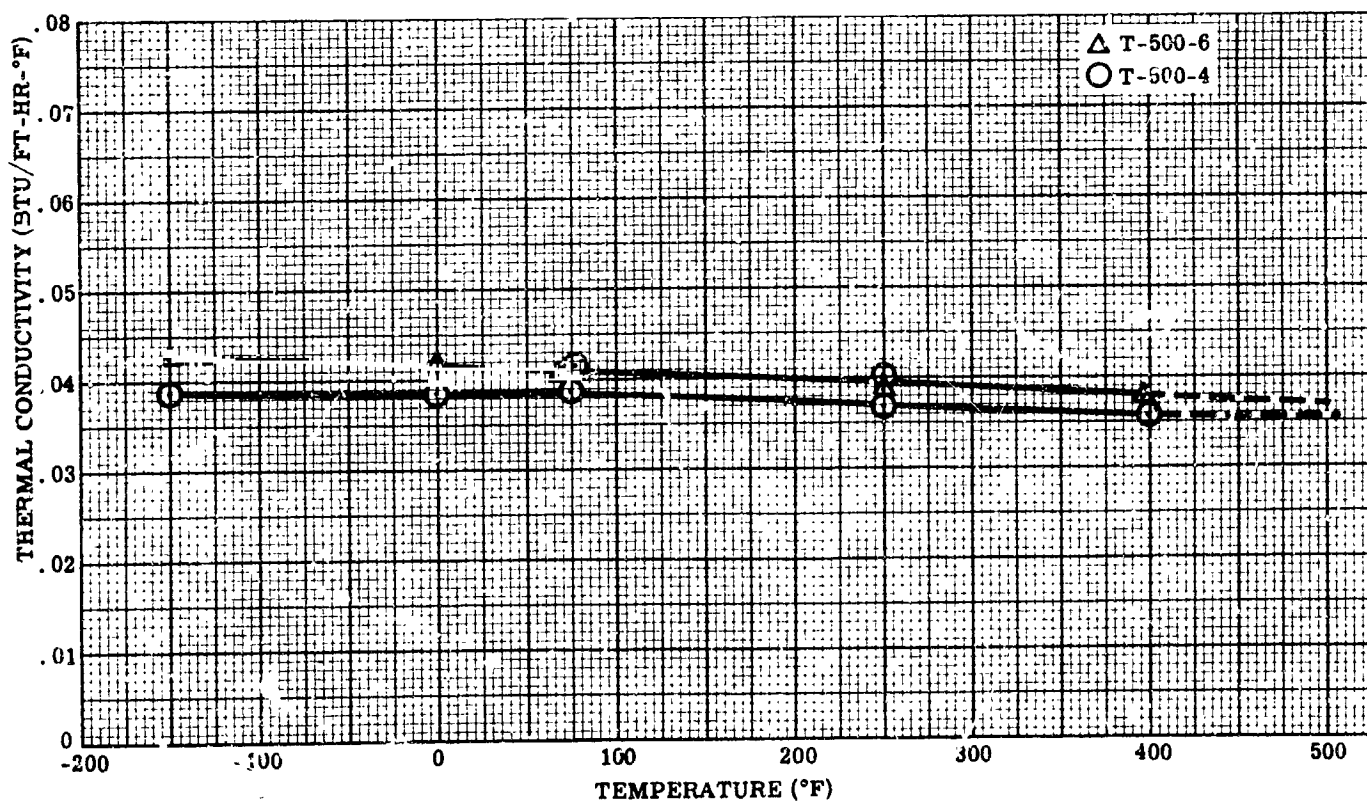


Figure 2-2. Thermal Conductivity of Charred "THERMO-LAG" T-500-4 and T-500-6



Material	Specimen No.	Test Temperature °F	Weight of Copper $\frac{W}{V_{cu}}$ GMS	Specific Heat of Copper $C_{p_{cu}}$ CAL/GM°C	Specimen Thickness 1, CM	Area Copper A_{cu} CM ²	Slope M	Conversion Factor	Thermal Conductivity k BTU/HR-FT-°F
T-500-4 Charred	10-G-1-4B-4	-150	340	.0940	.300	15.22	9360	242.0	.0376
T-500-4 Virgin	10-G-1-1-4	-150	340	.0940	.320	15.22	6970	242.0	.0533
T-500-4 Virgin	10-G-1-8-4	-150	340	.0940	.3225	15.22	6330	242.0	.0544
T-500-4 Virgin	10-G-1-16-4	0	340	.0940	.3185	15.22	5400	242.0	.0690
T-500-4 Virgin	10-G-1-6-4	0	340	.0940	.3165	15.22	5340	242.0	.0694
T-500-4 Charred	10-G-1-4C-4	0	340	.0940	.307	15.22	7740	242.0	.0466
T-500-4 Charred	10-G-1-4E-4	0	340	.0940	.400	15.22	7520	242.0	.0466
T-500-4 Charred	10-G-1-4A-4	77	340	.0940	.3145	15.22	8430	242.0	.0436
T-500-4 Charred	10-G-1-4B-4	77	340	.0940	.300	15.22	8430	242.0	.0418
T-500-4 Virgin	10-G-1-4-4	77	340	.0940	.3225	15.22	4730	242.0	.0798
T-500-4 Charred	10-G-1-19-4	250	340	.0941	.3045	15.22	8940	242.0	.0399
T-500-4 Charred	10-G-1-19A-4	250	340	.0941	.3110	15.22	9260	242.0	.0394
T-500-4 Charred	10-G-1-1-4	400	340	.0941	.3125	15.22	9460	242.0	.0369
T-500-4 Charred	10-G-1-9-4	400	340	.0945	.310	15.22	9960	242.0	.0366
T-500-4 Virgin	10-G-1-10-4	400	340	.0945	.3165	15.22	1500	242.0	.1135

Table 2-1. Test Results - Thermal Conductivity (Sheet 1 of 3)

Material	Specimen No.	Test Temperature F	Weight of Copper W_{cu} GMS	Specific Heat of Copper $C_{p_{cu}}$ CAL/GM C	Specimen Thickness l , CM	Area Copper A_{cu} CM ²	Slope M	Conversion Factor	Thermal Conductivity k BTU HR-FT- F
T-500-4 Virgin	10-G-1-17-4	400	340	.0945	.3235	15.22	2280	242.0	.0923
T-500-6 Charred	10-H-1-17A-5	-150	340	.0940	.3125	15.22	8540	242.0	.0429
T-500-6 Charred	10-H-1-17B-6	-150	340	.0940	.310	15.22	8180	242.0	.0434
T-500-6 Virgin	10-H-2-17-6	-150	340	.0940	.320	15.22	5281	242.0	.0710
T-500-6 Virgin	10-H-2-17-6A	-150	340	.0940	.320	15.22	4560	242.0	.0820
T-500-6 Virgin	10-G-2-5-6	77	340	.0940	.320	15.22	7050	242.0	.0532
T-500-6 Virgin	10-G-2-2-6	77	340	.0940	.310	15.22	5870	242.0	.0623
T-500-6 Virgin	10-G-2-4-6	250	340	.0941	.3275	15.22	5650	242.0	.0698
T-500-6 Virgin	10-G-2-9-6	250	340	.0941	.317	15.22	5240	242.0	.0717
T-500-6 Virgin	10-H-2-16-6	400	340	.0945	.320	15.22	7040	242.0	.0537
T-500-6 Virgin	10-G-2-10-6	400	340	.0945	.337	15.22	5650	242.0	.0699
T-500-6 Virgin	10-H-2-20-6	0	340	.0940	.3264	15.22	1150	242.0	.0566
T-500-6 Virgin	10-G-2-11-6	0	340	.0940	.3264	15.22	1660	242.0	.0543
T-500-6 Charred	10-H-2-15-6	0	340	.0940	.3175	15.22	8840	242.0	.0422
T-500-6 Charred	10-H-2-6-6	0	340	.0940	.3022	15.22	8170	242.0	0.433

Table 2-1. Test Results - Thermal Conductivity (Sheet 2 of 3)



Material	Specimen No.	Test Temperature °F	Weight of Copper W_{cu} GMS.	Specific Heat of Copper $C_{p_{cu}}$ CAL/GM °C	Specimen Thickness l , CM	Area Copper A_{cu} CM ²	Slope M	Conversion Factor	Thermal Conductivity k BTU HR-FT-°F
T-500-6 Charred	10-H-2-17-6	77	340	.0940	.3145	15.22	8175	242.0	.0452
T-500-6 Charred	10-H-2-19-6	77	340	.0940	.320	15.22	8840	242.0	.0422
T-500-6 Charred	10-G-2-8-6	250	340	.0941	.3045	15.22	9300	242.0	.0384
T-500-6 Charred	10-H-2-15-6	250	340	.0941	.3125	15.22	9260	242.0	.0395
T-500-6 Charred	10-H-2-19-6	400	340	.0945	.3085	15.22	10960	242.0	.0332
T-500-6 Charred	10-G-2-8-6	400	340	.0945	.310	15.22	9500	242.0	.0300

Table 2-1. Test Results - Thermal Conductivity (Sheet 3 of 3)

SPECIFIC HEAT. Species of virgin and charred "THERMO-LAG" materials, T-500-4 and T-500-6 were tested for specific heat values. The tests were conducted using a method of mixtures with a Parr calorimeter. A description of the material test specimen is illustrated in Figure A-2, Appendix A. Mean specific heats were calculated from the tests performed at temperatures above and below standard laboratory values by the equation

$$C_{ps} = \frac{W_t C_{pt} (T_{t1} - T_{t2})}{W_s (T_{s2} - T_{s1})}$$

where:

C_{ps} = Specific heat of samples

W_t = Weight of toluene (immersion liquid)

W_s = Total weight of sample

T_{t1} = Initial temperature of toluene

T_{s2} = Initial temperature of samples

For $T_{s2} = T_{t2}$, there exists equilibrium temperatures between samples and toluene.

The results of the conducted tests are recorded in Table 2-2 and illustrated in Figures 2-3 and 2-4. Figure 2-3 shows plots of the mean specific heat of virgin "THERMO-LAG" materials, T-500-4 and T-500-6 as a function of temperature. The charred "THERMO-LAG" materials, T-500-4 and T-500-6, as a function of temperature is given in Figure 2-4. The trends of the plotted data are typical for the types of material tested.

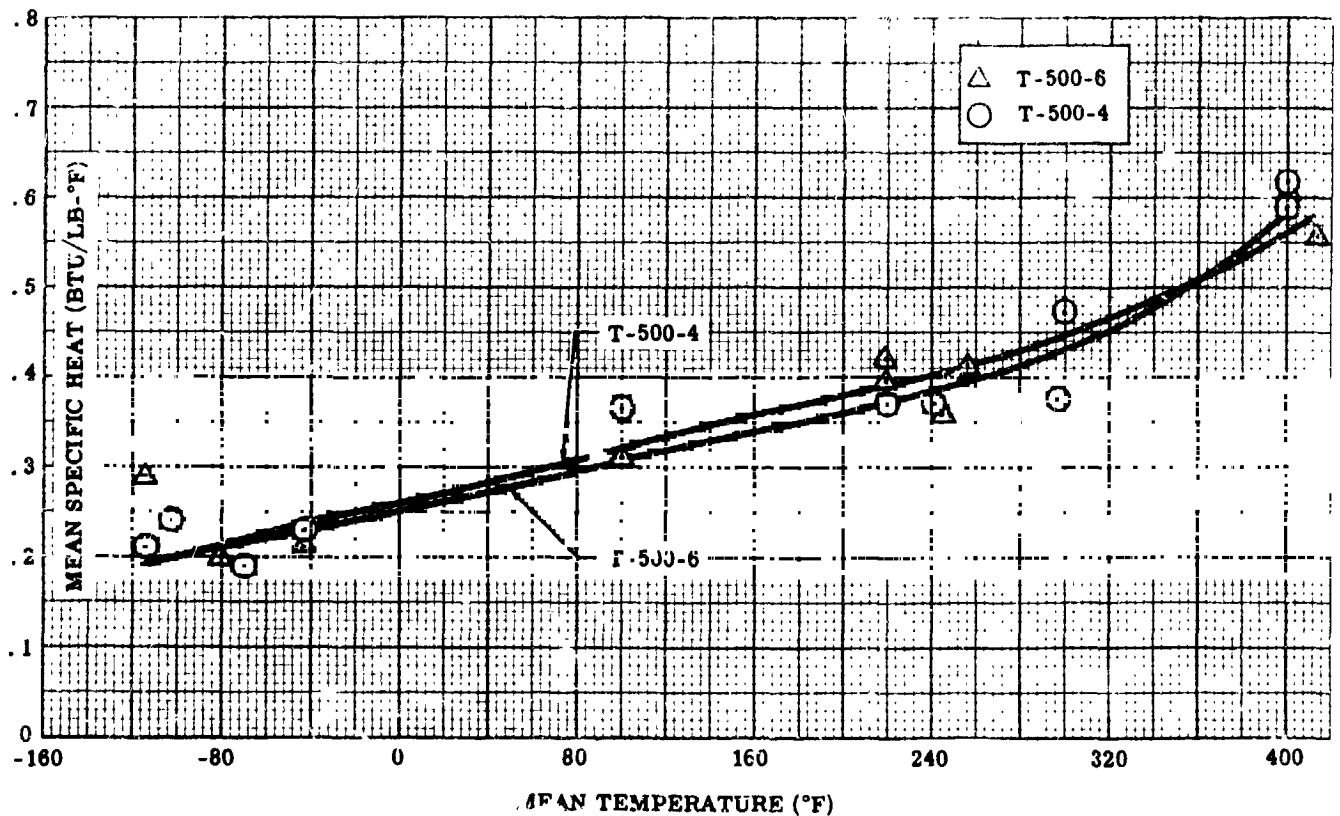


Figure 2-3. Mean Specific Heat of Virgin "THERMO-LAG" T-500-4 and T-500-6

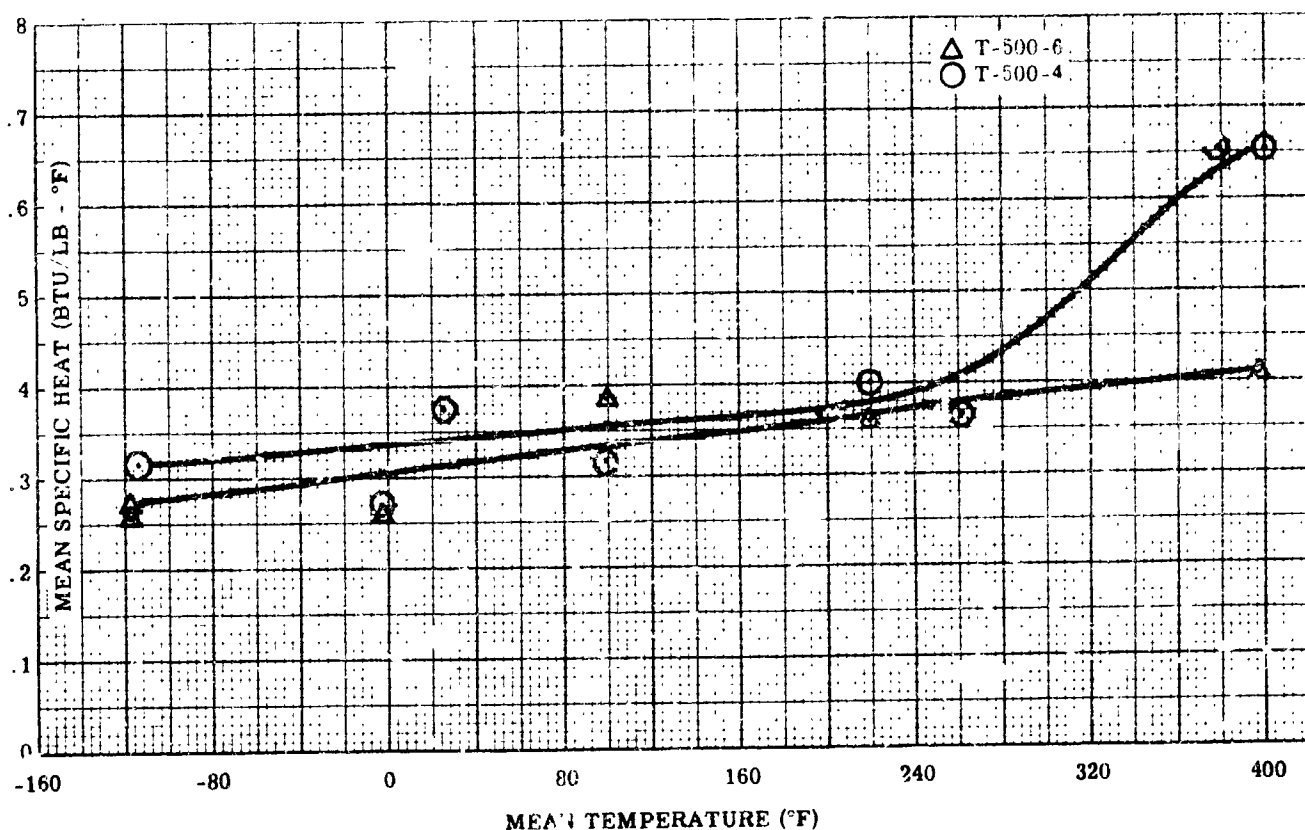


Figure 2-4. Mean Specific Heat of Charred "THERMO-LAG" T-500-4 and T-500-6

Material	Specimen Weight GMS	Initial Specimen Temperature °F	Initial Toluene Temperature °F	Equilibration Temperature °F	T_t °F	T_s °F	Toluene Weight GMS	C_{pt} BTU/LB-°F	Specific Heat of Specimen BTU/LB-°F
T-500-6 Charred	98.24	130	72.55	75.10	2.55	54.90	2091	.395	0.389 at 102
T-500-6 Charred	98.77	350	81.03	91.68	10.65	258.32	2122	.403	0.358 at 221
T-500-6 Charred	98.89	-295	81.72	70.01	11.71	365.01	2056	.403	0.271 at -113
T-500-6 Charred	98.60	-295	82.40	71.29	11.11	366.29	2099	.404	0.261 at -112
T-500-6 Charred	99.04	-89.5	91.10	86.64	4.46	176.14	2002	.403	0.208 at -2

Table 2-2. Test Results - Specific Heat (Sheet 1 of 3)

Material	Specimen Weight GMS	Initial Specimen Temperature °F	Initial Toluene Temperature °F	Equilibration Temperature °F	T_t °F	T_s °F	Toluene Weight GMS	C_p BTU/LB-°F	Specific Heat of Specimen BTU/LB-°F
T-500-4 Charred	99.74	350	78.57	90.75	12.18	259.25	2116	.402	0.400 at 200
T-500-4 Charred	98.20	429	76.36	90.88	14.52	338.12	2150	.401	0.377 at 260
T-500 Charred	99.91	130	76.03	76.03	2.00	51.97	2110	.401	0.326 at 104
T-500-4 Charred	99.52	130	75.35	77.24	1.89	52.76	2113	.401	0.305 at 103
T-500-4 Charred	98.69	-300	80.75	66.65	14.1	366.55	2045	.403	0.312 at -117
T-500-4 Charred	98.68	-295	81.51	67.70	13.81	362.70	2009	.403	0.312 at -116
T-500-4 Charred	98.16	-70	81.74	78.21	3.53	148.21	2085	.403	0.204 at 4
T-500-6 Virgin	94.70	500	70.28	85.36	15.08	414.64	1990	.402	0.308 at 292
T-500-6 Virgin	100.71	500	69.90	84.48	14.58	415.52	1960	.402	0.274 at 292
T-500-4 Virgin	99.40	496	70.05	93.46	17.41	402.54	2159	.403	0.378 at 294
T-500-4 Virgin	93.83	502	78.96	93.87	14.91	408.13	2045	.404	0.322 at 298
T-500-4 Virgin	104.04	-275	78.22	66.10	10.12	343.10	1983	.400	0.234 at -103
T-500-4 Virgin	98.32	-81	78.30	77.05	4.25	158.05	2060	.403	0.226 at -2
T-500-4 Virgin	97.46	-81	76.55	72.73	3.82	153.73	2043	.401	0.210 at -4
T-500-4 Virgin	95.60	-298	79.06	70.30	8.76	368.30	2081	.401	0.208 at -114
T-500-4 Virgin	94.03	-210	75.43	69.41	6.02	279.41	2070	.400	0.190 at -70

Table 2-2. Test Results - Specific Heat (Sheet 2 of 3)



Material	Specimen Weight GMS	Initial Specimen Temperature °F	Initial Toluene Temperature °F	Equilibration Temperature °F	T_t °F	T_s °F	Toluene Weight GMS	C_{pt} BTU/LB-°F	Specific Heat of Specimen BTU/LB-°F
T-500-6 Virgin	95.44	-221	73.80	67.00	6.80	288.00	2035	0.399	0.201 at -77
T-500-6 Virgin	95.70	-302	63.20	71.15	12.05	373.15	2104	0.402	0.286 at -116
T-500-6 Virgin	95.28	293	78.53	66.35	12.18	359.53	2080	0.400	0.296 at -114
T-500-6 Virgin	93.46	417	Toluene 81.80	63.20	15.60	319.80	Toluene 1917	Toluene .407	.406 at 257
T-500-6 Virgin	96.21	400	Toluene 79.58	66.31	13.27	307.15	Toluene 1910	Toluene .405	.353 at 245
T-500-6 Virgin	83.84	493	TCP 296	286	8	189	TCP 2430	TCP .490	.606 at 399
T-500-6 Virgin	83.85	516	TCP 308	300	8	202	TCP 2387	TCP .490	.553 at 415
T-500-4 Charred	98.2	429	Toluene 76.36	61.84	14.52	338.12	Toluene 2150	Toluene .401	.377 at 26
T-500-4 Charred	97.88	432	Toluene 77.65	63.52	14.13	340.22	Toluene 2152	Toluene .402	.367 at 262
T-500-4 Charred	195	431	TCP 313	301	12	106	TCP* 2295	TCP .490	.652 at 378
T-500-6 Charred	198.88	461	TCP 320	310	10	130	TCP 2150	TCP .490	.407 at 396
T-500-4 Virgin	85.5	400	Toluene 69.11	56.92	12.19	318.7	Toluene 2070	Toluene .401	.371 at 241
T-500-4 Virgin	90.46	495	Toluene 77.90	55.30	22.60	394.5	Toluene 1832	Toluene .406	.473 at 298
T-500-4 Virgin	99.4	496	Toluene 76.05	58.64	17.41	402.54	Toluene 2159	Toluene .403	.378 at 294
T-500-4 Virgin	129.69	496	TCP 302	290	12	182	TCP 2344	TCP .490	.583 at 405
T-500-4 Virgin	185.59	460	TCP 318	304	14	138	TCP 2297	TCP .490	.615 at 401
*Tritoluyyl Phosphate									

Table 2-2. Test Results - Specific Heat (Sheet 3 of 3)

DENSITY. Tests were performed to measure the density of virgin "THERMO-LAG" materials T-500-4 and T-500-6 in accordance with Method A of ASTM D792-50, using a laboratory gravitometer. A description of the material test specimen is illustrated in Figure A-3, Appendix A.

Twelve samples each of "THERMO-LAG" materials, T-500-4 and T-500-6 were subjected to density testing at temperatures of -150°F to 400°F . The results of these tests are presented in Figure 2-5. Within the range of experimental accuracy, the densities of the two compounds correspond and vary in almost a straight line relationship from about 68 LB/FT³ at -150°F to about 59 LB/FT³ at 400°F .

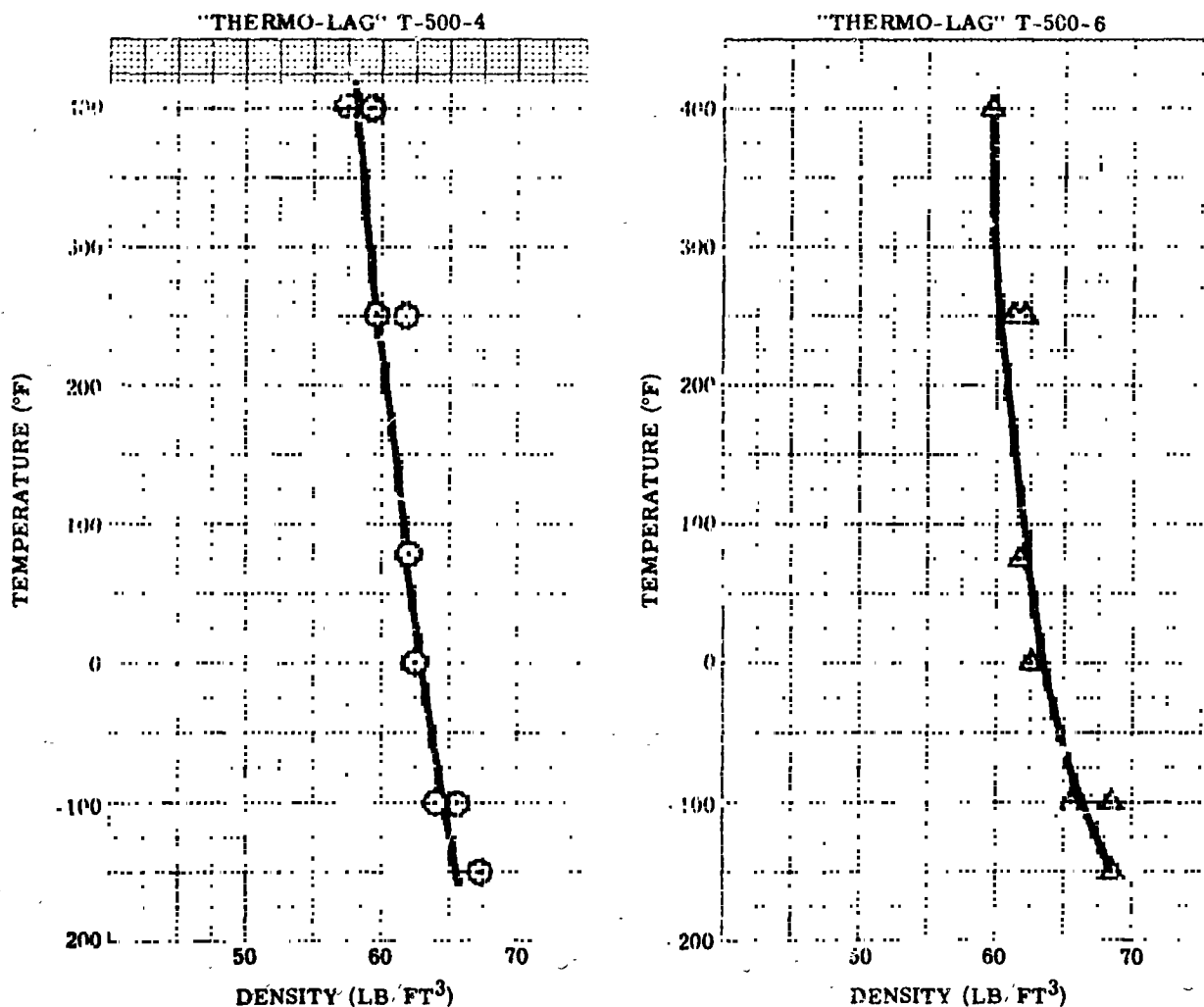


Figure 2-5. Density Versus Temperature for "THERMO-LAG" T-500-4 and T-500-6



VACUUM EXPOSURE. The effect of very low pressure (5×10^{-5} MM Hg), equivalent to that realized at an extreme altitude, on "THERMO-LAG" materials T-500-4 and T-500-6 was investigated. The material test specimens one inch square and 3/8-inch thick were contained in flasks which were connected to the high vacuum system and evacuated to the desired vacuum. For the high temperature tests, the flasks containing the specimens were surrounded by a constant temperature bath. A description of the material test specimen is illustrated in Figure A-4, Appendix A.

The weight loss per unit area of the "THERMO-LAG" materials for given periods of exposure, a given pressure, and for a range of temperatures was determined. The results of one through seven and 48-hour periods of exposure are recorded in Tables 2-3 and 2-4, respectively. The recorded data for the one to seven hourly tests are average values for three tests of very similar initial weights. The average value of two tests are recorded for the 48-hour periods of exposure. Test results for "THERMO-LAG" T-500-4 for 80°F and 250°F are illustrated in Figure 2-6. A study of the recorded data indicates a substantial portion of the total weight loss occurred within the first seven hours of exposure. A portion of this weight loss is attributed to loss of physically entrapped liquid volatiles.

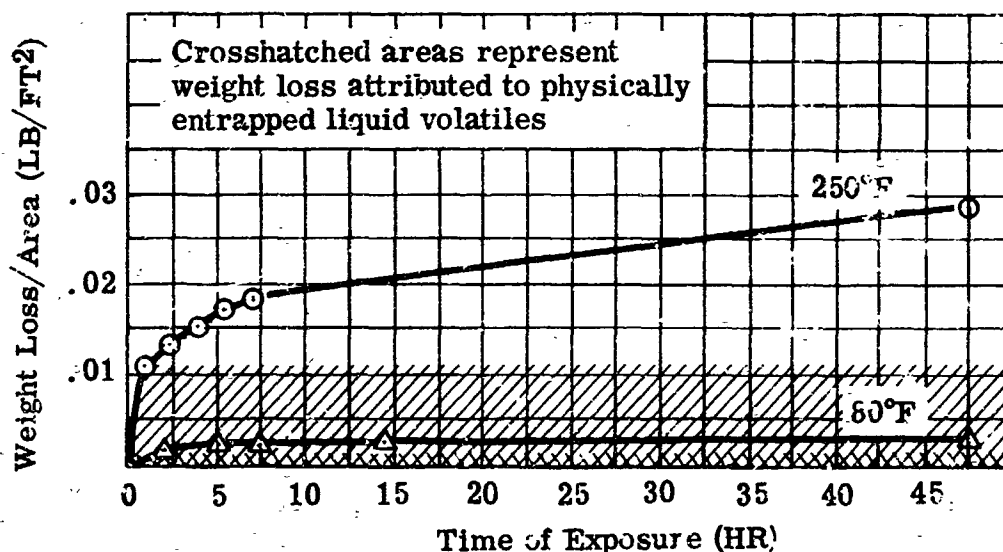


Figure 2-6. Effect of Temperature at Low Pressure (10^{-5} MM HG) on Weight Loss of "THERMO-LAG" T-500-4

Material	Test Duration Hours	Temperature °F	Sample Weight GMS		Weight Loss GM	Weight Loss GM/HR-FT ²
			Initial	Final		
T-500-4	1	80	5.931	5.936	.01059	.4273
	2	80	5.936	5.932	.00390	.0800
	3	80	5.932	5.930	.00280	.0383
	4	80	5.930	5.927	.00200	.0205
	5	80	5.927	5.925	.00177	.0145
	6	80	5.925	5.924	.00113	.0077
	7	80	5.924	5.923	.00097	.0057
T-500-4	1	250	5.8625	5.8279	.13460	5.5267
	2	250	5.8279	5.8306	.01760	.49733
	3	250	5.8306	5.7843	.01530	.20933
	4	250	5.7843	5.7434	.01153	.11833
	5	250	5.7434	5.7677	.00910	.07467
	6	250	5.7677	5.7603	.00733	.05010
	7	250	5.7603	5.7597	.00667	.03823

Table 2-3. Weight Loss of "THERMO-LAG" T-500-4
at Low Pressure for Exposed Time Periods



Material	Specimen Number	Test Duration Hours	Temp-erature °F	Sample Weight		Surface Area FT ²	Weight Loss LB/FT ²
				Initial Weight LBS	Final Weight LBS		
T-500-4	10-G-1-4a	48	30	.01533	.01526	.026	.0026
T-500-4	10-G-1-4b	48	80	.01471	.01464	.026	.0025
T-500-4	10-G-1-4c	48	150	.01593	.01574	.026	.0067
T-500-4	10-G-1-4d	48	200	.01556	.01530	.026	.0093
T-500-6	10-H-2-6a	48	80	.017493	.01736	.026	.0027
T-500-6	10-H-2-6b	48	80	.01819	.01812	.026	.0028
T-500-6	10-H-2-6c	48	150	.01726	.01697	.026	.0112
T-500-6	10-H-2-6d	48	150	.01835	.01806	.026	.0114
T-500-6	10-H-2-6e	48	200	.01708	.01670	.026	.0183
T-500-4	10-G-1-4e	48	250	.01565	.01494	.026	.028
T-500-6	10-H-2-6g	48	250	.01705	.0162	.026	.034

Table 2-4. Effect of Low Pressure and Temperature for 48 Hours on "THERMO-LAG" T-500

THERMAL-CHEMICAL CHARACTERISTICS.

PHASE CHANGE OF VIRGIN MATERIAL. A qualitative investigation using differential thermal analysis was performed to determine whether phase changes occur prior to sublimation, to determine if the phase changes are endothermic or exothermic, and to determine the approximate temperature of sublimation. The apparatus used in the determination was a Deltatherm Differential Thermal Analyzer and an external furnace. Both "THERMO-LAG" T-500-5 and T-500-4 were analyzed, using approximately one gram of pulverized material for each test.

The results of the analysis for "THERMO-LAG" T-500-4 and T-500-6, where heated rates of 35.6, 41, and 50°F/minute were imposed are illustrated in Figures 2-7 and 2-18. Both materials were analyzed in an inert atmosphere. A study of recorded data indicates that two distinct phase changes occurred for each test. The first endotherm indicates a phase change in the crystal structure of the subliming material. The second endotherm indicates the temperature of sublimation of the subliming material taken as the point at which the slope change is first observed. The sublimation temperature, as indicated in the illustrations, is on the average about 560°F for T-500-4, and 545°F for T-500-6.

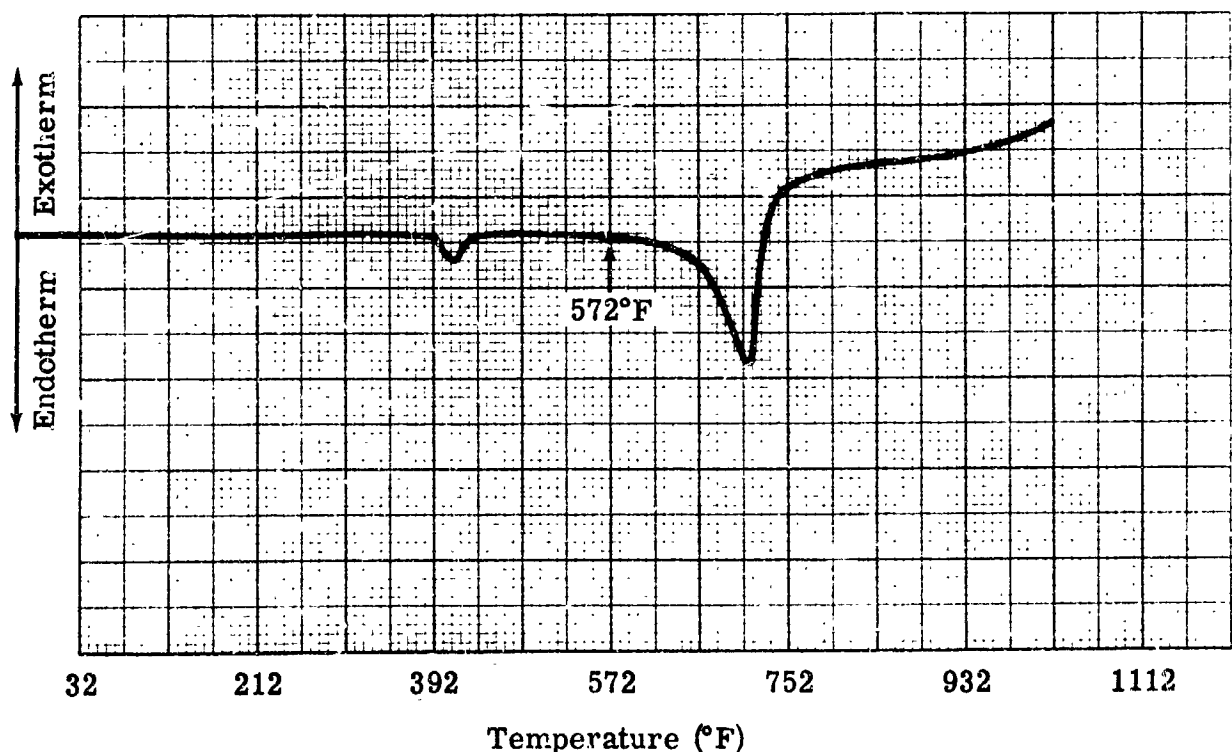


Figure 2-7. Differential Thermal Analysis of "THERMO-LAG" T-500-4 for a Heated Rate of 35.6°F per Minute

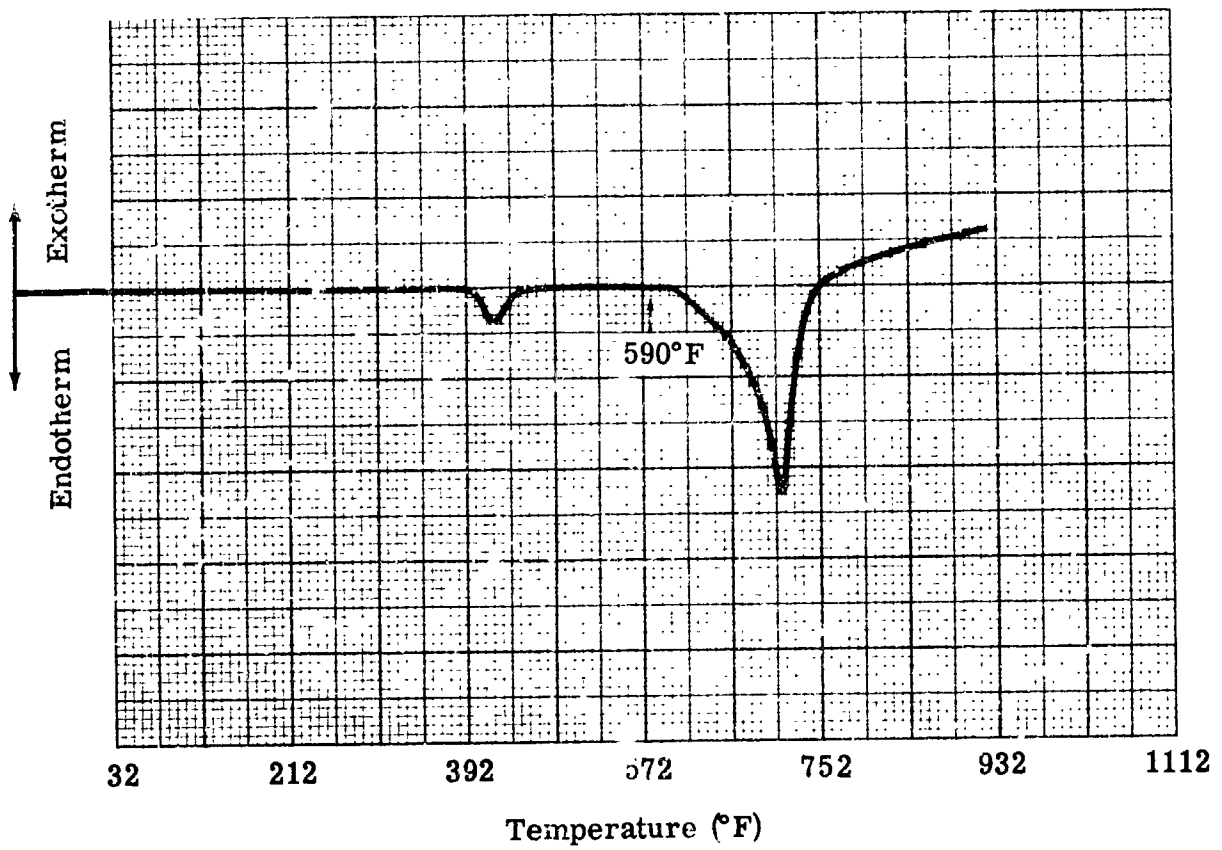


Figure 2-8. Differential Thermal Analysis of "THERMO-LAG" T-500-4 for a Heated Rate of 35.6°F per Minute

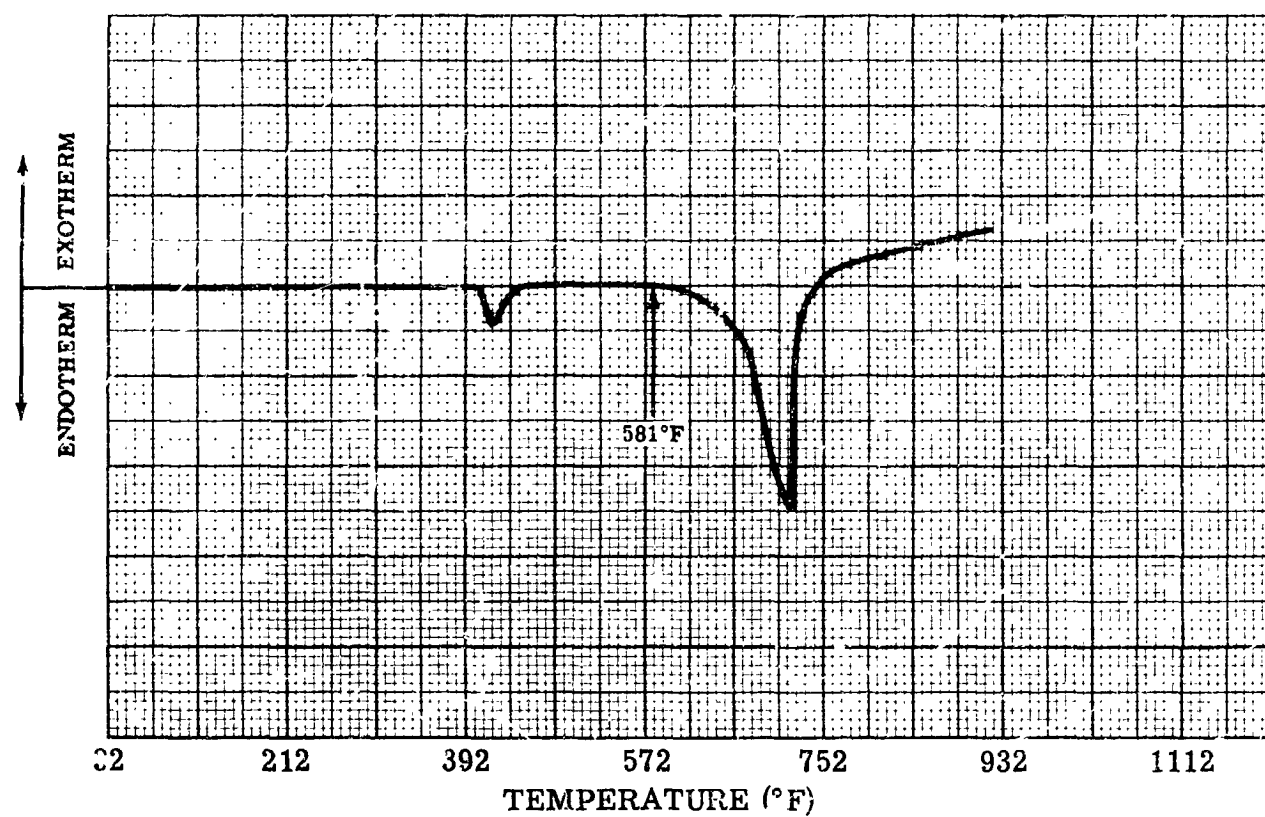


Figure 2-9. Differential Thermal Analysis of "THERMO-LAG" T-500-4 for a Heated Rate of 35.6°F per Minute

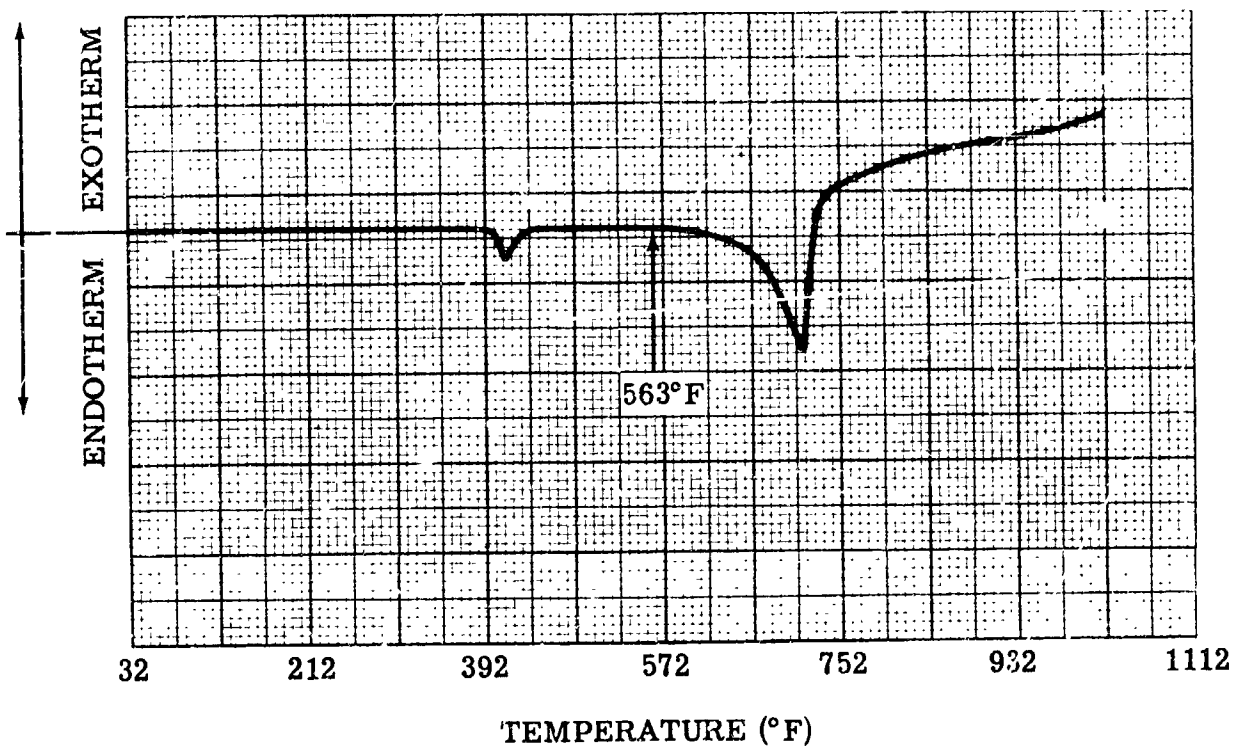


Figure 2-10. Differential Thermal Analysis of "THERMO-LAG" T-500-4 for a Heated Rate of 35.6°F per Minute

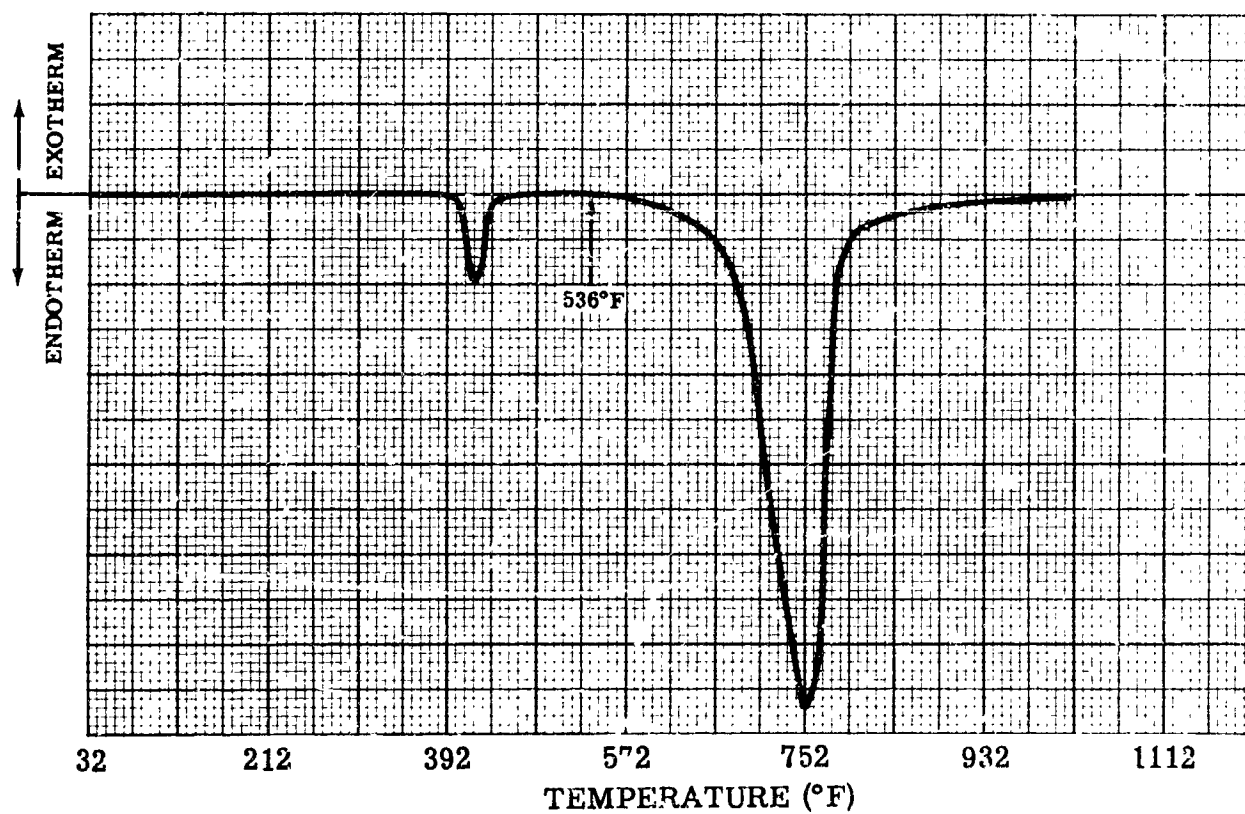


Figure 2-11. Differential Thermal Analysis of "THERMO-LAG" T-500-4 for a Heated Rate of 41°F per Minute

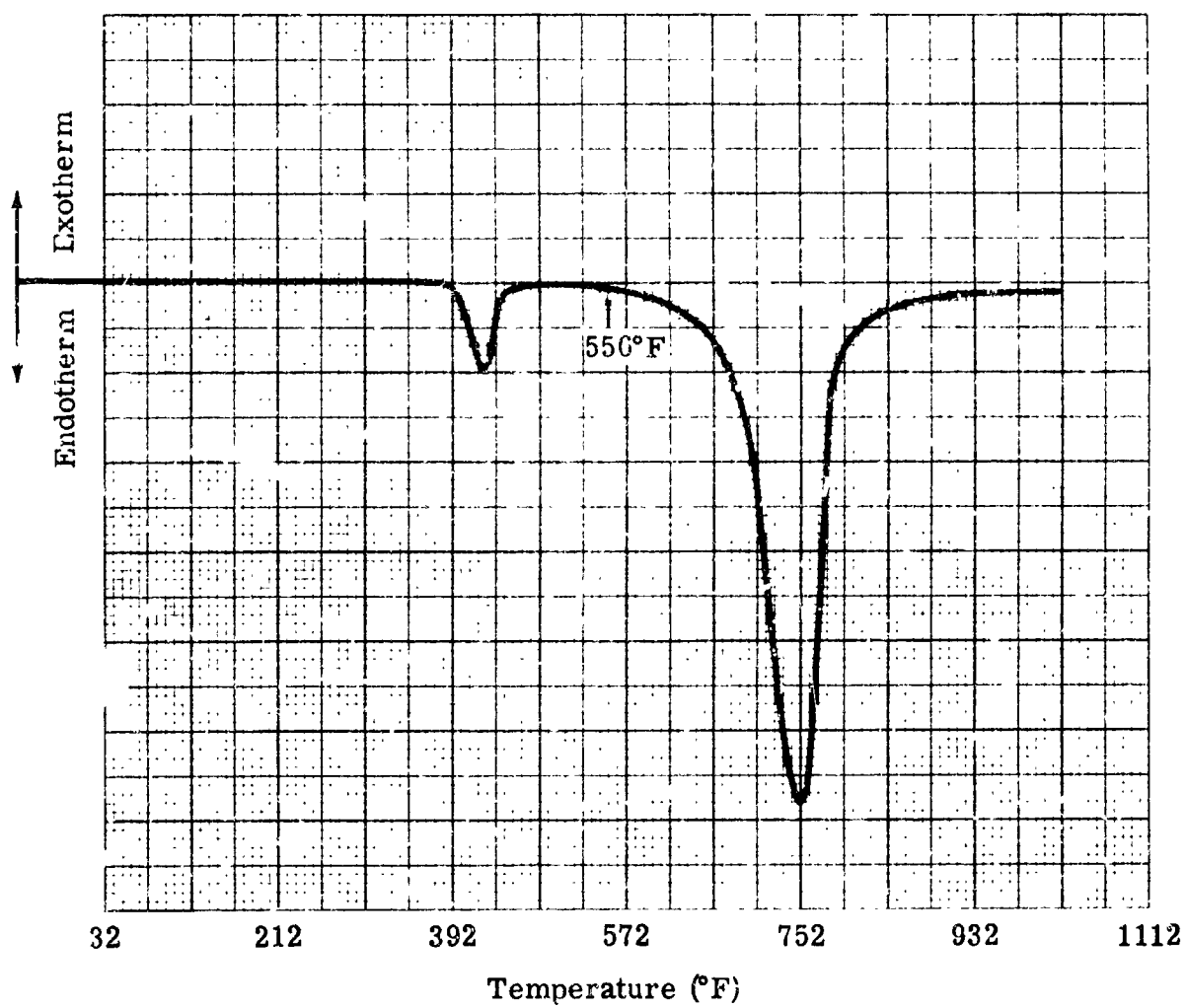


Figure 2-12. Differential Thermal Analysis of "THERMO-LAG" T-500-4 for a Heated Rate of 41°F per Minute

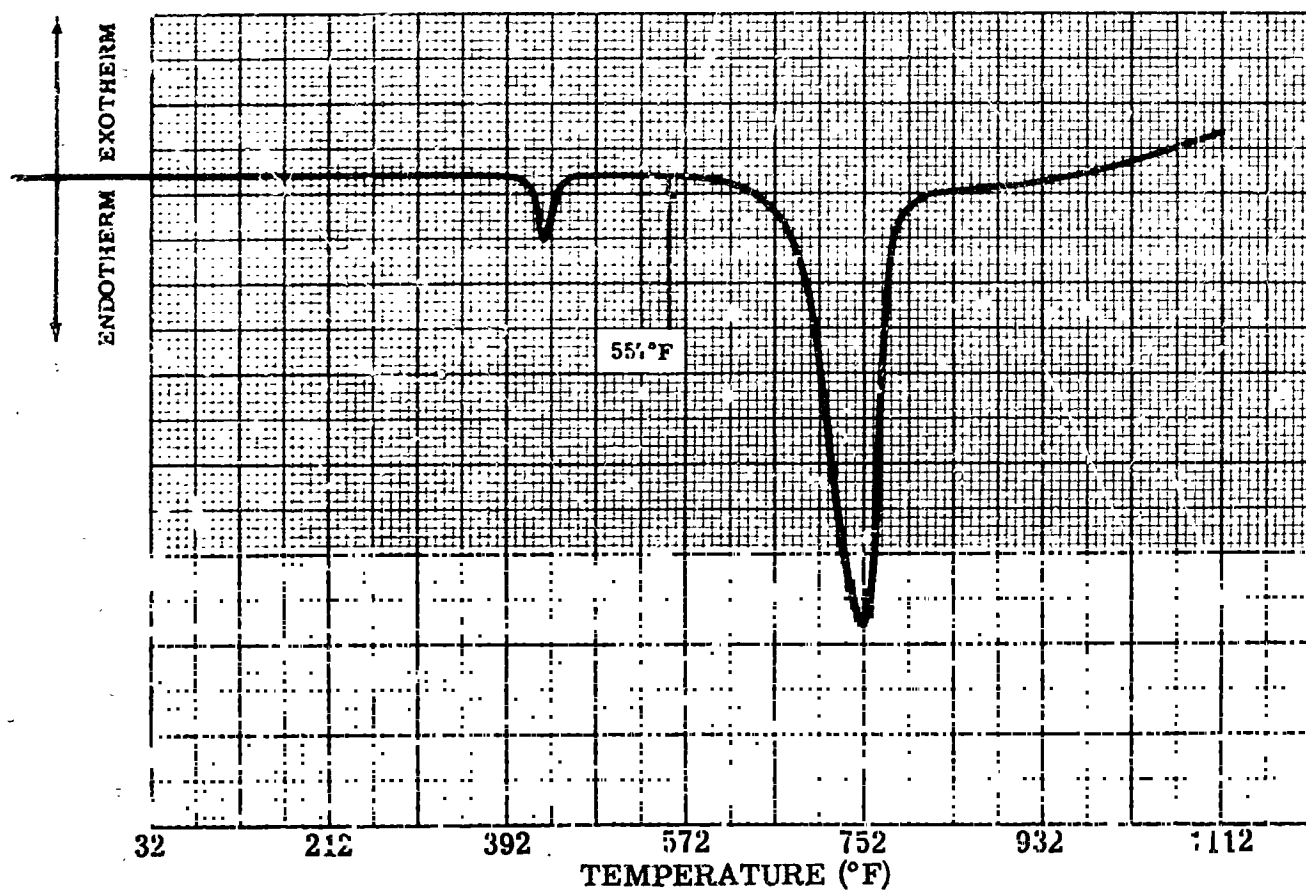


Figure 2-13. Differential Thermal Analysis of "THERMO-LAG" T-500-4 for a Heated Rate of 41°F per Minute

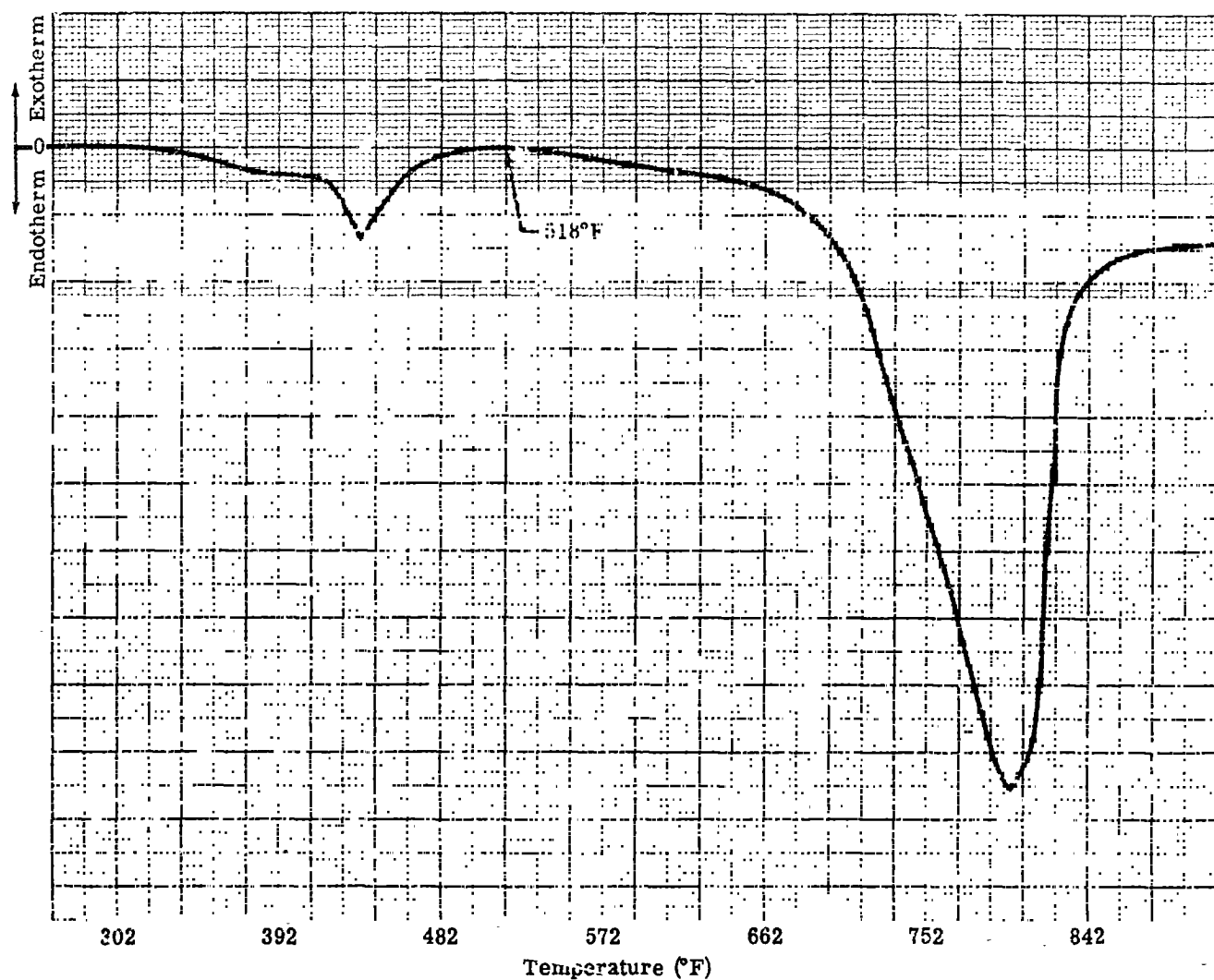


Figure 2-14. Differential Thermal Analysis of "THERMO-LAG" T-500-4 for a Heated Rate of 50°F per Minute

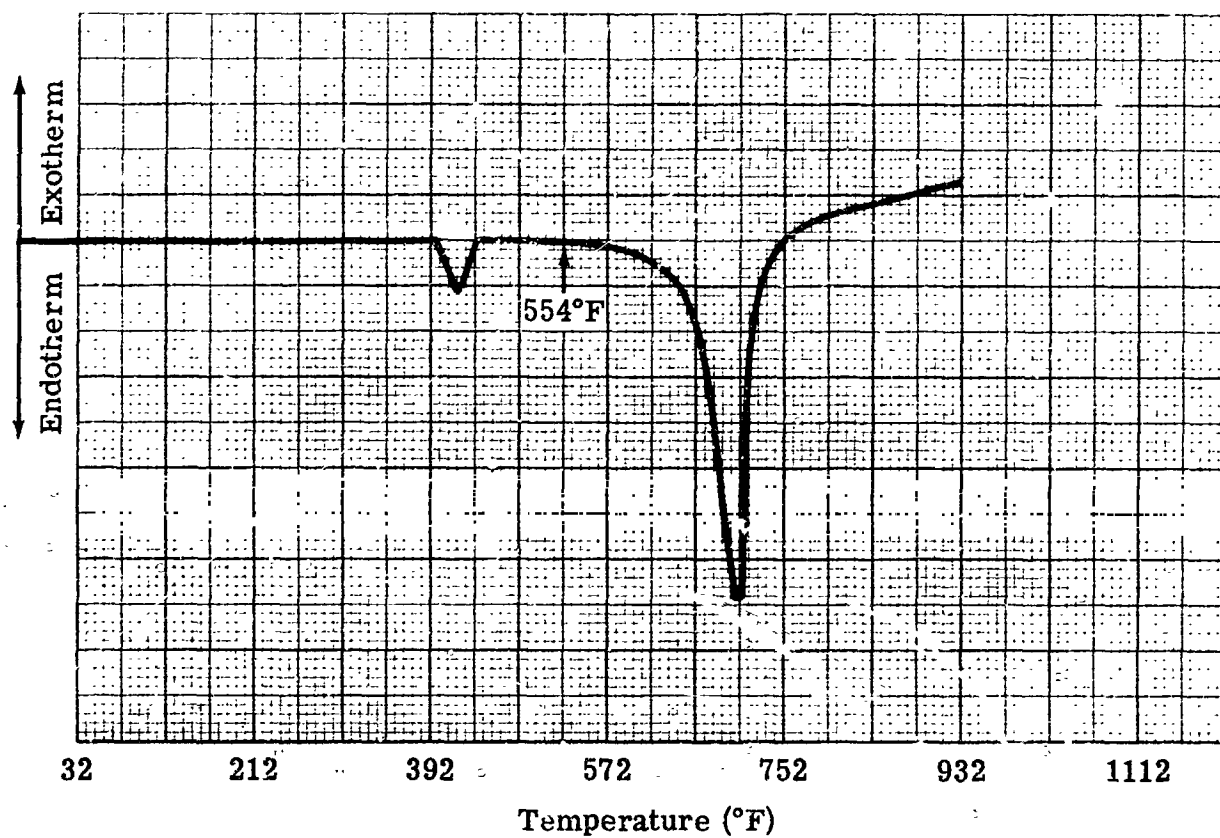


Figure 2-15. Differential Thermal Analysis of "THERMO-LAG" T-500-6 for a Heated Rate of 35.6°F per Minute

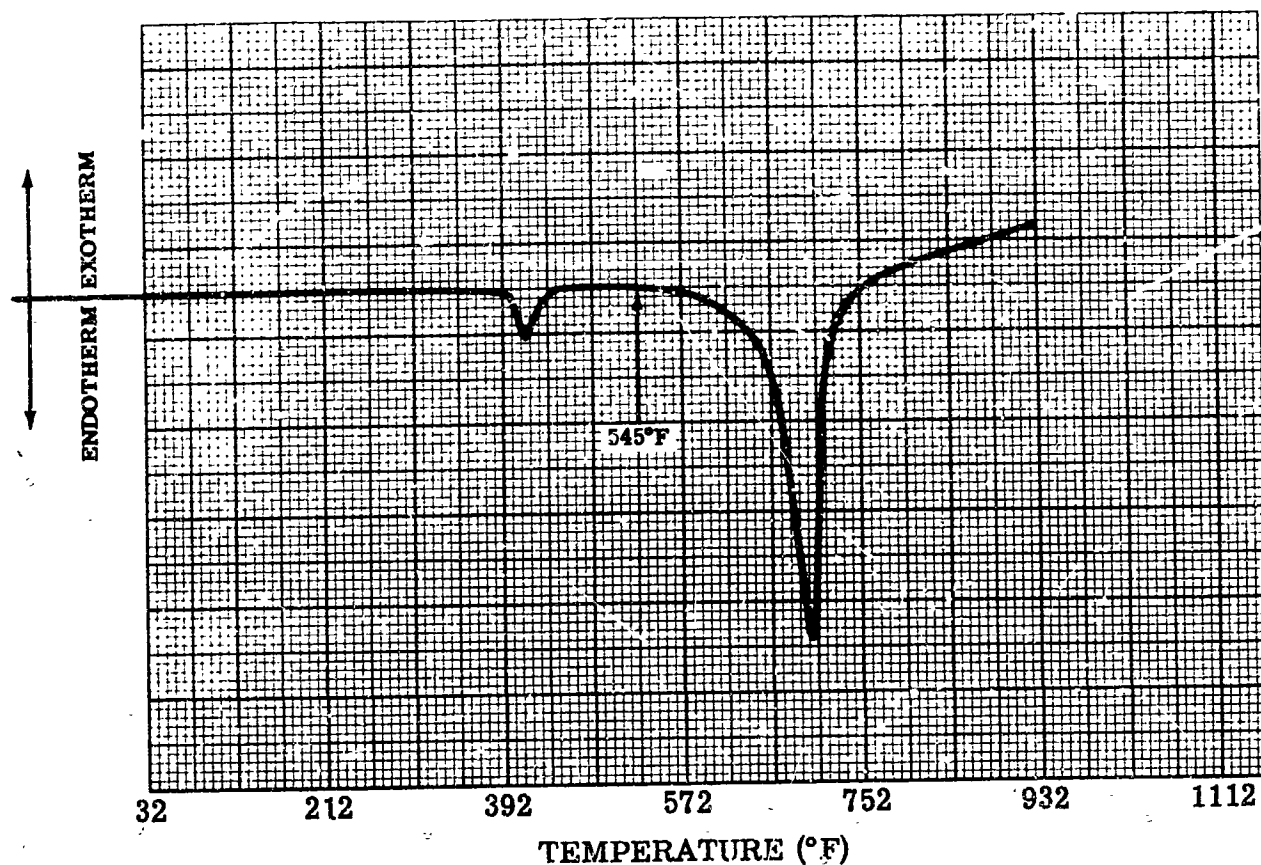


Figure 2-16. Differential Thermal Analysis of "THERMO-LAG" T-500-6 for a Heated Rate of 35.6°F per Minute

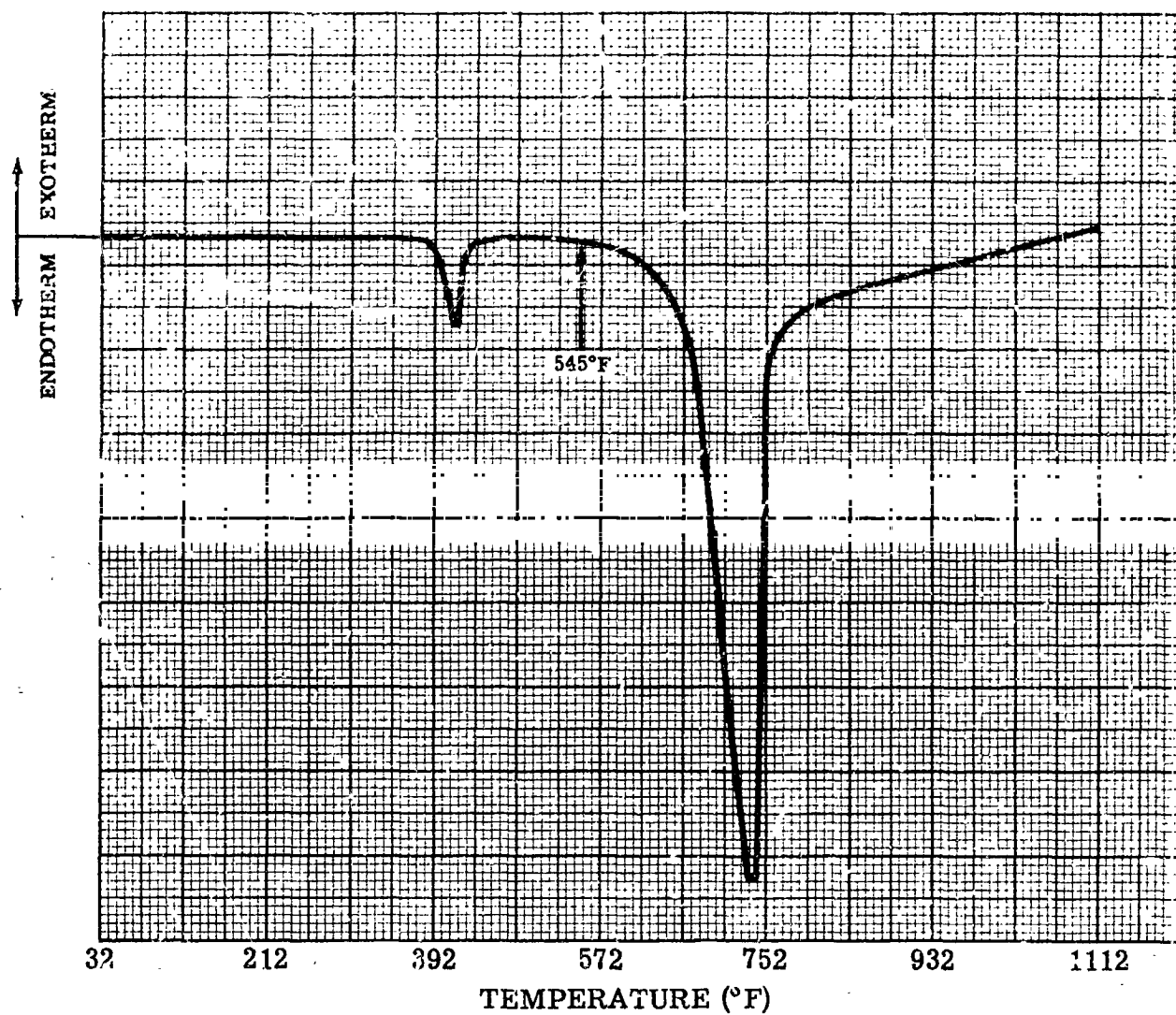


Figure 2-17. Differential Thermal Analysis of "THERMO-LAG" T-500-6 for a Heated Rate of 41°F per Minute

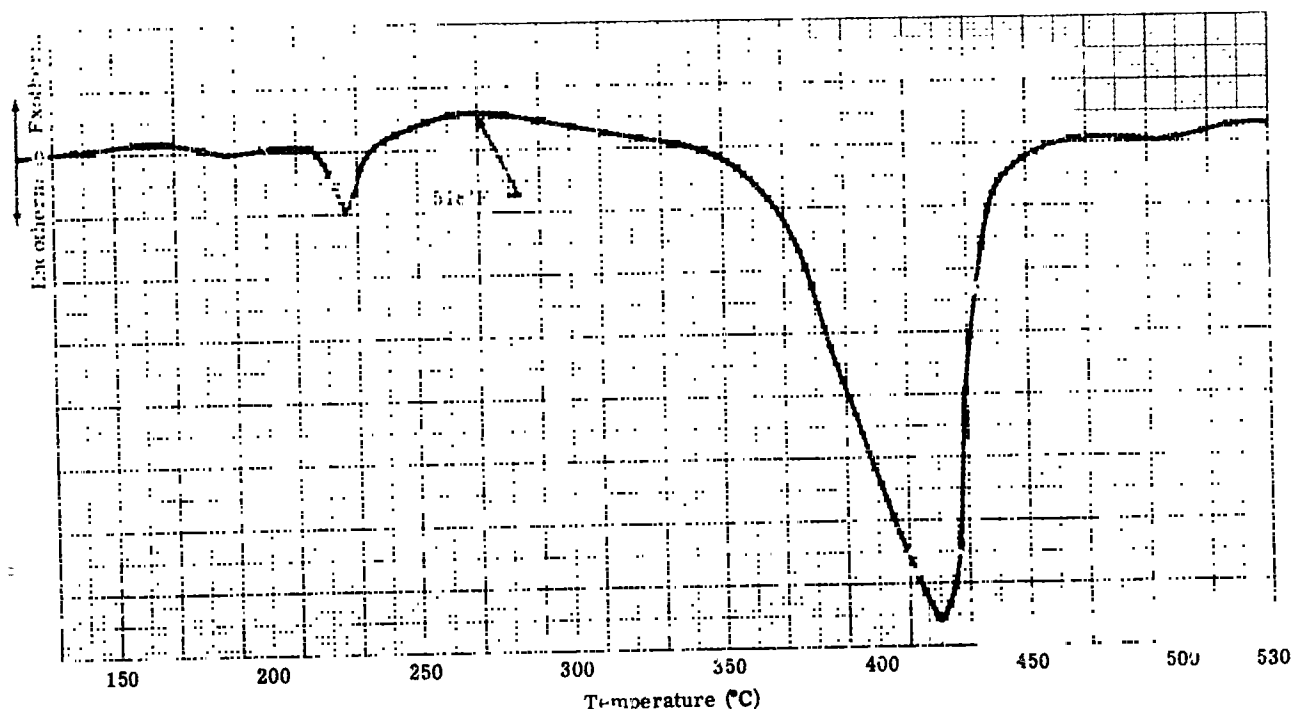


Figure 2-18. Differential Thermal Analysis of "THERMO-LAG" T-500-6 for a Heated Rate of 50°C per Minute

THERMOGRAVIMETRIC ANALYSIS. A qualitative investigation using thermogravimetric analysis was performed to determine the rate that "THERMO-LAG" T-500 material loses weight during the sublimation process at a constant temperature rise, and to determine the temperature range at which the maximum weight loss of "THERMO-LAG" T-500 occurs. The apparatus used in the determination was a Deltatherm Thermogravimetric Analyzer, which incorporates an electronic balance and an external furnace. Both "THERMO-LAG" materials, T-500-4 and T-500-6, were analyzed using approximately three to four milligrams of pulverized material for each test.

The test results for "THERMO-LAG" T-500-4, where two or more heated rates of 35.6, 41, and 50°F/minute were imposed are illustrated in Figures 2-19 through 2-25. Figures 2-26 through 2-31 illustrate the test results for "THERMO-LAG" T-500-6 for the same imposed heated rates. Figure 2-32 illustrates the fractional weight loss for T-500-4 powdered material. Due to the powdered state of the material, a larger weight loss occurred for the imposed heated rate than would have occurred for a molded state of material.

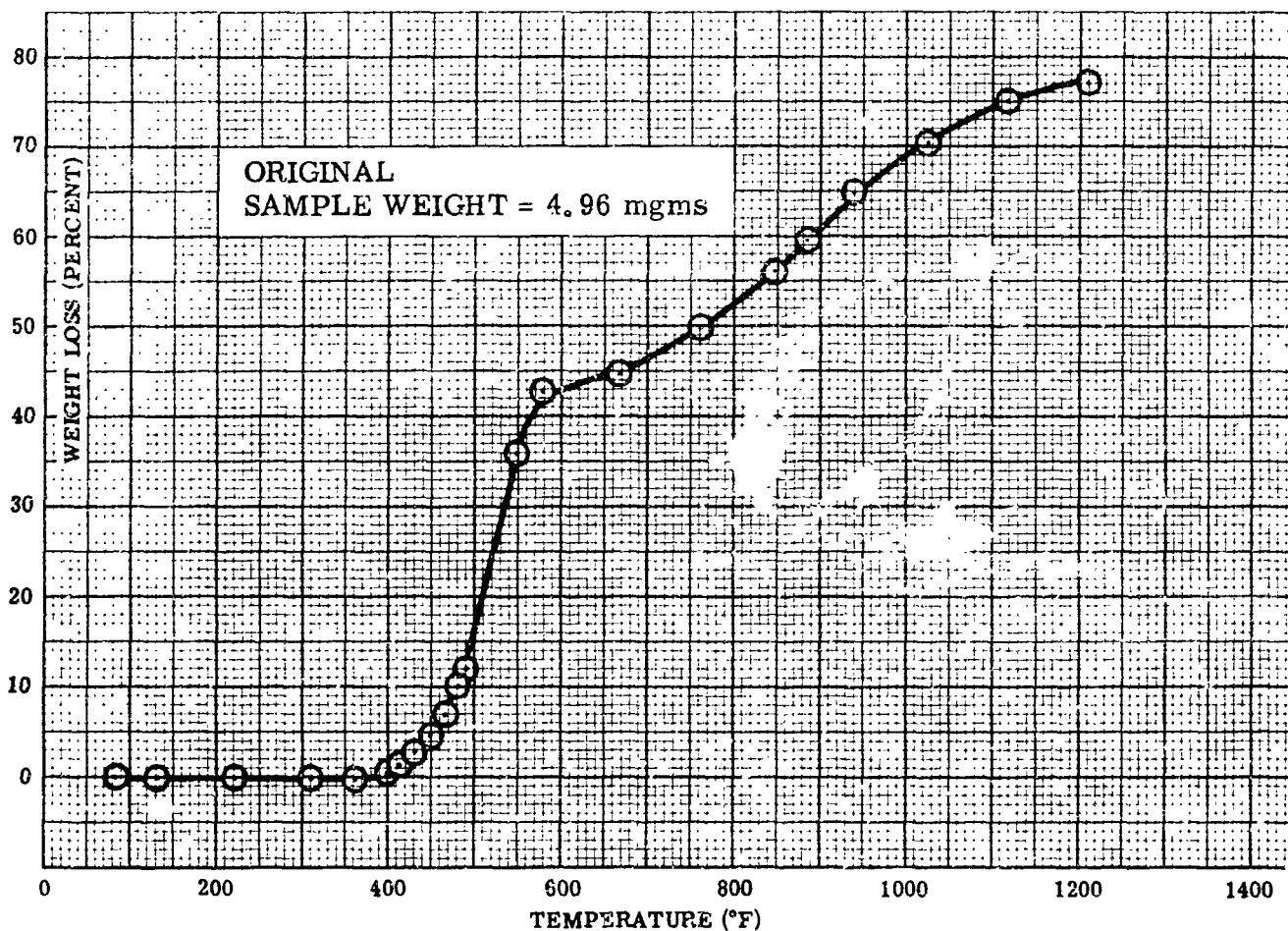


Figure 2-19. Thermogravimetric Analysis of "THERMO-LAC" T-500-4 for a Heated Rate of 35.6°F per Minute

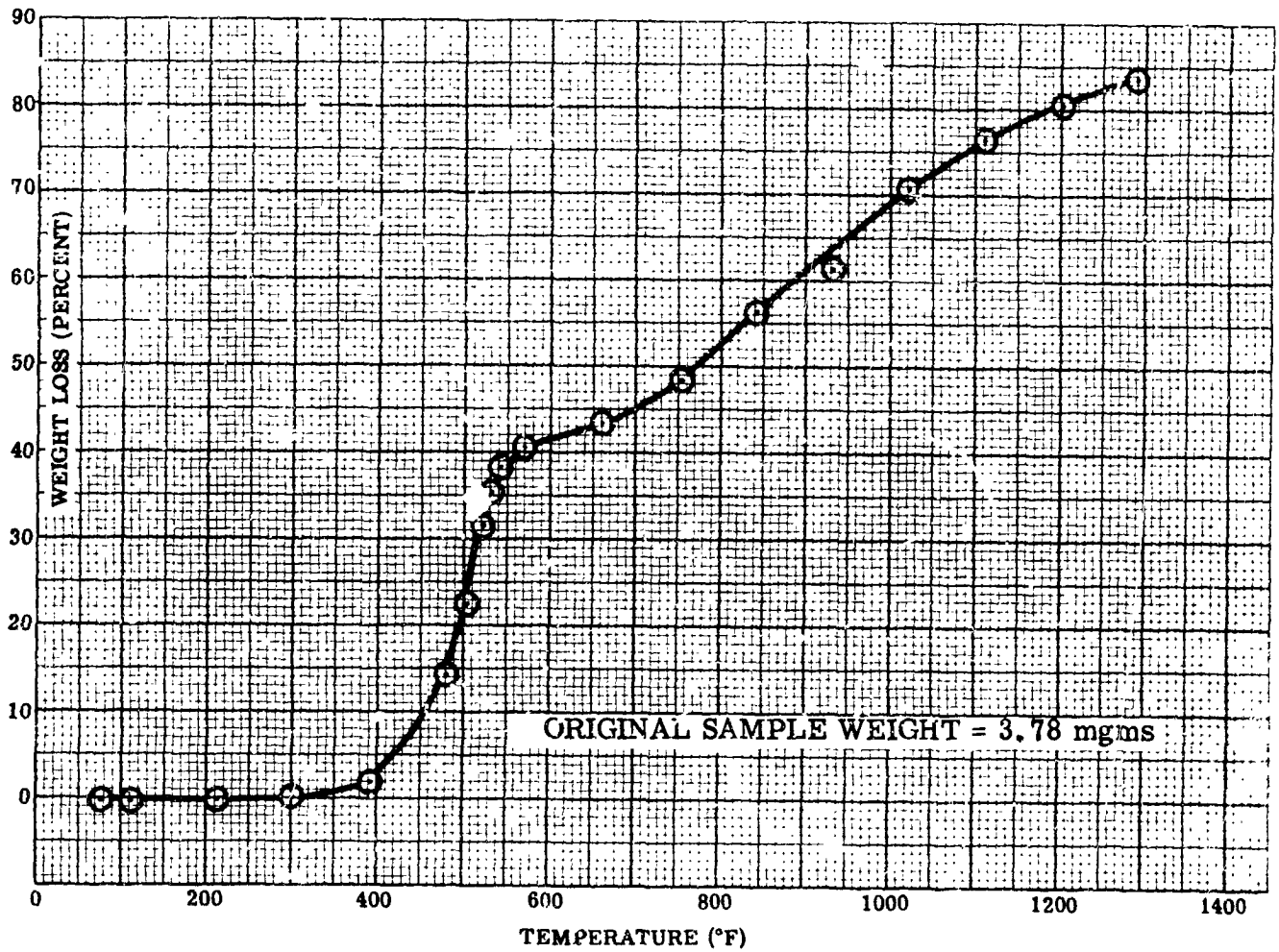


Figure 2-20. Thermogravimetric Analysis of "THERMO-LAG" T-500-4 for a Heated Rate of 35.6°F per Minute

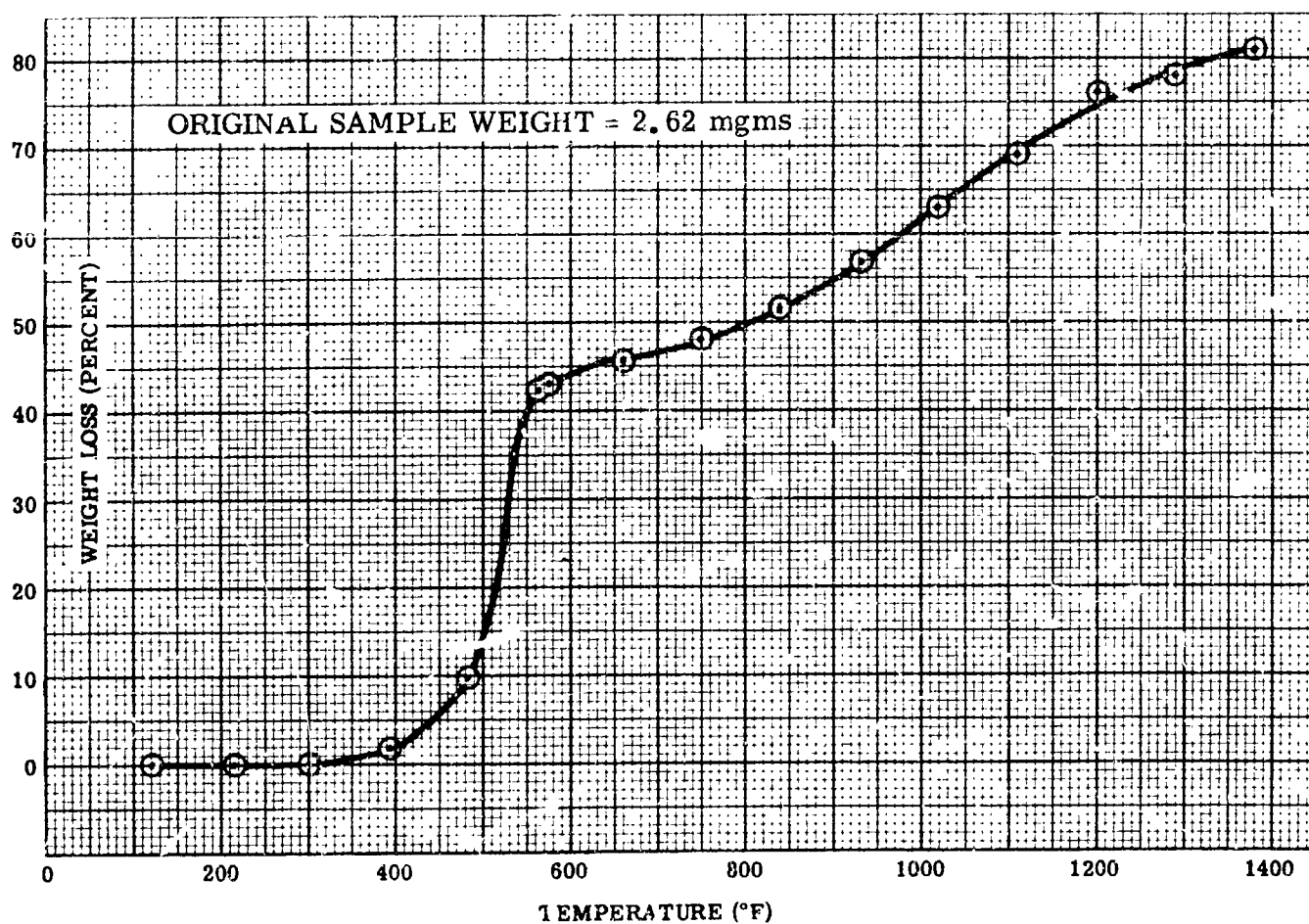


Figure 2-21. Thermogravimetric Analysis of "THERMO-LAG" T-500-4 for a Heated Rate of 41°F per Minute

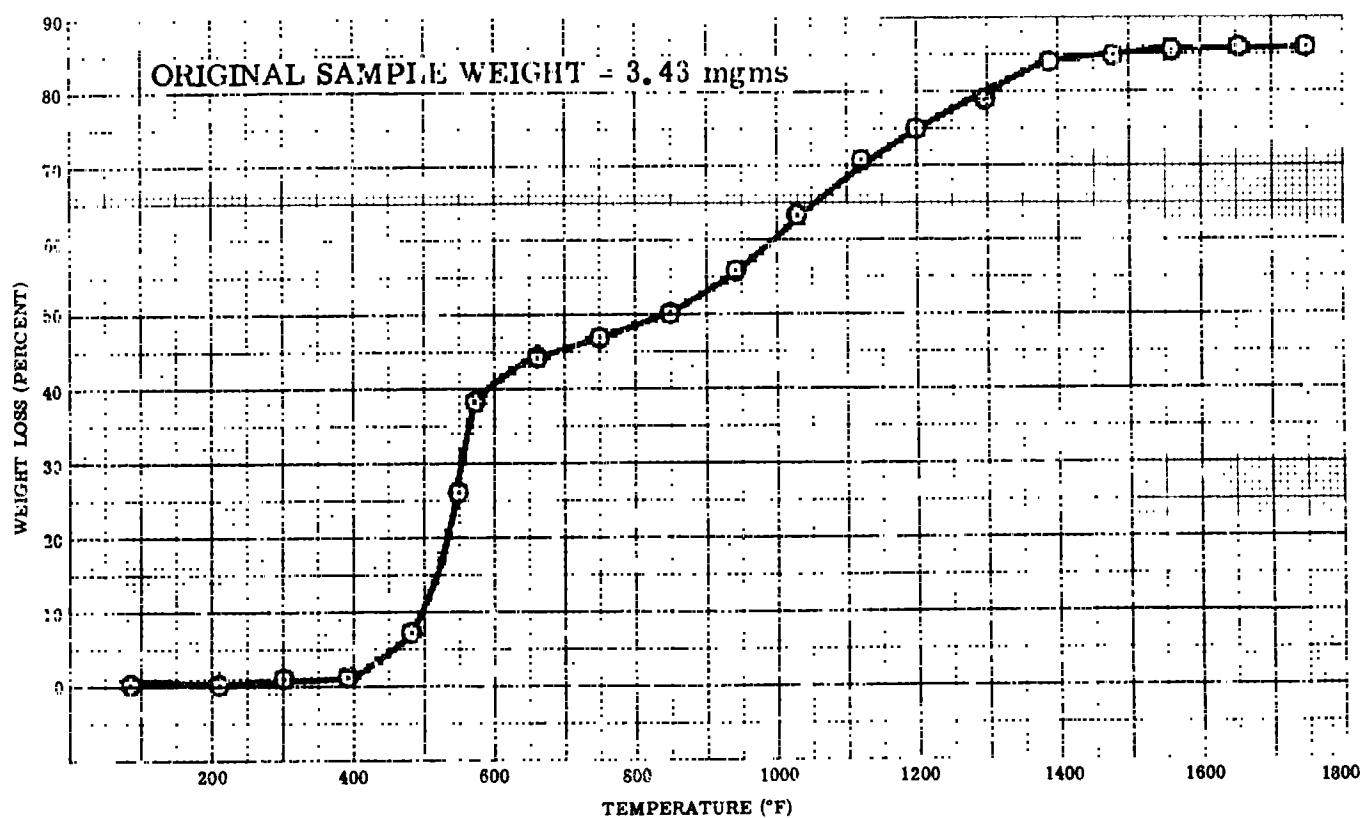


Figure 2-22. Thermogravimetric Analysis of "THERMO-LAG" T-500-4 for a Heated Rate of 41°F per Minute

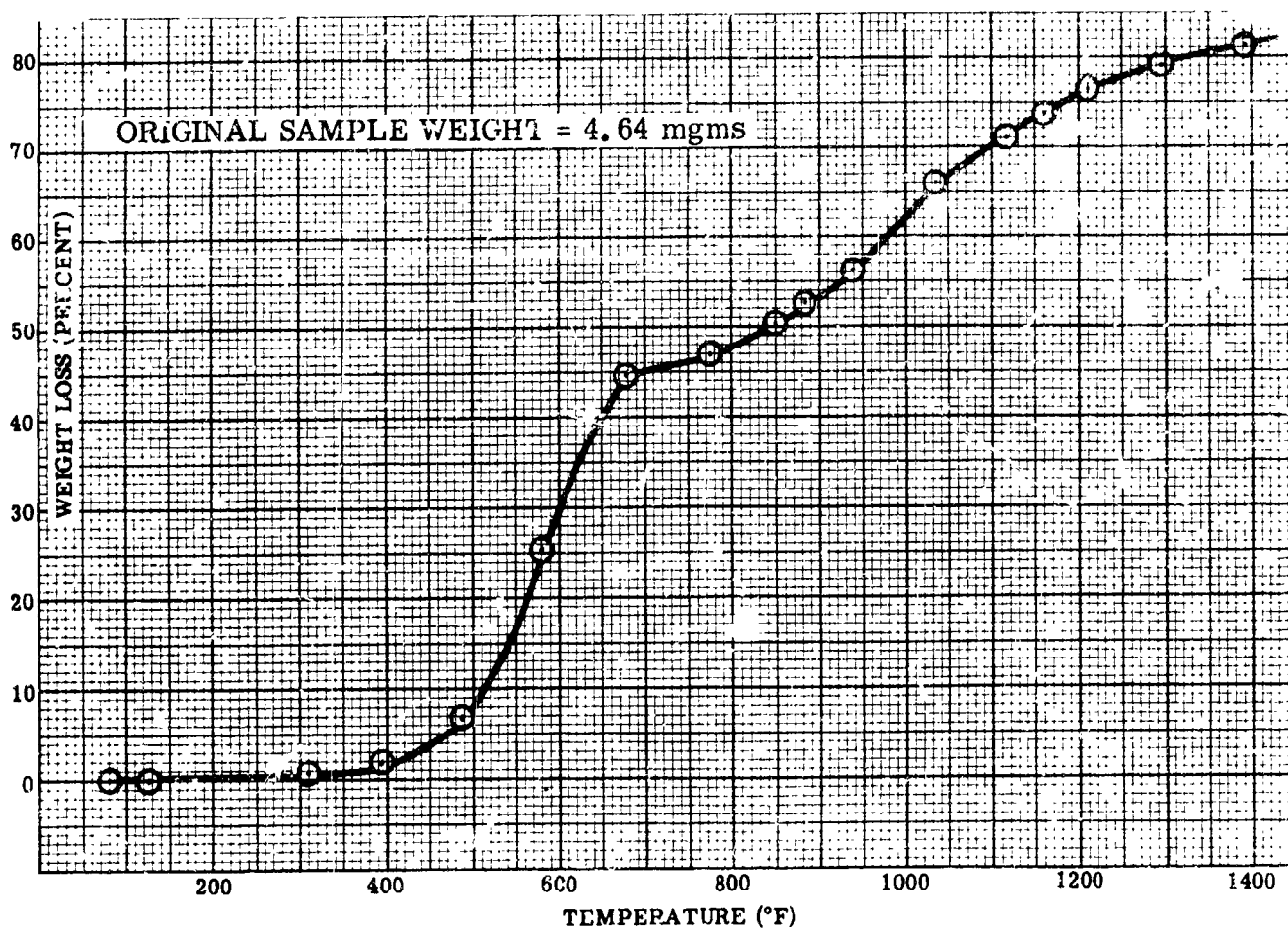


Figure 2-23. Thermogravimetric Analysis of "THERMO-LAG" T-500-4 for a Heated Rate of 41°F per Minute

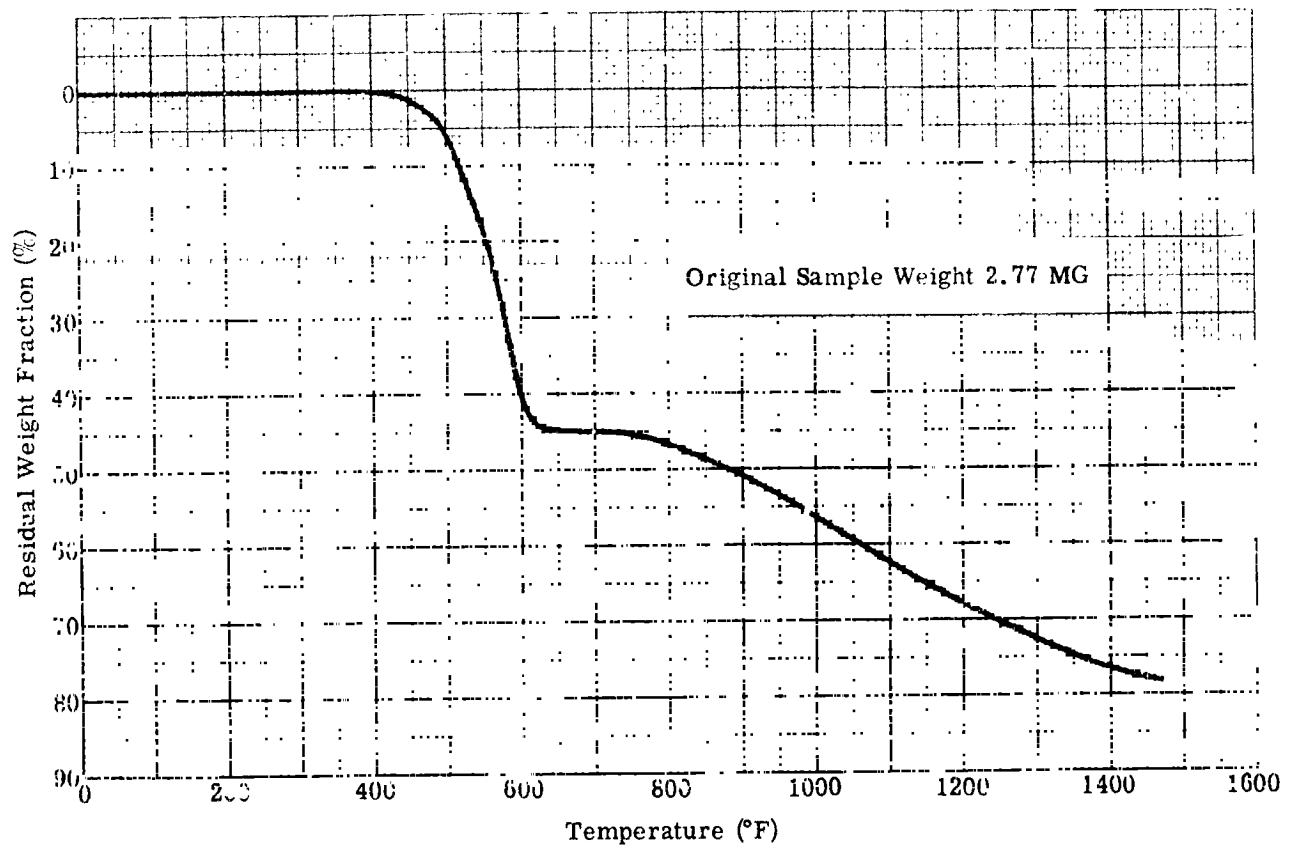


Figure 2-24. Thermogravimetric Analysis of "THERMO-LAG" T-500-4 for a Heated Rate of 50°F per Minute

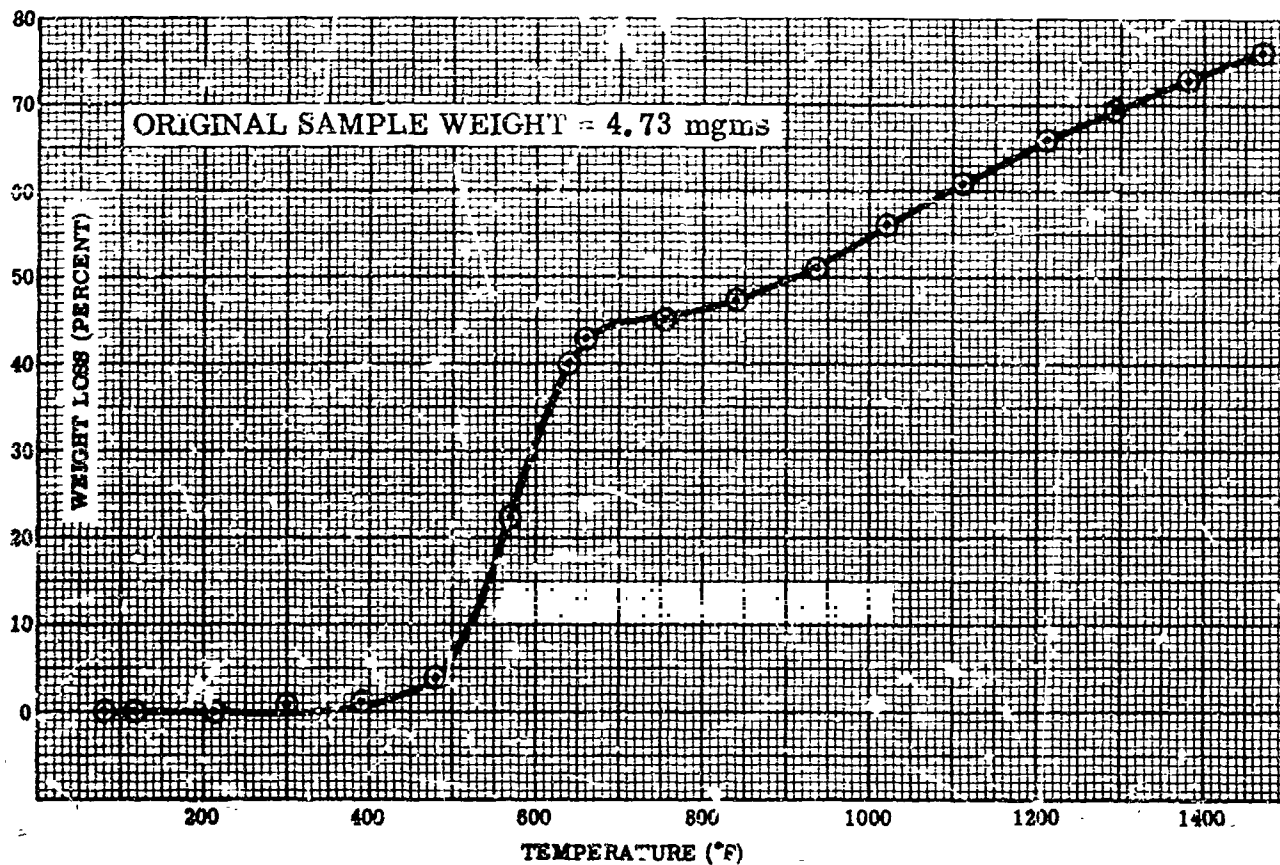


Figure 2-25. Thermogravimetric Analysis of "THERMO-LAG" T-500-4 for a Heated Rate of 50°F per Minute

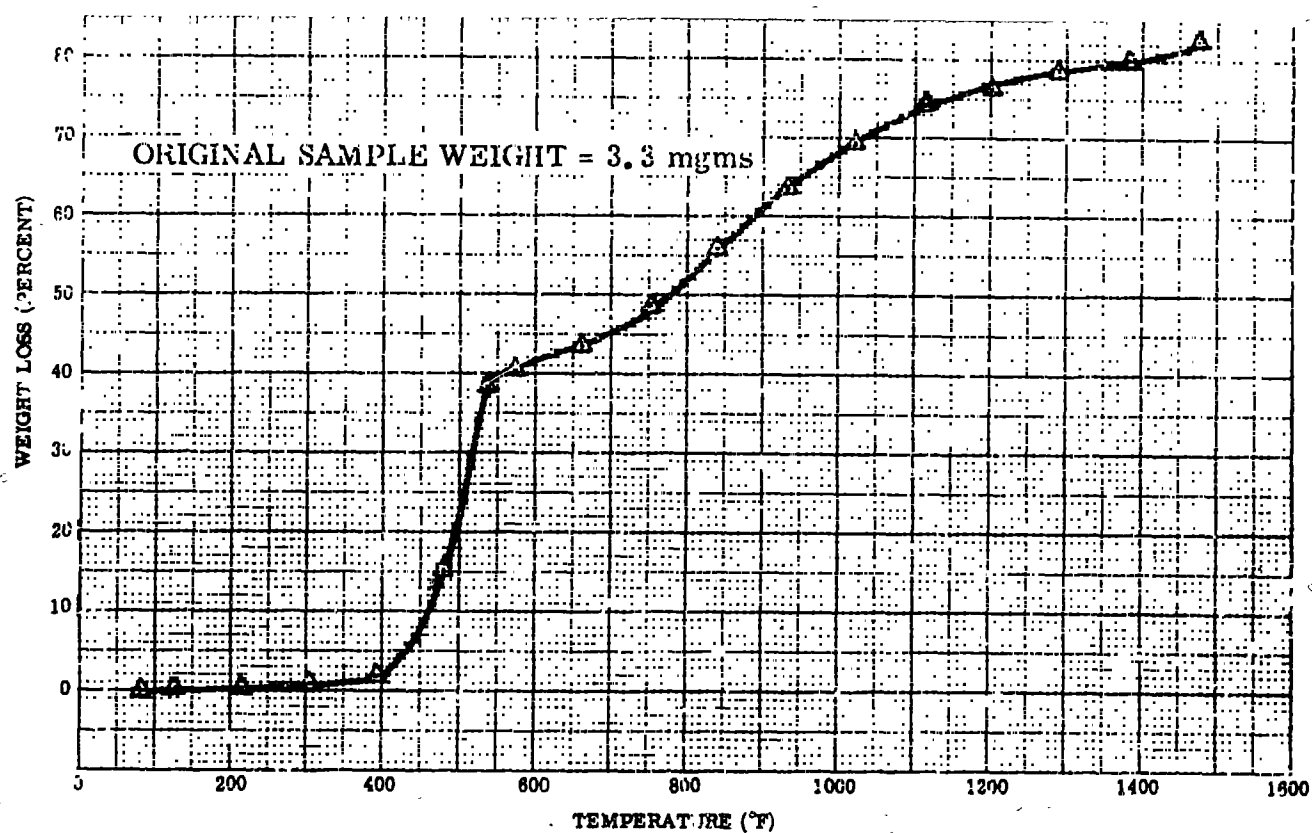


Figure 2-20. Thermogravimetric Analysis of "THERMO-LAG" T-500-6 for a Heated Rate of 35.6°F per Minute

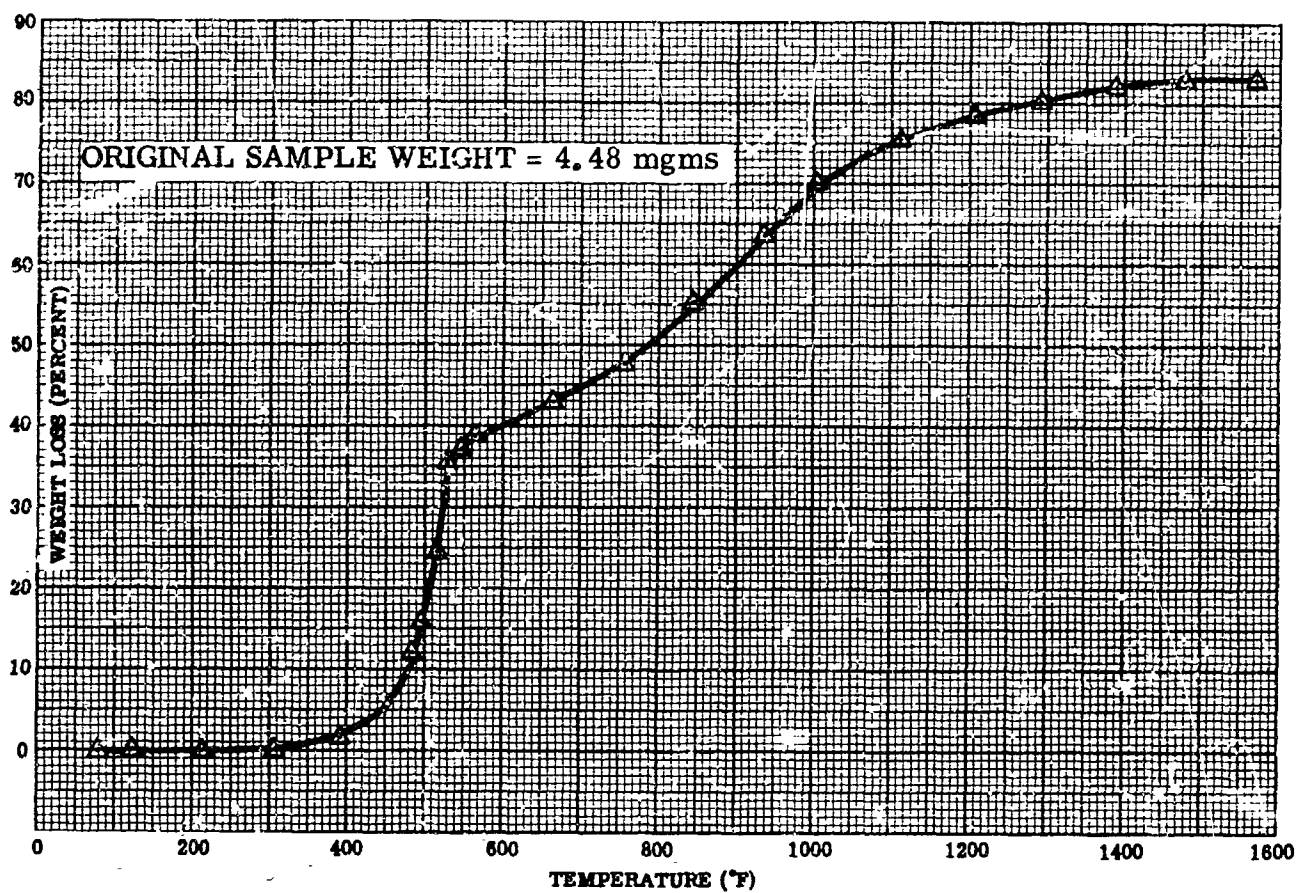


Figure 2-27. Thermogravimetric Analysis of "THERMO-LAG" T-500-6 for a Heated Rate of 35.6°F per Minute

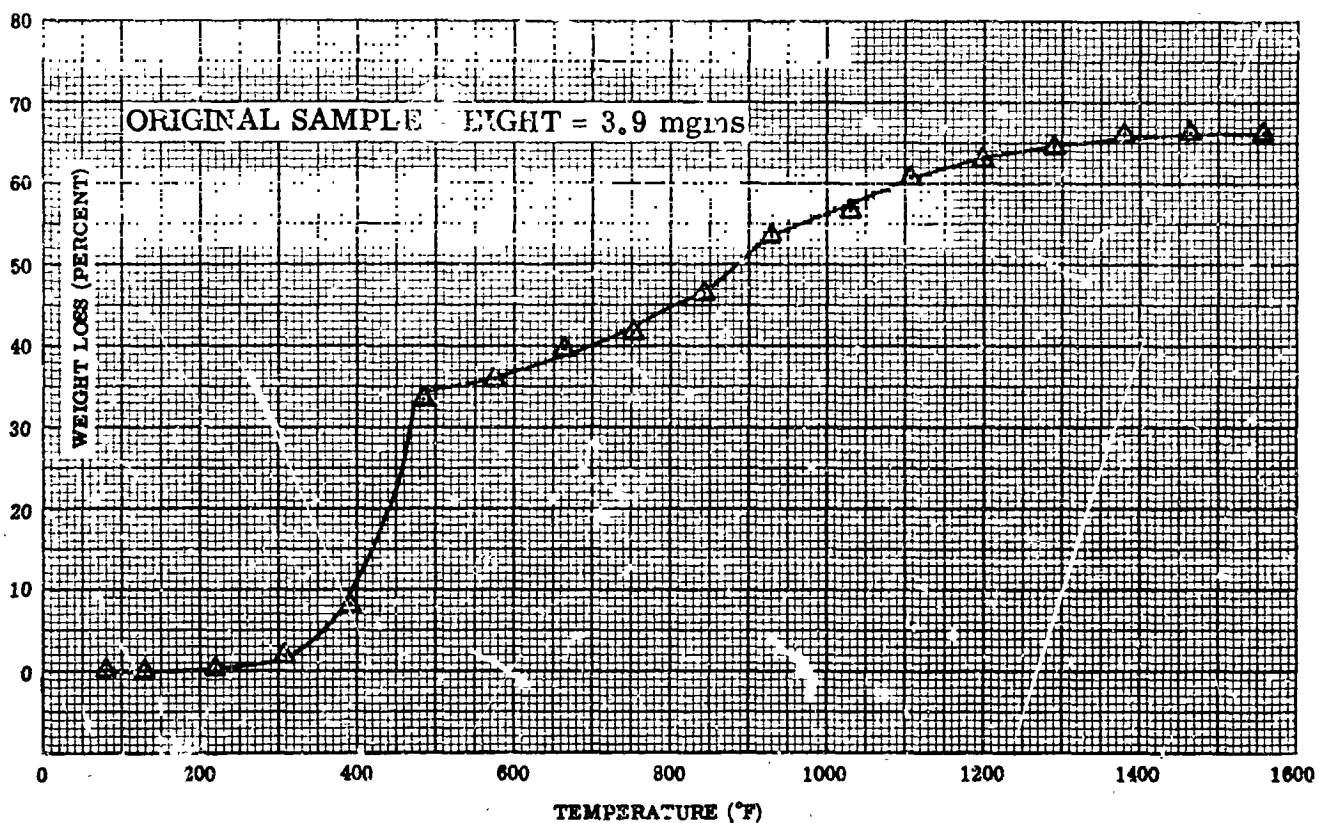


Figure 2-28. Thermogravimetric Analysis of "THERMO-LAG" T-500-6 for a Heated Rate of 41°F per Minute

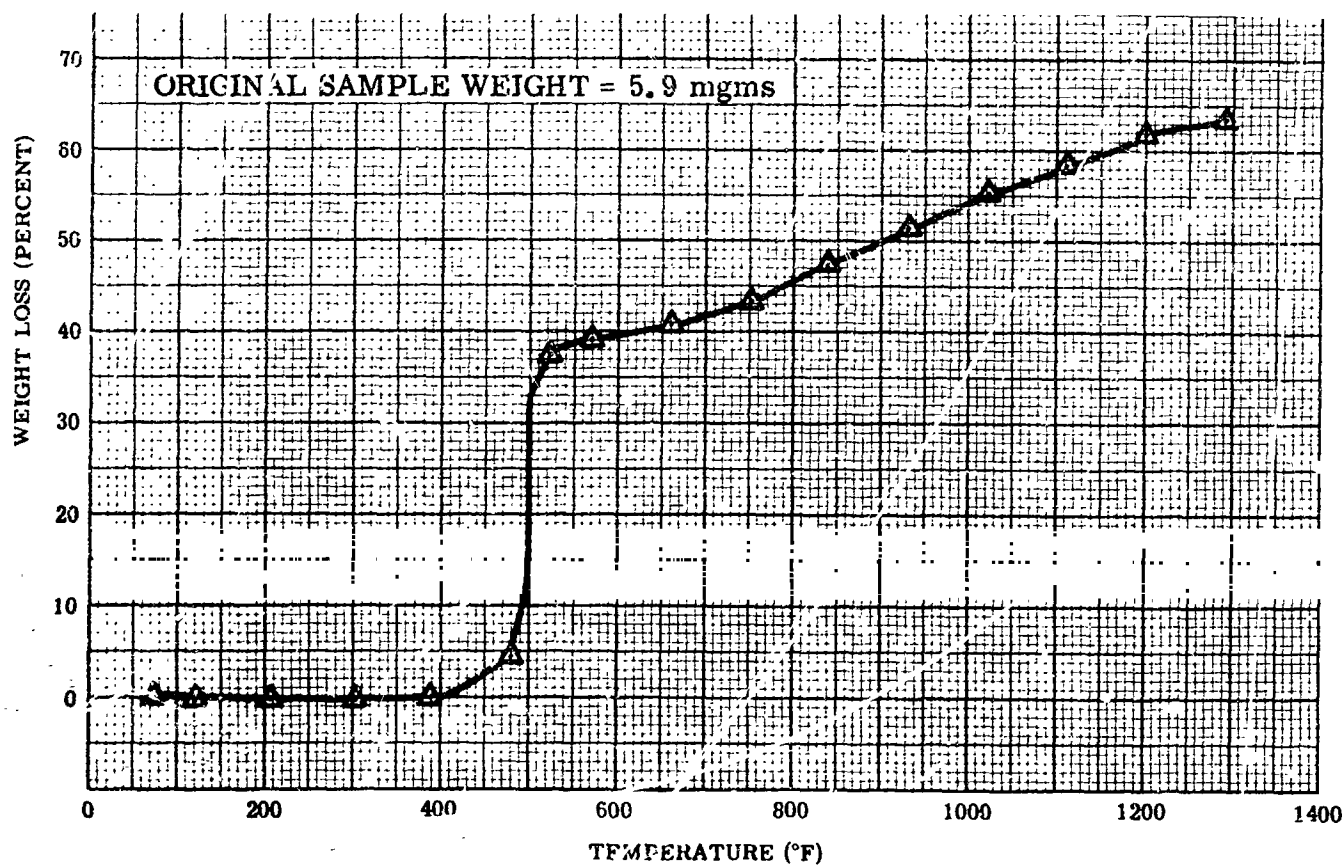


Figure 2-29. Thermogravimetric Analysis of "THERMO-LAG" T-500-6 for a Heated Rate of 41°F per Minute

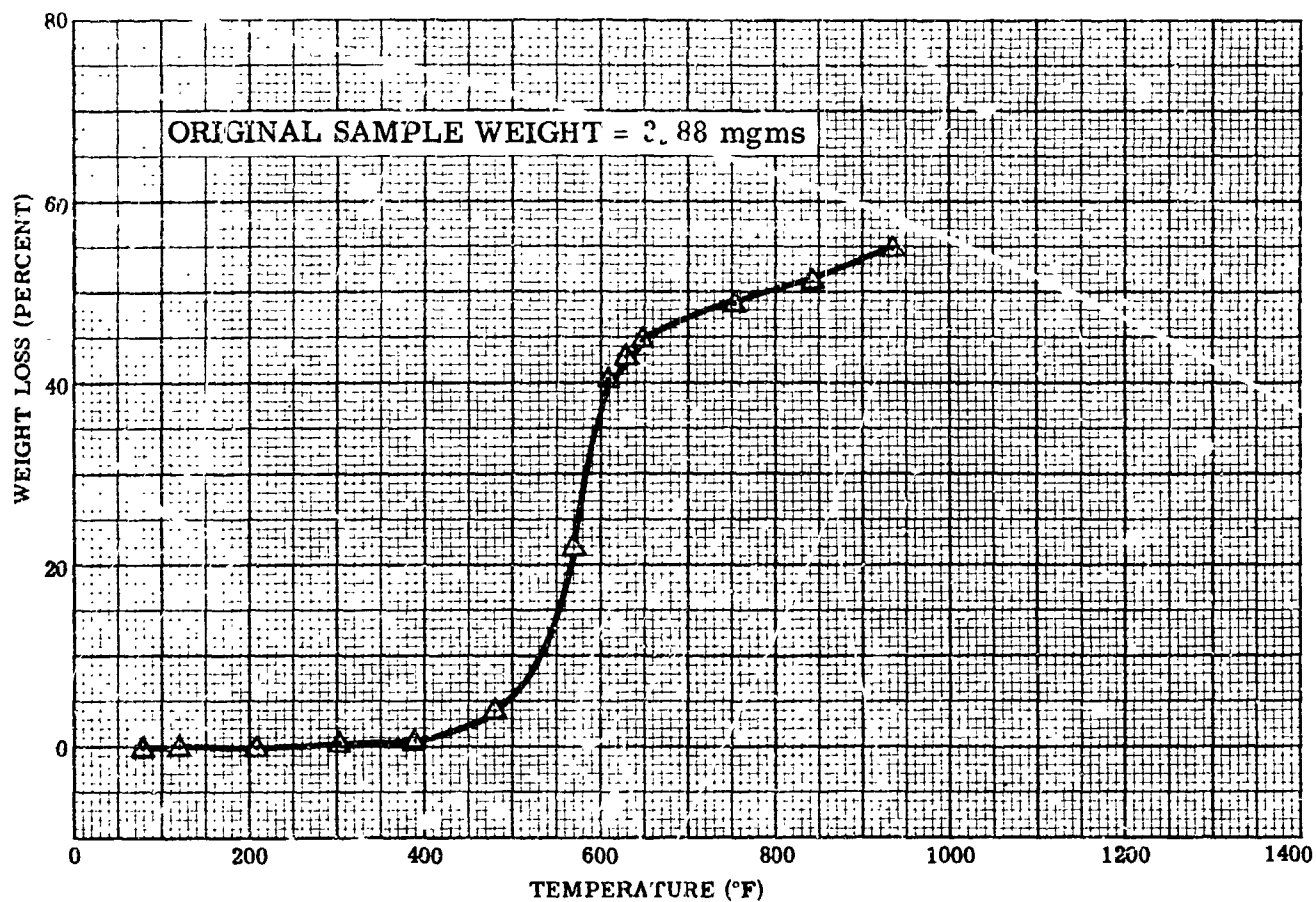


Figure 2-30. Thermogravimetric Analysis of "THERMO-LAG" T-500-6 for a Heated Rate of 50°F per Minute

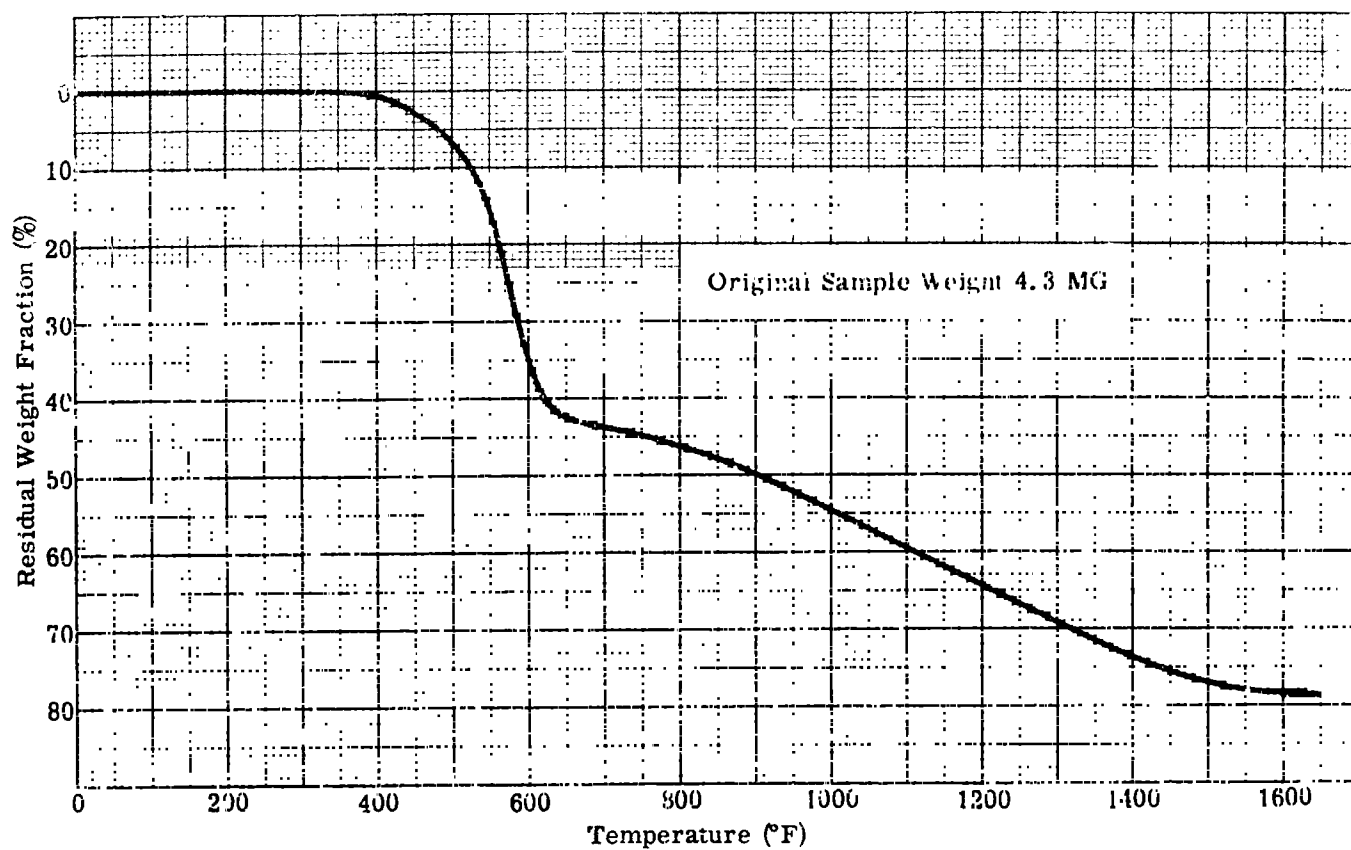


Figure 2-31. Thermogravimetric Analysis of "THERMO-LAG" T-500-6 for a Heated Rate of 50°F per Minute

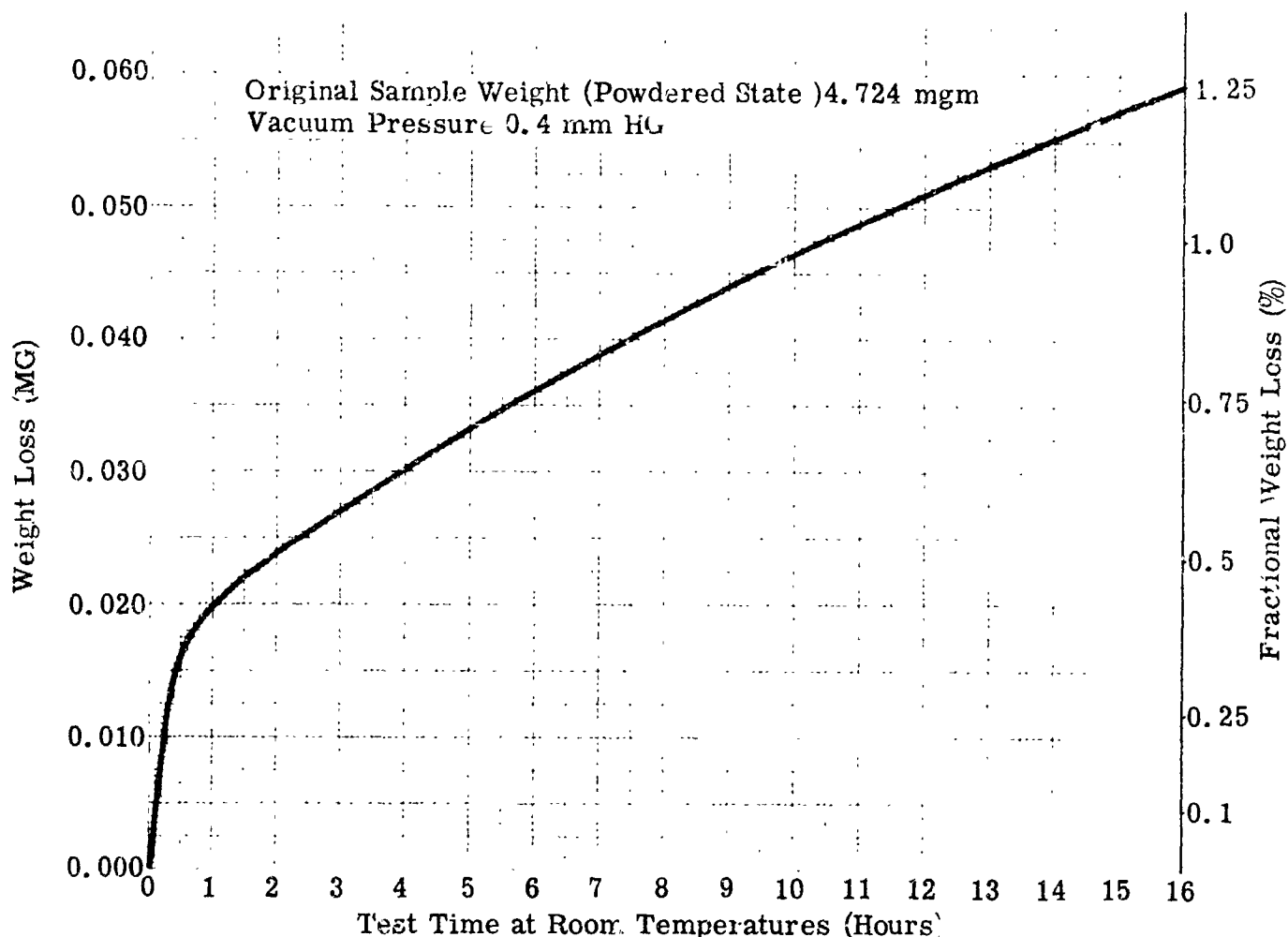


Figure 2-32. Thermogravimetric Analysis of "THERMO-LAG" T-500-4 Fractional Weight Loss

GAS CHROMATOGRAPH STUDIES. Experiments are being undertaken to analyze the composition of the gases produced from the char formation of heated "THERMO-LAG" T-500. The determination of the produced gases will enable an analysis to be performed on the debris layer mechanism.

The experiment utilizes a glass quartz tube which contains a known quantity of "THERMO-LAG" T-500-4 heated at a specific temperature by radiant lamps. The quartz tube is evacuated to remove the air, and the gases produced are collected in a deflated balloon.

The gases collected are passed through a gas chromatograph for separation of the various fractions. The retention time is recorded. The gases that condensed in the tube are collected and fractionized by distillation. No results are reported at this time. Effort will be made for the reporting of data during the next reporting period.



SECTION III

MECHANICAL PROPERTY AND PARAMETER EVALUATION

MECHANICAL PROPERTIES (PRECAST AND SPRAYED MATERIALS) TENSILE STRENGTH.

To facilitate testing at temperature extremes where utilization of conventional extensometers are not possible, tab-end specimens were used to determine the tensile properties of "THERMO-LAG" materials T-500-4 and T-500-6. The material test specimen models are illustrated in Figure A-5. (See Appendix A.)

The tensile strength was determined by using the recorded data in the standard stress equation

$$\sigma_{ULT} = \frac{P_{ULT}}{A}$$

where:

σ_{ULT} = Tensile Strength, (LB/IN.²)

P_{ULT} = Maximum load carried, (LB)

A = Cross-sectional area of sample, (IN.²)

The initial modulus stress was determined by using the recorded data in the equation

$$E = \frac{\sigma_E}{\epsilon_E}$$

where:

E = Initial modulus (LB/IN.²)

σ_E = Stress at elastic limit, (LB/IN.²)

ϵ_E = Strain at elastic limit (IN./IN.)

The elongation of the specimen, ϵ_{max} , (IN./IN.) was determined from the equation

where:

$$\epsilon_{max} = \frac{\Delta_{max}}{\text{Gage Length}}$$

Δ_{max} = Total increase in length (IN.) of gage length.

Twelve tests were performed on each of the "THERMO-LAG" materials T-500-4 and T-500-6. The results of these tests are illustrated in Figures 3-1 through 3-3 and recorded in Table 3-1. The tensile strength data for materials T-500-4 and T-500-6 are plotted in Figure 3-1. The data indicates the increase in tensile strength contributed by the laminated fibers in the material. Figure 3-2 shows the initial modulus of the T-500 materials. The tensile elongations of the T-500 materials are illustrated in Figure 3-3. The T-500-6 data indicates the effect the low-ductile strength laminated fibers have upon elongation.

The tests indicate that due to the metal tabs, there was a stress concentration at the ends of the "THERMO-LAG" specimens. At elevated temperatures, the materials were ductile enough to dissipate this effect and failures then occurred in the middle of the test specimens. At the cryogenic temperature region, the failures occurred in the bond between the specimen and the tab-end which accounts for the low recorded values of tensile strength and elongation illustrated in Figures 3-1 and 3-3, respectively. These tests were conducted during the selection phase for a desirable bonding material. Additional tensile tests will be conducted during the next six months with a selected bonding material.

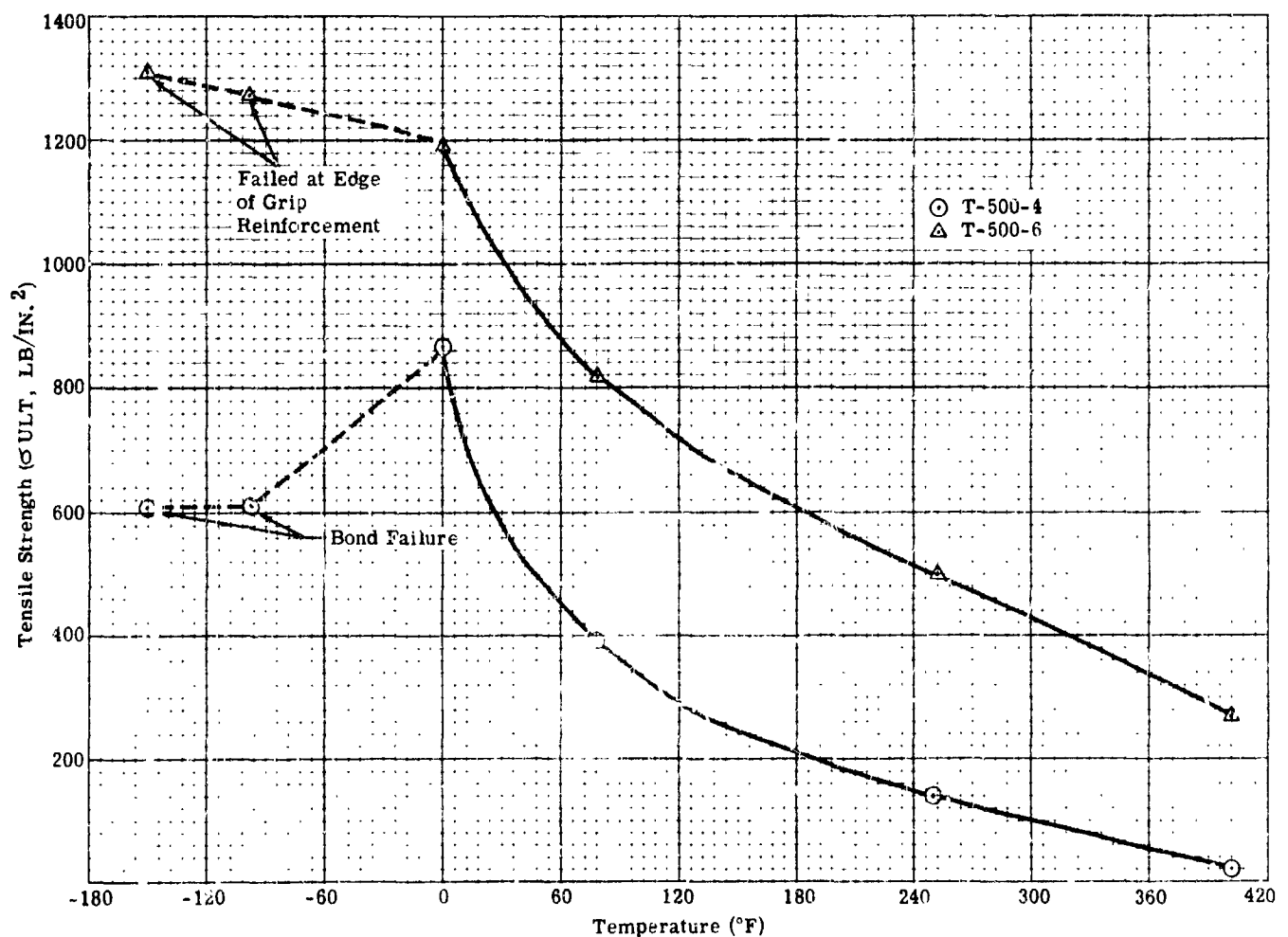


Figure 3-1. Tensile Strength of "THERMO-LAG" T-500-4 and T-500-6

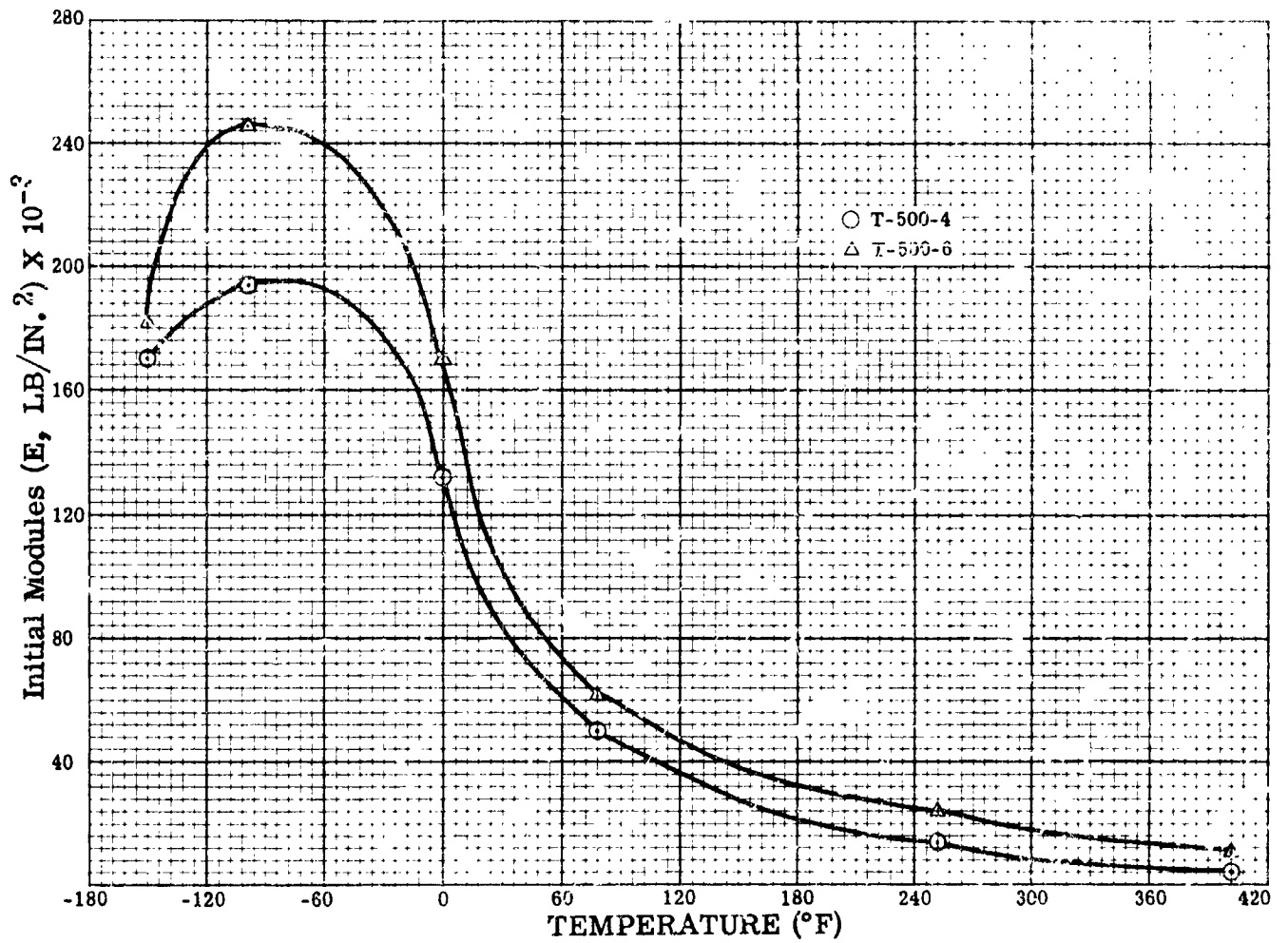


Figure 3-2. Initial Modulus of "THERMO-LAG" T-500-4 and T-500-6

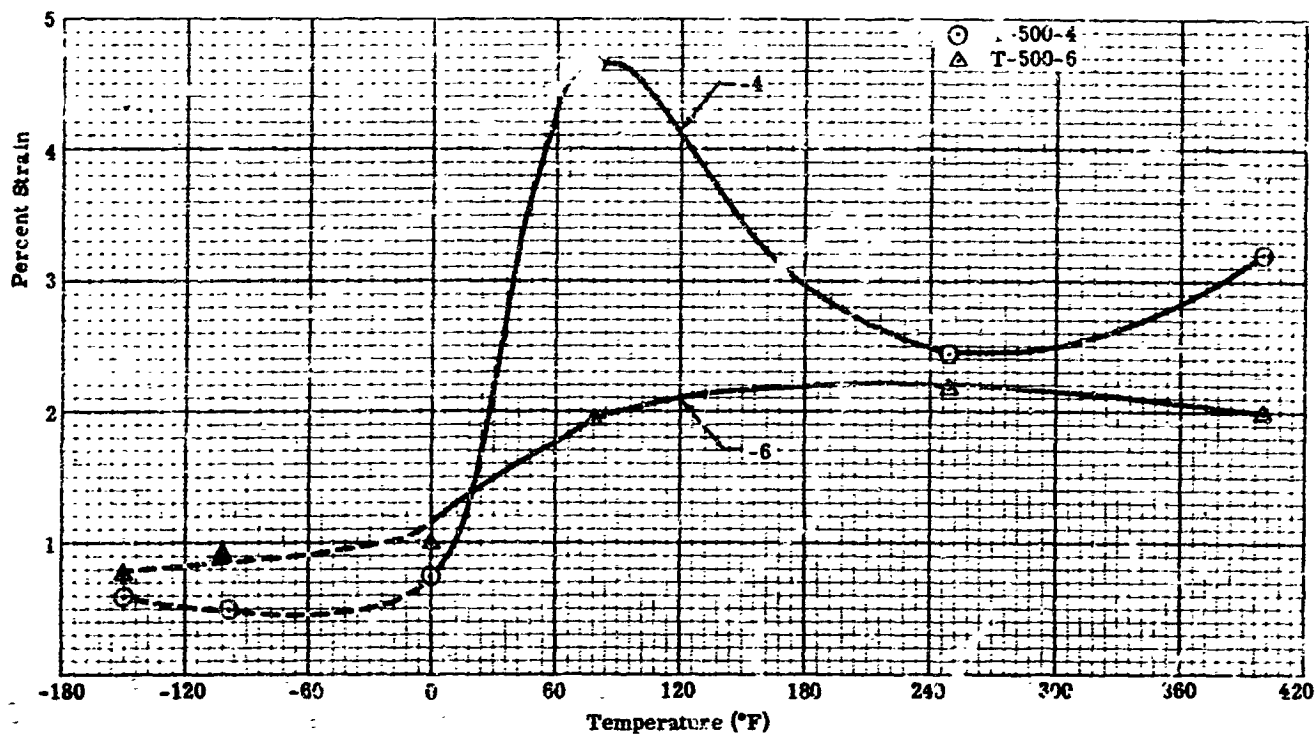


Figure 3-3. Tensile Elongation of "THERMO-LAG" T-500-4 and T-500-6

Sample No.	TEMPERATURE (°F)	Ultimate Strength (PSI)	Initial Modulus (PSI)	Elongation (IN/IN)
1-P-1-16-6	400	280	11,000	.020
1-P-1-17-6	400	260	10,800	.020
1-P-1-13-6	250	475	23,200	.021
1-F-1-15-6	250	525	24,000	.023

Table 3-1. Tensile Strength of "THERMO-LAG" T-500-4 and T-500-6



Sample No.	TEMPERATURE (°F)	Ultimate Strength (PSI)	Initial Modulus (PSI)	Elongation (IN/IN)
1-P-1-11-6	80	790	62,200	.019
1-P-1-4-6	80	850	63,000	.020
1-P-1-1-6	0	1250	166,000	.010
1-P-1-2-6	0	1130	175,000	.010
1-P-1-8-6	-100	1260	244,000	.009
1-P-1-6-6	-100	1290	246,000	.009
1-P-1-10-6	-150	1310	184,000	.008
1-P-1-19-6	-150	1315	183,000	.007
1-P-1-8-4	400	20	2,300	.040
1-P-1-2-4	400	23	3,600	.024
1-P-1-4-4	250	145	16,300	.021
1-P-1-5-4	250	135	9,850	.028
1-P-1-9-4	80	380	51,300	.049
1-P-1-8-4	80	400	50,500	.044
1-P-1-7-4	0	850	114,000	.009
1-P-1-11-4	0	880	148,000	.006
1-P-1-13-4	-100	620	198,000	.005
1-P-1-14-4	-100	600	191,000	.005
1-P-1-15-4	-150	630	168,000	.004
1-P-1-17-4	-150	580	173,000	.008

Table 3-1. Tensile Strength of "THERMO-LAG" T-500-4 and T-500-6 (Continued)

FLEXURAL STRENGTH.

The flexural properties of "THERMO-LAG" T-500 were determined in accordance with ASTM D 790-61A. The material test specimens were rectangular bars of 4.0 x 0.25 x 0.5 inches. (See Appendix A, Figure A-6.)

The maximum flexural strength σ in the outer fiber was determined by using the recorded data in the standard stress equation

$$\sigma = \frac{3PL}{bd^2}$$

where:

σ = Maximum flexural stress, (LB/FT²)

P = Applied load, (LB)

L = Span of specimen (FT)

b = Width of specimen, (FT)

d = Depth of specimen (FT)

Fourteen tests were performed on each of the "THERMO-LAG" materials T-500-4 and T-500-6. Seven of these were under cryogenic conditions. Results of the tests are plotted in Figure 3-4 and recorded in Table 3-2. The laminated fibers in T-500-6 contribute to its higher flexural strength for the entire range of test temperatures.

Figure 3-4 also records the ultraviolet test data of T-500-4 and T-500-6. Three tests were performed on each material. The recorded data indicates that ultraviolet exposure shows no effect at room temperature on the flexural stress property of "THERMO-LAG" T-500 materials.

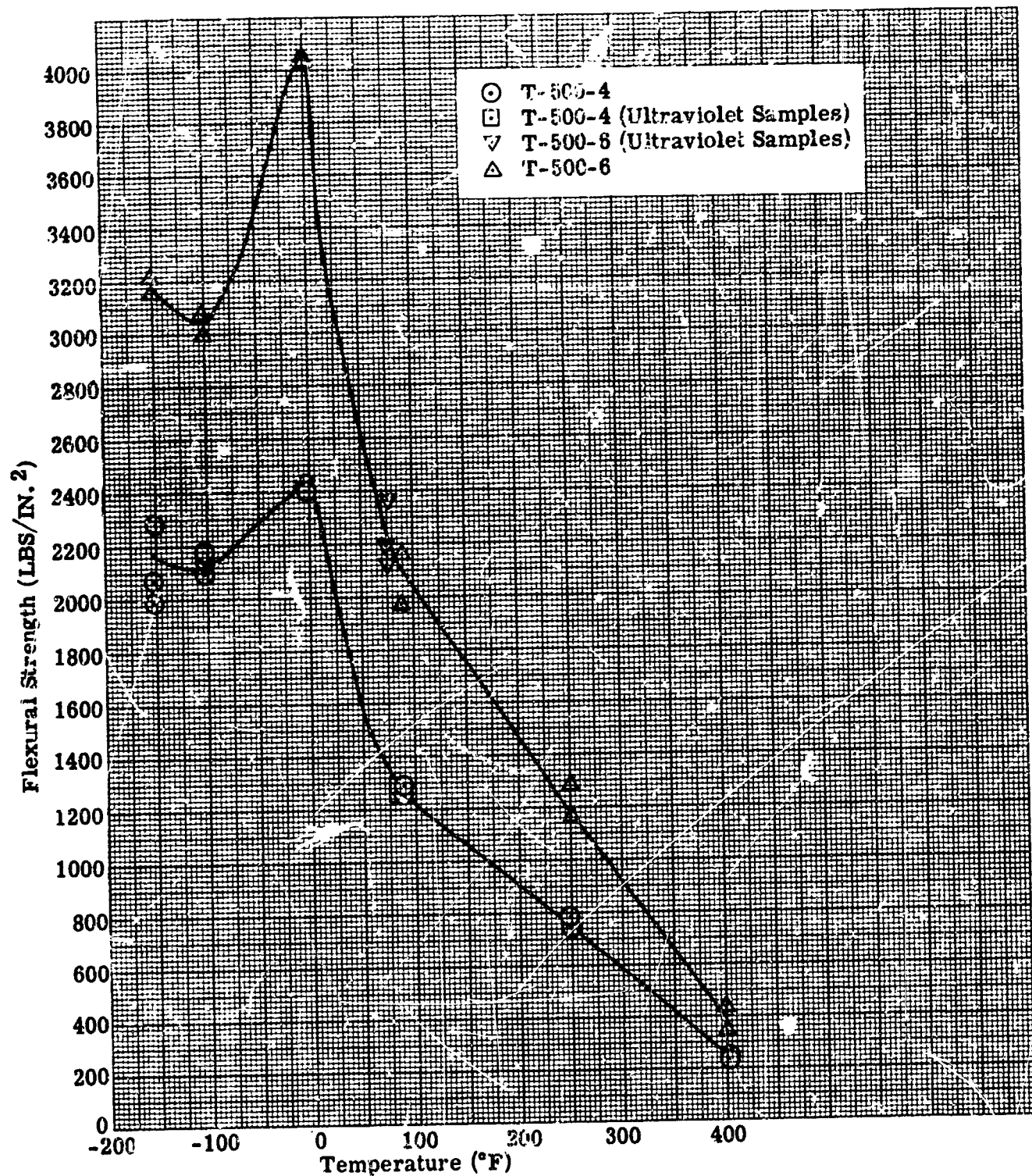


Figure 3-4. Flexural Strength of "THERMO-LAG" T-500-4 and T-500-6

Material	Specimen No.	Temperature (°F)	Specimen Depth (Inches)	Specimen Width (Inches)	Specimen Span (Inches)	Travel Rate Cross-head (IN./MIN)	Maximum Load (LBS)	Flexural Strength LB/IN ²
T-500-6 Virgin	3-H-2-2-5	90	.251	.500	2.687	.05	15	1930
T-500-6 Virgin	3-H-2-7-6	90	.252	.501	2.687	.05	17	2150
T-500-4 Virgin	3-G-1-11-4	90	.252	.498	2.687	.05	10	1230
T-500-4 Virgin	3-G-1-5-4	90	.253	.496	2.687	.05	10	1272
T-500-6 Virgin	8-H-1-1-6	250	.250	.504	2.687	.05	9	1150
T-500-6 Virgin	8-H-1-6-6	250	.250	.504	2.687	.05	10	1279
T-500-4 Virgin	3-G-1-7-4	250	.252	.500	2.687	.05	6	762
T-500-4 Virgin	3-G-1-12-4	250	.249	.495	2.687	.05	5.5	723
T-500-6 Virgin	3-H-2-5-6	400	.251	.498	2.687	.05	2.5	322
T-500-6 Virgin	8-H-1-2-6	400	.251	.502	2.687	.05	3.5	450
T-500-4 Virgin	3-G-1-1-4	400	.251	.493	2.687	.05	2	257
T-500-4 Virgin	3-G-1-3-4	400	.253	.504	2.687	.05	2	250
T-500-4 Virgin	3-07-C-1-4	0	.247	.503	4.0305	.05	18.2	2389
T-500-4 Virgin	3-08-C-1-4	0	.25	.492	4.0305	.05	18.2	2382
T-500-4 Virgin	3-09-C-1-4	-100	.257	.501	4.0305	.05	17.1	2082
T-500-4 Virgin	3-11-C-1-4	-100	.254	.497	4.0305	.05	17.0	2135
T-500-4 Virgin	3-03-C-1-4	-100	.253	.496	4.0305	.05	17.0	2161

Table 3-2. Test Results - Flexural Strength



Material	Specimen No.	Temperature (°F)	Specimen Depth (Inches)	Specimen Width (Inches)	Specimen Span (Inches)	Travel Rate Cross-head IN./MIN	Maximum Load (LBS)	Flexural Strength LB/IN ²
T-500-4 Virgin	3-12-C-1-4	-150	.247	.498	4.0305	.05	17.4	2270
T-500-4 Virgin	3-06-C-1-4	-150	.250	.509	4.0305	.05	15.7	1990
T-500-4 Virgin	3-6-C-1-4	-150	.252	.504	4.0305	.05	16.4	2066
T-500-6 Virgin	3-02-C-1-6	0	.249	.500	4.0305	.05	30.7	3991
T-500-6 Virgin	3-05-C-1-6	0	.248	.500	4.0305	.05	30.5	3991
T-500-6 Virgin	3-H-2-6-6	-100	.250	.501	4.0305	.05	23.75	3058
T-500-6 Virgin	3-H-2-12-6	-100	.249	.501	4.0305	.05	23.0	2981
T-500-6 Virgin	3-G-1-9-6	-150	.250	.501	4.0305	.05	24.85	3200
T-500-6 Virgin	3-H-2-9-6	-150	.250	.502	4.0305	.05	24.5	3145
Ultraviolet Tests								
T-500-4	8-G-1-5-4	77	.250	.502	4.0305	.05	9.7	1245
T-500-4	8-G-1-4-4	77	.250	.505	4.0305	.05	9.75	1244
T-500-4	8-G-1-6-4	77	.250	.504	4.0305	.05	9.75	1248
T-500-6	8-H-1-3-6	77	.254	.501	4.0305	.05	17.0	2121
T-500-6	8-H-1-4-6	77	.254	.500	4.0305	.05	17.4	2171
T-500-6	8-H-1-5-6	77	.250	.502	4.0305	.05	18.4	2362

Table 3-2. Test Results - Flexural Strength (Continued)

SHEAR STRENGTH.

Shear stress properties in the direction normal to the flat face surface of "THERMO-LAG" T-500 specimens were determined in accordance with ASTM D 732-46, using the punch type shear tool. The material test specimens were 0.25 inch thick blocks two inches square with a 5/16 inch hole drilled in the center to accommodate the bolt on the shear tool. (See Appendix A, Figure A-8.)

The punched shear stress was determined by using the recorded data in the standard stress equation

$$S = \frac{L}{A}$$

where:

S = Shear strength (LB/IN.²)

L = Load carried (LB)

A = Area of sheared edge (FT²)

Area of sheared edge = $X C$

C = Circumference of the punched tool (πd), where diameter of punch tool was one inch.

X = Thickness of specimen

Thirteen tests were performed on each of the "THERMO-LAG" materials T-500-4 and T-500-6. Six tests for T-500-4 and seven tests for T-500-6 were performed at cryogenic conditions. The results of these tests for both materials are plotted in Figure 3-5 and recorded in Table 3-3. The reported shear strength is higher for the T-500-6 material at room and elevated temperatures as it is in the direction normal to the laminated fibers.

For the range of temperatures tested, the results indicate that the T-500-6 material contains about the appropriate percentage of laminated fibers. They contribute to its higher normal shear stress at elevated temperatures and cause only slightly lower values than the normal T-500 material at cryogenic temperatures. The shear stress tests normal to the structural fibers of the material are to simulate approximately the effects that impingement of micrometeorites, dust, and water particles would have on a heat shield material.

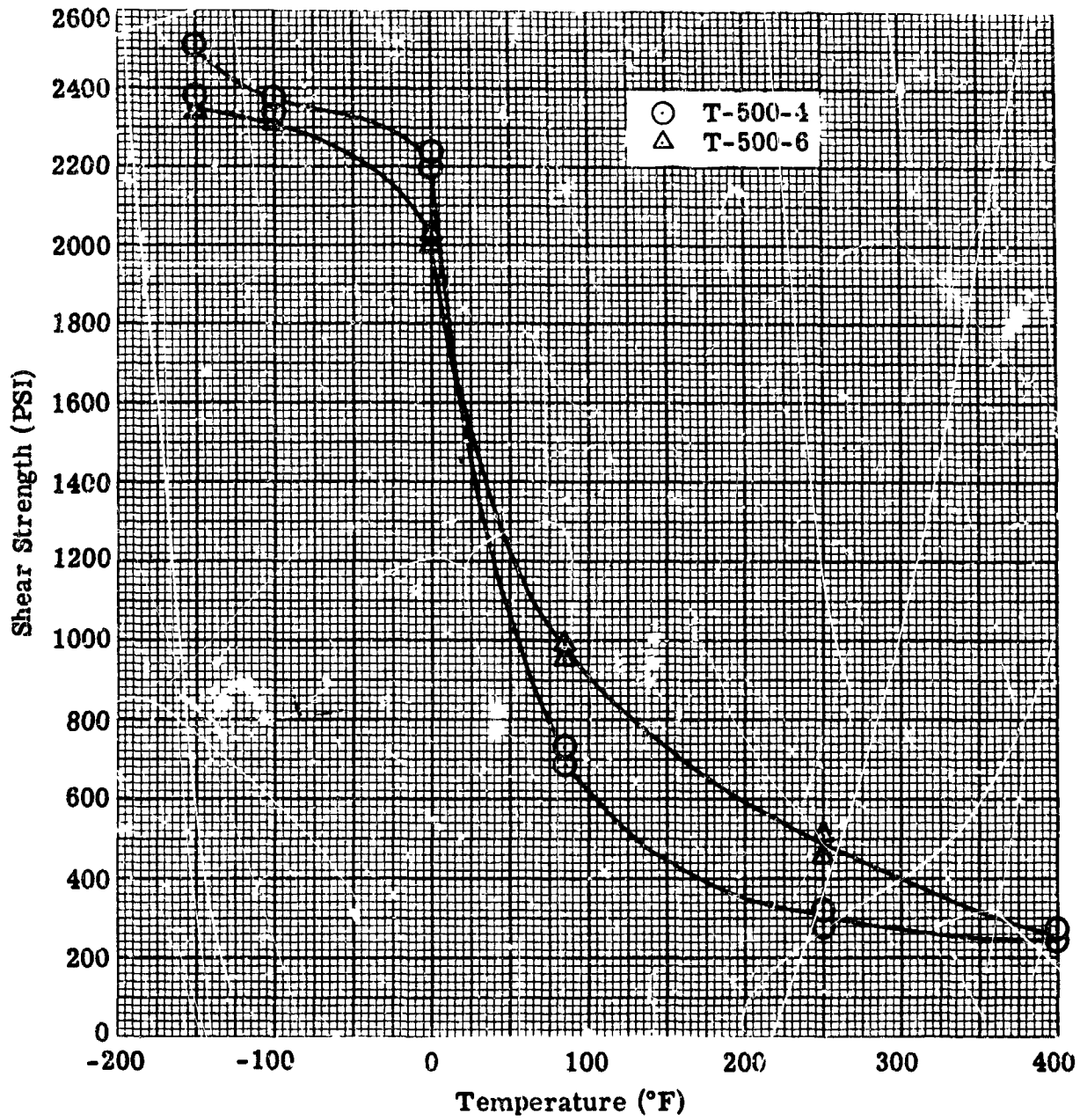


Figure 3-5. Shear Strength of "THERMO-LAG" T-500-4 and T-500-6

Sample No.	Temperature (°F)	Thickness (IN.)	Area of Shear (IN. ²)	Max Load (LB)	Shear Strength (PSI)
2-M-1-1-6	86	.251	$\pi(.251) = .787$	775	984.0
2-M-1-1A-6	86	.248	.779	750	952.0
2-M-1-12-6	250	.248	.782	365	467.0
2-M-1-11-6	250	.2465	.773	370	479.0
2-M-1-6-6	250	.247	.775	400	516.0
2-M-1-2-6	400	.2495	.783	190	243.0
2-M-1-9-6	400	.248	.779	215	276.0
2-H-2-3-4	84	.254	.797	585	734.0
2-H-2-4-4	85	.250	.787	540	686.0
2-H-2-11-4	250	.252	.792	220	277.5
2-H-2-4-4	250	.255	.800	260	325.0
2-H-2-1-4	400	.253	.795	215	270.0
2-H-2-7-4	400	.251	.787	200	254.0

Table 3-3. Shear Strength of "THERMO-LAG" T-500-4 and T-500-6



COEFFICIENT OF THERMAL EXPANSION.

Tests to determine the coefficient of thermal expansion for "THERMO-LAG" T-500 were performed on both virgin and charred materials in accordance with ASTM D 696-44. A description of the material test specimen is illustrated in Figure A-9. (See Appendix A.)

The coefficient of thermal expansion, α , (IN/IN.°C) was calculated from the measured data between the test temperature and 30°C above and below test temperature using the equation

$$\alpha = \frac{\Delta L}{L \Delta T}$$

where:

ΔL = Average of three changes in length of the samples due to heating, (inches).

L = Length of test sample at test temperature, (inches).

ΔT = Temperature difference, °C, over which the changes in length of the specimen are measured.

Eight tests were performed on each of the virgin "THERMO-LAG" materials T-500-4 and T-500-6. The results of these tests are illustrated in Figure 3-6 and recorded in Table 3-4. Study of the plotted data indicates certain behavior characteristics of the two materials.

1. A phase transition from the glass to the elastic state in the temperature regime below room value for T-500-6.
2. The erratic behavior above 250°F indicates where T-500-4 began to behave as a viscous fluid.

For the charred "THERMO-LAG" ten tests were performed on each of the materials T-500-4 and T-500-6. The results of the tests are plotted in Figure 3-7 and recorded in Table 3-4.

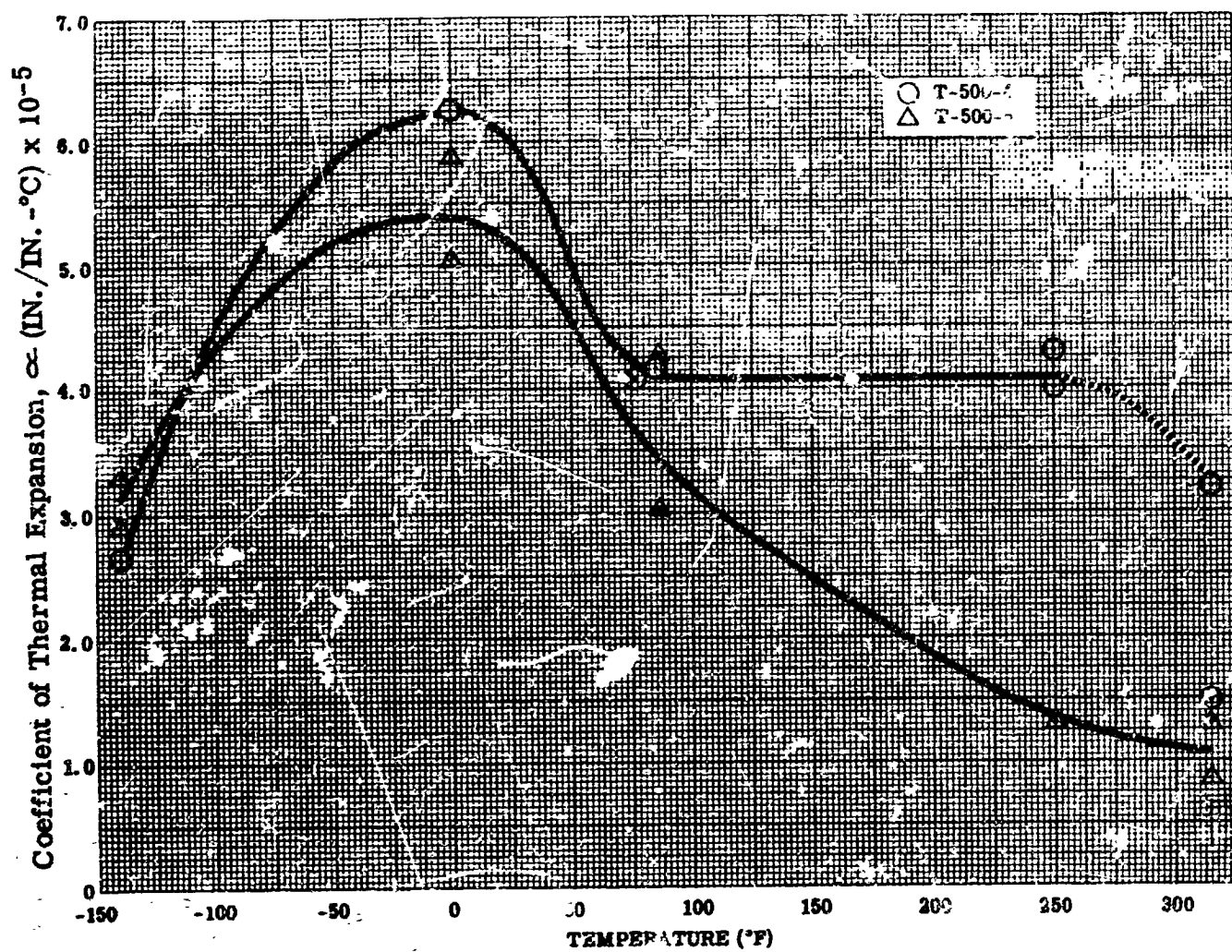


Figure 3-6. Coefficient of Thermal Expansion at Virgin "THERMO-LAG" Materials T-500-4 and T-500-6

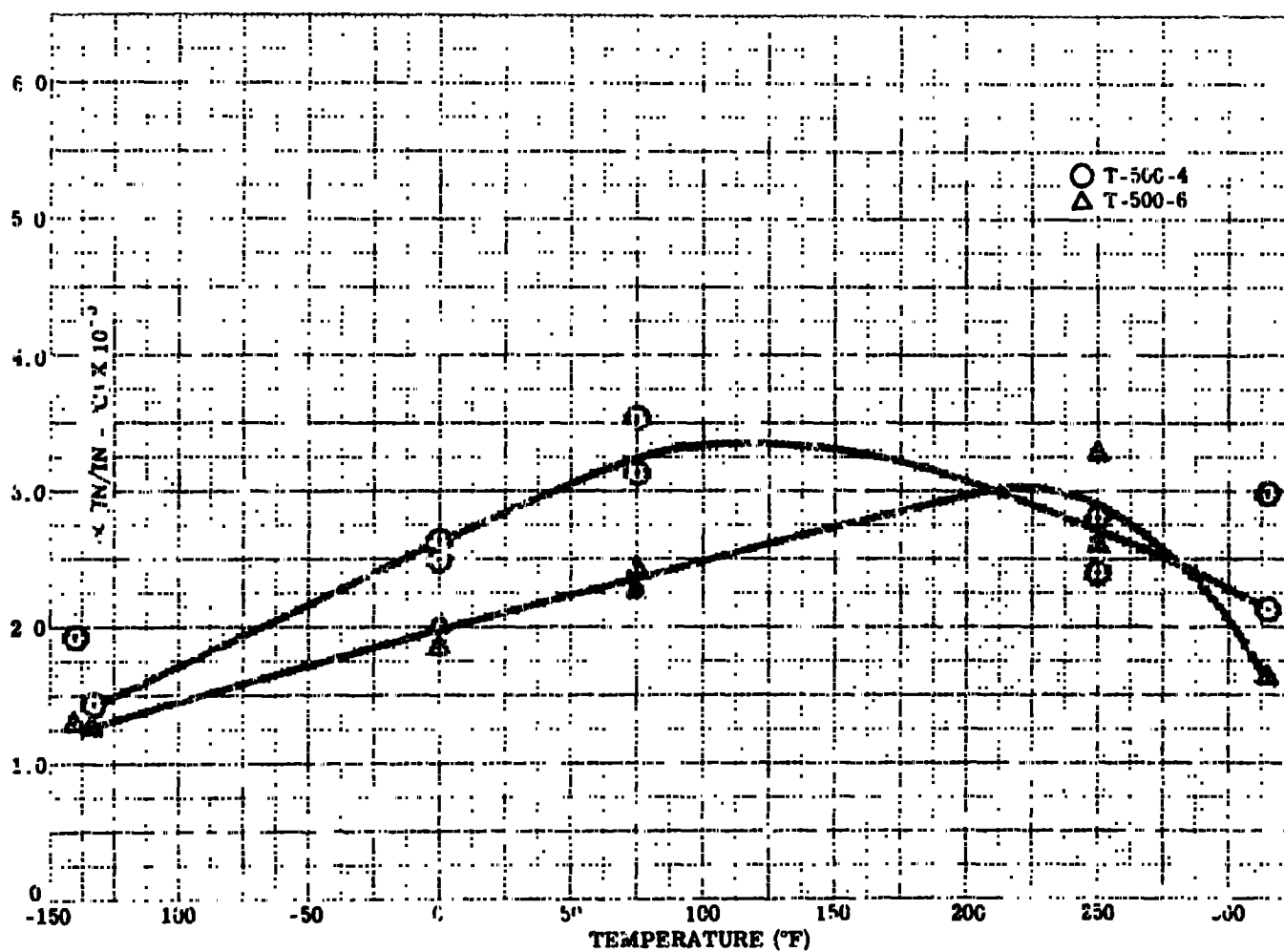


Figure 3-7. Coefficient of Thermal Expansion of "THERMO-LAG" T-500-4 and T-500-6

Material	Sample No.	Temperature		L inches	ΔL inches	ΔT °C	$a = \frac{\Delta L}{L \times \Delta T}$ IN/IN.-°C
		°C	°F				
T-500-4 charred	13-G-1-21-1	-95	-139	2.0174	.00117	30	1.93×10^{-5}
T-500-4 charred	13-G-1-30-4	-91	-132	2.0410	.00087	30	1.42×10^{-5}
T-500-4 charred	13-G-1-22-4	-18	0	2.0468	.00161	30	2.62×10^{-5}
T-500-4 charred	13-G-1-23-4	-18	0	2.0550	.00154	30	2.50×10^{-5}
T-500-4 charred	13-G-1-25-4	24	75	2.0497	.00218	30	3.55×10^{-5}
T-500-4 charred	13-G-1-29-4	24	75	2.0535	.00193	30	3.13×10^{-5}
T-500-4 charred	13-G-1-24-4	121	250	2.0583	.00148	30	2.40×10^{-5}
T-500-4 charred	13-G-1-26-4	121	250	2.0620	.00175	30	2.83×10^{-5}
T-500-4 charred	13-G-1-28-4	157	315	2.0710	.00132	30	2.12×10^{-5}
T-500-4 charred	13-G-1-27-4	157	315	2.0640	.00186	30	3.00×10^{-5}
T-500-4	13-G-1-13-4	-96	-141	2.0722	.00206	30	3.31×10^{-5}
T-500-4	13-G-1-16-4	-96	-141	2.0384	.00162	30	2.65×10^{-5}
T-500-4	13-G-1-6-4	-18	0	2.0719	.00339	30	6.26×10^{-5}
T-500-4	13-G-1-1-4	-18	0	2.0629	.00285	30	6.22×10^{-5}

Table 3-4. Coefficient of Thermal Expansion Test Results



Material	Sample No.	Temperature		L inches	ΔL inches	ΔT °C	$\alpha = \frac{\Delta L}{L \times \Delta T}$ IN./IN.-°C
		°C	°F				
T-500-4	13-G-1-4-4	24	76	2.0780	.00255	30	4.09×10^{-5}
T-500-4	13-G-1-17-4	30	86	2.0801	.00260	30	4.17×10^{-5}
T-500-4	13-G-1-20-4	157	315	2.0588	.00392	30	1.489×10^{-5}
T-500-4	13-G-1-14-4	157	315	2.0735	.00030	30	$.482 \times 10^{-5}$
T-500-4	13-G-1-11-4	157	315	2.0897	.00201	30	3.21×10^{-5}
T-500-6 charred	13-G-1-27-6	-94	-137	2.0549	.00078	30	1.27×10^{-5}
T-500-6 charred	13-G-1-22-6	-96	-141	2.0821	.00090	30	1.29×10^{-5}
T-500-6 charred	13-G-1-25-6	-18	0	2.0817	.00116	30	1.86×10^{-5}
T-500-6 charred	13-G-1-8-6	-18	0	2.0814	.00332	30	5.32×10^{-5}
T-500-6 charred	13-G-1-24-6	24	75	2.0730	.00141	30	2.27×10^{-5}
T-500-6 charred	13-G-1-26-6	24	75	2.0738	.00152	30	2.44×10^{-5}
T-500-6 charred	13-G-1-23-6	121	250	2.0587	.00162	30	2.62×10^{-5}
T-500-6 charred	13-G-1-21-6	121	250	2.0551	.00203	30	3.29×10^{-5}
T-500-6 charred	13-G-1-12-6	157	315	2.0588	.00103	30	1.67×10^{-5}

Table 3-4. Coefficient of Thermal Expansion Test Results (Continued)

Material	Sample No.	Temperature		L inches	ΔL inches	ΔT °C	$\alpha = \frac{\Delta L}{L \times \Delta T}$ IN./IN.-°C
		°C	°F				
T-500-6 charred	13-G-1-7-6	157	315	2.0461	.00098	30	1.60×10^{-5}
T-500-6	13-G-1-15-6	-96	-141	2.0496	.00179	30	2.91×10^{-5}
T-500-6	13-G-1-7-6	-96	-141	2.0636	.00205	30	3.31×10^{-5}
T-500-6	13-G-1-10-6	-18	0	2.1000	.00318	30	5.05×10^{-5}
T-500-6	13-G-1-9-6	-18	0	2.0827	.00367	30	5.87×10^{-5}
T-500-6	13-G-1-3-6	30	86	2.0854	.00180	30	3.04×10^{-5}
T-500-6	13-G-1-6-6	30	86	2.0975	.00270	30	4.29×10^{-5}
T-500-6	13-G-1-17-6	157	315	2.1030	.00081	30	1.284×10^{-5}
T-500-6	13-G-1-14-6	157	315	2.0809	.00052	30	$.833 \times 10^{-5}$
T-500-4	13-G-1-2-4	121	250	2.0933	.0027	30	4.30×10^{-5}
T-500-4	13-G-1-9-4	121	250	2.0867	.0025	30	4.0×10^{-5}
T-500-6	13-H-2-1-6	121	250	2.074	.0009	30	1.45×10^{-5}
T-500-6	13-H-2-10-6	121	250	2.0625	.0008	30	1.29×10^{-5}

Table 3-4. Coefficient of Thermal Expansion Test Results (Continued)



SECTION IV

SPACE PERFORMANCE EVALUATION

RE-ENTRY SIMULATION (CONVECTION AND RADIATION).

TEST DESIGN.

This test program was designed to use existing techniques for the evaluation of existing thermodynamic performance of "THERMO-LAG" T-500 material. Work progressed in this test phase which was also designed to separate the different effects of the environment upon the material so that the test data could be correlated with theoretical predictions. The flight simulation tests conducted during the reporting period were:

1. Variation of oxygen concentration in plasma stream.
2. Ablation performance under purely radiative heat loads.
3. Flight simulation for sidewall heating at low pressures.

VARIATION OF OXYGEN CONCENTRATION IN PLASMA STREAM.

To provide an order of magnitude estimate of the importance of debris layer-oxygen chemical kinetics upon the rate and extent of debris layer recession in relation to the variation of oxygen concentration in the plasma stream, the test matrix recorded in Table 4-1 was used. The test matrix contains the conditions and durations of tests performed on the model geometries.

Table 4-2 identifies each test and summarizes the preliminary results. The preliminary data reduction results which indicate the trends of parameter interdependency associated with an oxidizing debris are presented in Figures 4-1 and 4-2. A summary of the data reduction and the approach to the analysis of debris layer recession is discussed in the following paragraphs.

The surface recession of a carbonaceous debris layer under the influence of a high energy fluid stream was determined by experimental analysis. Consideration was given to:

1. Oxidation of carbon with oxygen in the fluid stream.
2. Erosion from shear stress exerted mostly by the displaced nitrogen.

The empirical expression was a function of the carbon surface temperature. Experimental data indicates that recession by oxidation prevails at carbon surface temperatures of approximately 3300°F and below, with erosion by shear stresses predominant at the higher values.

Predicted values of the reaction rate are tabulated in Table 4-3. Table 4-4 records experimental data and the results of the analysis. Investigation of the tabulated test data indicates that only the rate controlling term of the applied Arrhenius equation was used to determine the values of R_I and n .

A check of the analytical values was made from a cross plot of recession versus surface temperature as a function of test time. Variation of the parameter from 0 to 1.0 (the term R_I is independent of surface temperature) resulted in almost equivalent values of R_I . The values of R_I listed in Table 4-4 were predicted for the order of reaction, $n = 0.25$. The existing variations in the predicted reaction rate constants are 15 percent.

Use of the predicted values for the terms R_I and n allowed the rate controlling equation to be expressed in the generalized form

$$\frac{dx}{dt} = 100 (P_{O_2})^{0.25} e^{-\frac{36200}{T_w}}$$

for the instantaneous reaction rate, and

$$\int_{x_1}^{x_2} dx = 100 (P_{O_2})^{0.25} \int_{t_1}^{t_2} e^{-36200/T_w(t)} dt$$

for the over-all recession.

A correlation of debris recession for the oxidation test is illustrated in Figure 4-3. The ranges of variables at which the diffusion controlling equation and the recession due to oxidation effect equation can be expected to give good correlation to measured recession rates are:

$$T_w (^{\circ}F) = 3000 - 3300$$

$$P_{O_2}, \text{ stag (atm)} = 0.0001 - 0.006$$

$$H_{\text{stag}} (\text{BTU/lb.}) = 5000 - 15000$$

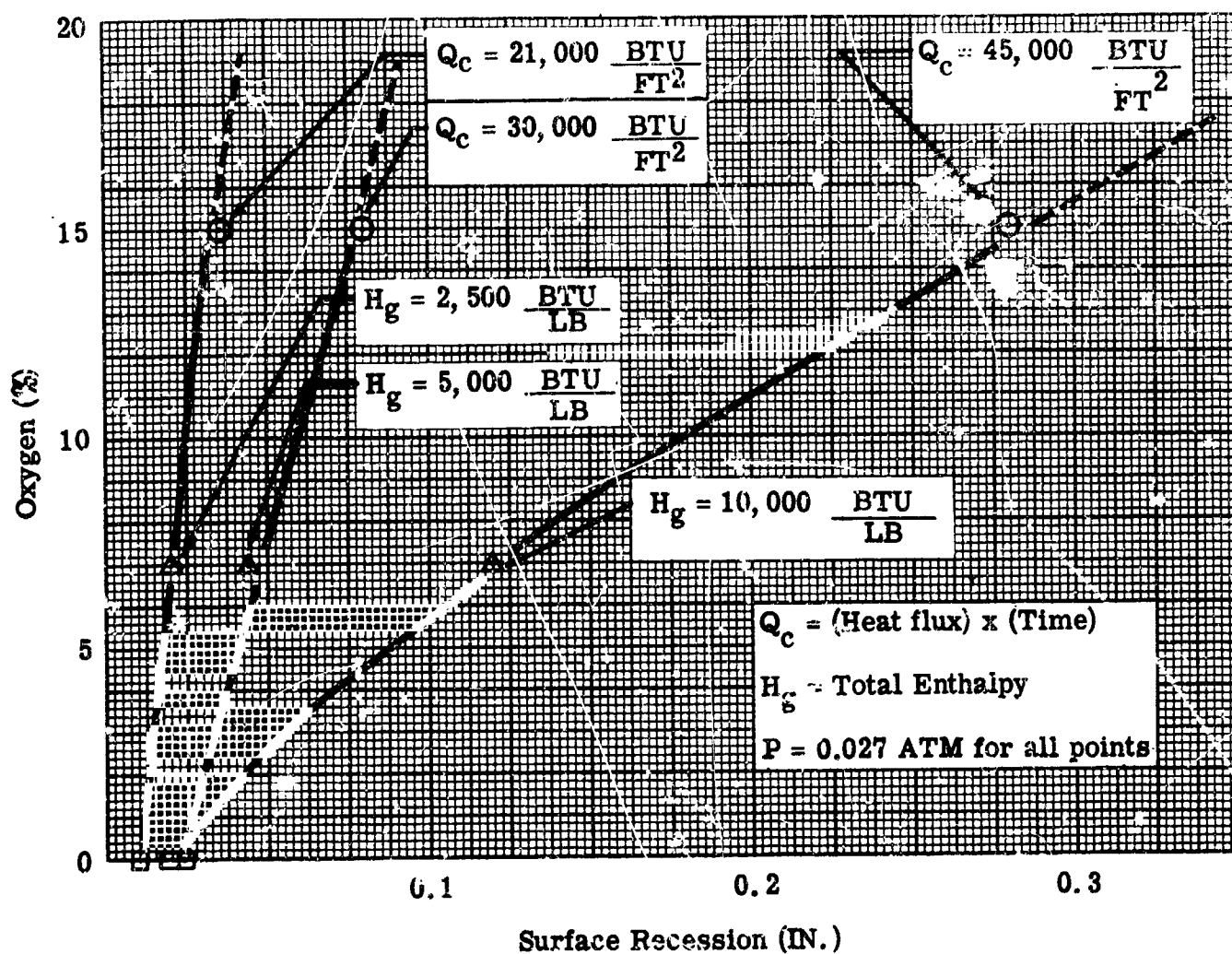


Figure 4-1. Percentage of Oxygen Versus Surface Recession for "THERMO-LAG" T-500

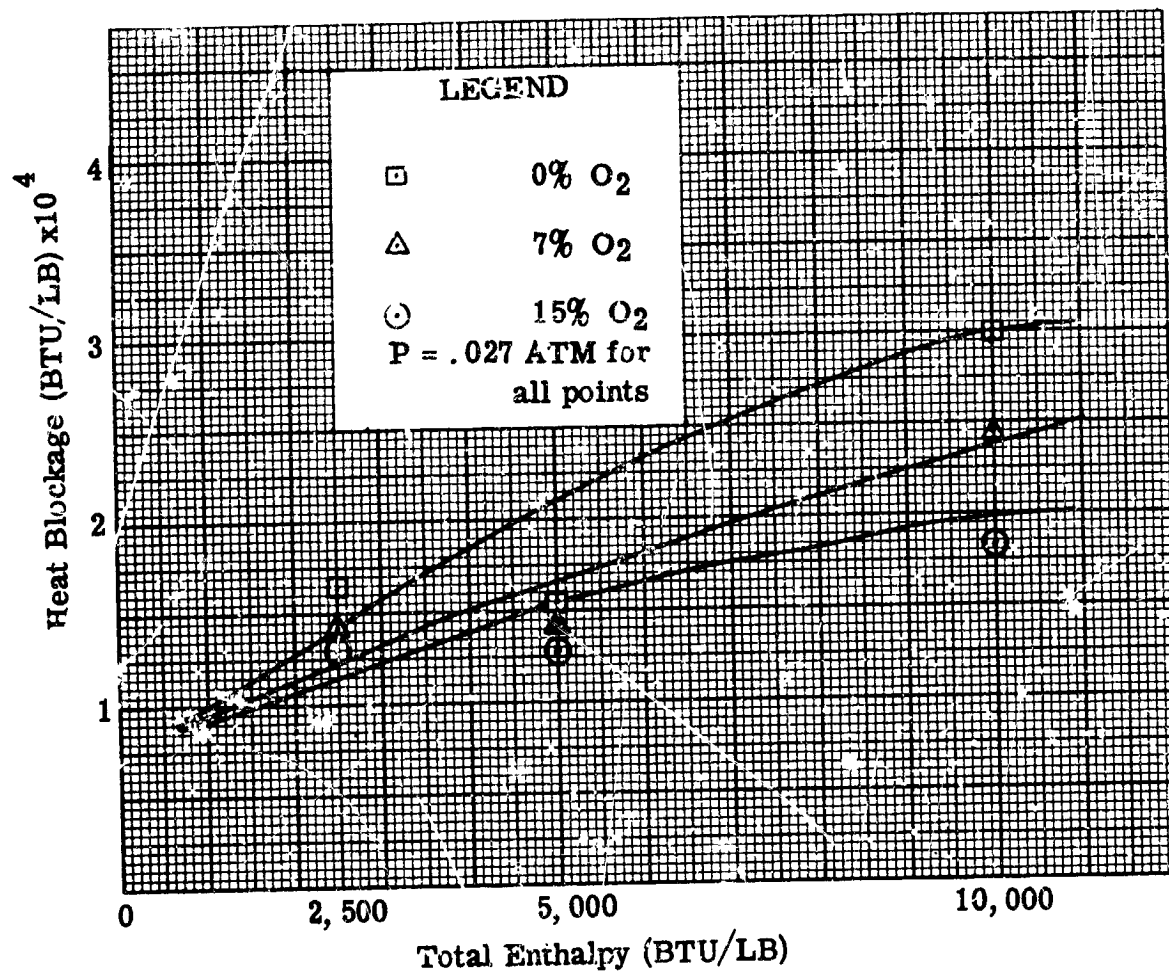


Figure 4-2. Heat Blockage Versus Total Enthalpy for "THERMO-LAG" T-500

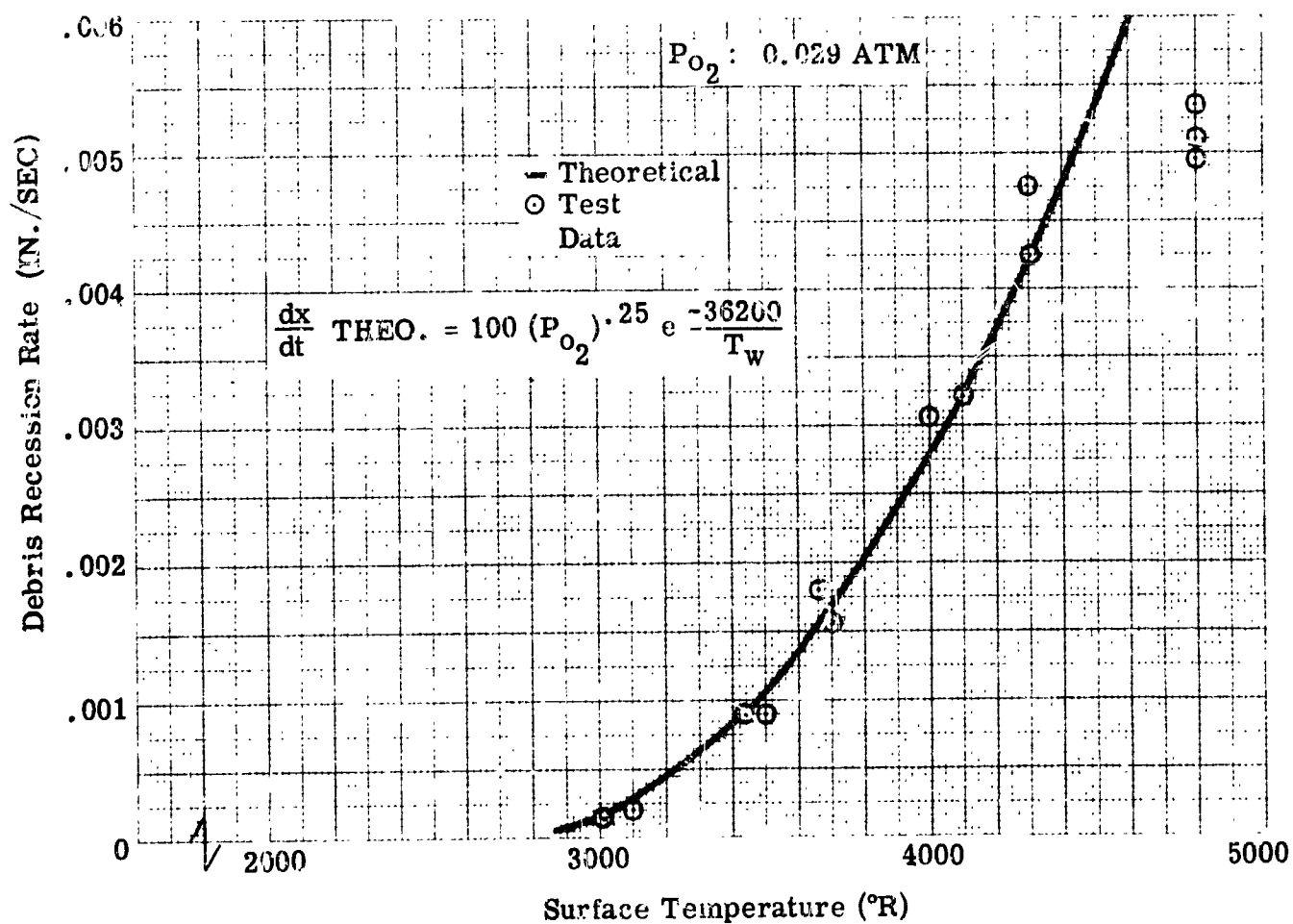


Figure 4-3. Correlation of Debris Recession Data

Oxygen (Percent)	15 7 0	420 420 420	Test Point 3 4 5	Model No. O-14 O-15 O-16	Total Enthalpy (BTU/LB) 2,500 2,500 2,500	Nose Radius (IN.) 1 1 1	Cold Wall Stagnation Heat Flux (BTU/FT ² -SEC) 50 50 50	Stagnation Pressure (ATM) .026 .026 .026
Oxygen (Percent)	15 7 0	300 300 300	8 9 10	O-9 O-11 O-13	5,000 5,000 5,000	1 1 1	110 110 110	.026 .026 .026
Oxygen (Percent)	15 7 0	150 150 150	15 16 17	O-1 O-2 O-3	10,000 10,000 10,000	5/8 5/8 5/8	300 300 300	.0290 .0290 .0290
Oxygen (Percent)	15 7 0	300 300 300	21 22 23	O-6 O-7 O-8	15,000 15,000 15,000	1 1 1	110 110 110	.00267 .00267 .00267

Table 4-1. Variation of Oxygen Concentration Tests



Model No.	0-1	0-2	0-3	0-6	0-7	0-8	0-9	0-11	0-13	0-14	0-15	0-16
Nose Radius (IN.)	5/8	5/8	5/8	1	1	1	1	1	1	1	1	1
Run Duration (SEC)	150	150	150	500	300	300	300	300	300	420	420	420
Stagnation Enthalpy (BTU/LB)	9,897	10,055	10,165	14,988	15,115	15,052	4,965	5,000	5,110	2,488	2,510	2,505
Test Point No.	15	15	17	21	22	23	8	9	10	3	4	5
Stagnation Cold Wall Heat Flux (BTU/FT ² -SEC)	297	295	301	108	105	107	109	107	109	18.7	46.8	47.9
Percent O ₂	15	7	0	15	7	0	15	7	0	15	7	0
Model Stagnation Pressure (ATM)	.0291	.0289	.0291	.0267	.0265	.0266	.026	.0253	.026	.0255	.0258	.0256
Recession to Debris Layer (IN.)	.28	.117	.022	.268	.156	.033	.068	.042	.026	.035	.033	.009
Debris Thickness (IN.)	.08	.17	.20	.17	.25	.27	.30	.30	.51	.19	.13	.17
Total Recession (IN.)	.360	.287	.222	.438	.406	.333	.368	.342	.336	.223	.215	.179
Surface Temp. = 0.9 (°F)	3,510	3,300	2,380	3,180	3,110	3,050	2,910	2,780	2,620	2,010	1,920	1,780
Enthalpy Difference (BTU/LB)	8,730	9,000	9,405	13,968	14,185	14,077	4,035	4,110	4,270	1,943	1,895	1,925
Hot Wall Heat Flux (BTU/FT ² -SEC)	262	265	279	101	98.3	100	88.7	88	91	36	35.3	36.8
Cold Wall Heat Blockage (BTU/LB)	18,550	23,300	30,500	11,100	11,500	14,150	13,550	14,000	14,600	13,650	13,750	16,900

Table 4-2. Stagnation Point Oxidation Results

Temperature (°F)	Log ₁₀ K _p , CO	Log ₁₀ K _p , CO ₂
1800	8.2	7.5
2500	7.5	5.0
4000	6.3	2.5
5500	5.6	1.2

Table 4-3. Equilibrium Constants for CO and CO₂ at Elevated Temperatures



Model No.	Stagnation Enthalpy (BTU/LB)	Partial Oxygen Pressure (ATM)	Surface Temperature (°F)	Recession Rate (IN./SEC)	Rate Constant (R) (IN./SEC-ATM. 25)	Order of Reaction (n)
0-1	10,000	.00435	3,912	2.5×10^{-3}	100	.25
0-2	10,000	.00203	3,912	2×10^{-3}	97	.25
0-1	10,000	.00435	3,732	1.6×10^{-3}	101	.25
0-2	10,000	.00203	3,732	1.2×10^{-3}	97	.25
0-1	10,000	.00435	3,552	1.0×10^{-3}	102	.25
0-2	10,000	.00203	3,552	$.8 \times 10^{-3}$	103	.25
0-1	10,000	.00435	3,192	$.28 \times 10^{-3}$	94	.25
0-2	10,000	.00203	3,192	$.24 \times 10^{-3}$	93	.25
0-6	15,000	.00039	3,732	$.94 \times 10^{-3}$	107	.25
0-7	15,000	.00018	3,732	$.71 \times 10^{-3}$	98	.25
0-6	15,000	.00039	3,552	$.501 \times 10^{-3}$	95	.25
0-7	15,000	.00018	3,552	$.46 \times 10^{-3}$	102	.25
0-6	15,000	.00039	3,462	$.43 \times 10^{-3}$	103	.25
0-7	15,000	.00018	3,462	$.33 \times 10^{-3}$	100	.25
0-9	5,000	.0039	3,372	$.61 \times 10^{-3}$	100	.25
0-11	5,000	.0018	3,232	$.26 \times 10^{-3}$	98	.25

Table 4-4. Experimental Data and Results of Oxidation Tests

RADIATION AND ABLATION TESTS.

To simulate the relative performance of "THERMO-LAG" T-500 under purely radiative heat loads, a series of tests were proposed. The test matrix is given in Table 4-5. The first series of these tests designed to complement the plasma-arc test series at heat fluxes of 7.4 and 9 BTU/FT²-SEC were performed. These radiant heat fluxes are within the scope of the aft body sidewall heat fluxes of a superorbital manned spacecraft. The results of the tests are recorded in Figures 4-4 and 4-5.

The tests to simulate the radiative mode of the performance of "THERMO-LAG" T-500 eliminates its transpiration cooling phenomenon. The heat blockage is a function of re-radiation, sensible heating of gases in the debris layer, chemical reactions in the debris layer, and latent heat of sublimation of the compound.

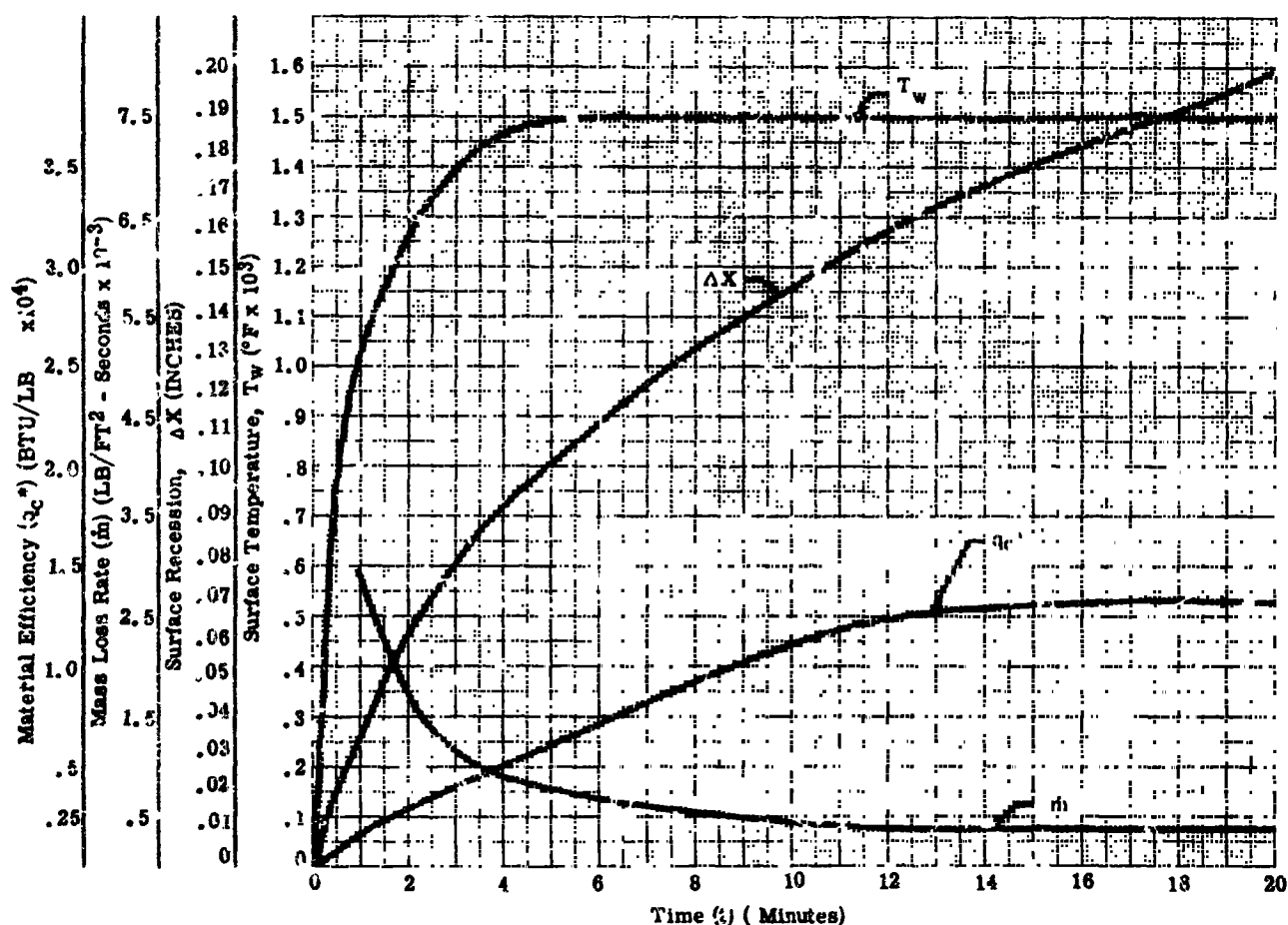


Figure 4-4. Composition - Radiation Ablation for Radiation Heat Flux of 7.4 BTU/FT²-SEC

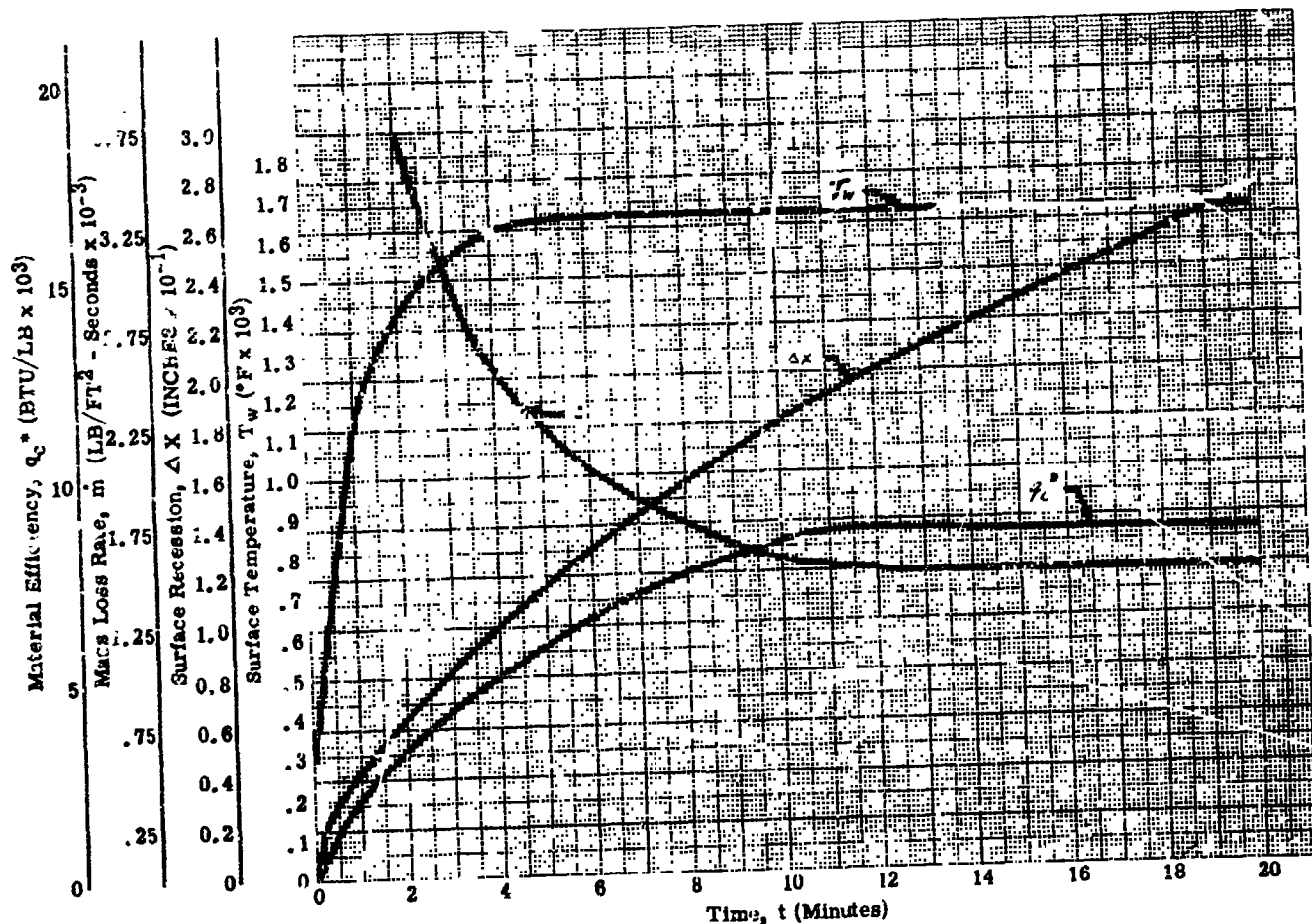


Figure 4-5. Composition - Radiation Ablation for Radiation Heat Flux of 9 BTU/FT²-SEC

Number of Tests	Sample Thickness (IN.)	Cold Wall Heat Flux (BTU/FT ² -SEC)
3	0.31	1
3	0.31	5
3	0.31	10
3	0.51	15
3	0.51	20
3	0.51	25
3	0.51	30
3	0.76	35
3	0.76	40
3	0.76	45
3	0.76	50

Table 4-5. Test Matrix - Ablation Radiation

AIR-ARC FLIGHT SIMULATION TESTS.

Flight simulation tests were conducted for selected portions of Apollo type re-entry trajectories. The test points are illustrated in Figure 4-6. The test matrix containing conditions, durations of test runs, and model geometry are recorded in Tables 4-6 through 4-8.

Figures 4-7 through 4-15 illustrate some results of reduced data of these tests. The thermal performance of "THERMO-LAG" T-500 at the stagnation region under transient conditions is presented in Figures 4-7 through 4-12 for a representative number of models tested. The sidewall transient histories for some "THERMO-LAG" models are presented in Figure 4-13.

Heat blockage, based upon model hot wall heat flux, is presented in Figure 4-14 for a representative number of models tested. The results presented in the figure are indicative of material performance under steady-state ablation. Test results indicated by triangles are the results of about ten models tested at the same conditions, while results indicated by circles represent a single model tested. Figure 4-14 was used to evaluate the transpiration cooling factor, $\alpha = 0.6$, for "THERMO-LAG" T-500.

Figure 4-15 indicates the reduction in stagnation point heat transfer coefficient due to mass injection that was determined from the tests. Since the results were evaluated from stagnation point data, they are taken to be applicable to laminar flow only. The theoretical relationship for laminar-turbulent transpiration cooling factor was used for the curve representing the heat transfer coefficient reduction for a turbulent boundary layer.

A summarization of all performed air-arc flight simulation tests of "THERMO-LAG" T-500 models is recorded in Table 4-9 and is illustrated in Figure 4-16 through 4-92. These figures present the non-reduced air-arc plasmajet substrate temperature-time history data for the model sidewalls. The stagnation temperature of the model for the test duration is plotted in the upper right-hand corner of some figures. Recorded in Table 4-9 is the conducted test matrix containing test conditions, model geometry, durations of test runs, measured material recession, cold and hot wall heat blockage, and sidewall material history. The test matrices can be identified by the prefix number of the models tested. The M series are the low pressure tests. The O series are the oxygen concentration tests. The D, E, GG and HH series are the material investigation series. The N series, constituting the bulk of the performed tests, fulfill the required low pressure, oxidation, transient, and pressure effect test matrices.

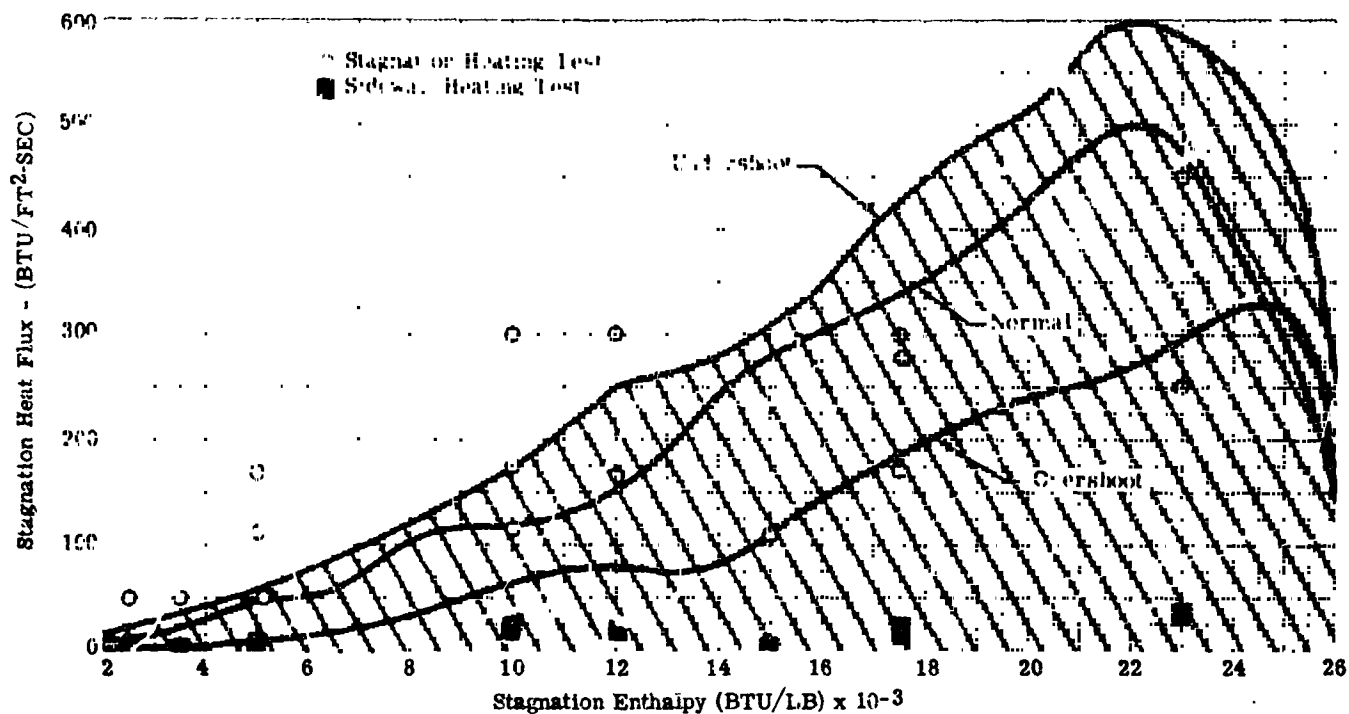


Figure 4-6. Flight Simulation Test Points for Apollo Type Re-Entry Trajectories

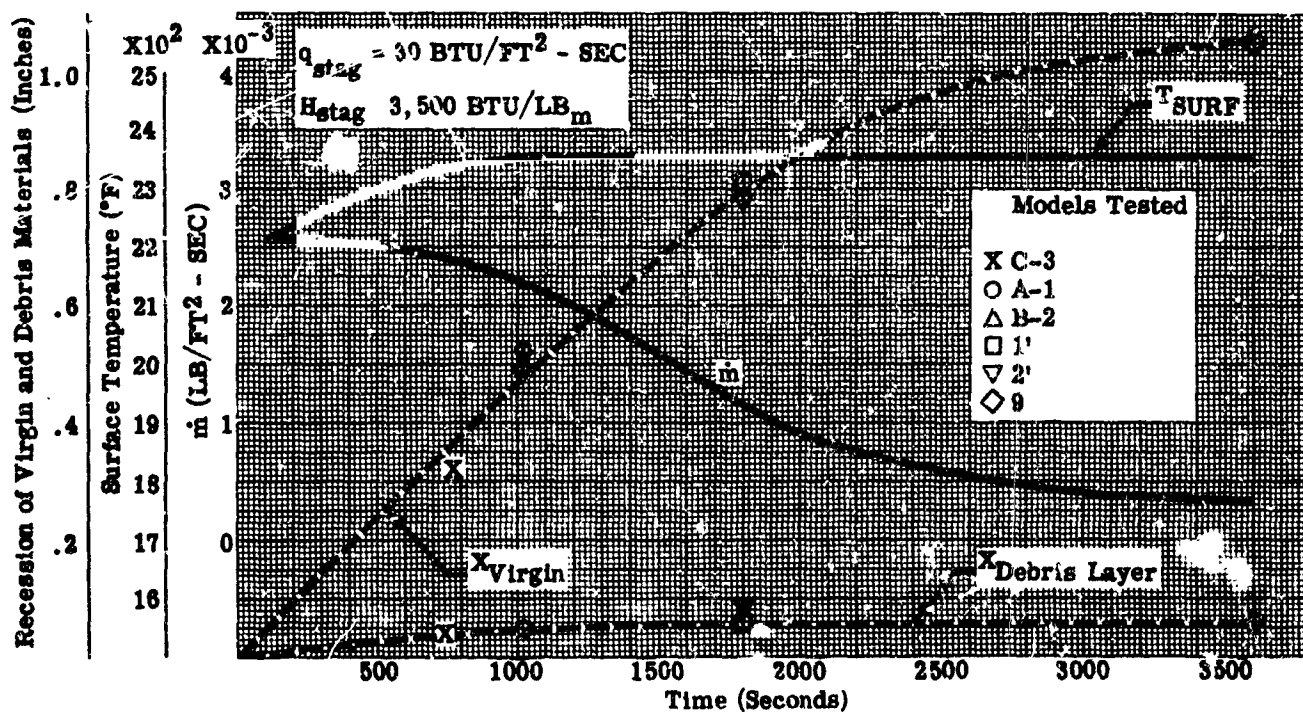


Figure 4-7. Transient Histories

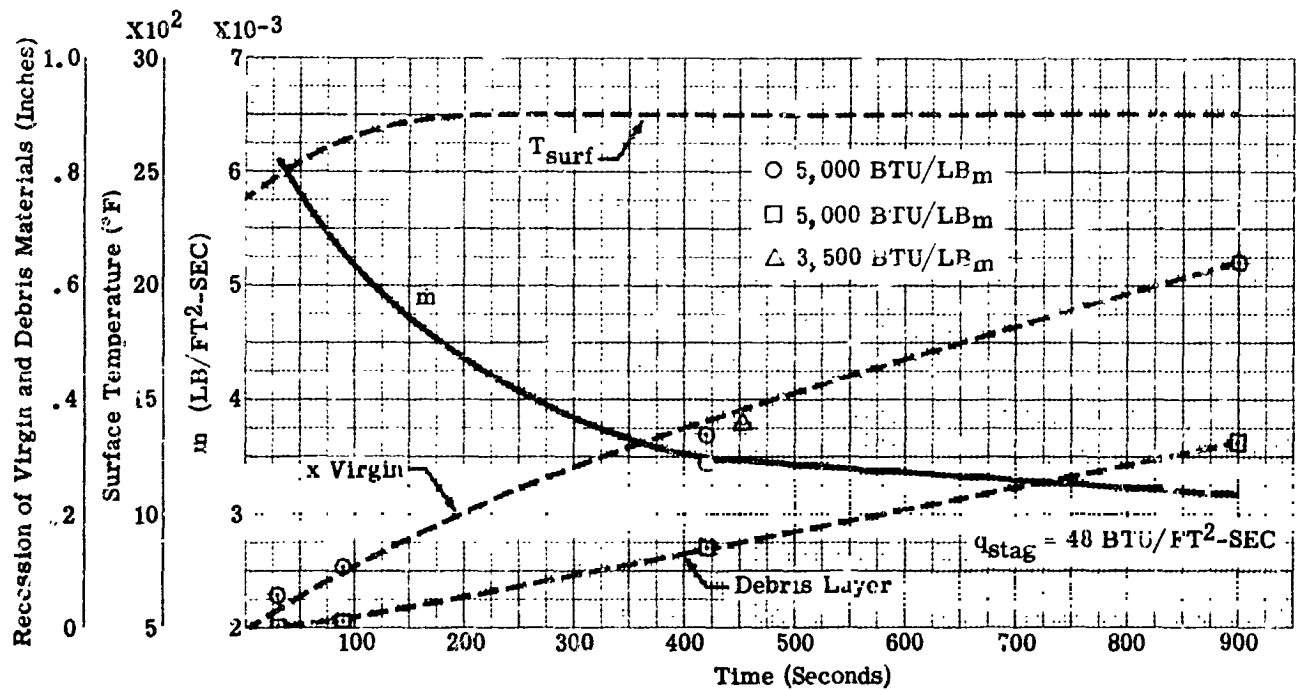


Figure 4-8. Transient Histories

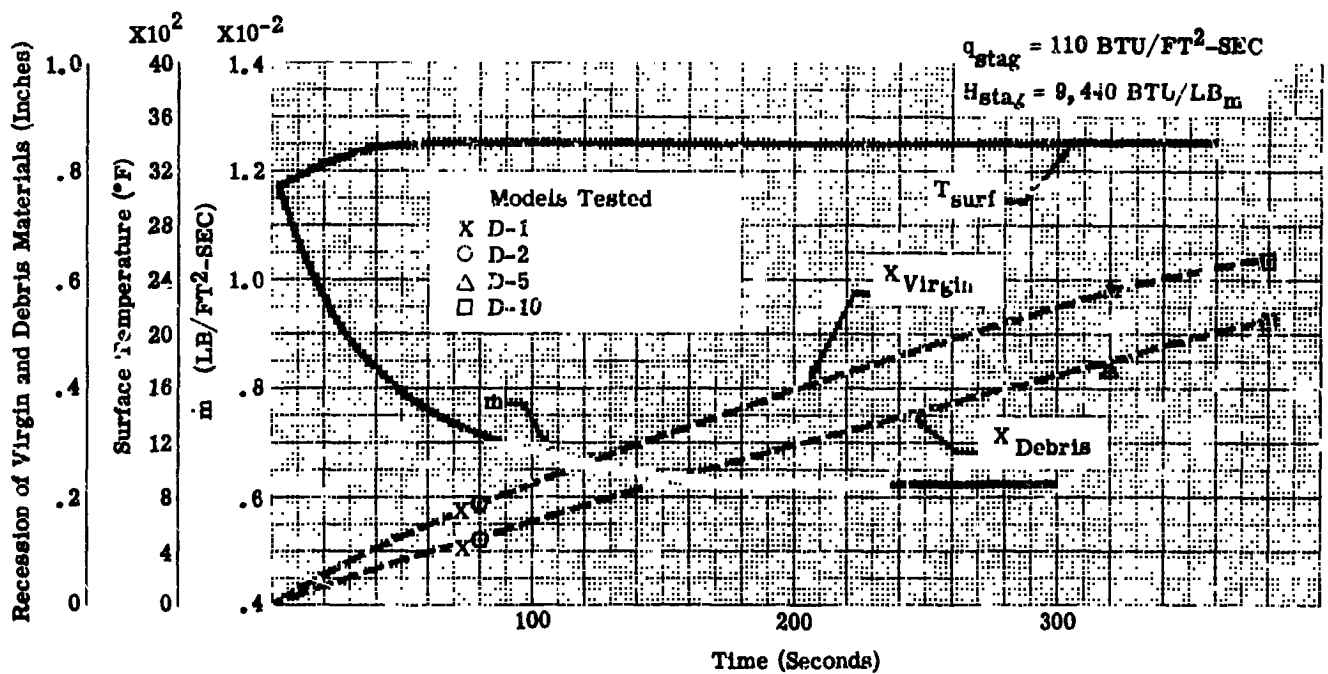


Figure 4-9. Transient Histories

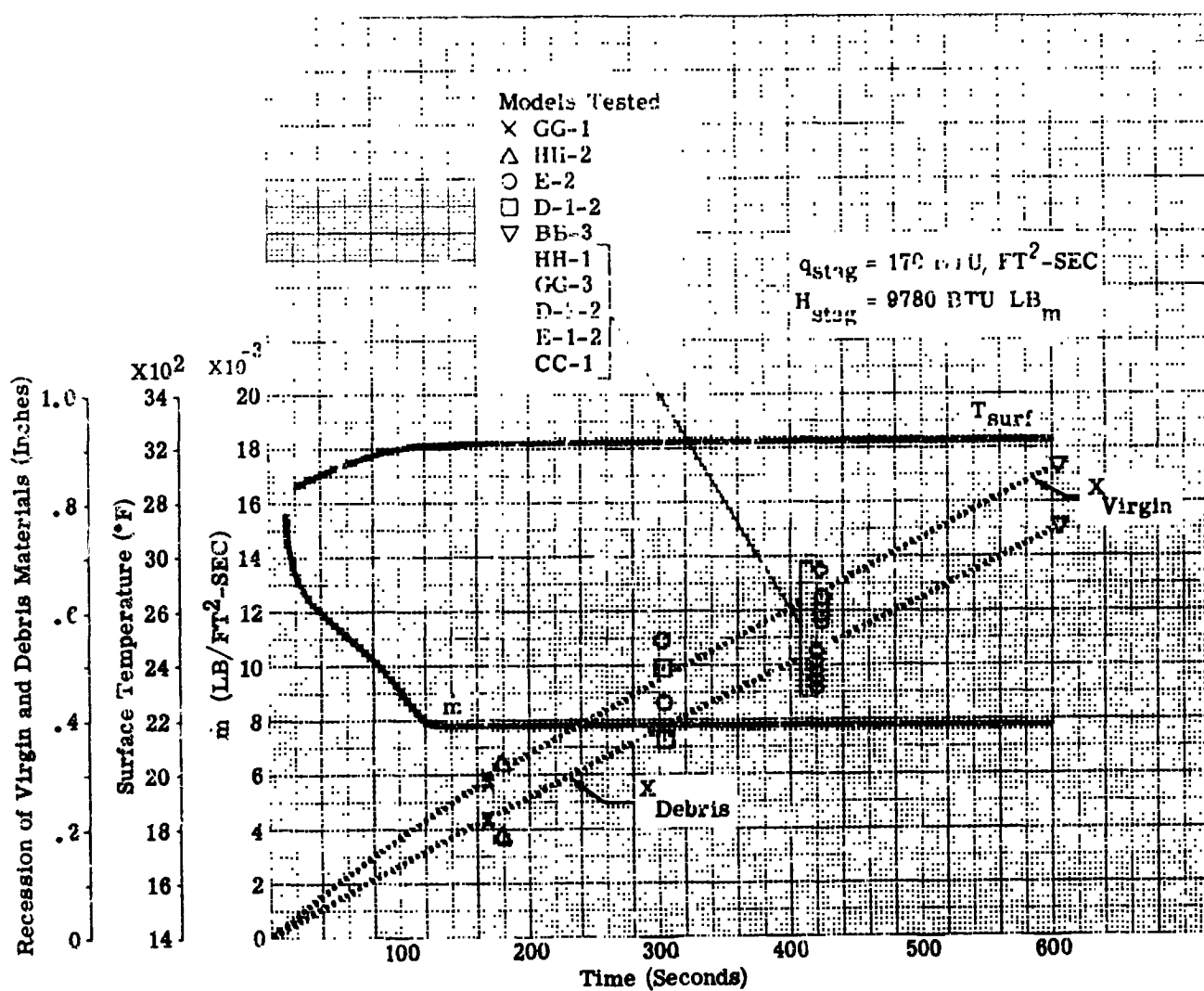


Figure 4-10. Transient Histories

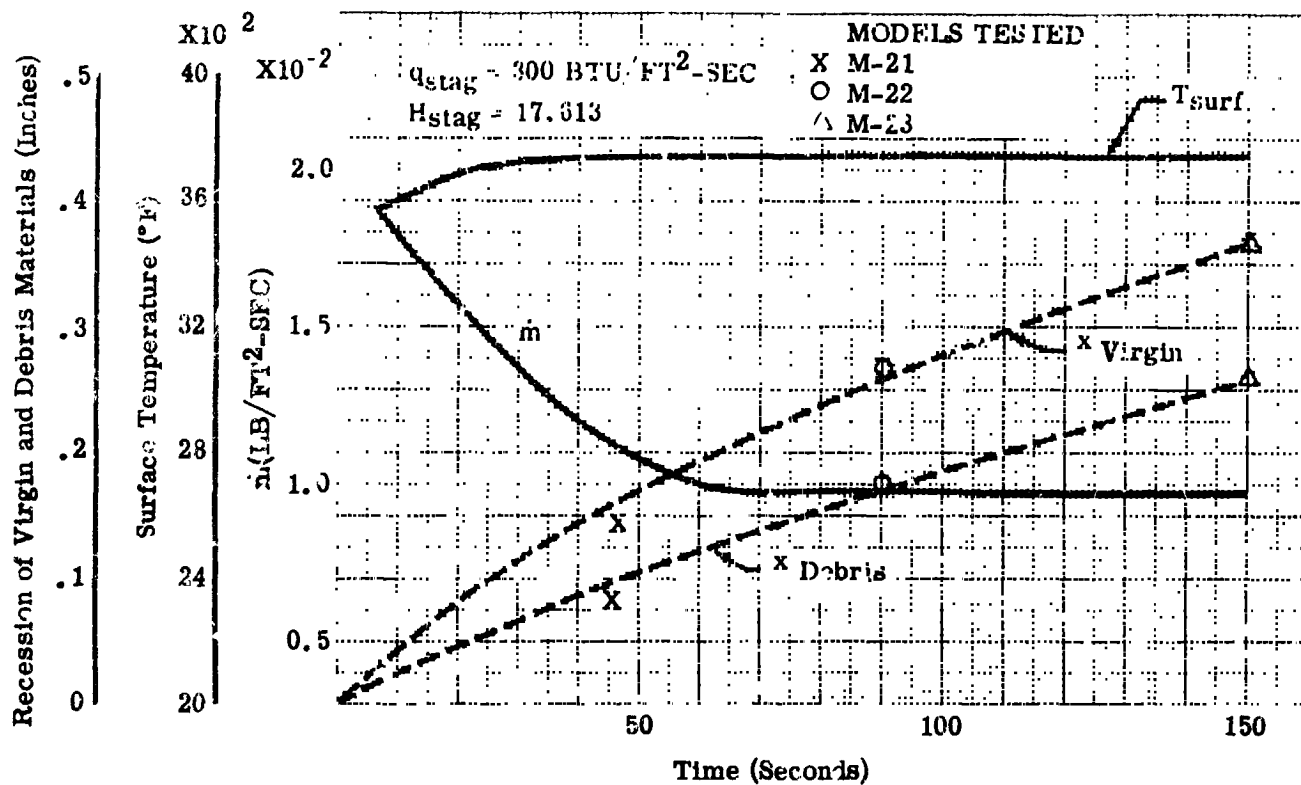


Figure 4-11. Transient Histories

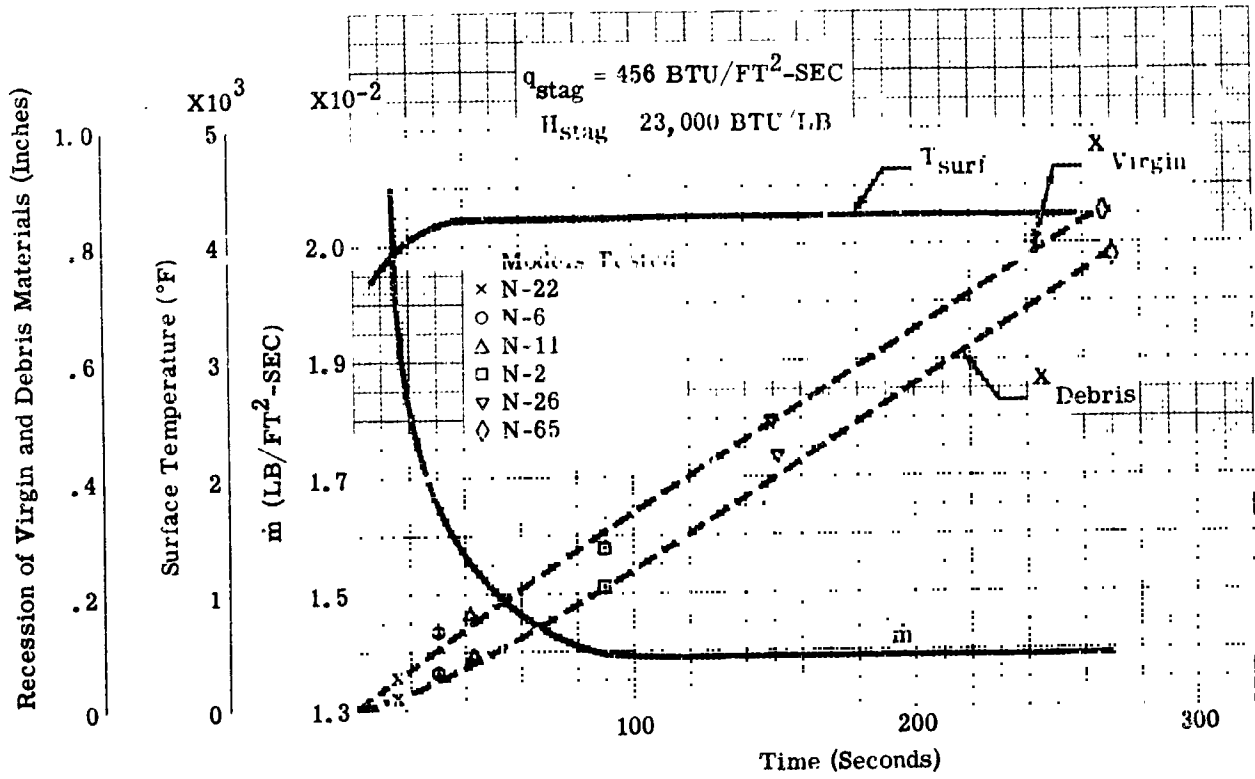


Figure 4-12. Transient Histories

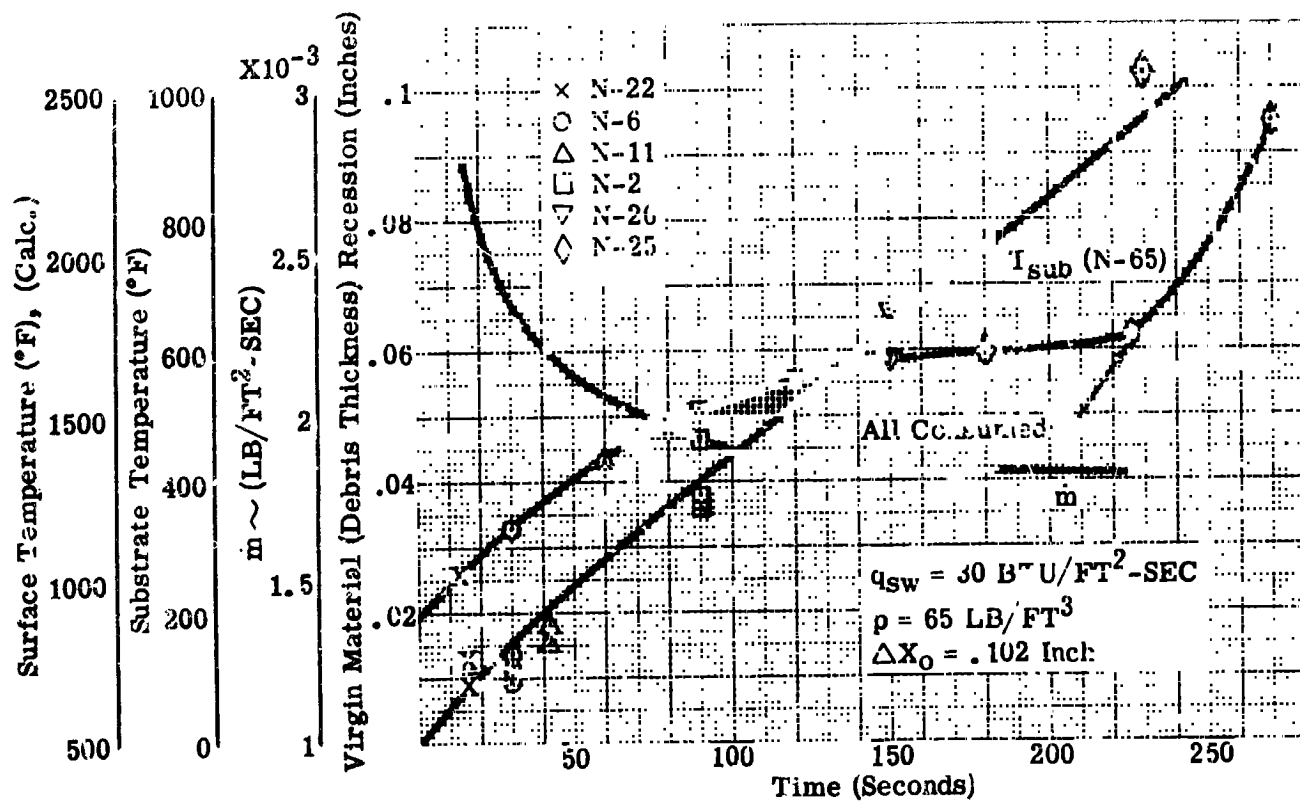


Figure 4-13. Sidewall Transient History

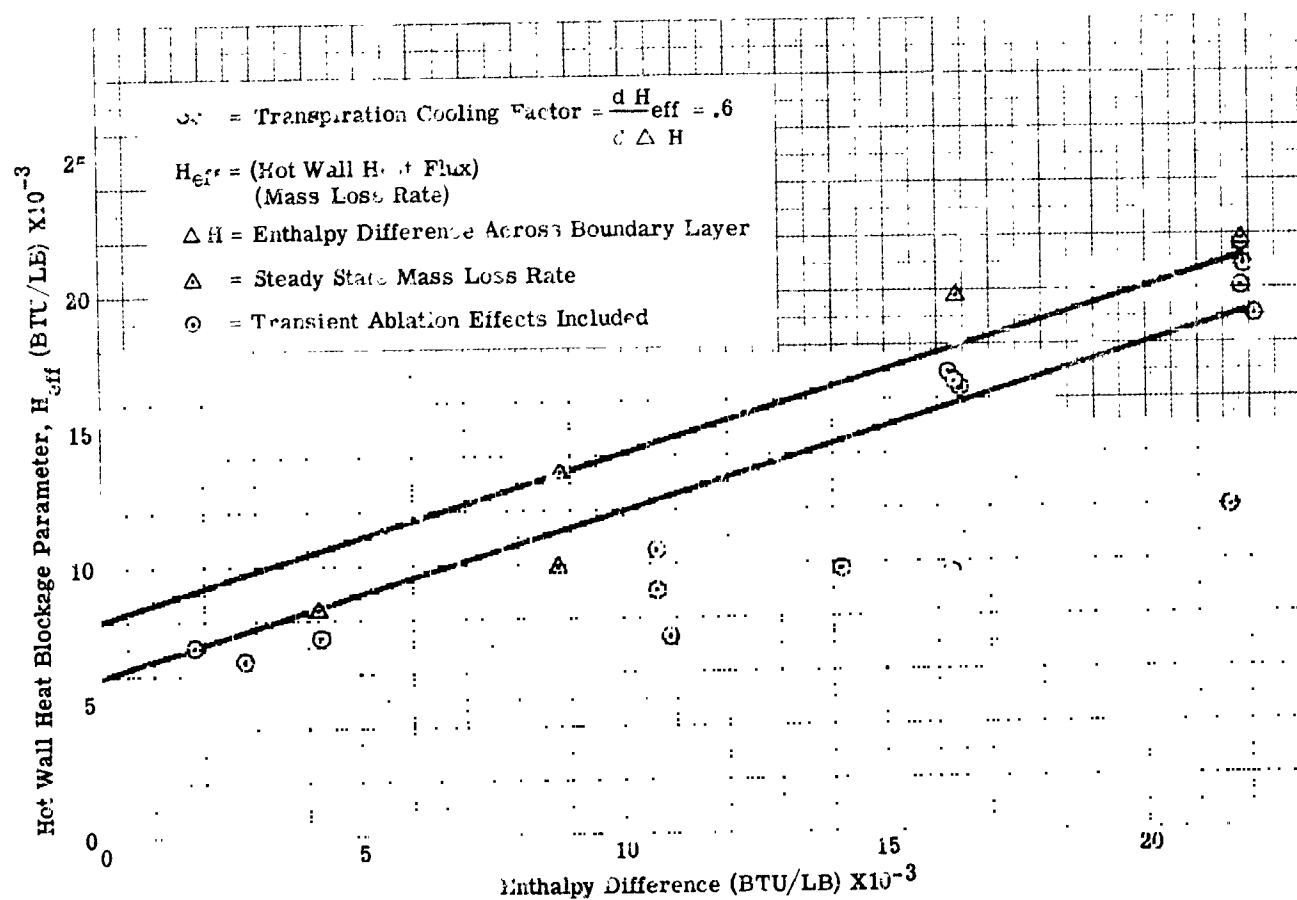


Figure 4-14. Stagnation Point Experimental Hot Wall Heat Blockage of "THERMO-LAG" T-500-7

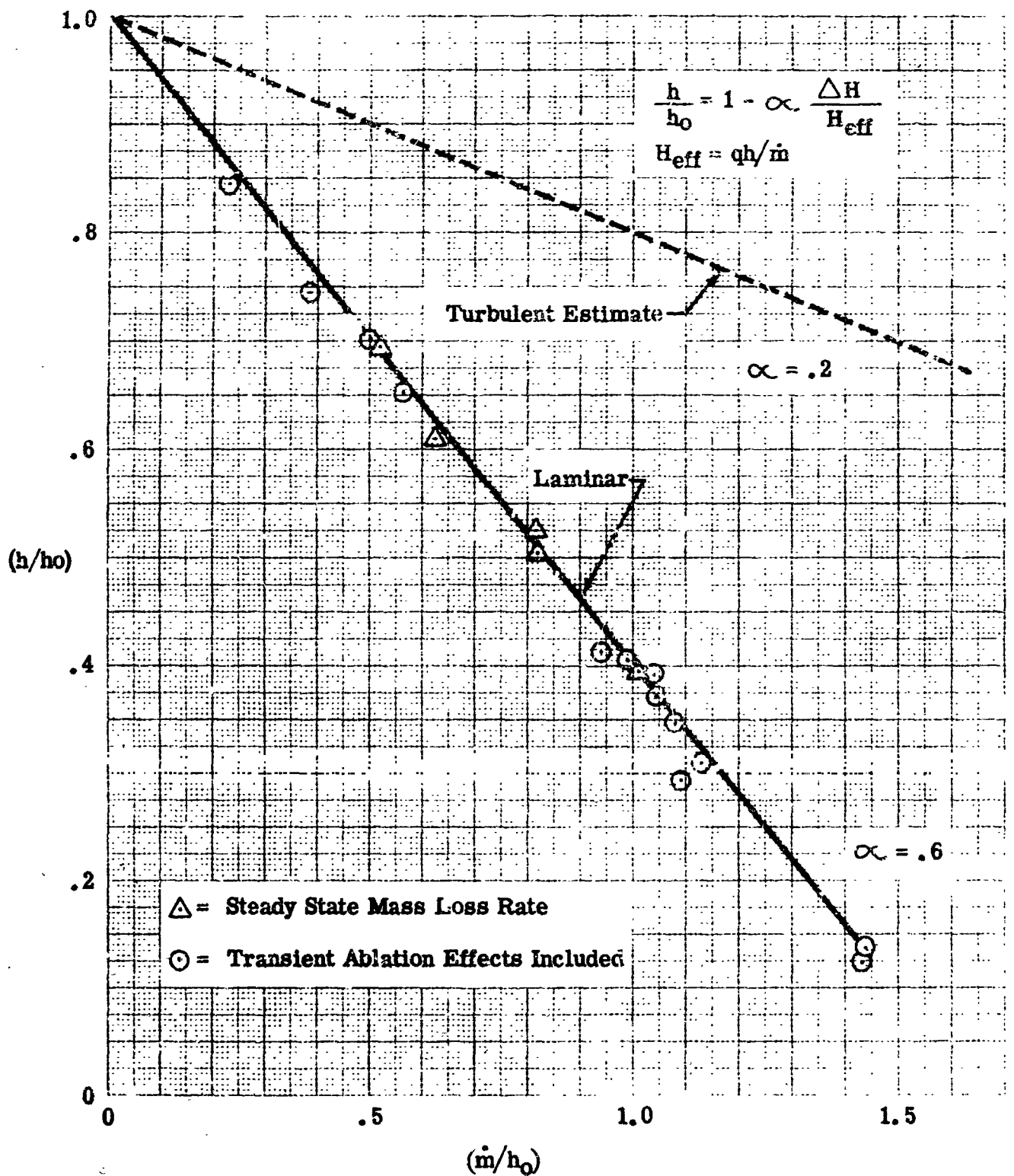


Figure 4-15. Stagnation Point Experimental Results of Reduced Heat Transfer Transfer Coefficient



Model Type and Series	Stagnation Pressure (ATM)	Stagnation Heat Flux (BTU/FT ² - SEC)	Desired Sidewall Heat Flux (BTU/FT ² - SEC)	Stagnation Enthalpy (BTU/LB)
1-1/4 IN. hc (N)	.024	300	24 - 36	12,000
1-1/4 IN. hc	.006	165	13.6 - 20	12,000
1-1/4 IN. hc	.024	165	13.6 - 20	12,000
2 IN. hc	.005	170	13.6 - 20	17,500
2 IN. ff	.020	170	13.6 - 20	17,500
2 IN. hc	.020	310	25 - 37	17,500
2 IN. hc	.009	305	25 - 37	25,000
2 IN. ff	.030	305	25 - 37	25,000
2 IN. hc	.030	550	44 - 66	25,000

Table 4-6. Test Matrix - Evaluation of Pressure Effect on Stagnation Point Ablation Kinetics

Model Type and Series	Stagnation Pressure (ATM)	Stagnation Heat Flux (BTU/FT ² - SEC)	Desired Sidewall Heat Flux (BTU/FT ² - SEC)	Stagnation Enthalpy (BTU/LB)
2 IN. hc	.03	50	4-6	2,500
2 IN. hc	.015	50	4-6	3,500
2 IN. hc	.007	50	4-6	5,000
2 IN. hc	.03	110	8.8-12.8	5,000
2 IN. hc	.007	110	8.8-12.8	10,000
2 IN. hc	.003	110	8.8-12.8	15,000
2 IN. hc	.06	170	13.6-19.2	5,000
2 IN. hc	.016	170	13.6-19.2	10,000
2 IN. hc	.005	170	13.6-19.2	17,500
1-1/4 IN. hc	.03	300	24-36	10,000
1-1/4 IN. hc	.024	300	24-36	12,000
1-1/4 IN. hc	.01	300	24-36	17,500

Table 4-7. Test Matrix - Variation of Enthalpy and Pressure With Constant Heat Flux

Model Type and Series	Stagnation Pressure (ATM)	Stagnation Heat Flux (BTU/FT ² - SEC)	Sidewall Heat Flux BTU/FT ² - SEC)	Stagnation Enthalpy (BTU/LB)	Duration (SEC)
2 IN. hc (N)	.015	35	6	3,500	750
2 IN. hc	.015	35	6.5	3,500	1,020
2 IN. hc	.015	35	6.5	3,500	1,800
2 IN. hc	.015	35	6.5	3,500	3,600
2 IN. hc	.0076	48	8.6	5,000	30
2 IN. hc	.0076	48	8.6	5,000	90
2 IN. hc	.0076	48	8.6	5,000	420
2 IN. hc (GG)	.007	110	17.6	10,000	75
2 IN. hc	.007	110	17.6	10,500	45
2 IN. hc	.007	110	17.6	10,500	90
2 IN. hc	.007	110	17.6	10,500	250
2 IN. hc	.016	170	23.8	10,500	170
2 IN. hc	.016	170	23.8	10,500	300
2 IN. hc	.016	170	23.8	10,500	420
1-1/4 IN. hc (M)	.01	300	30	17,500	15
1-1/4 IN. hc	.01	300	30	17,500	45
1-1/4 IN. hc	.01	300	30	17,500	90
1-1/4 IN. hc	.01	300	30	17,500	250
2 IN. hc (N)	.027	456	36.3	23,000	15
2 IN. hc	.027	456	36.3	23,000	30
2 IN. hc	.027	456	36.3	23,000	42
2 IN. hc	.027	456	36.3	23,000	90
2 IN. hc	.027	456	36.3	23,000	150
2 IN. hc	.027	456	36.3	23,000	270

Table 4-8. Transient Test Matrix

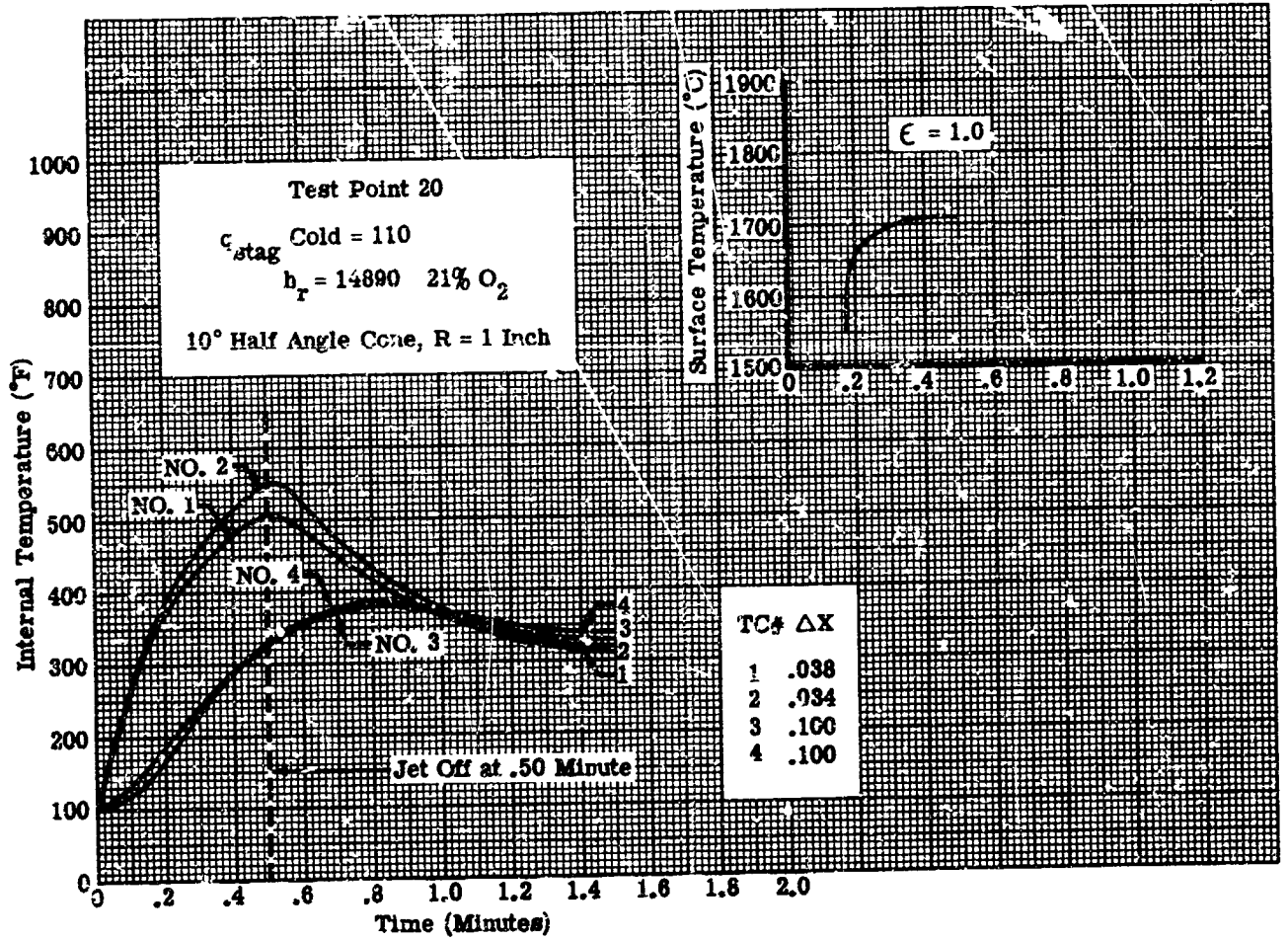


Figure 4-16. Temperature-Time History of Model M-6

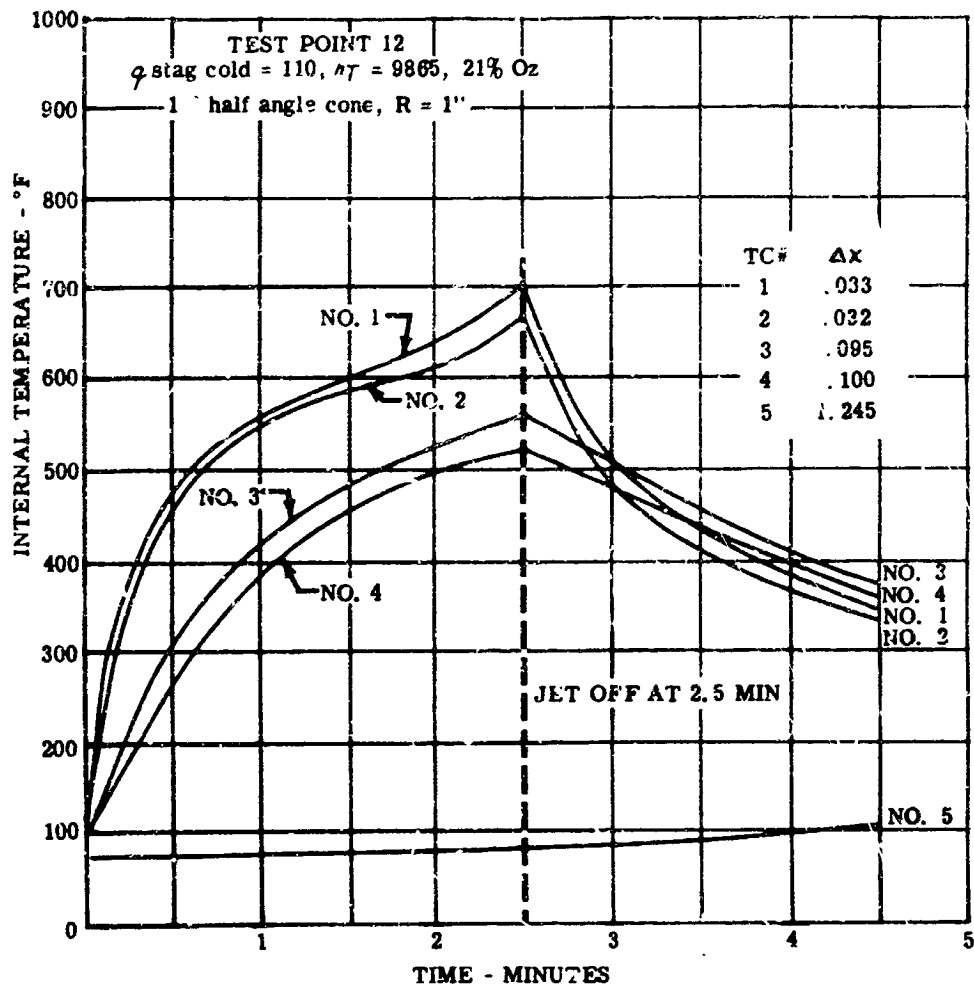


Figure 4-17. Temperature-Time History of Model M-9

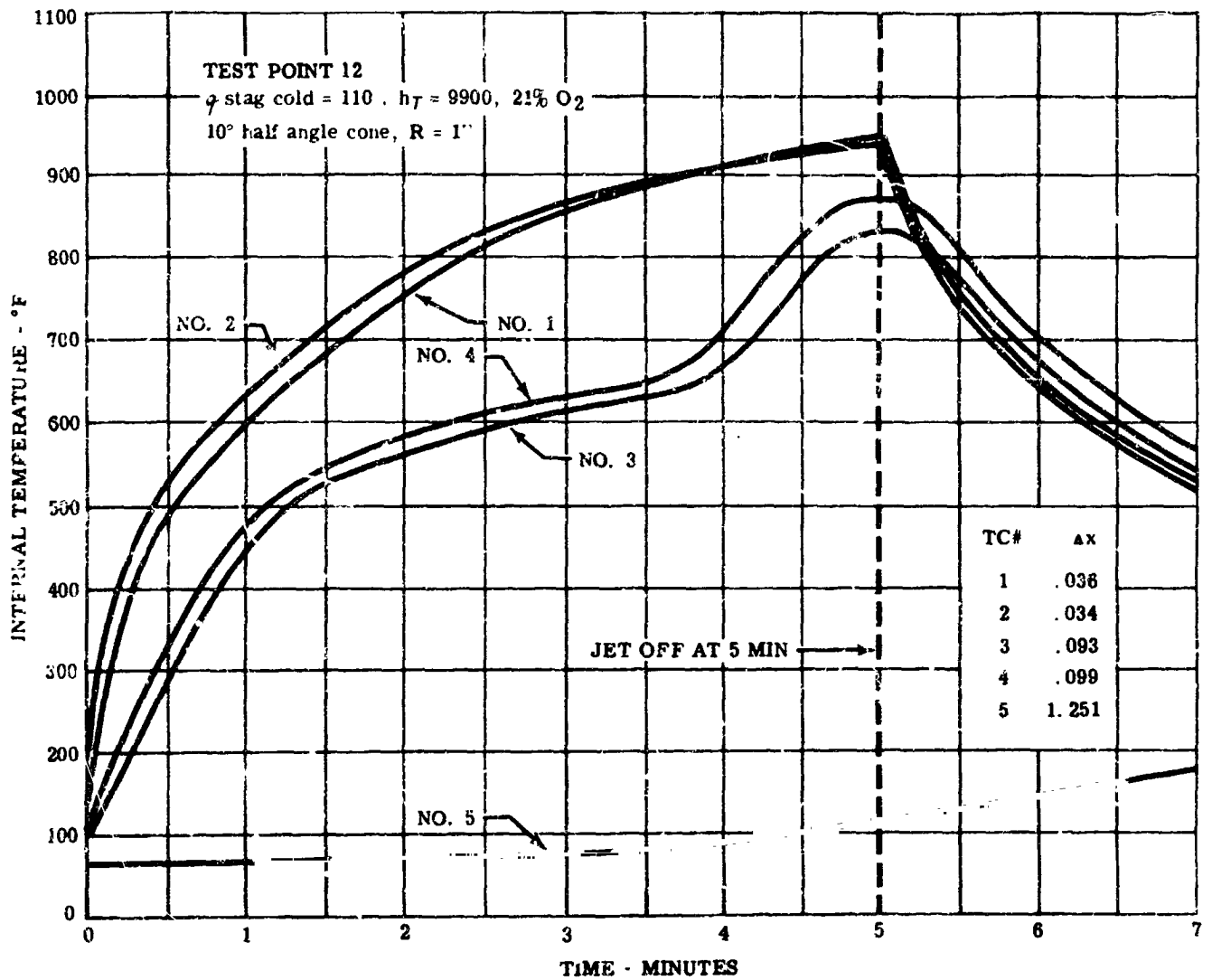


Figure 4-18. Temperature-Time History of Model M-10

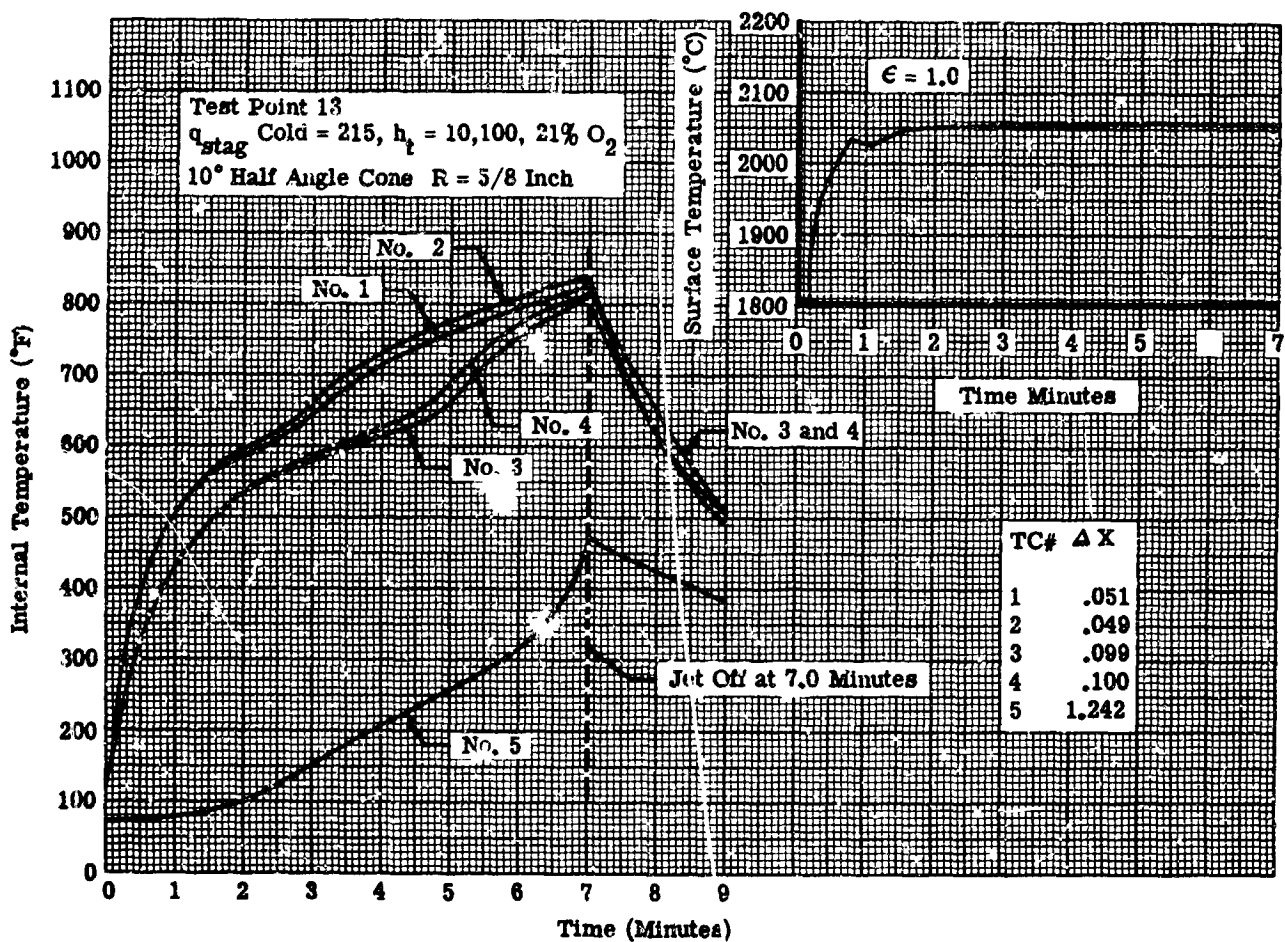


Figure 4-19. Temperature-Time History of Model M-18

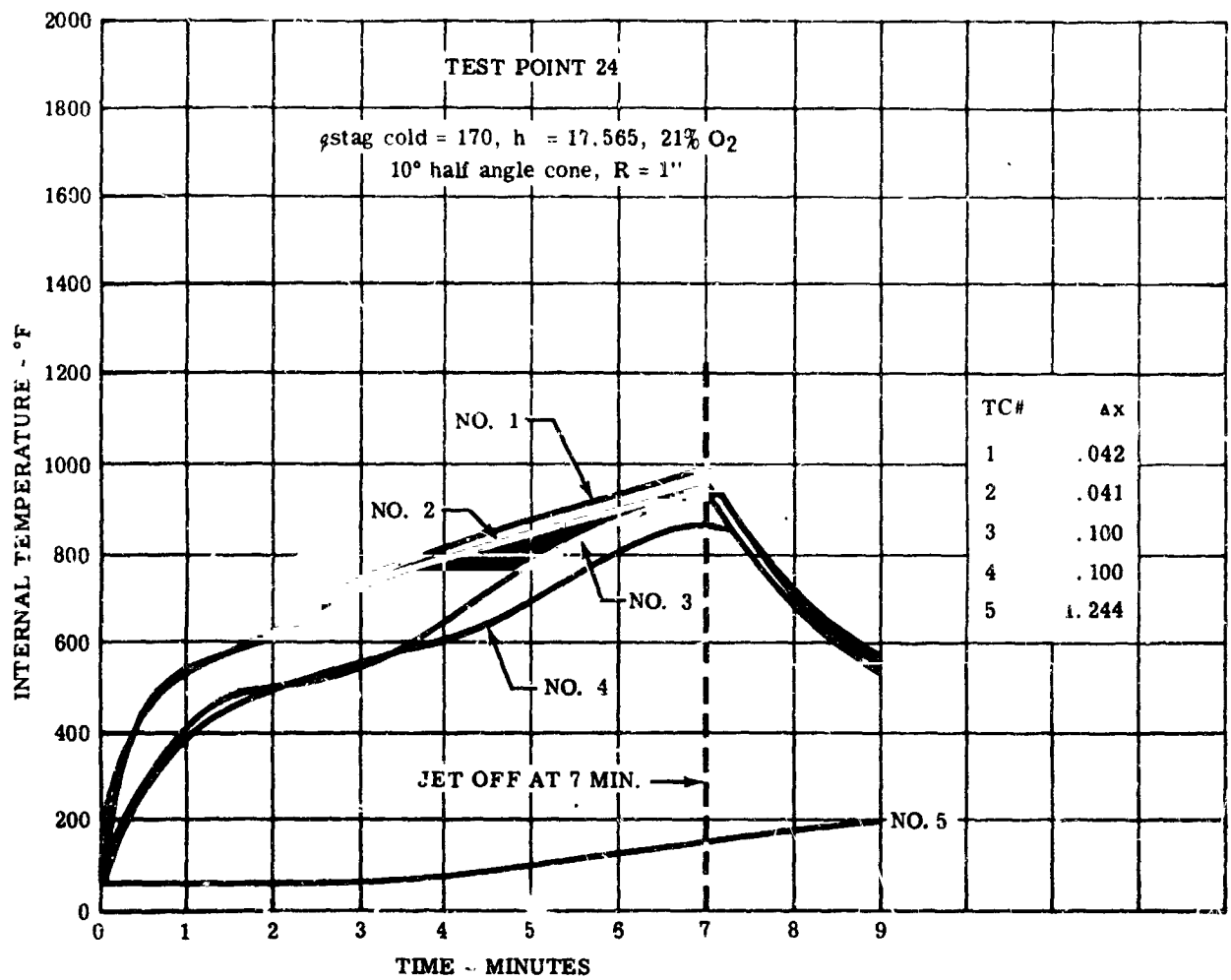


Figure 4-20. Temperature-Time History of Model M-16

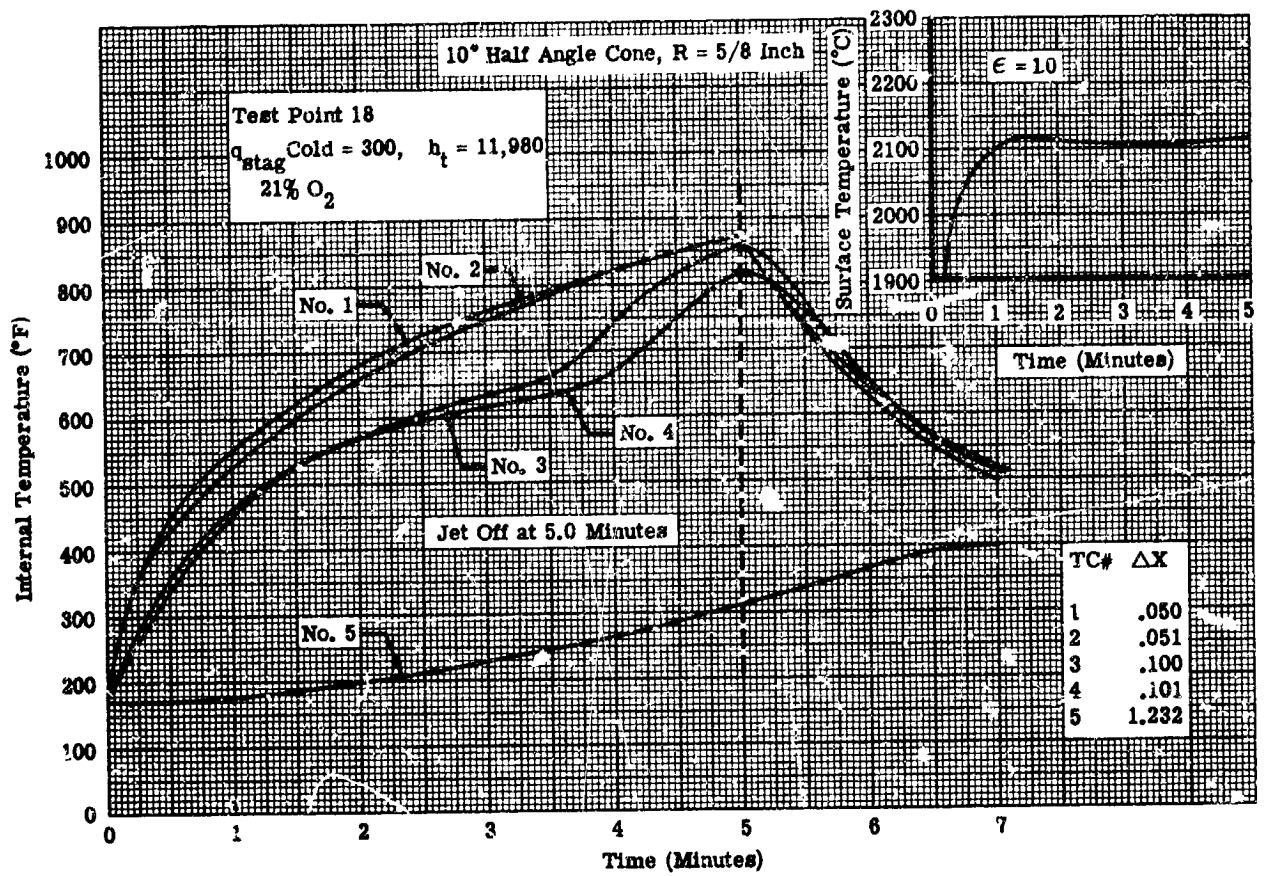


Figure 4-21. Temperature-Time History of Model M-19

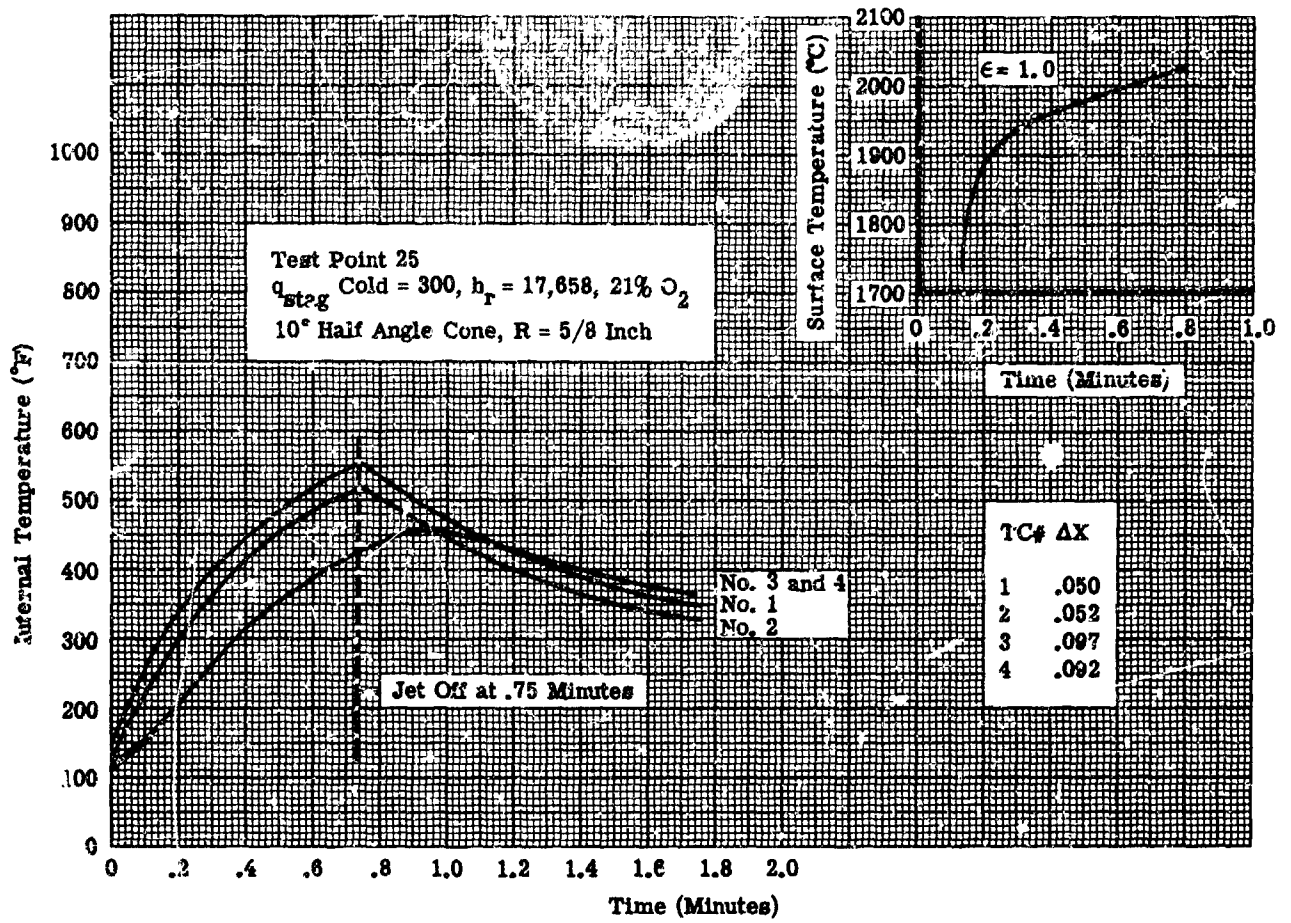


Figure 4-22. Temperature-Time History of Model M-21

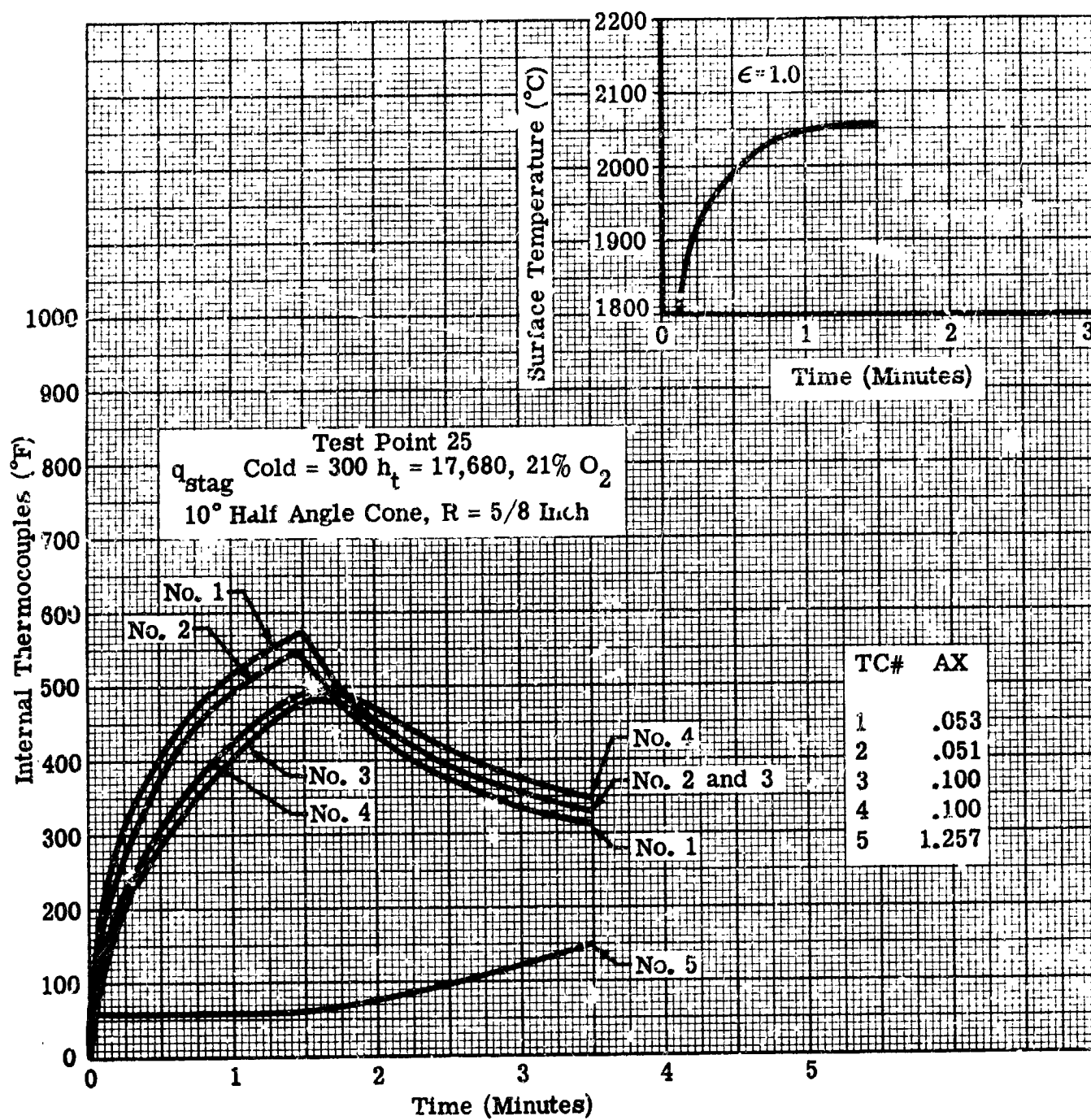


Figure 4-23. Temperature-Time History of Model M-22

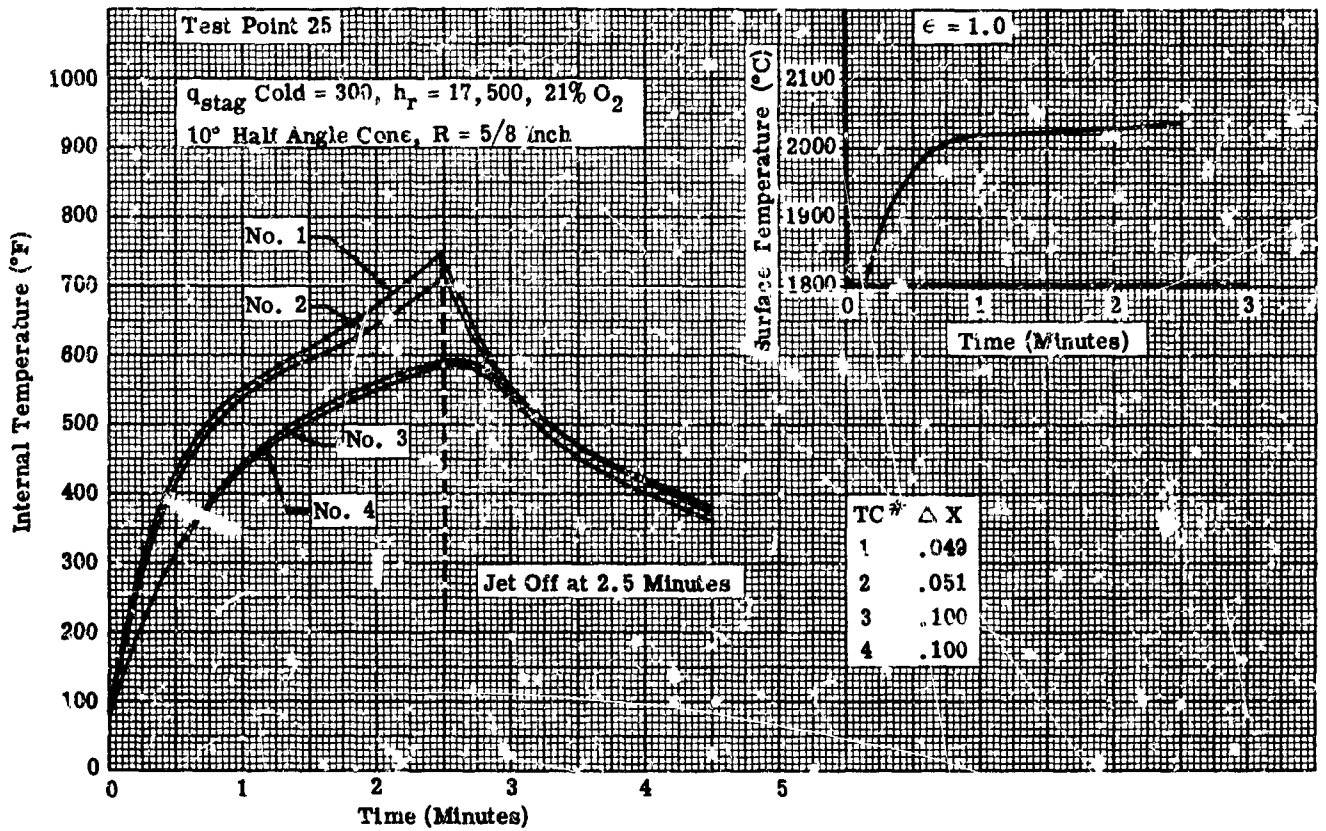


Figure 4-24. Temperature-Time History of Model M-23

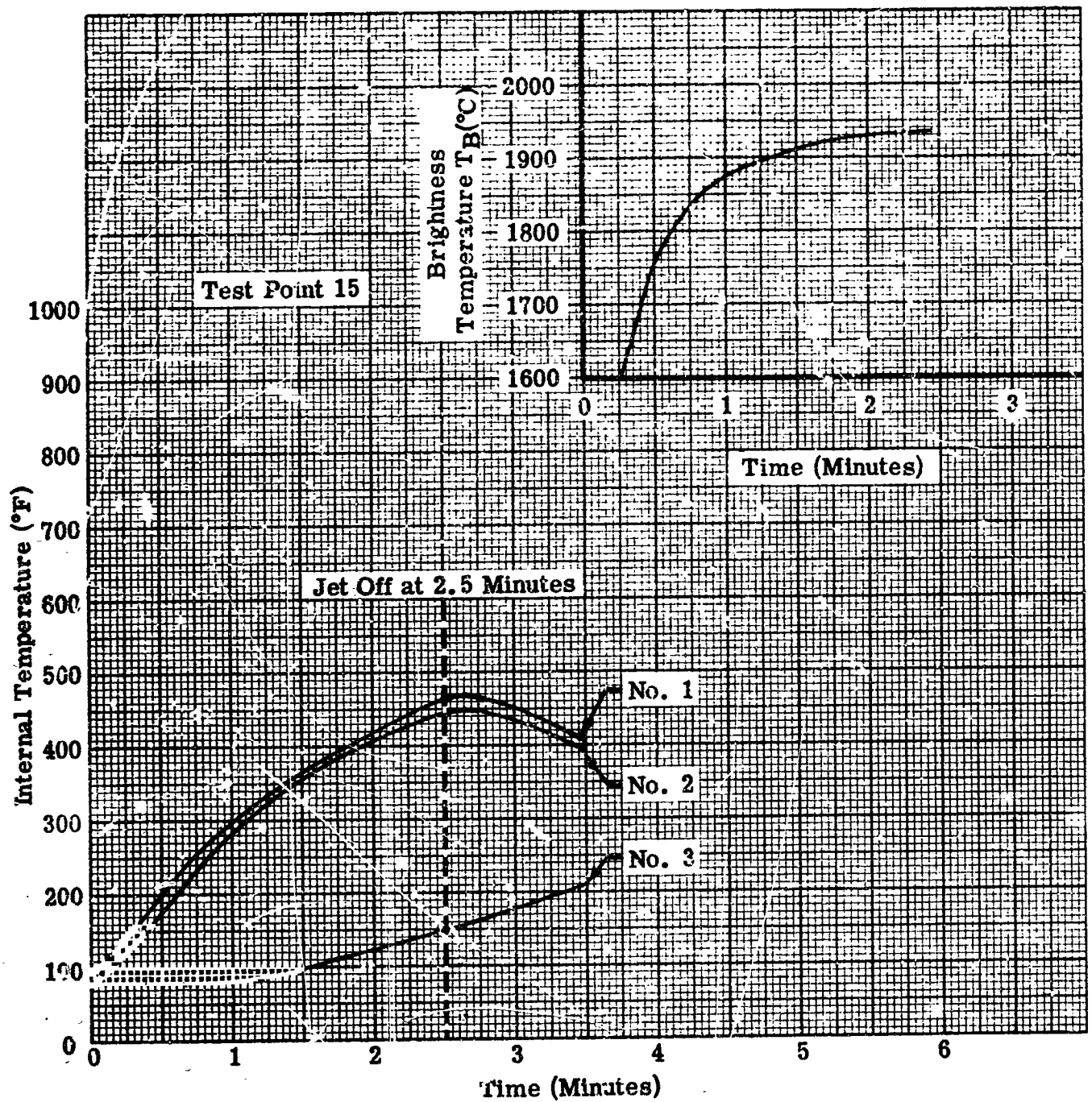


Figure 4-25. Temperature-Time History of Model O-1

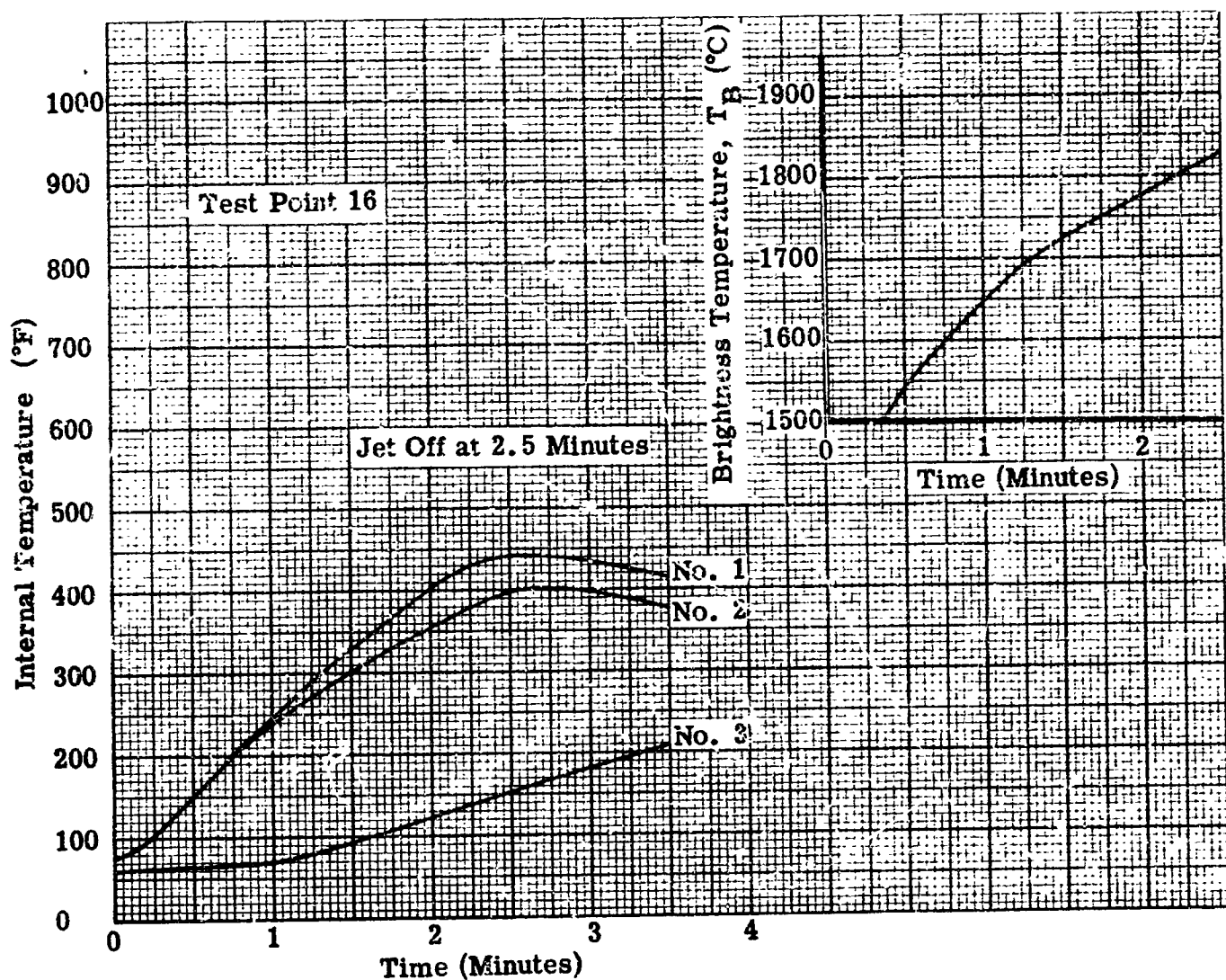


Figure 4-26. Temperature-Time History of Model O-2

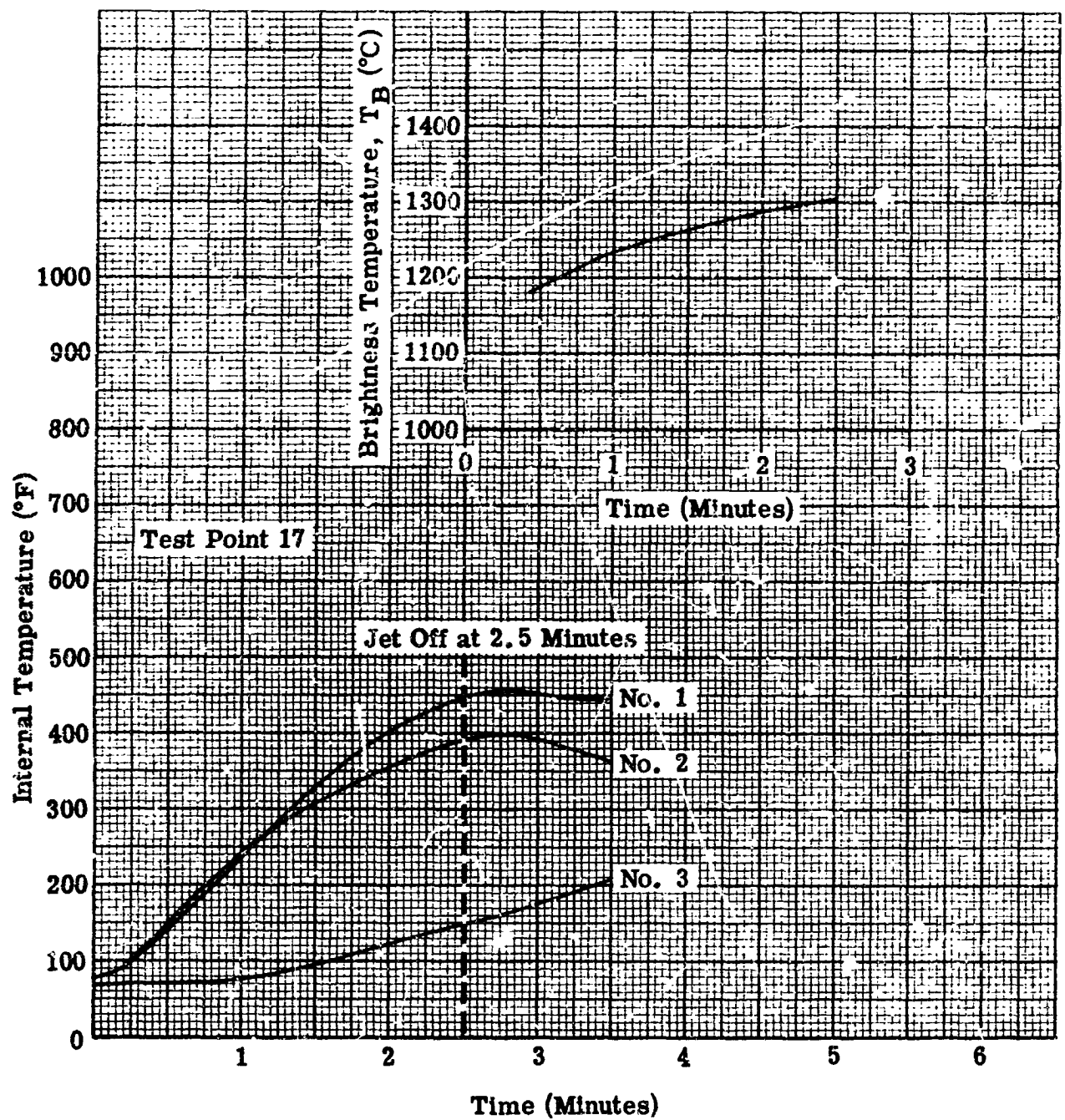


Figure 4-27. Temperature-Time History of Model O-3

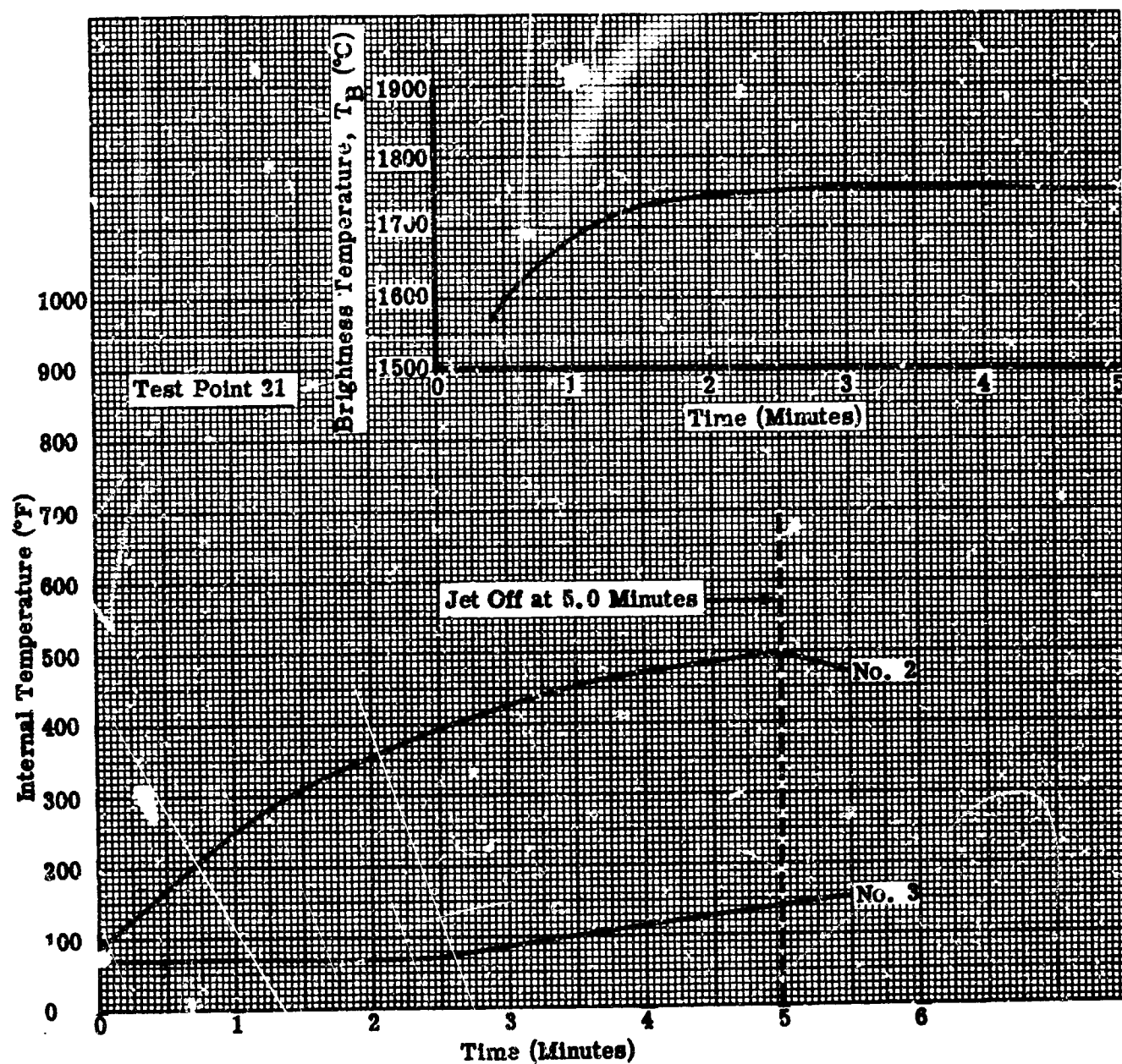


Figure 4-28. Temperature-Time History of Model Q-6

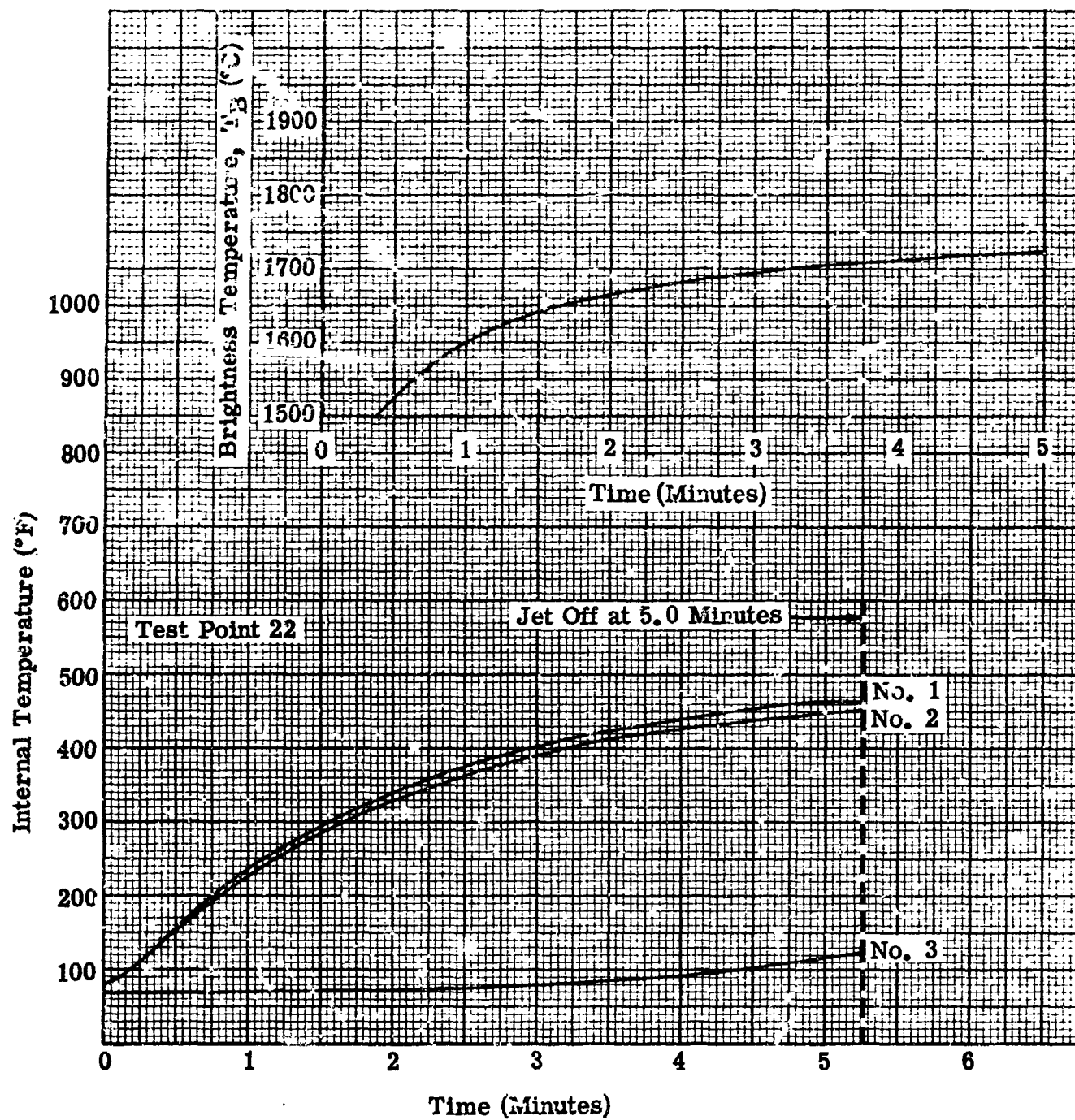


Figure 4-29. Temperature-Time History of Model O-7

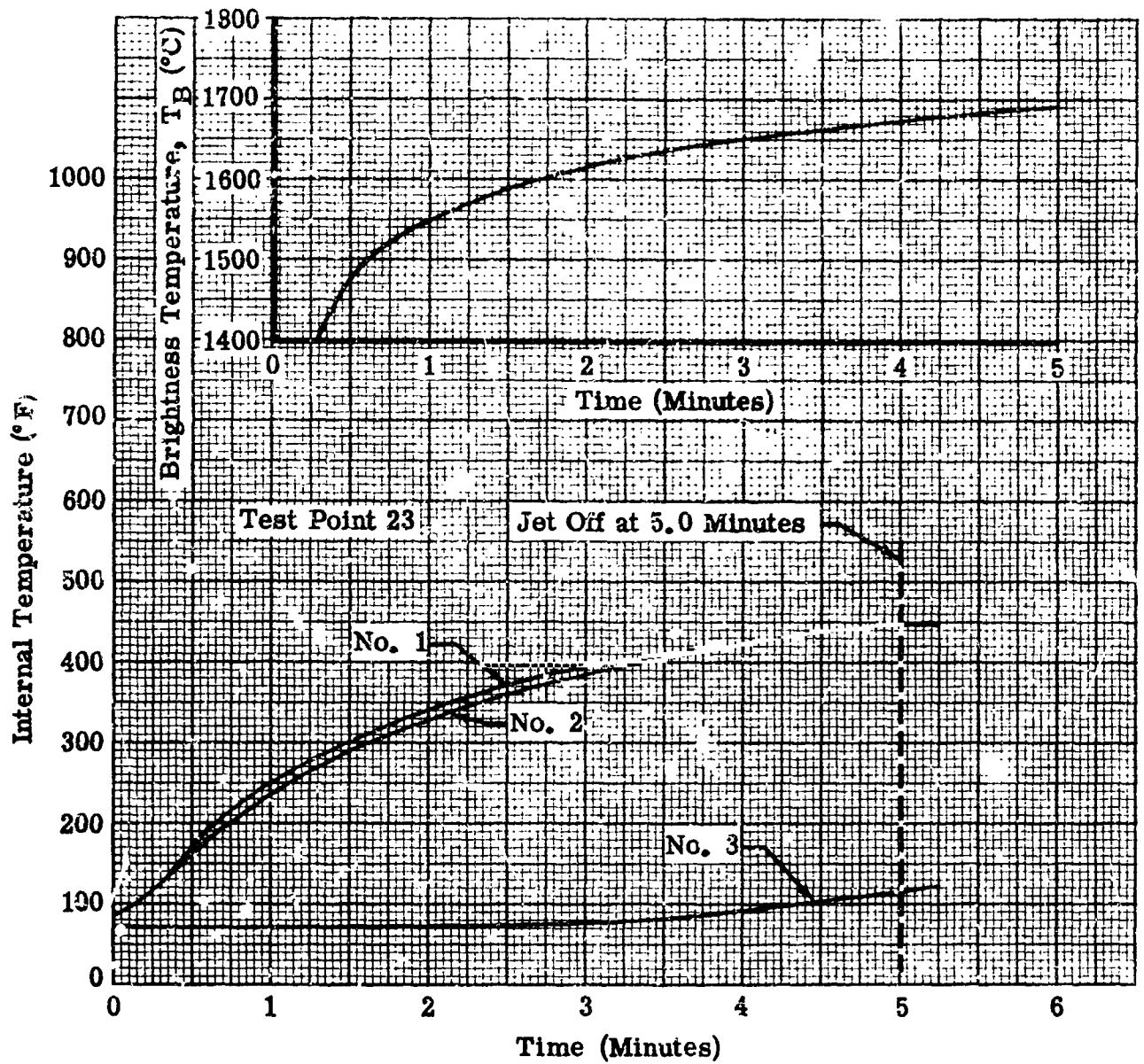


Figure 4-30. Temperature-Time History of Model O-8

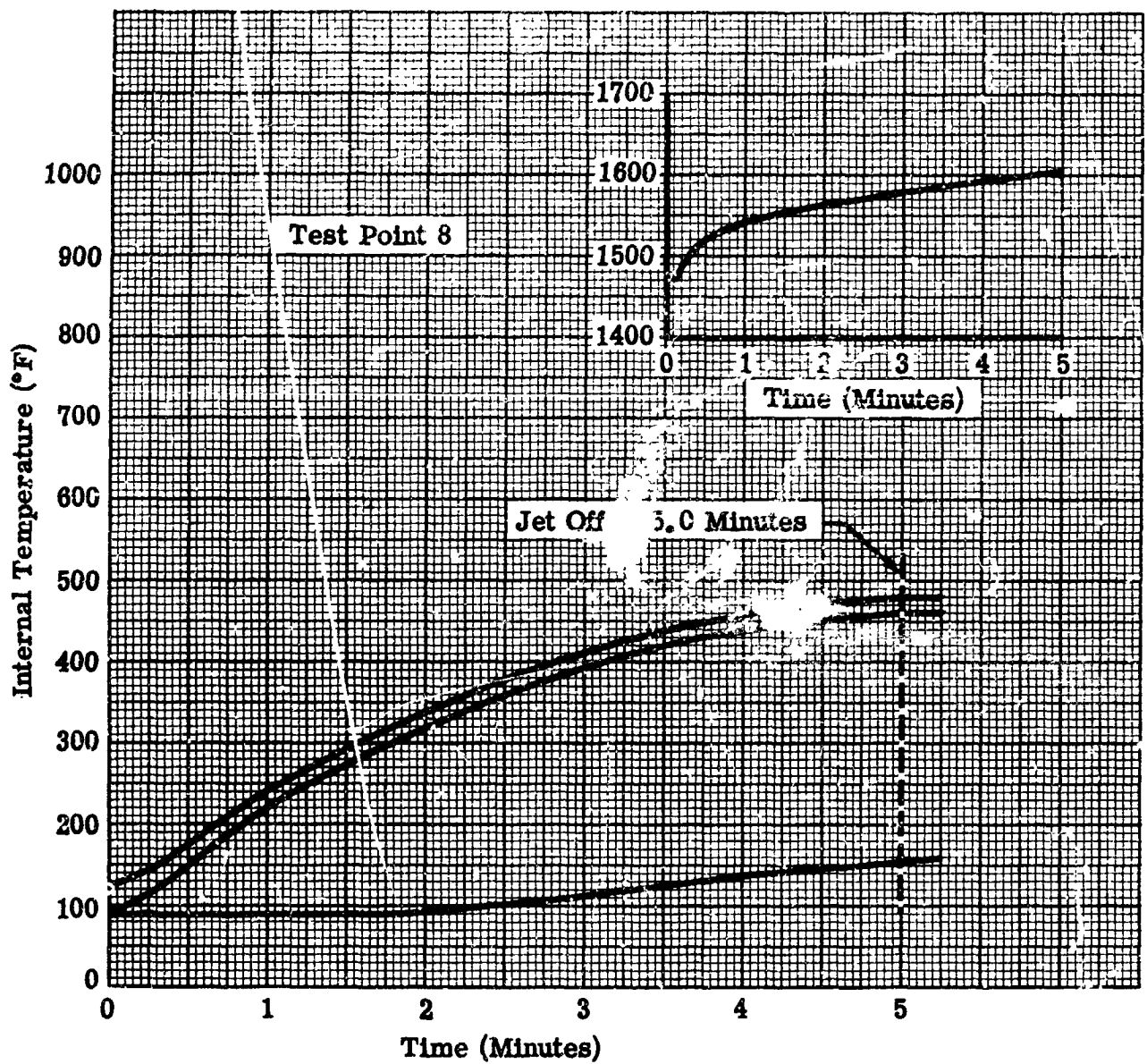


Figure 4-31. Temperature-Time History of Model O-9

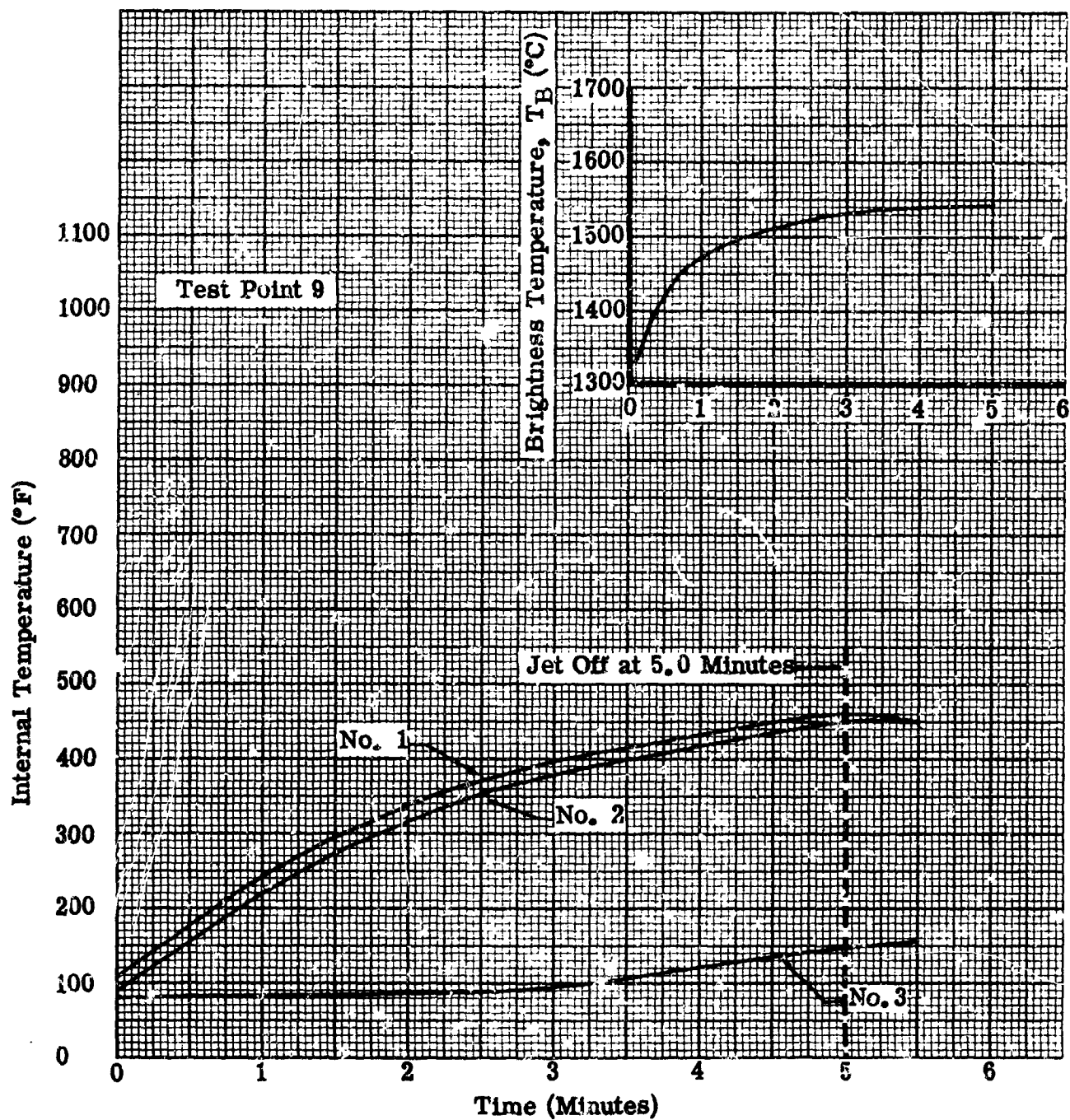


Figure 4-32. Temperature-Time History of Model O-11

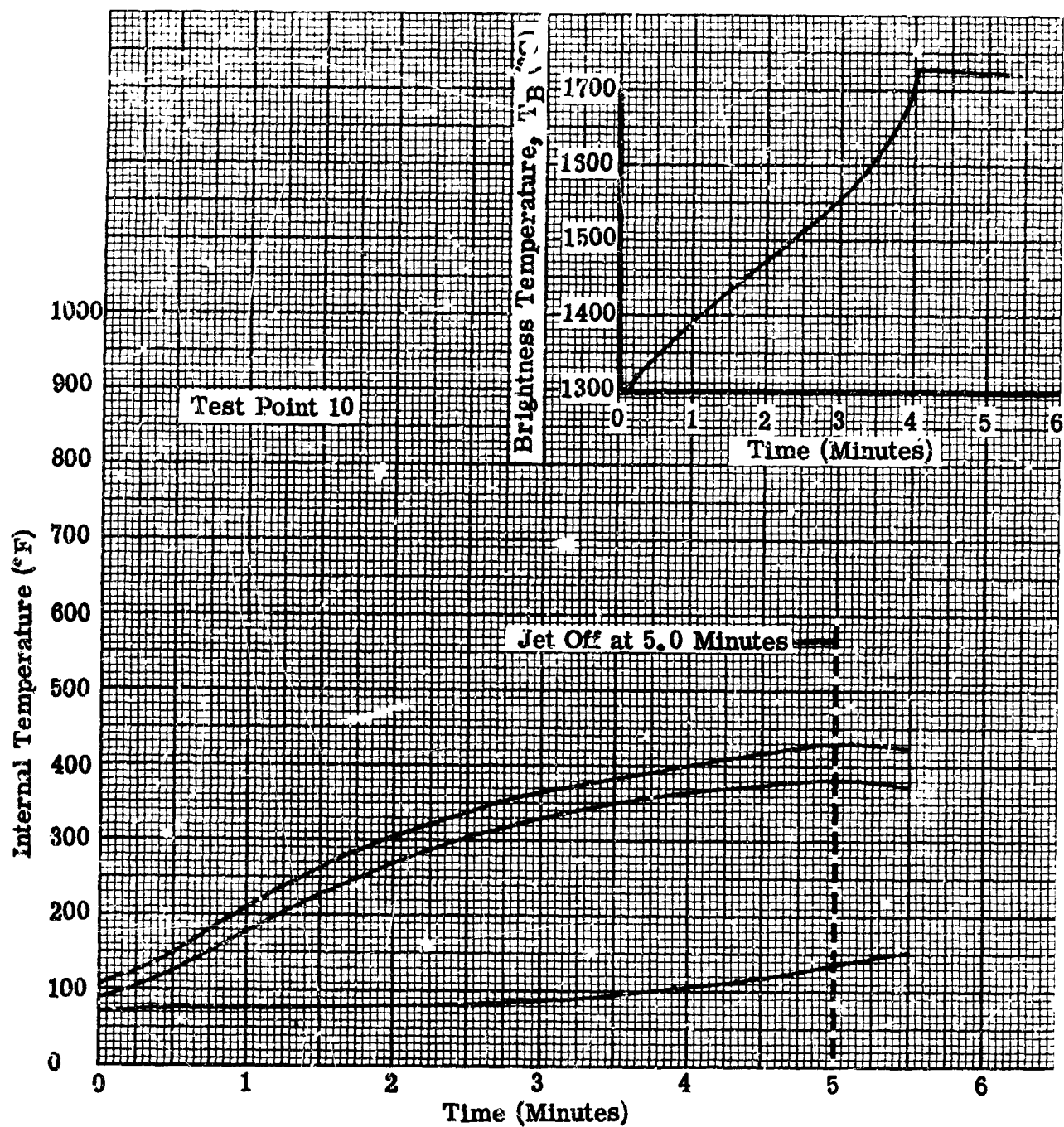


Figure 4-33. Temperature-Time History of Model O-13

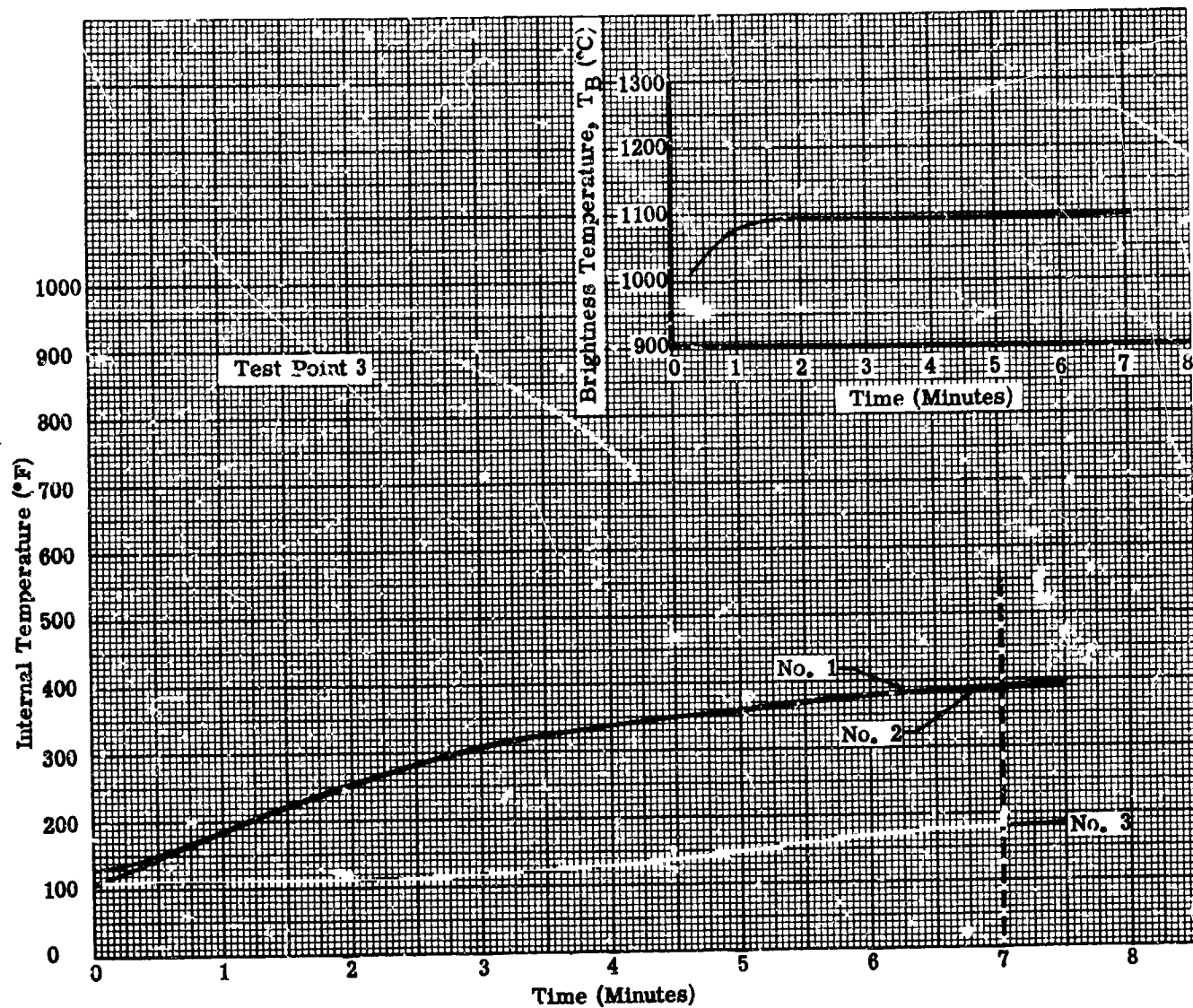


Figure 4-34. Temperature-Time History of Model O-14

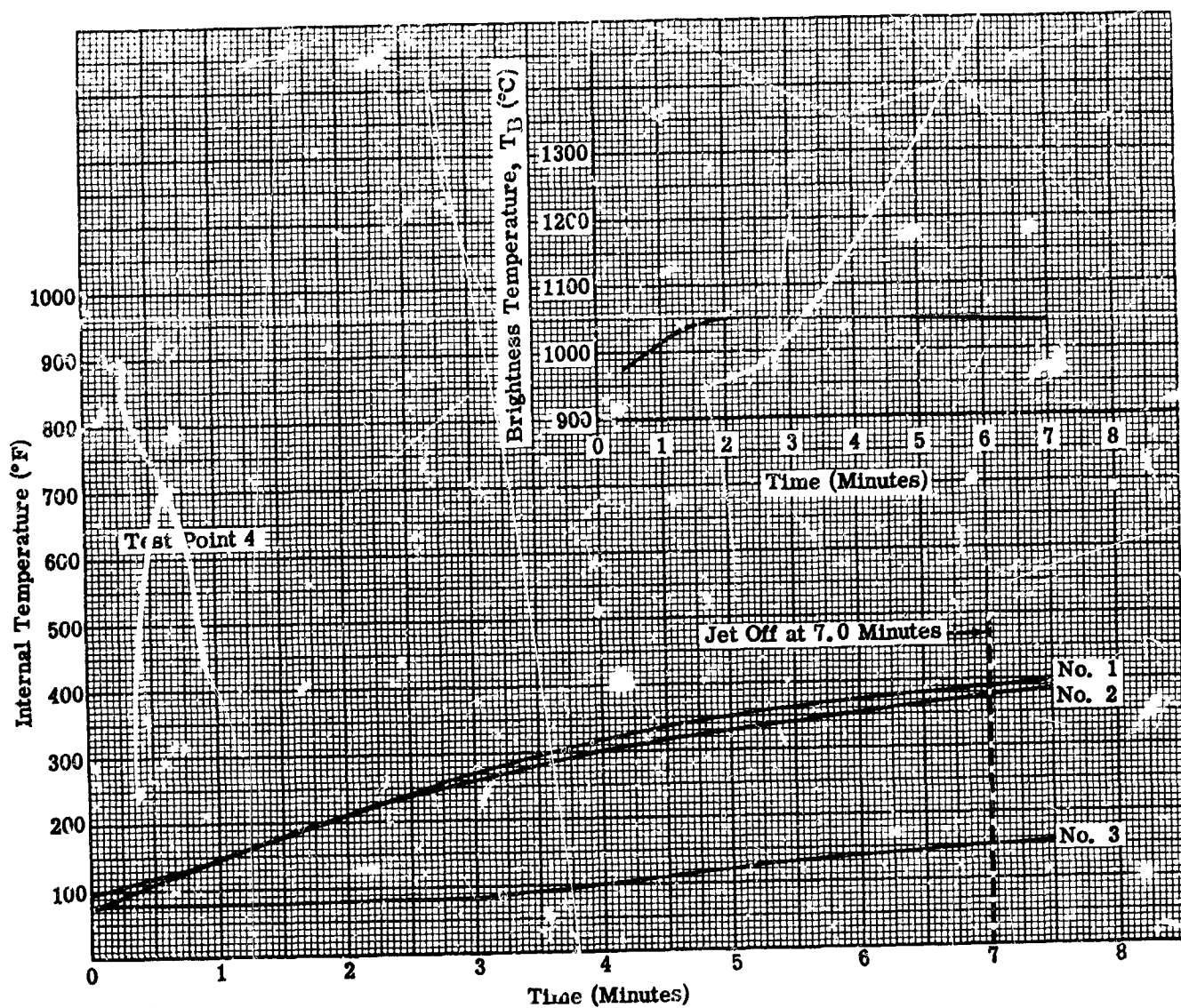


Figure 4-35. Temperature-Time History of Model O-15

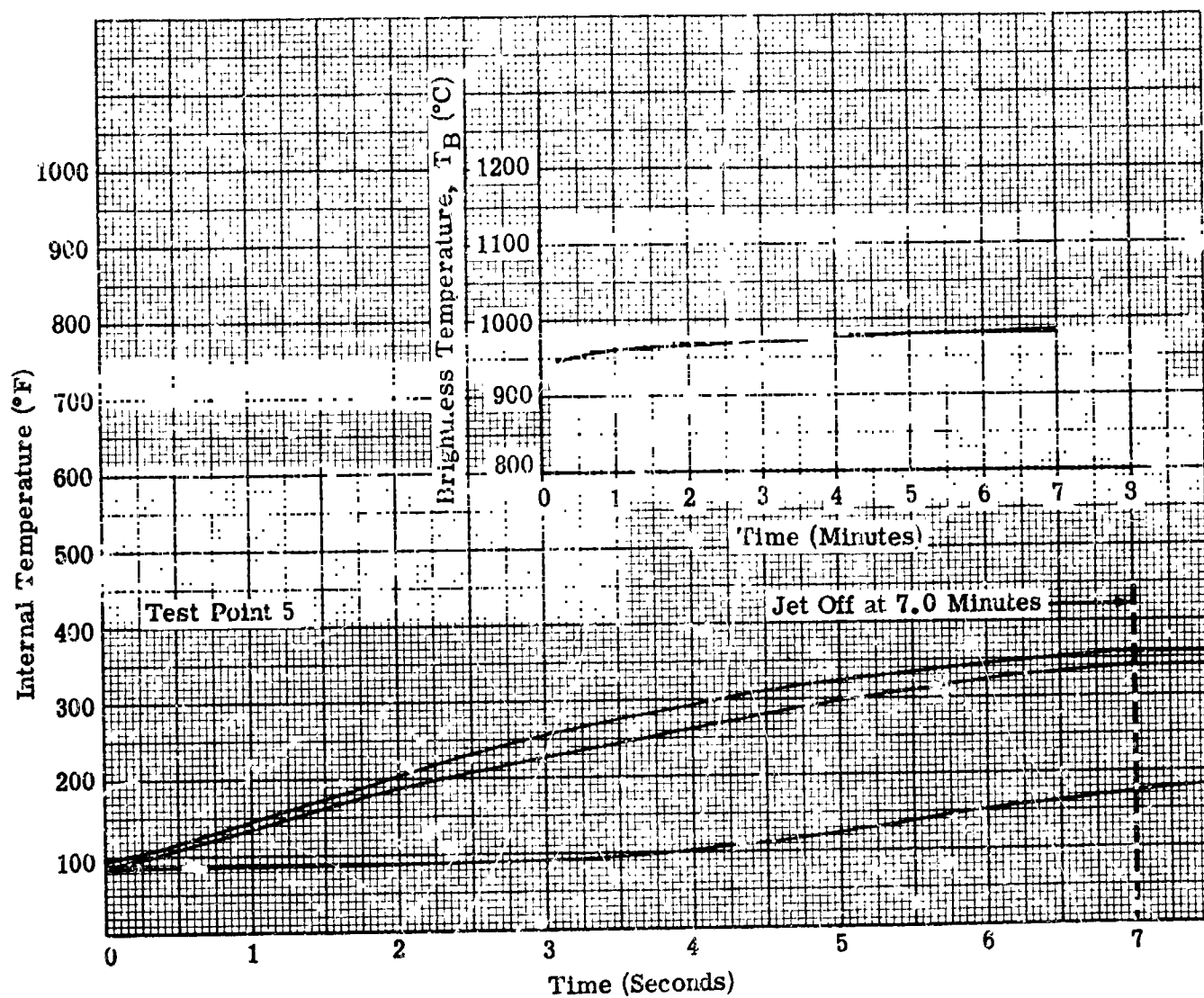


Figure 4-36. Temperature-Time History of Model O-16

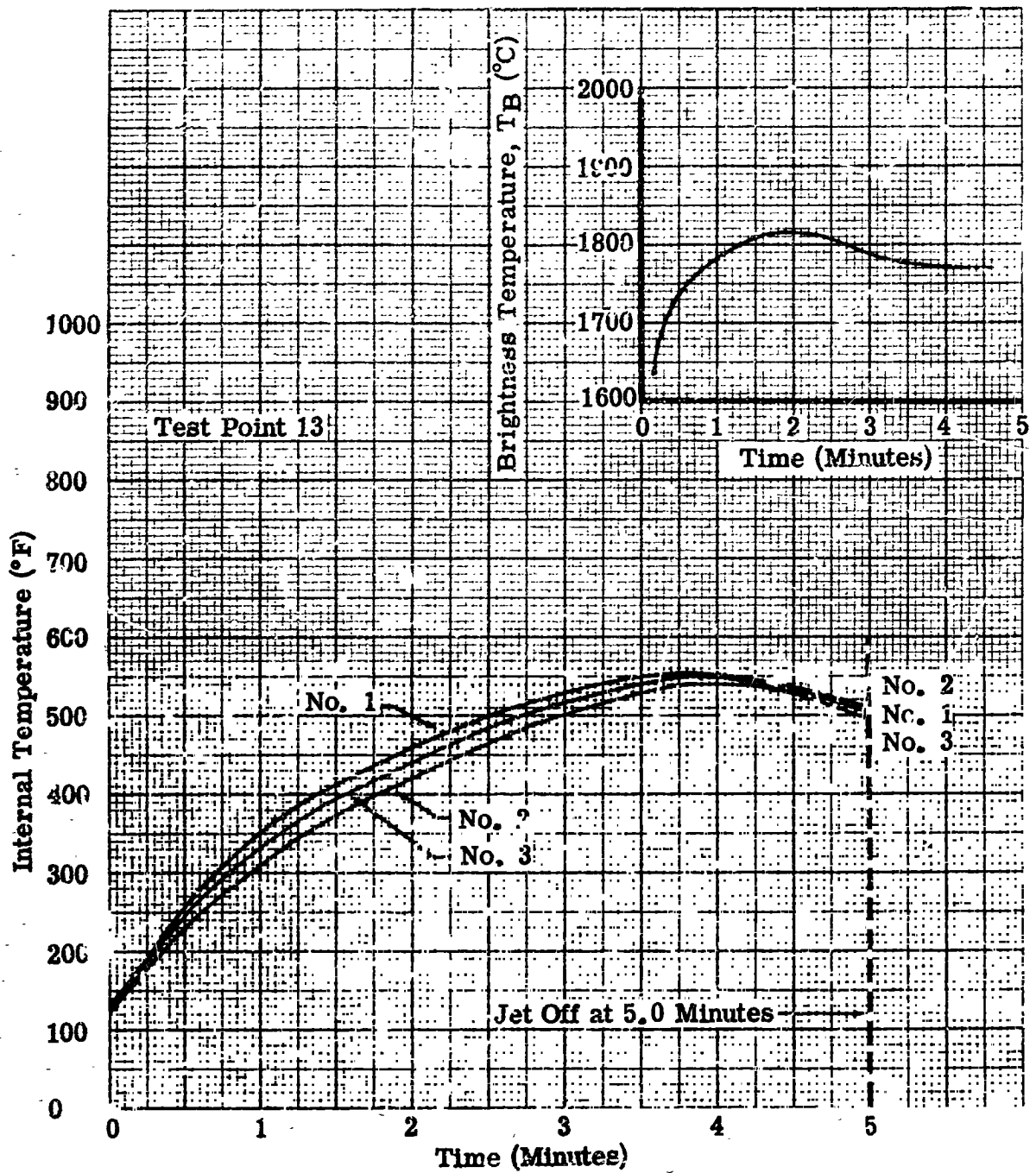


Figure 4-37. Temperature-Time History of Model D-12

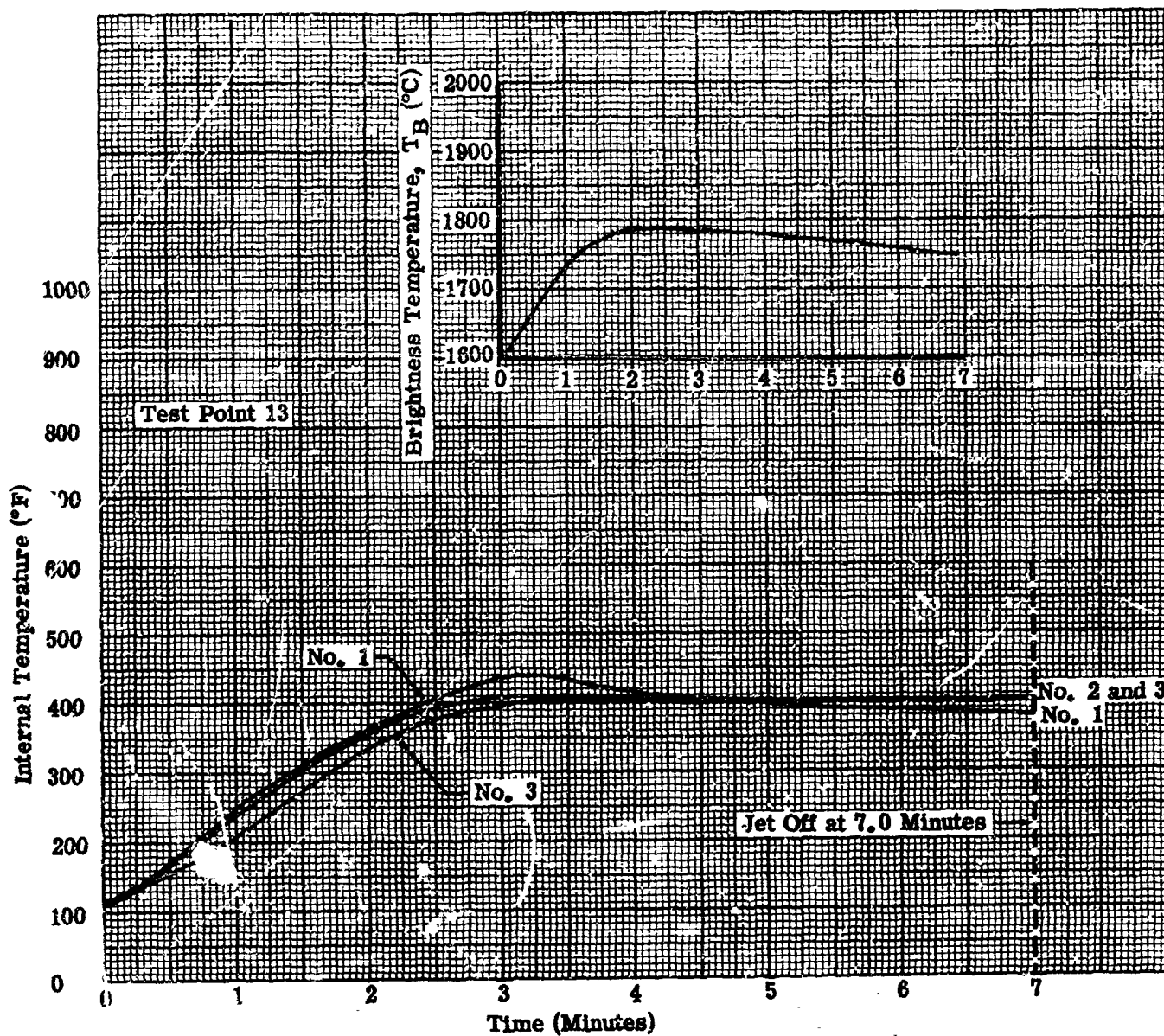


Figure 4-38. Temperature-Time History of Model D-2-2

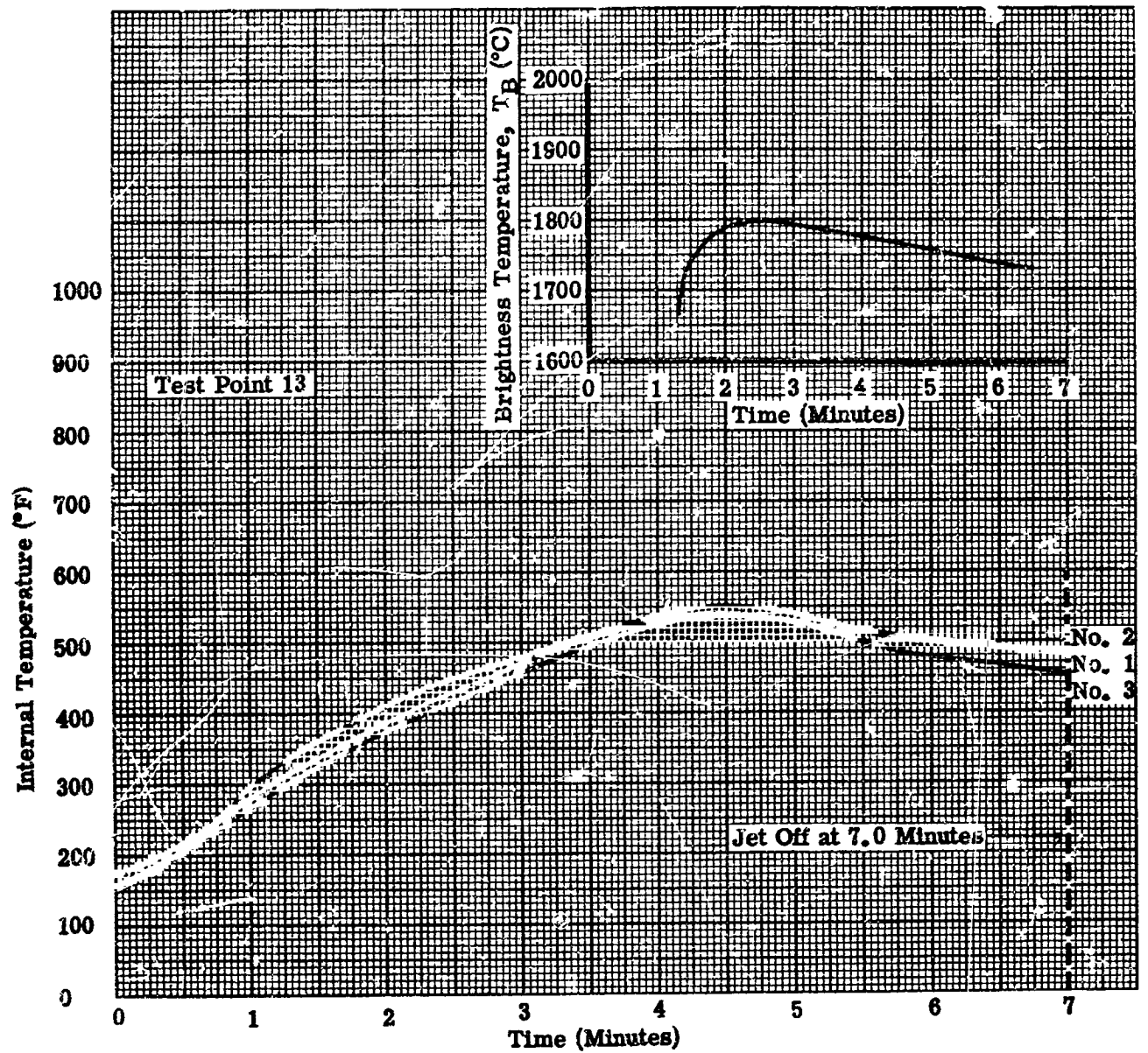


Figure 4-39. Temperature-Time History of Model E-12

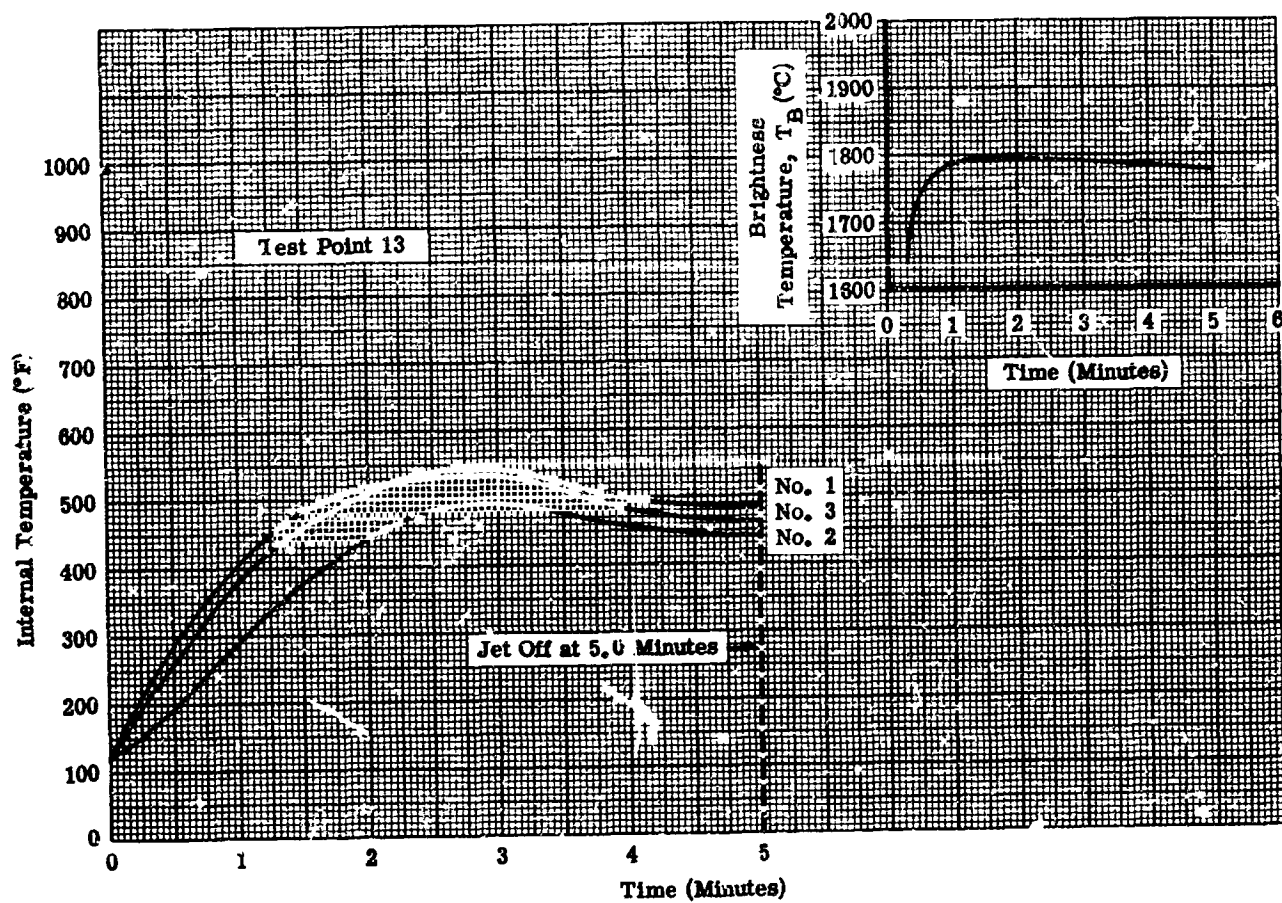


Figure 4-40. Temperature-Time History of Model E-22

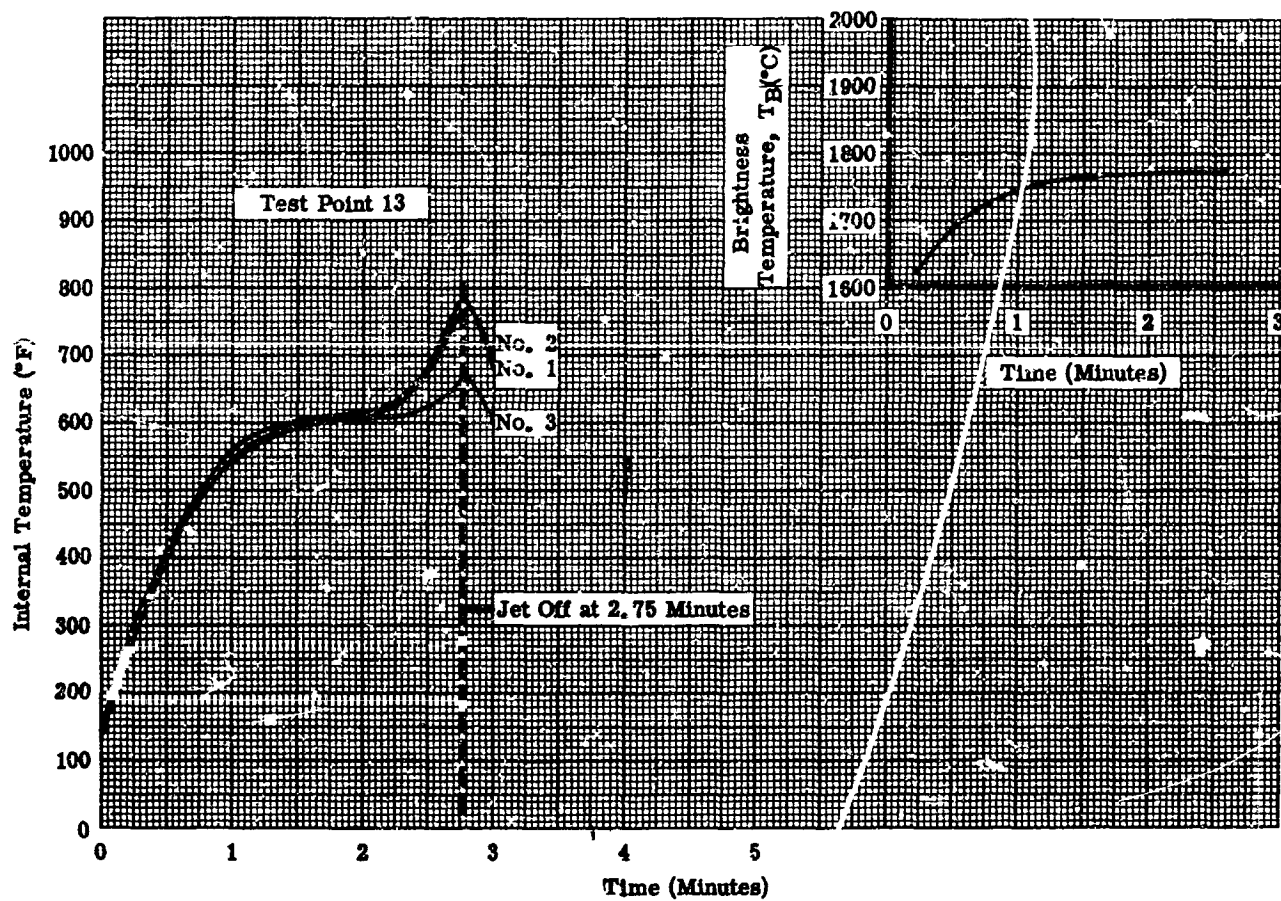


Figure 4-41. Temperature-Time History of Model GG-1

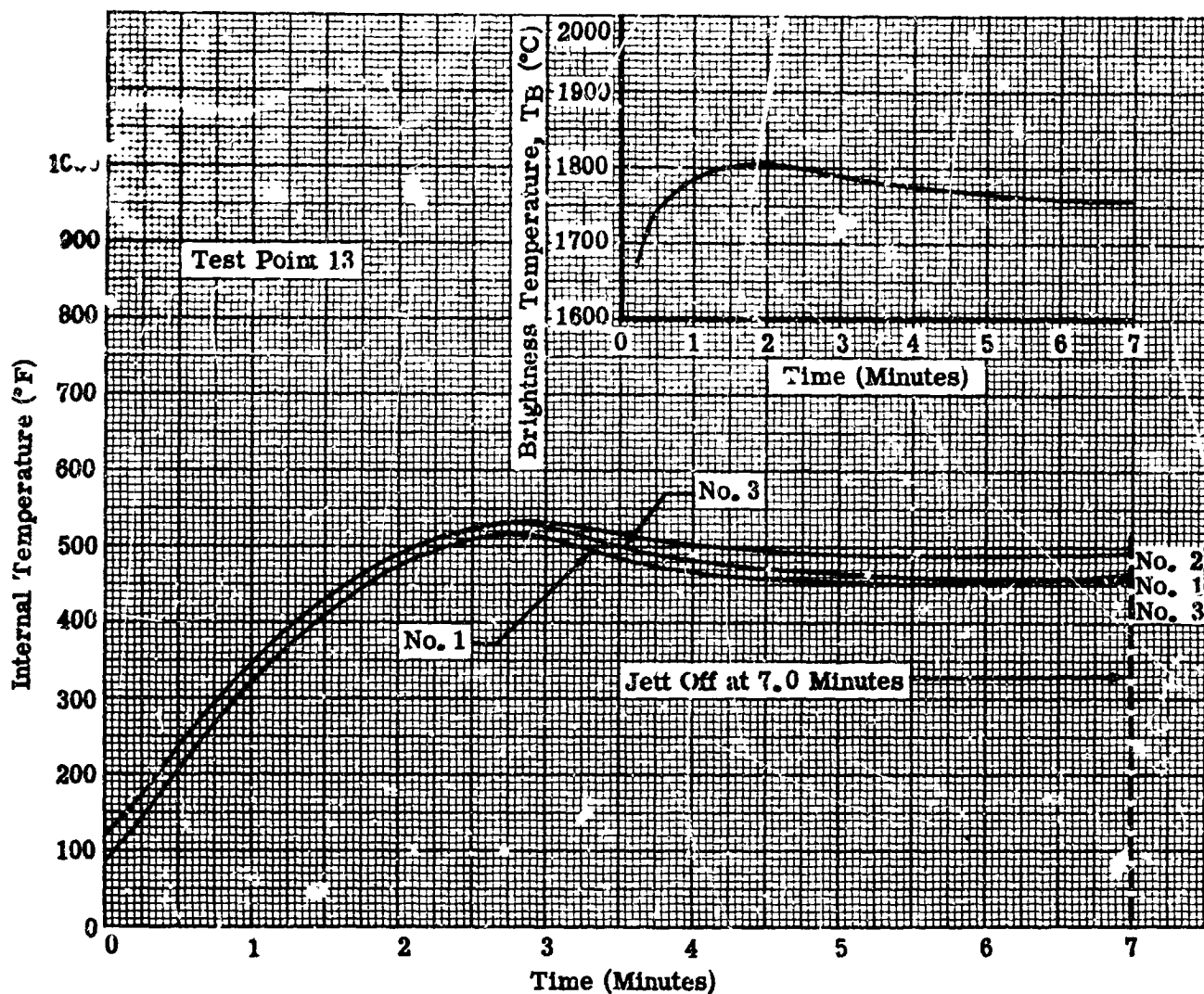


Figure 4-42. Temperature-Time History of Model GG-3

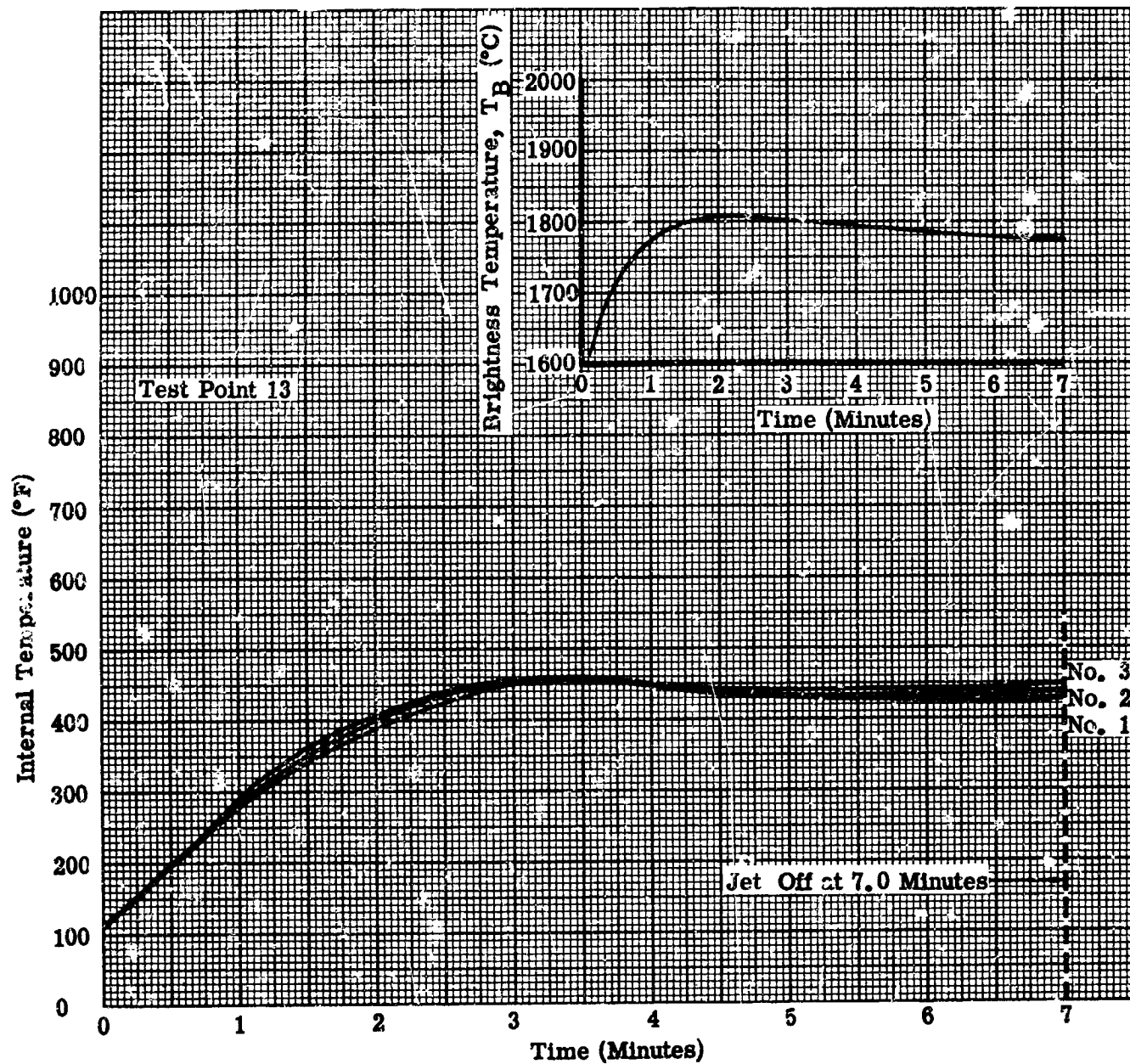


Figure 4-43. Temperature-Time History of Model HH-1

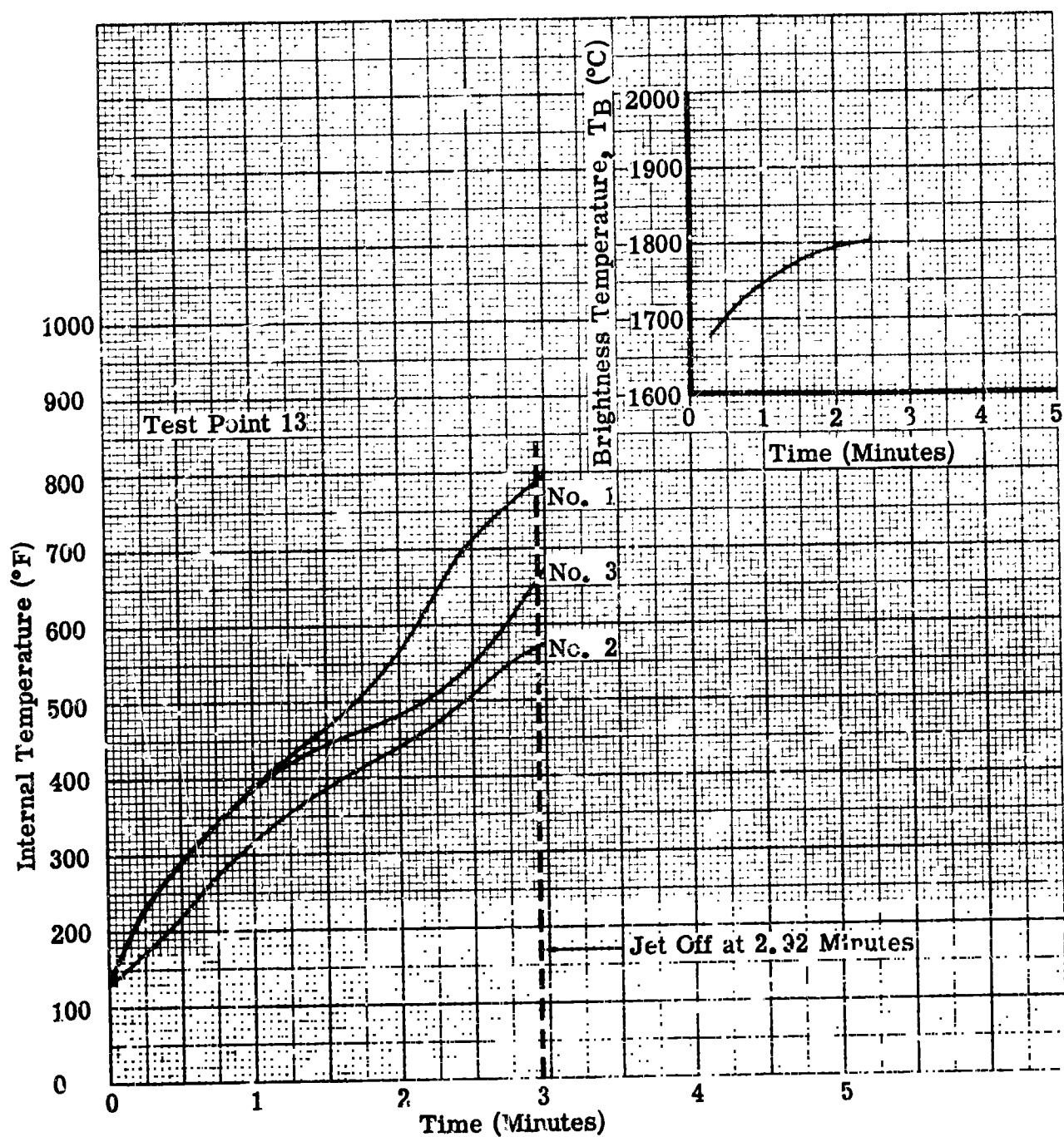


Figure 4-44. Temperature-Time History of Model HH-2

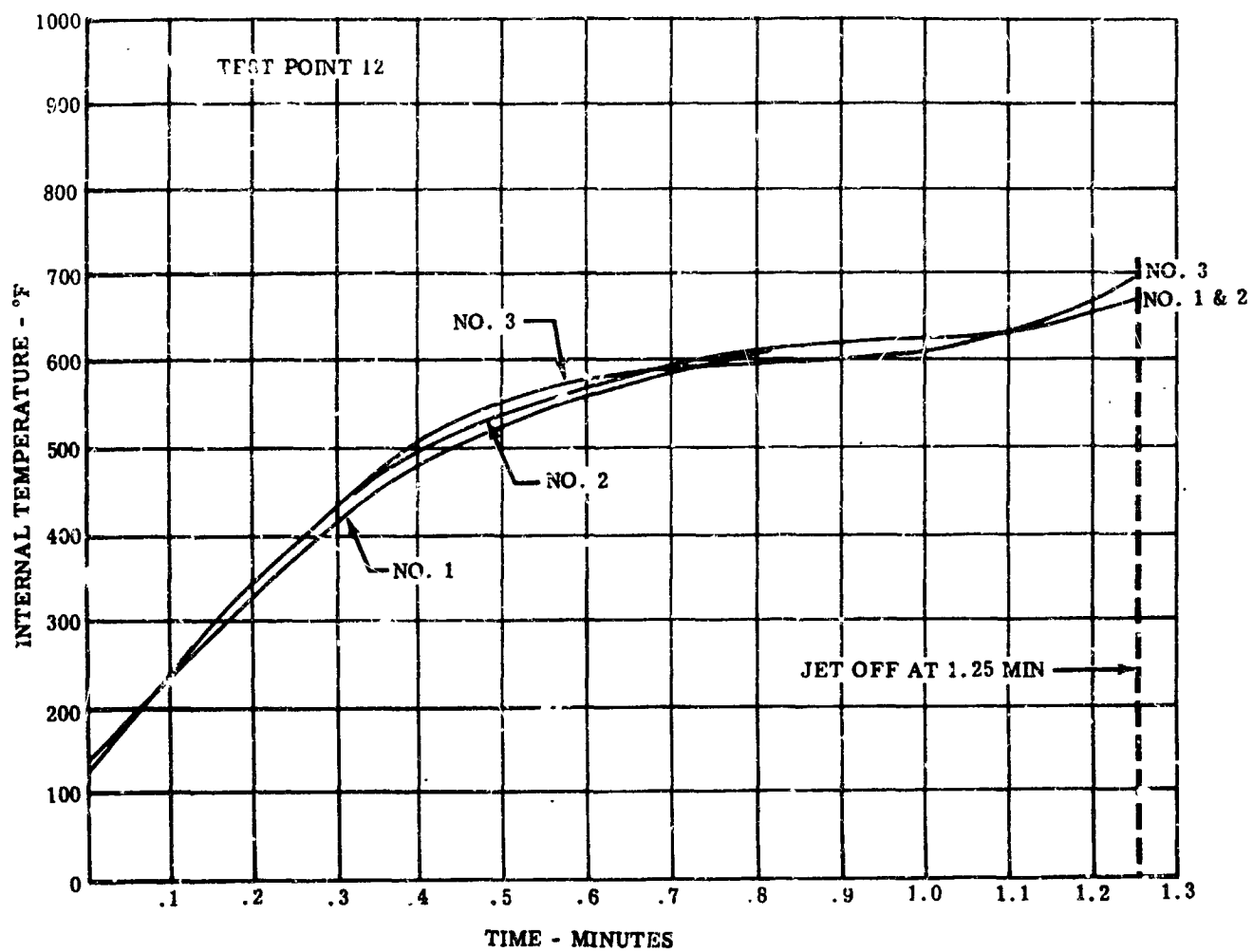


Figure 4-45. Temperature-Time History of Model D-1

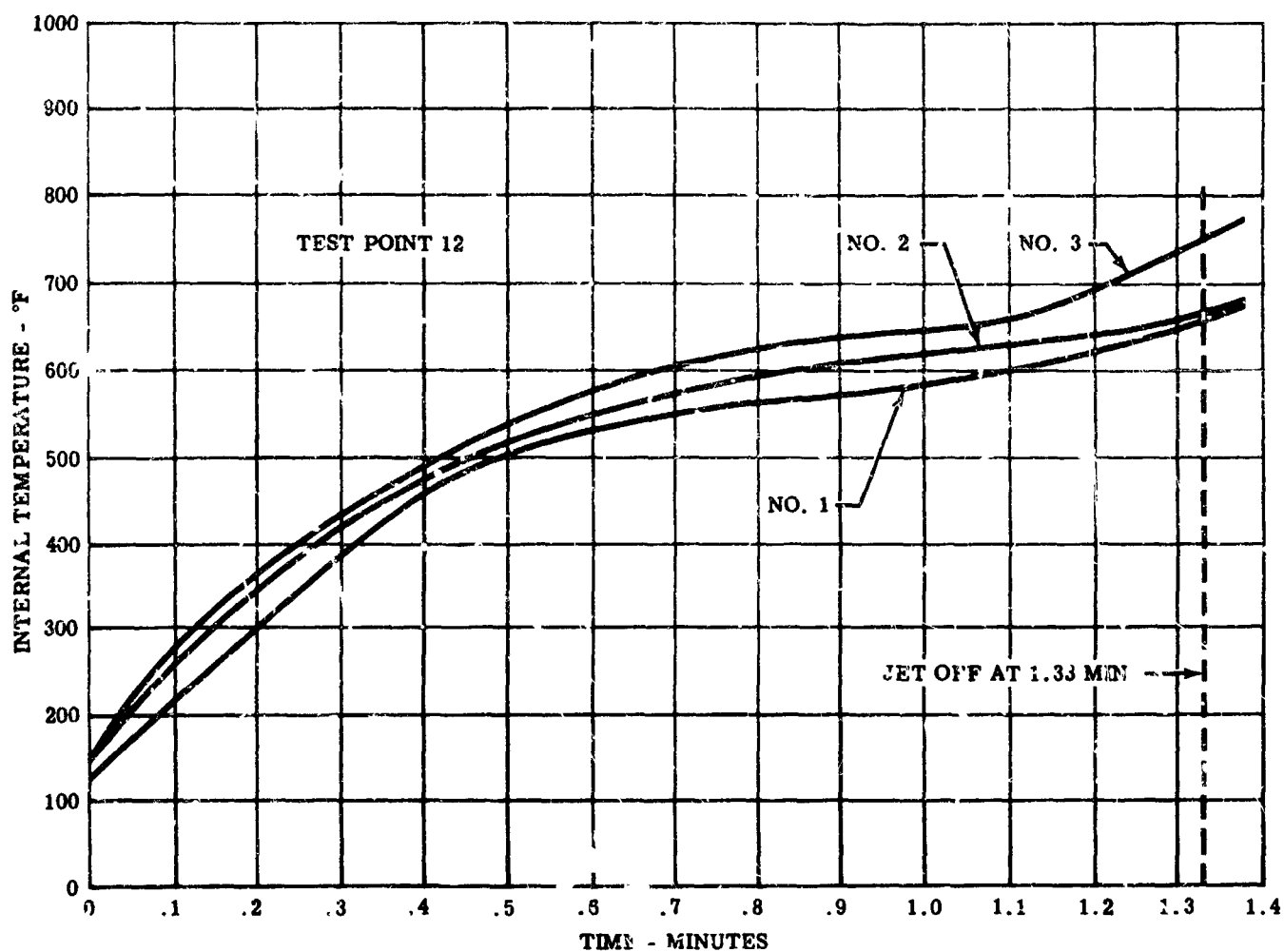


Figure 4-46. Temperature-Time History of Model D-2

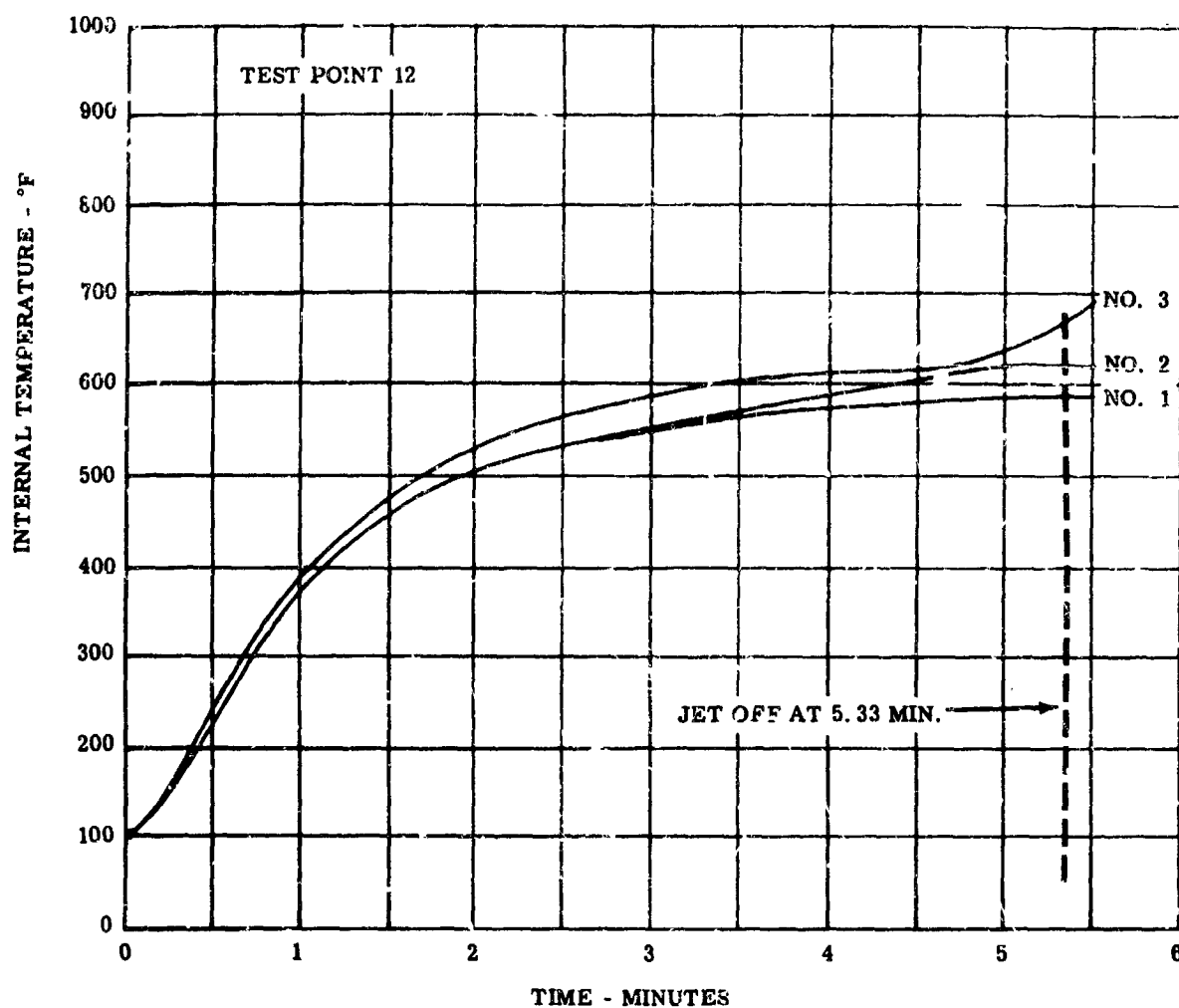


Figure 4-47. Temperature-Time History of Model D-9

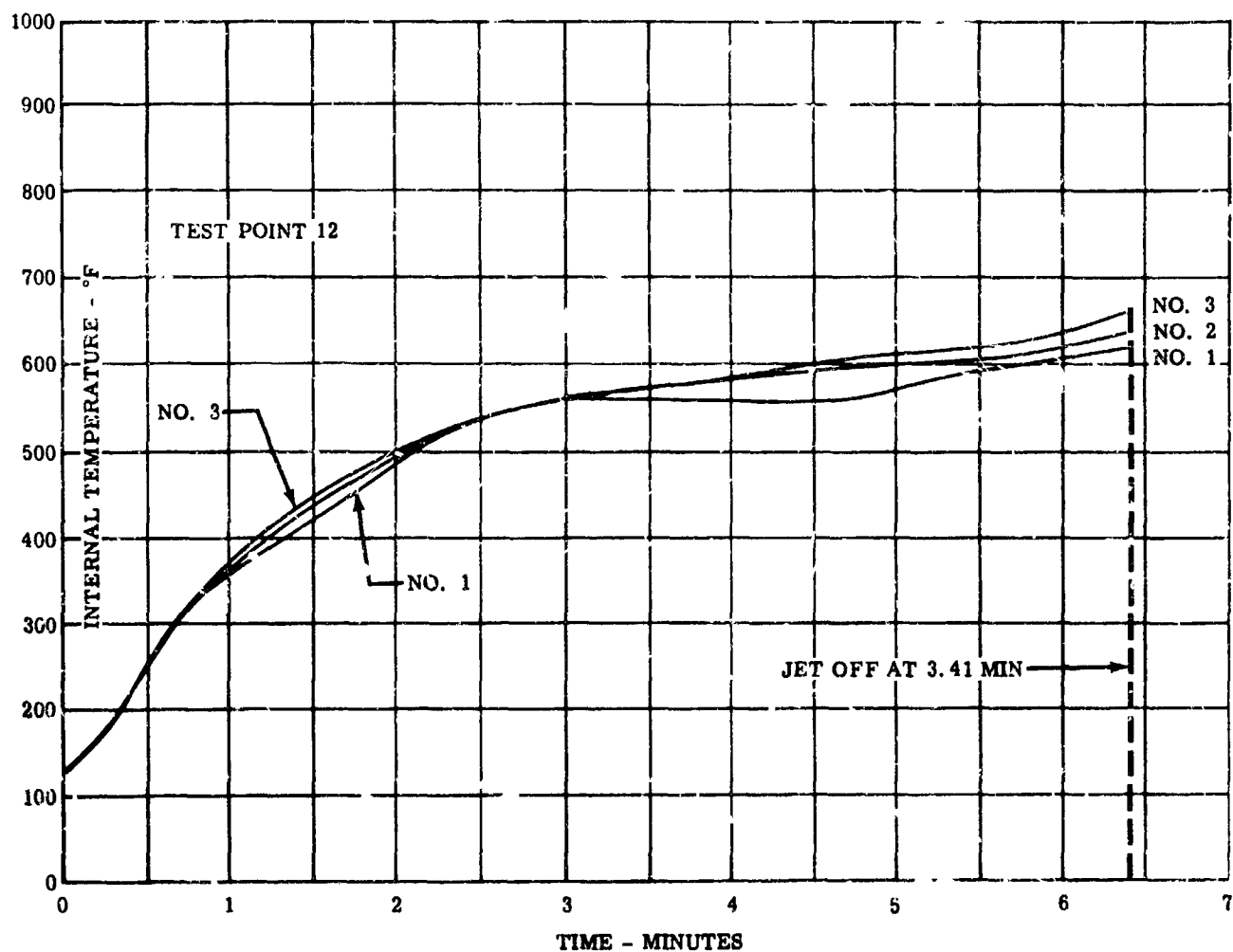


Figure 4-48. Temperature-Time History of Model D-10

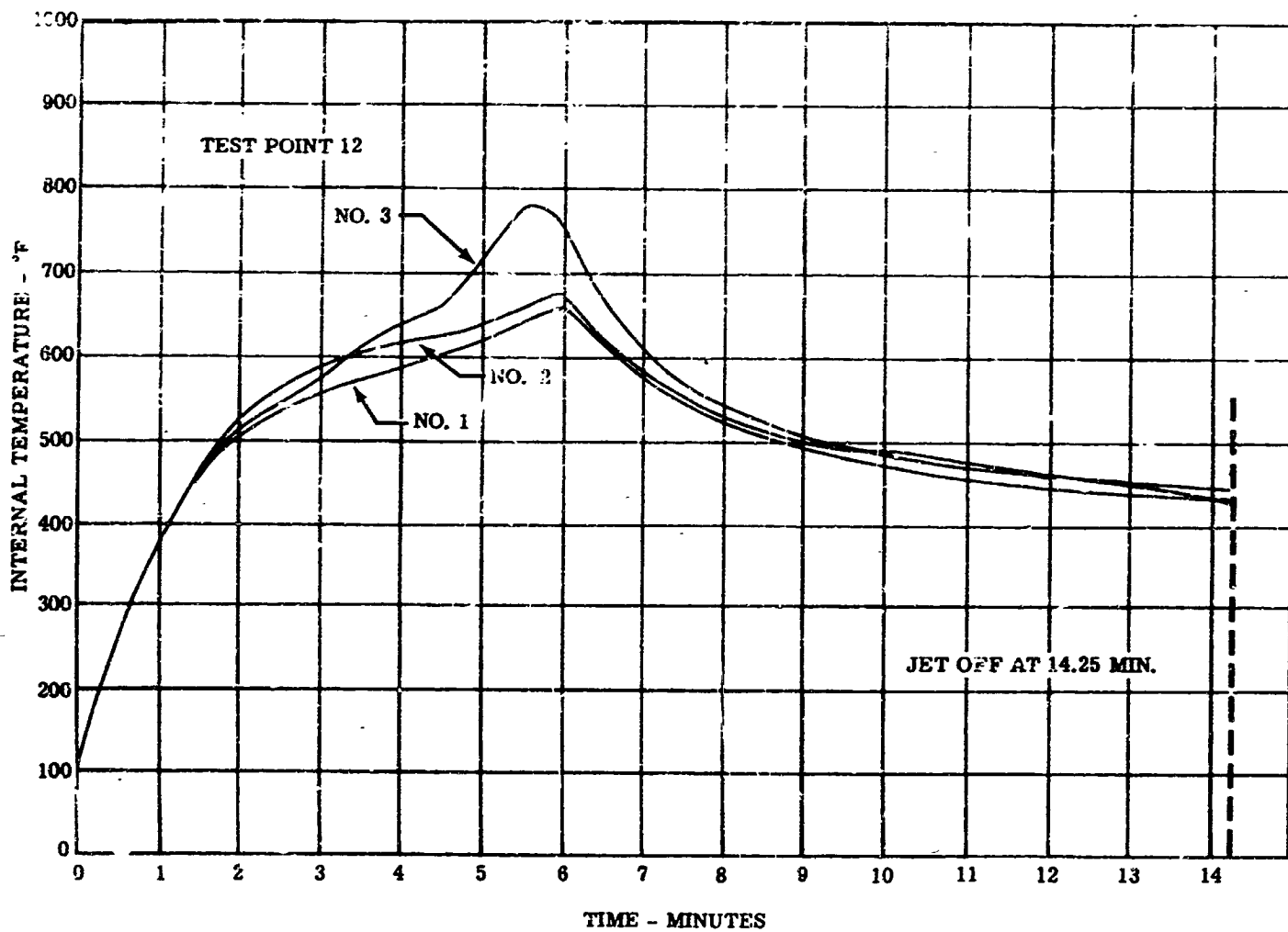


Figure 4-49. Temperature-Time History of Model D-11

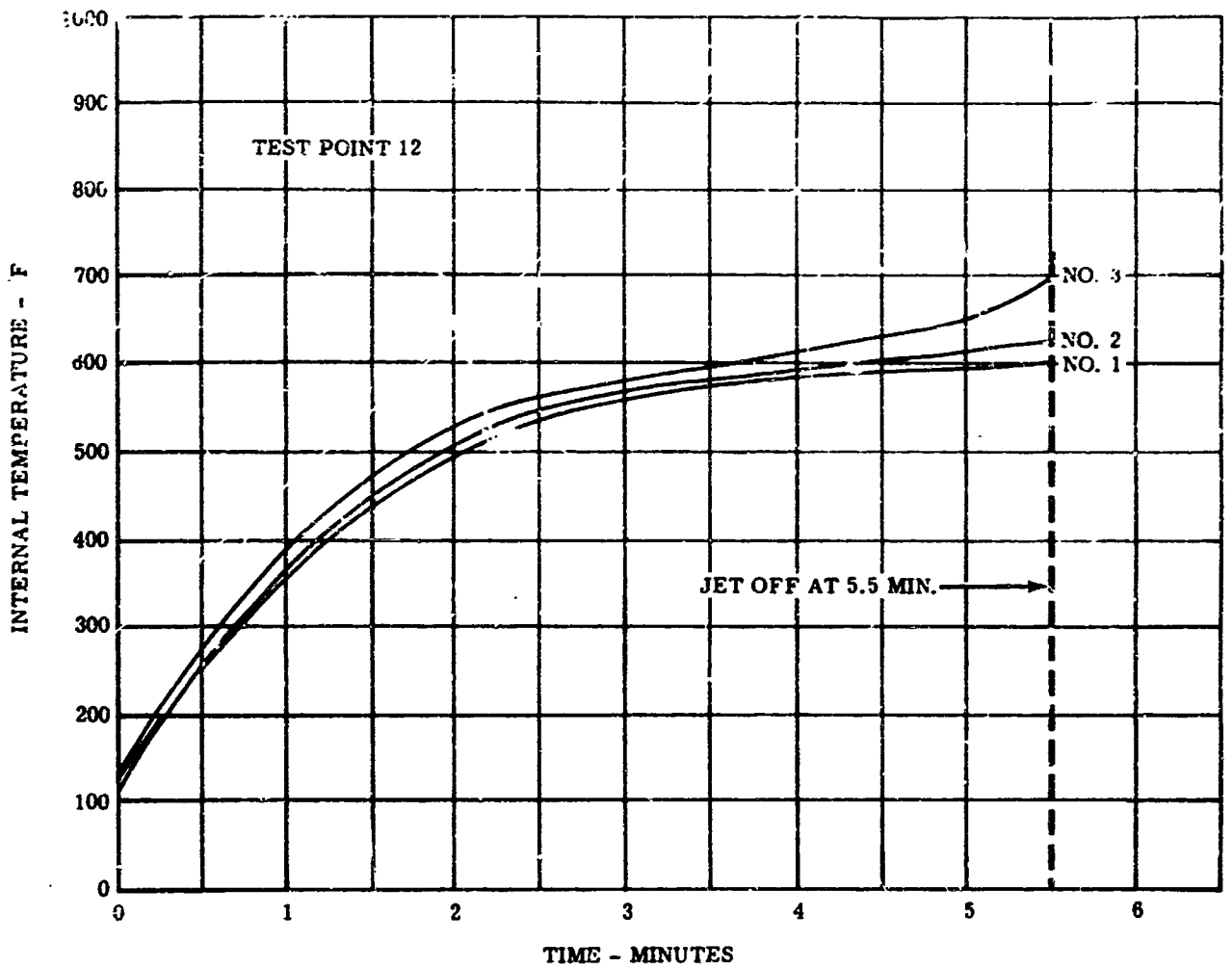


Figure 4-50. Temperature-Time History of Model D-12

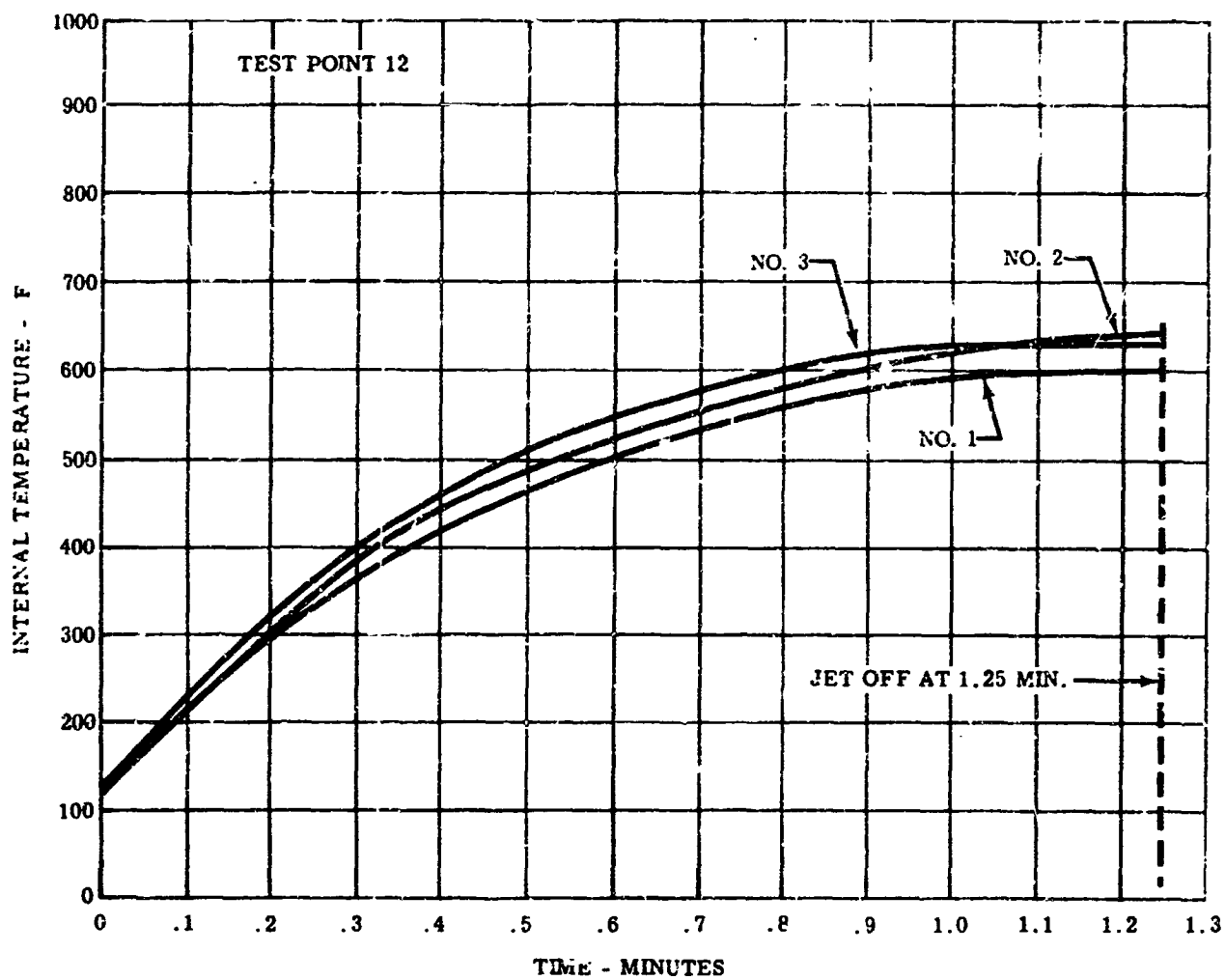


Figure 4-51. Temperature-Time History of Model E-15

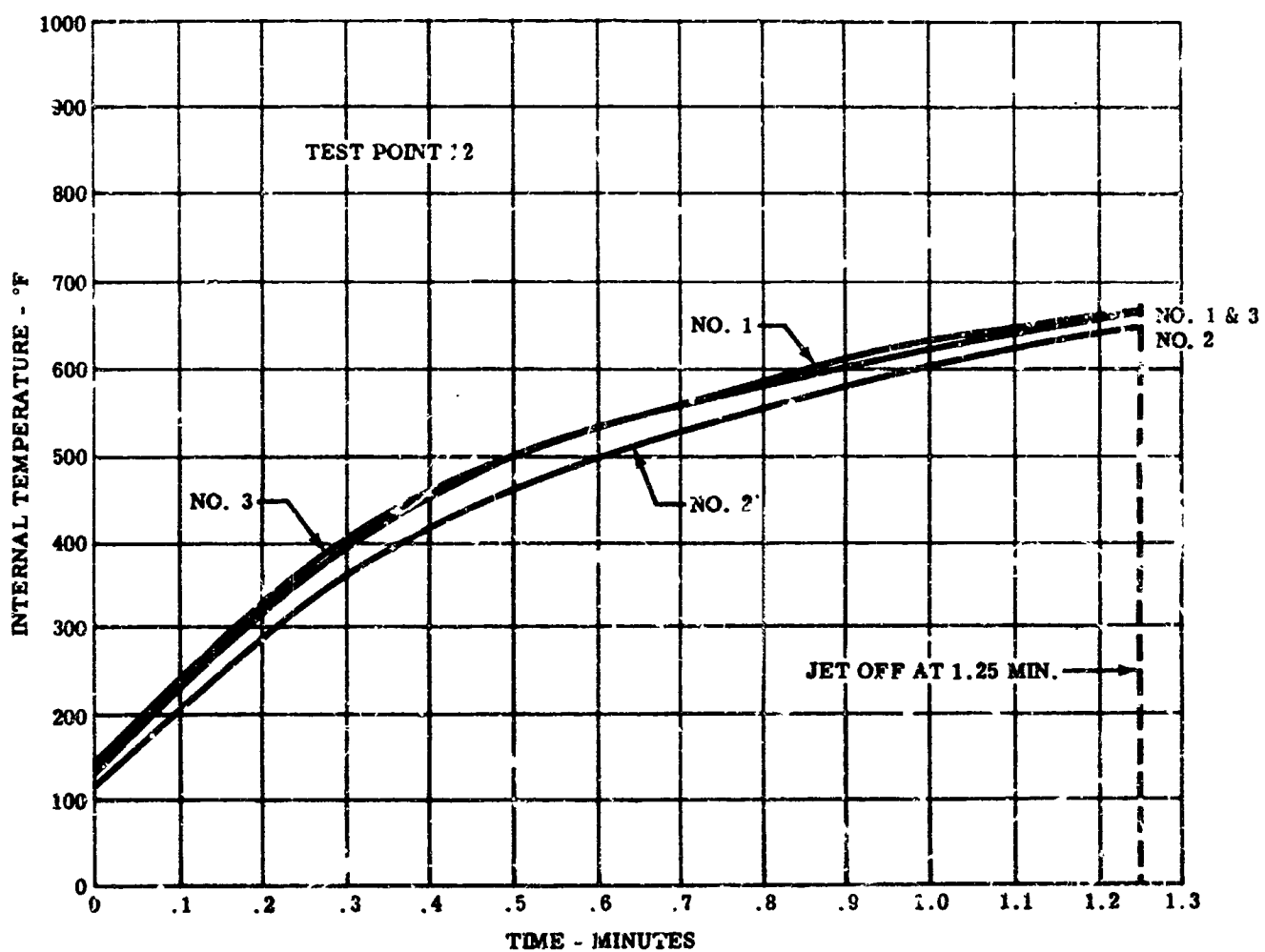


Figure 4-52. Temperature-Time History of Model E-16

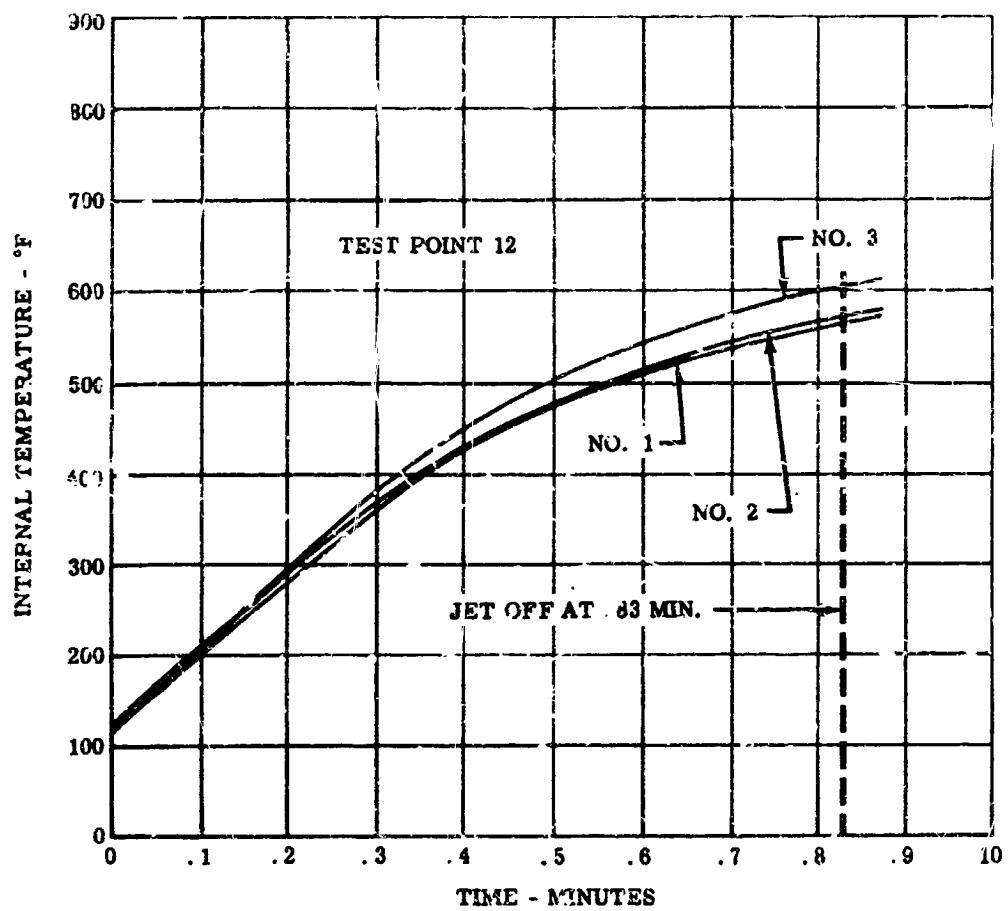


Figure 4-53. Temperature-Time History of Model E-17

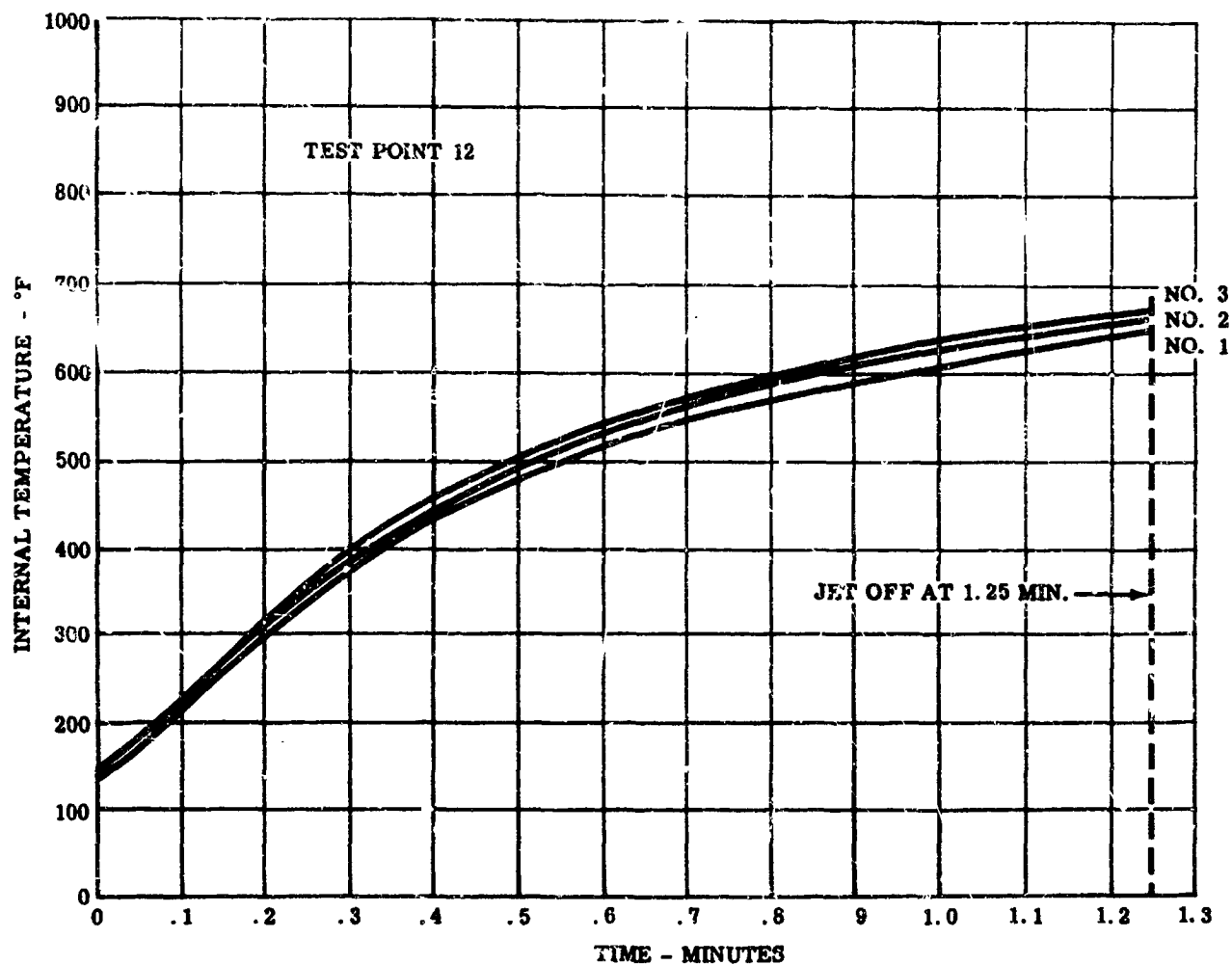


Figure 4-54. Temperature-Time History of Model E-18

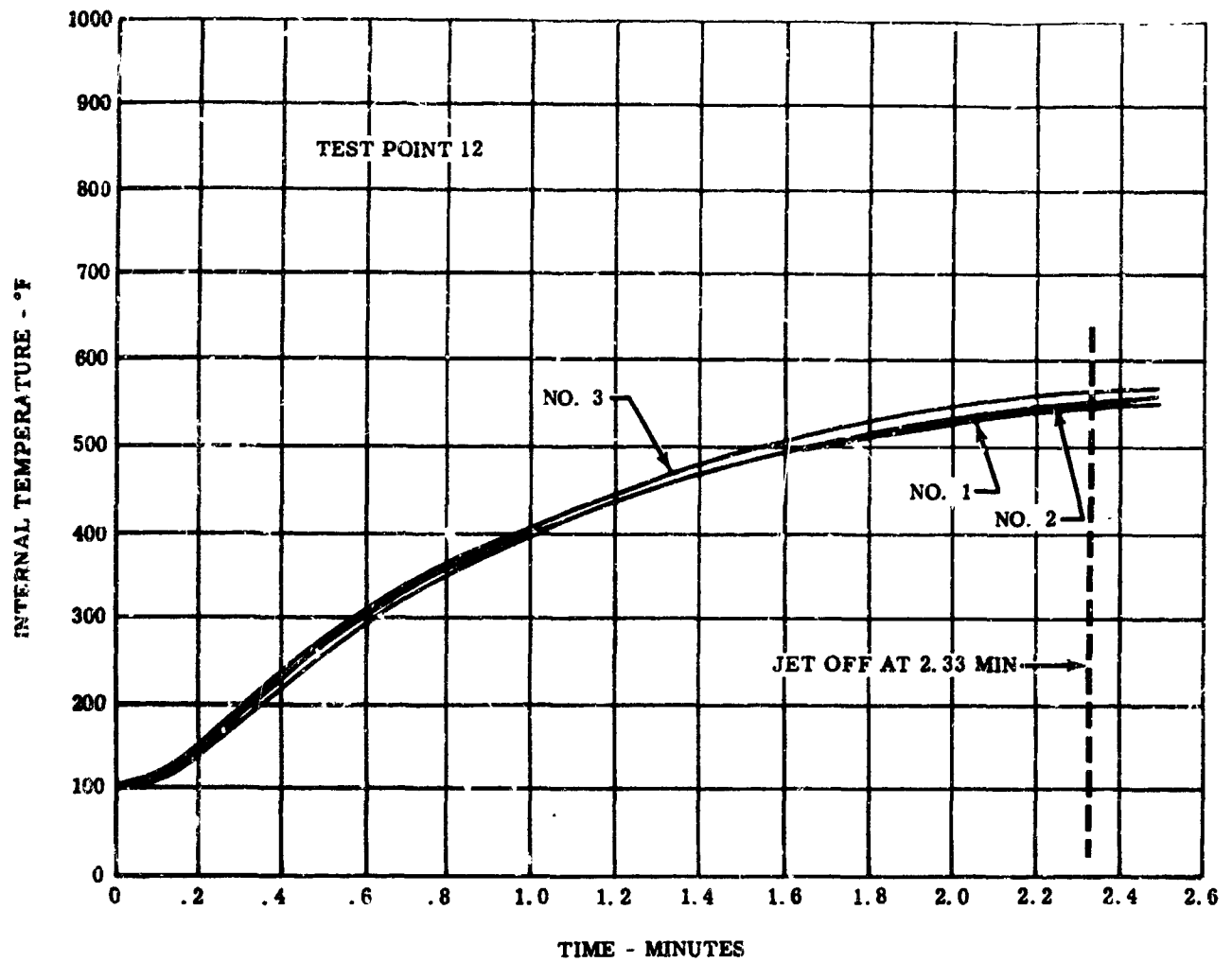


Figure 4-55. Temperature-Time History of Model GG-1A

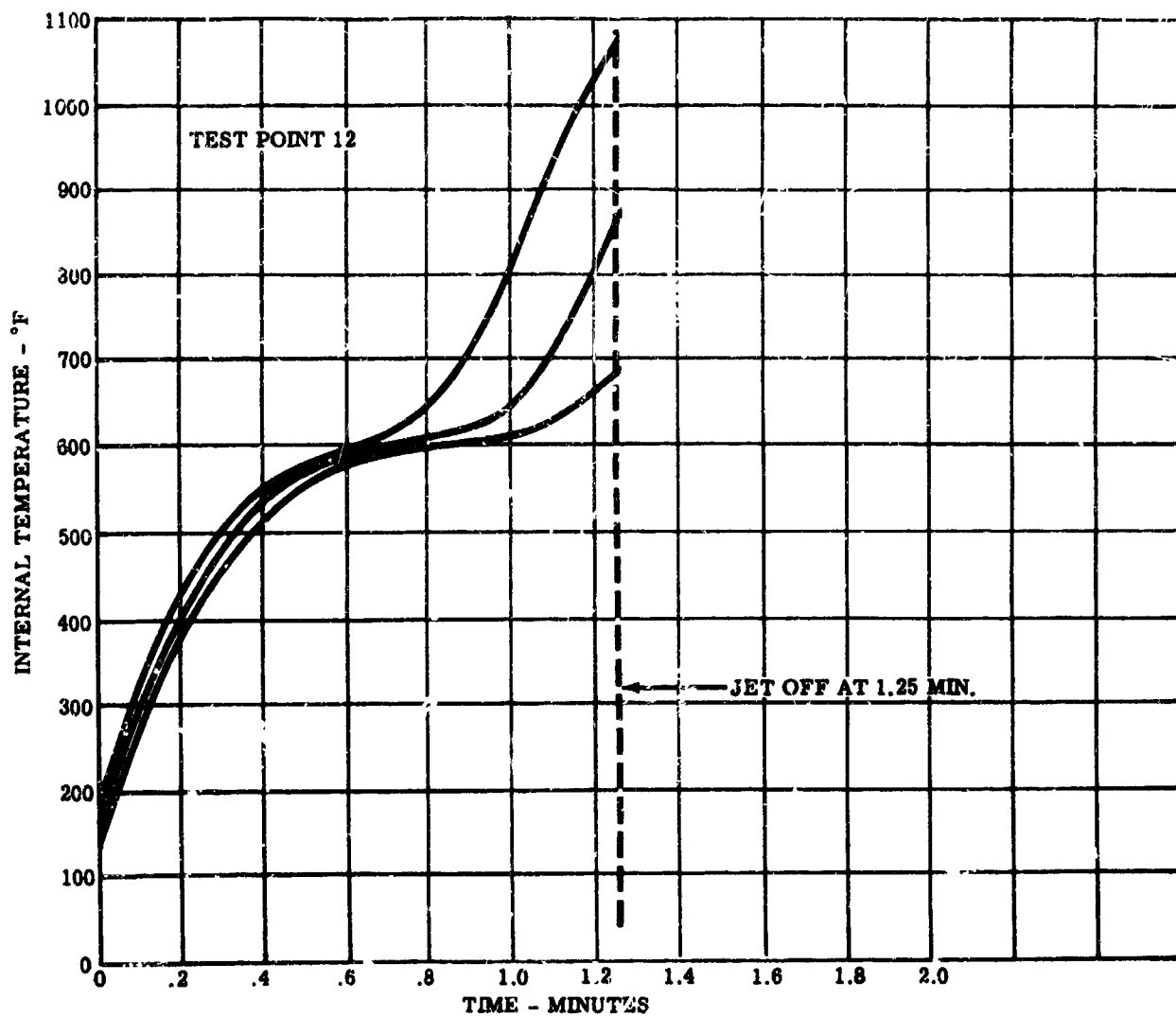


Figure 4-56. Temperature-Time History of Model GG-2

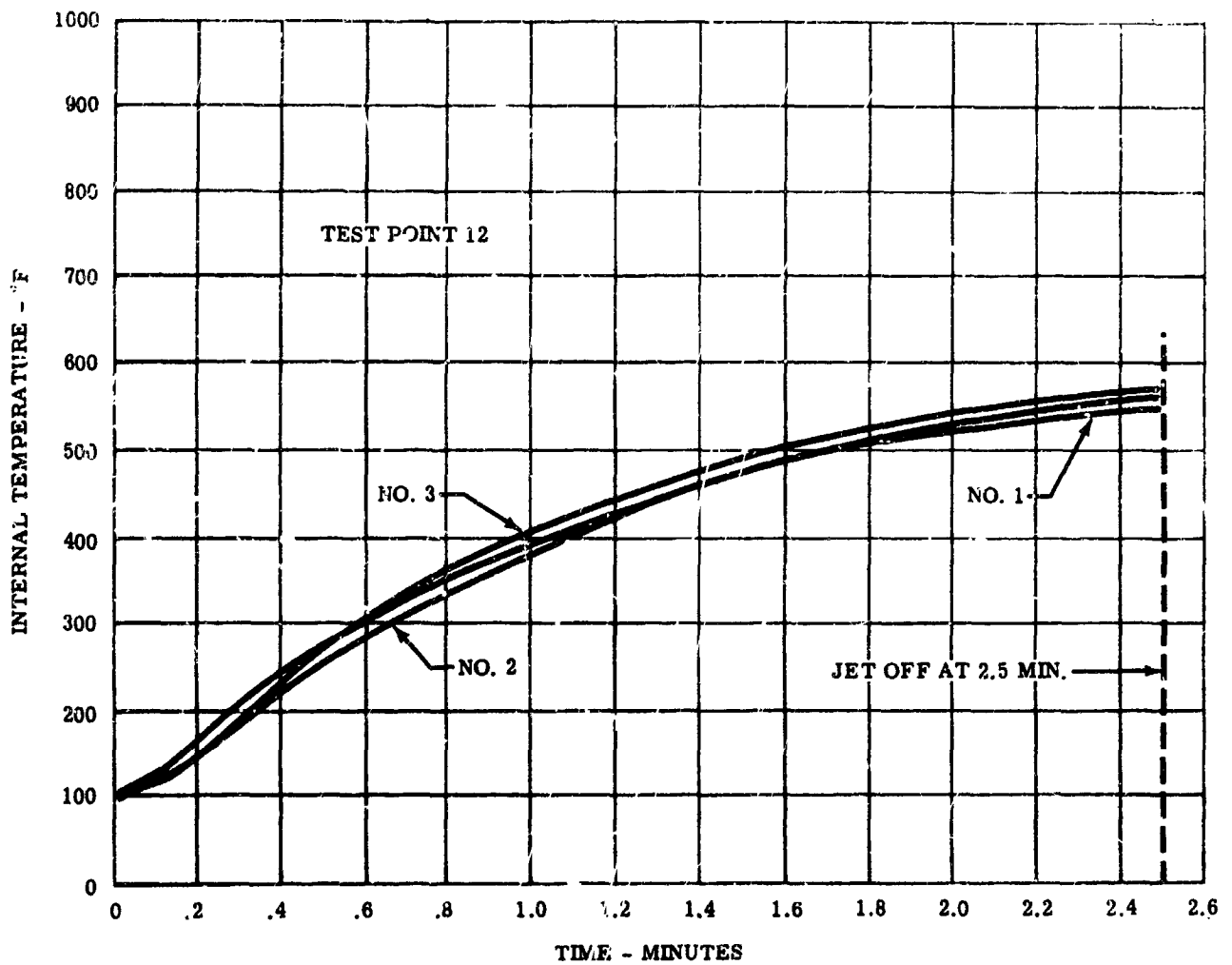


Figure 4-57. Temperature-Time History of Model GG-4

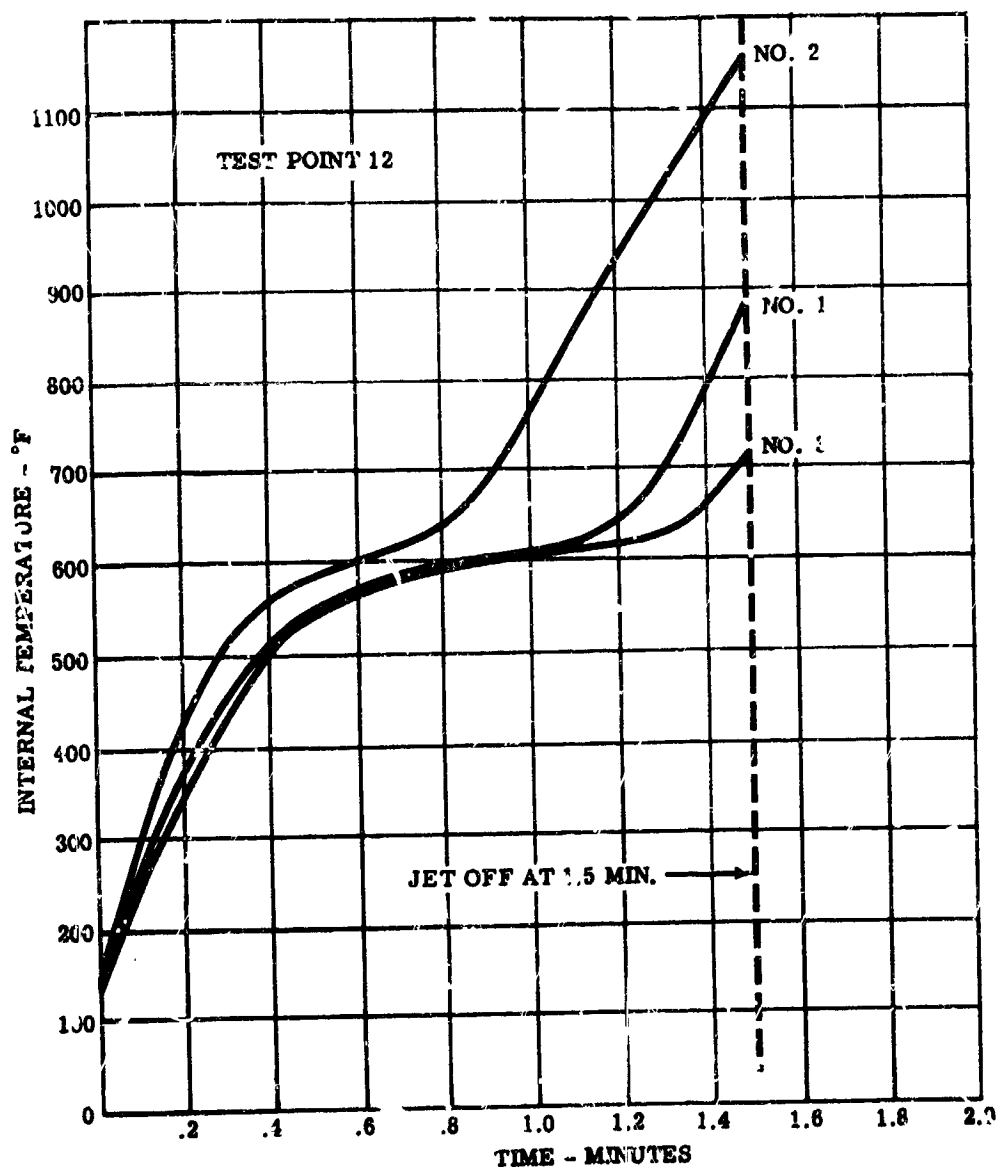


Figure 4-58. Temperature-Time History of Model GG-5

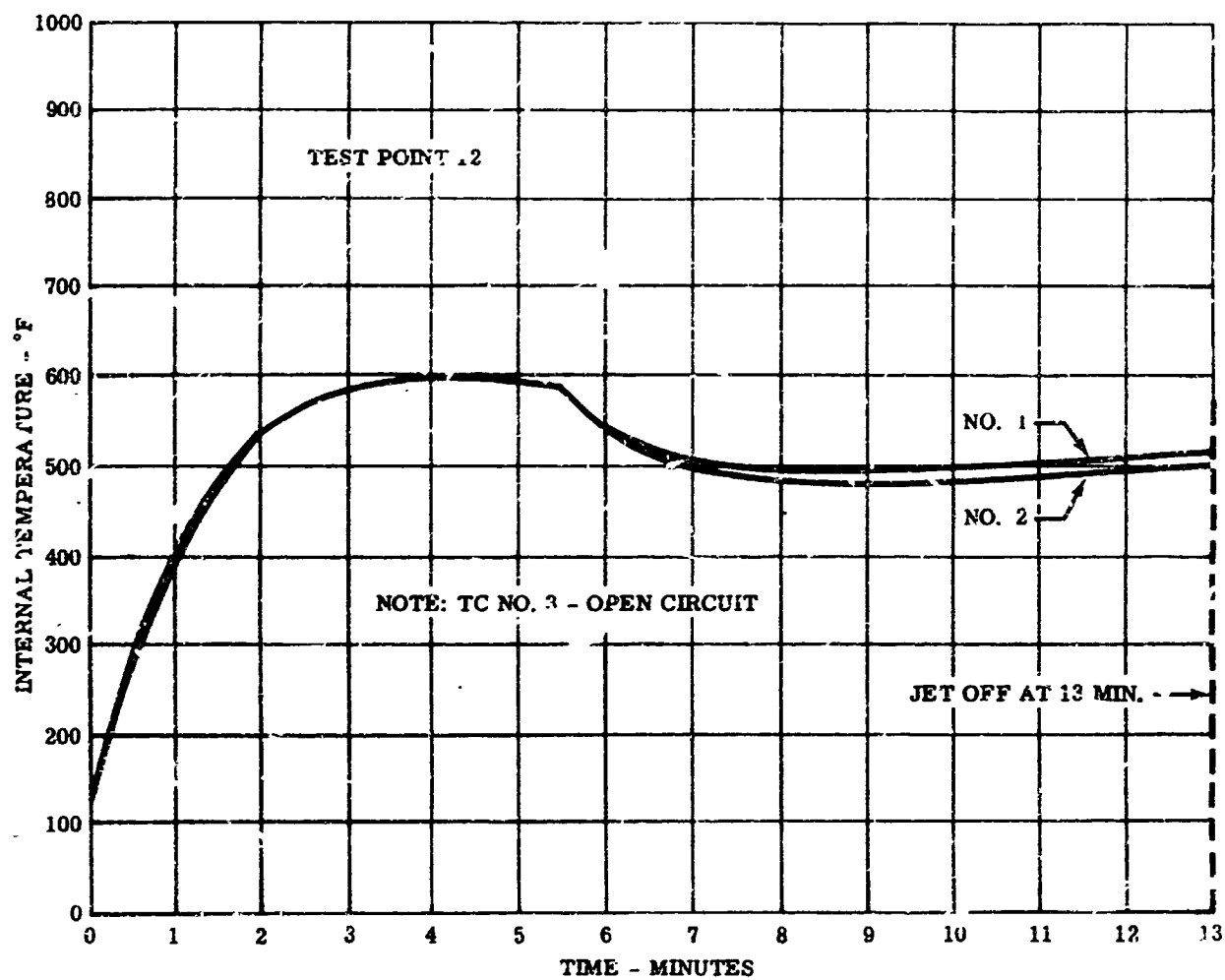


Figure 4-59. Temperature-Time History of Model GG-7

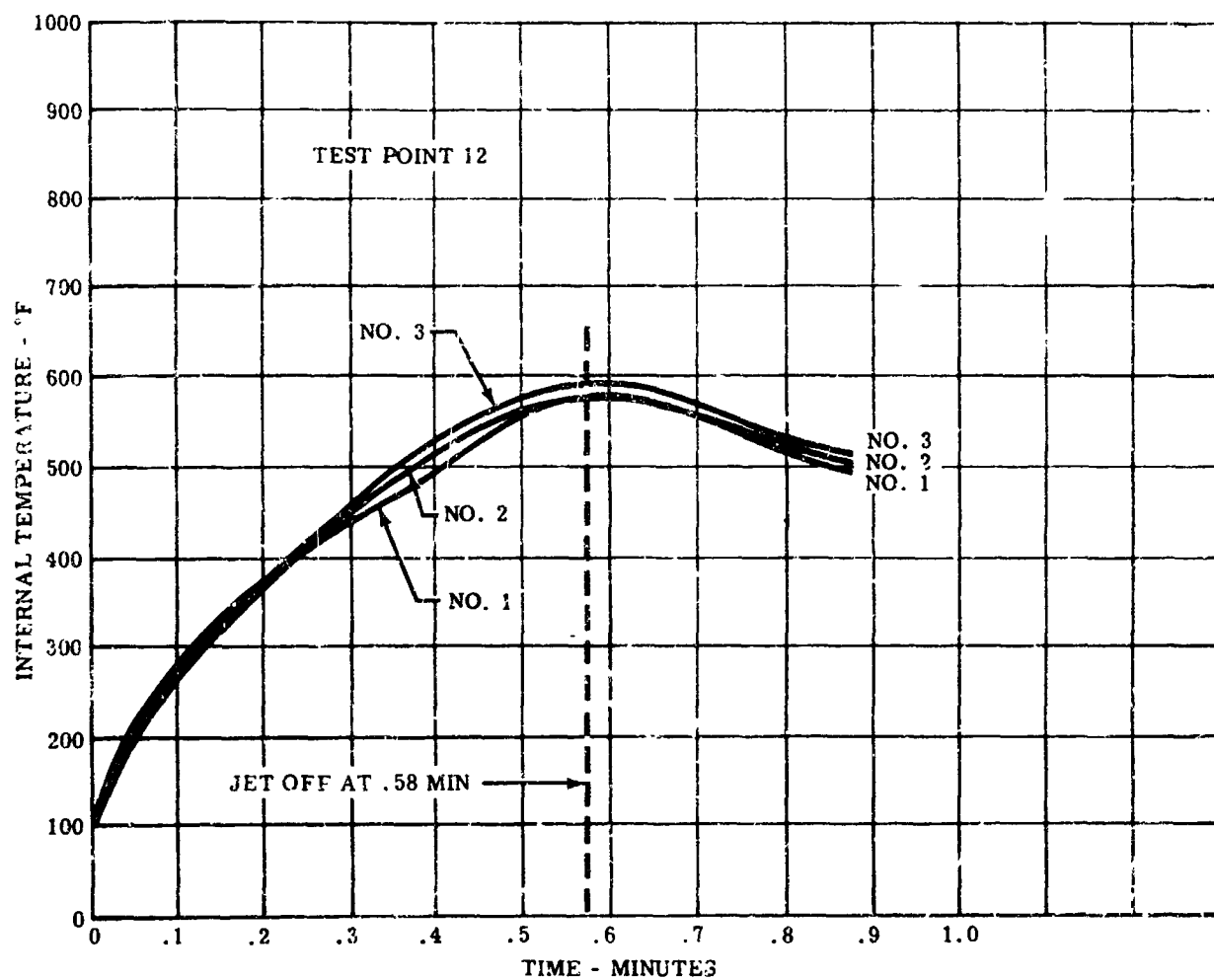


Figure 4-60. Temperature-Time History of Model GG-8

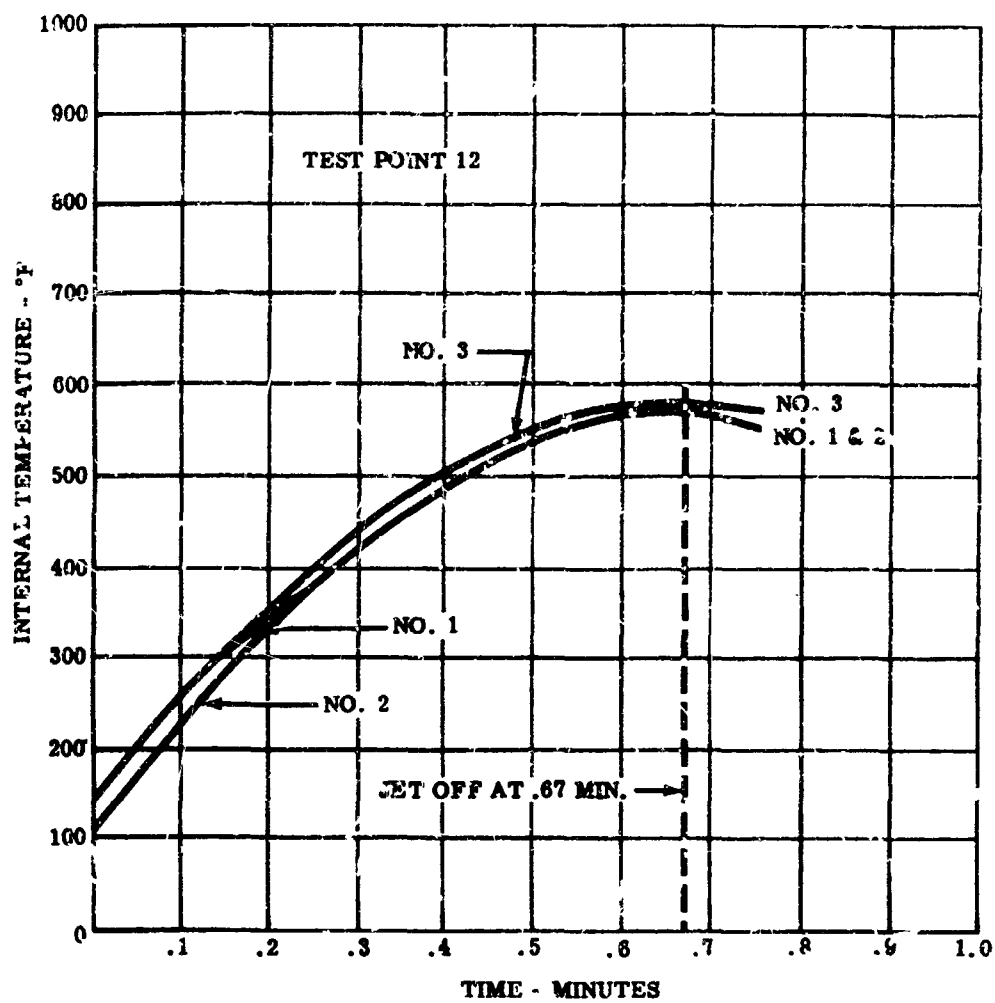


Figure 4-61. Temperature-Time History of Model GG-9

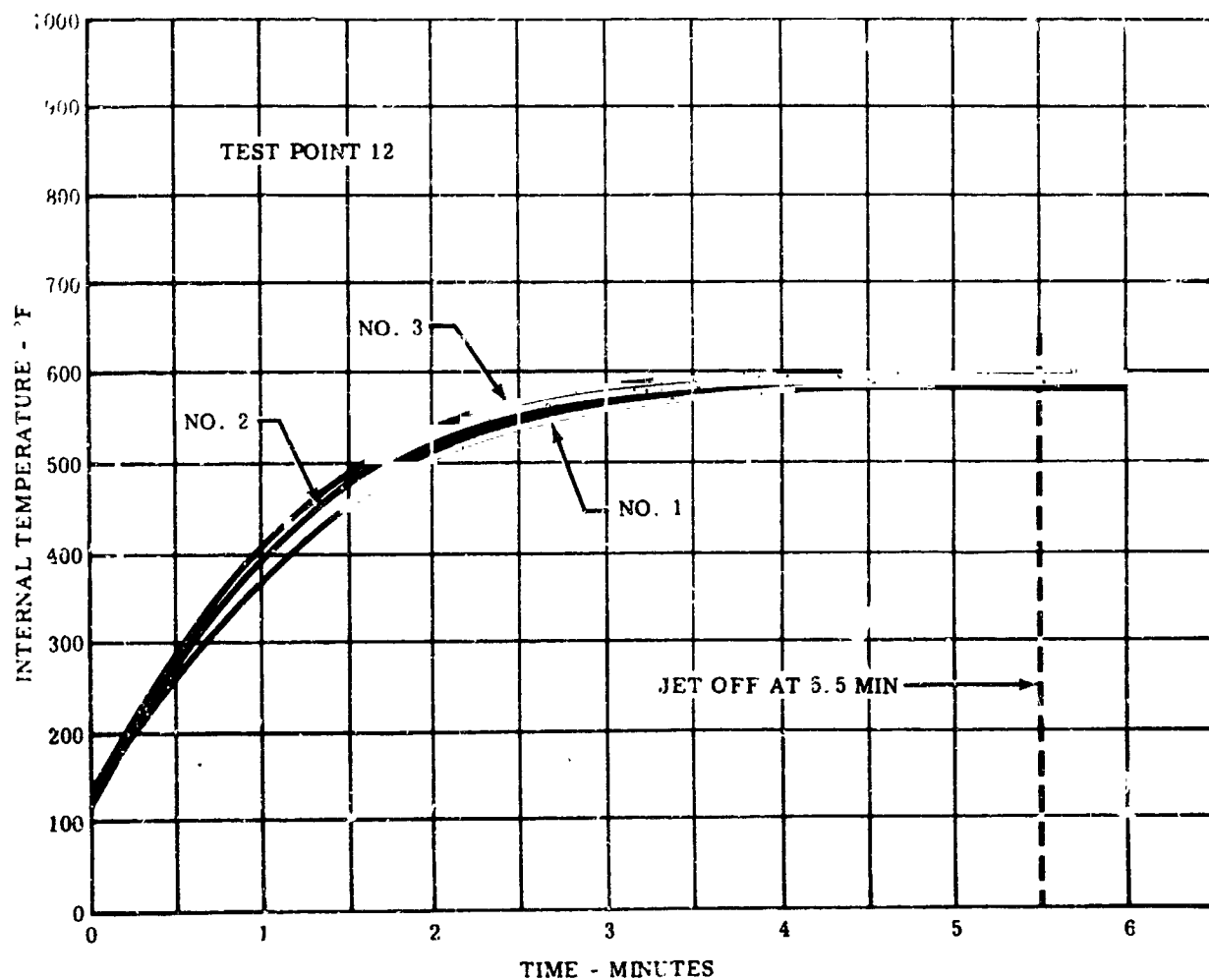


Figure 4-62. Temperature-Time History of Model GG-11

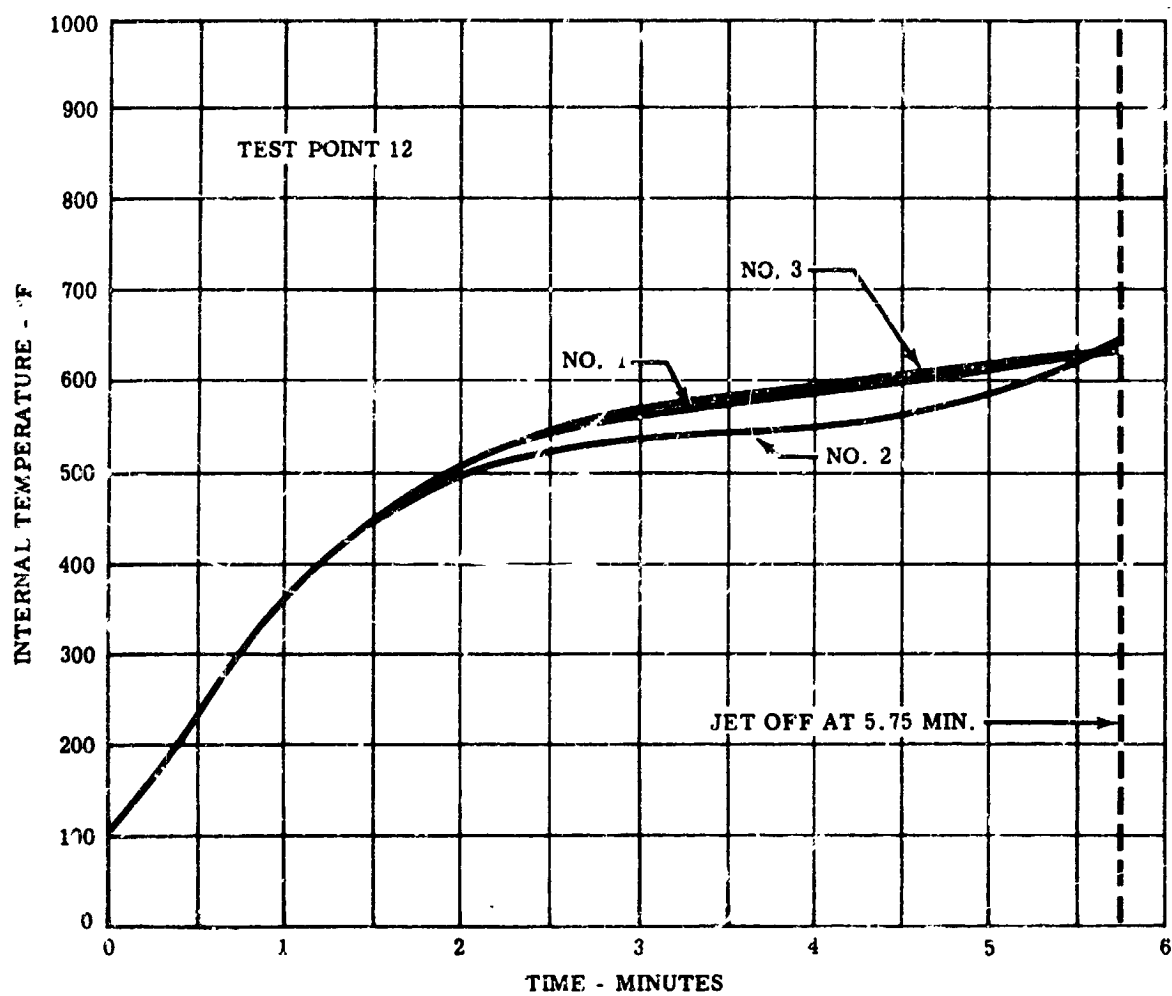


Figure 4-63. Temperature-Time History of Model D-2-4

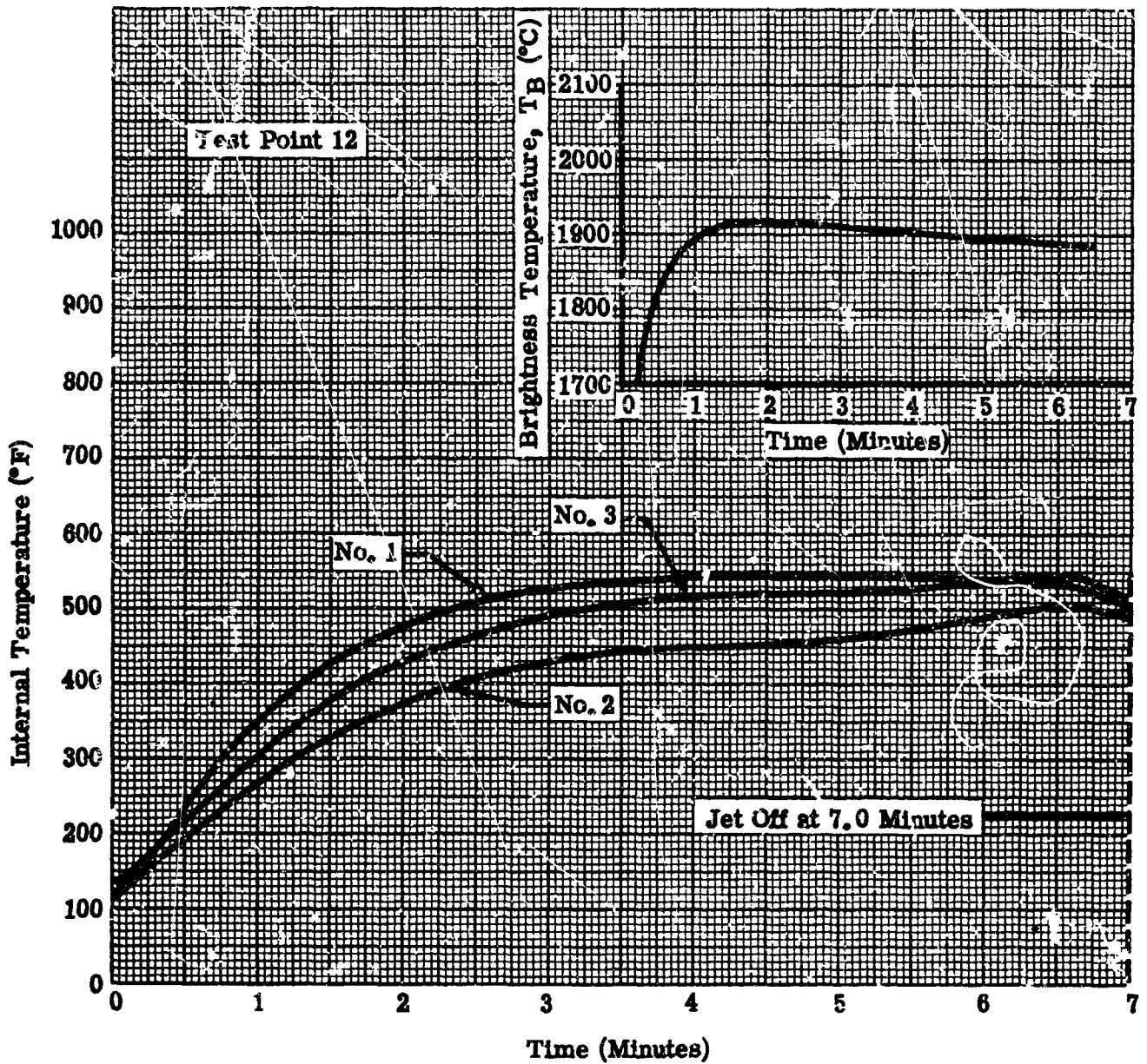


Figure 4-64. Temperature-Time History of Model D-2-5

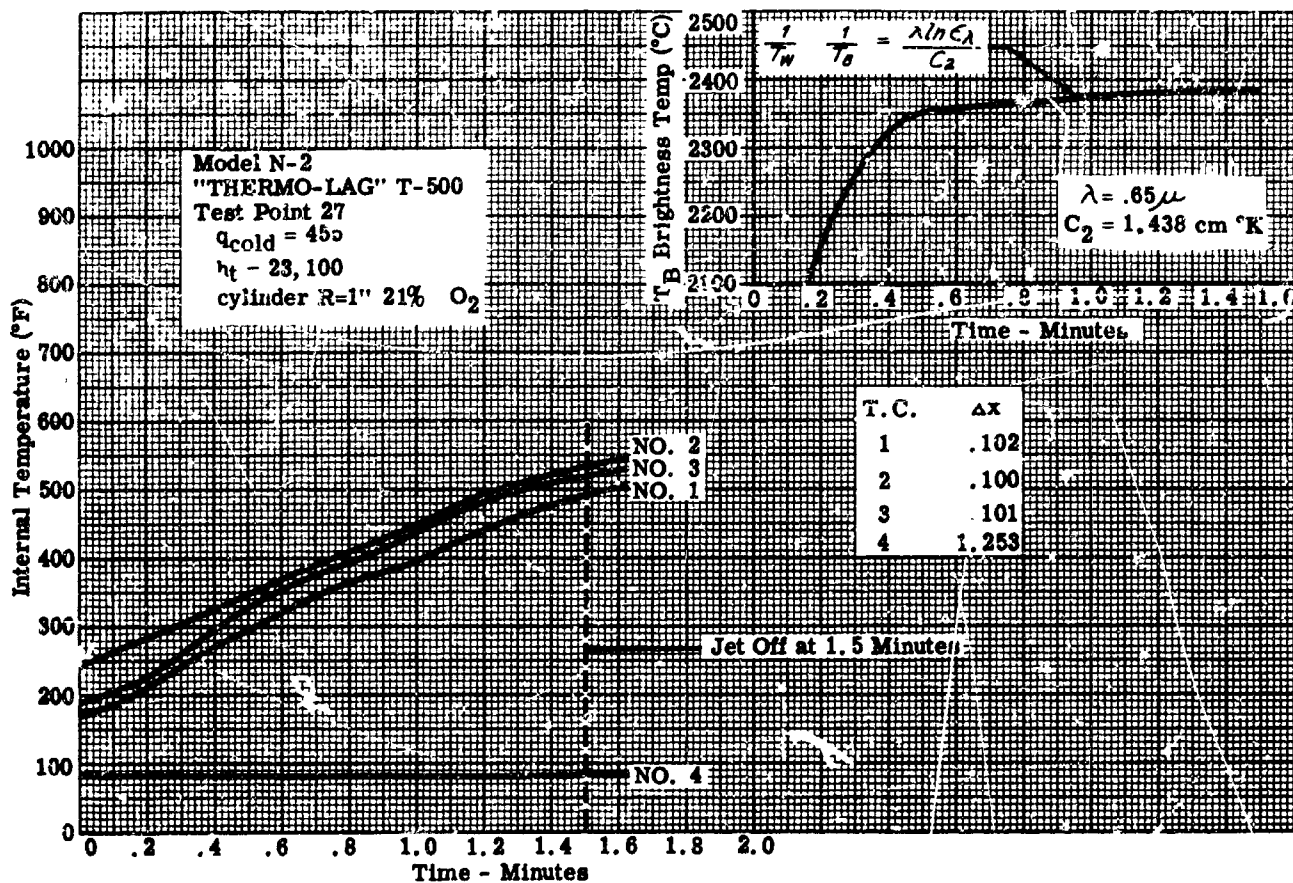


Figure 4-65. Temperature-Time History of Model N-2

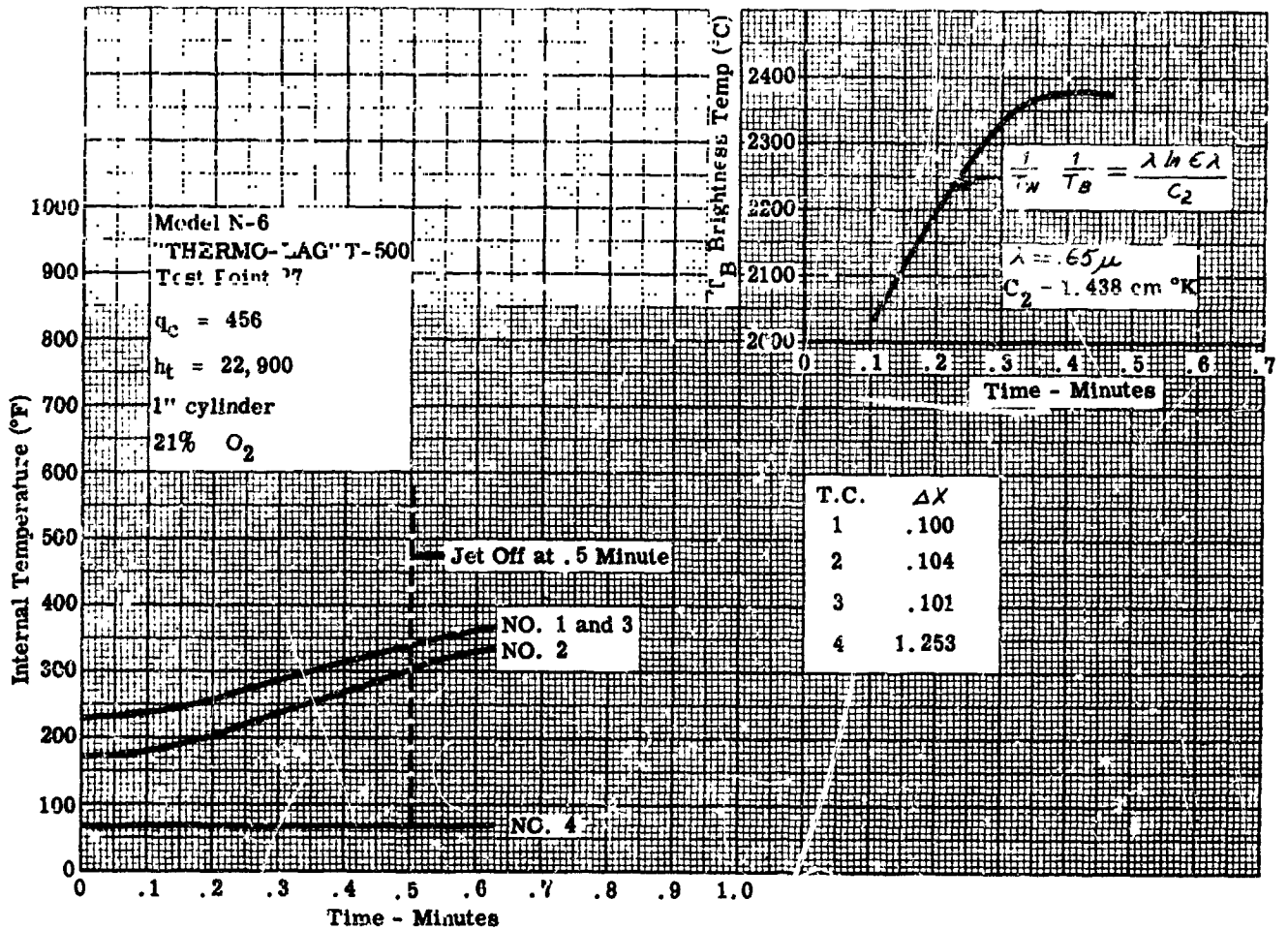


Figure 4-66. Temperature-Time History of Model N-6

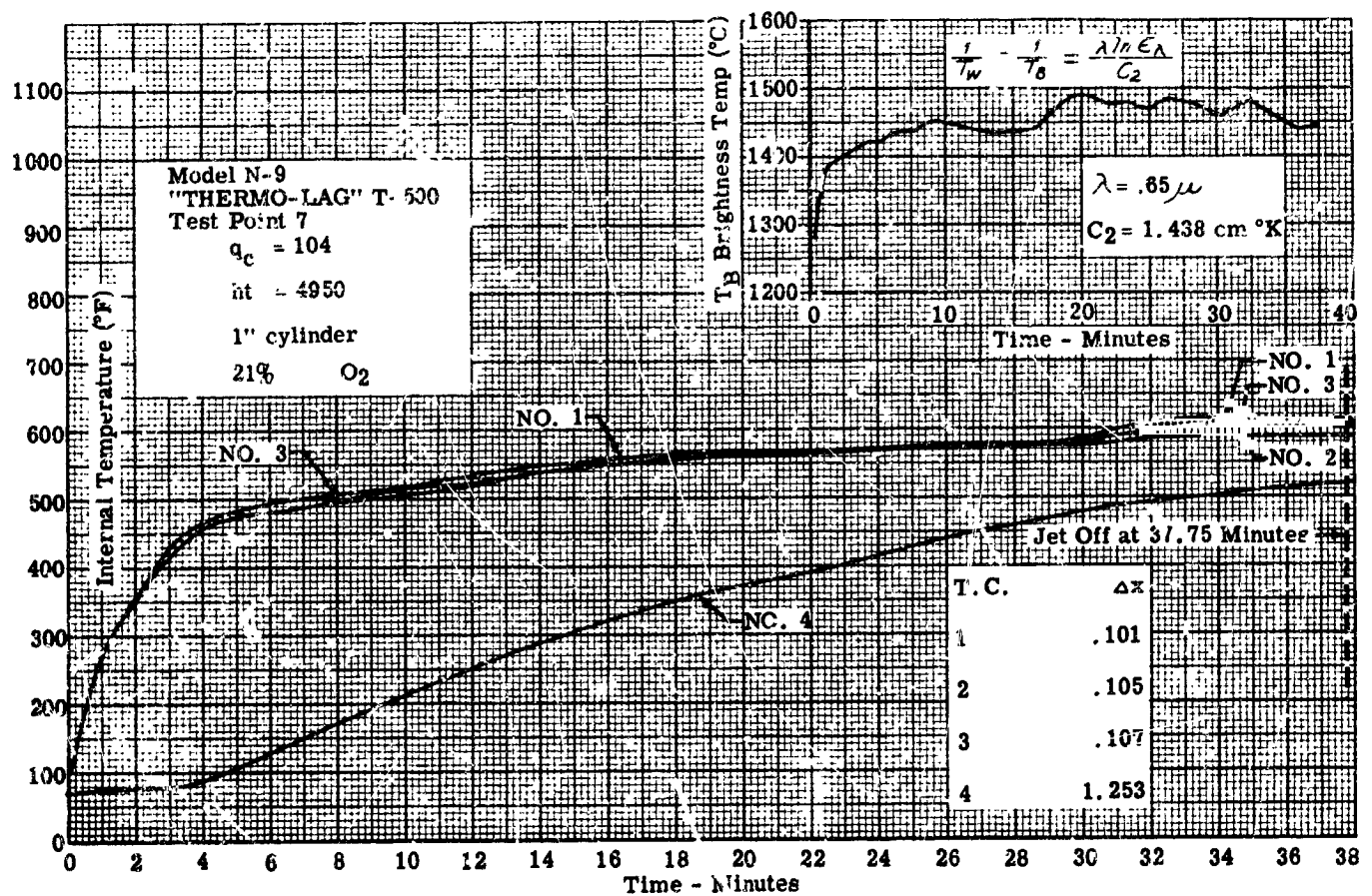


Figure 4-67. Temperature-Time History of Model N-9

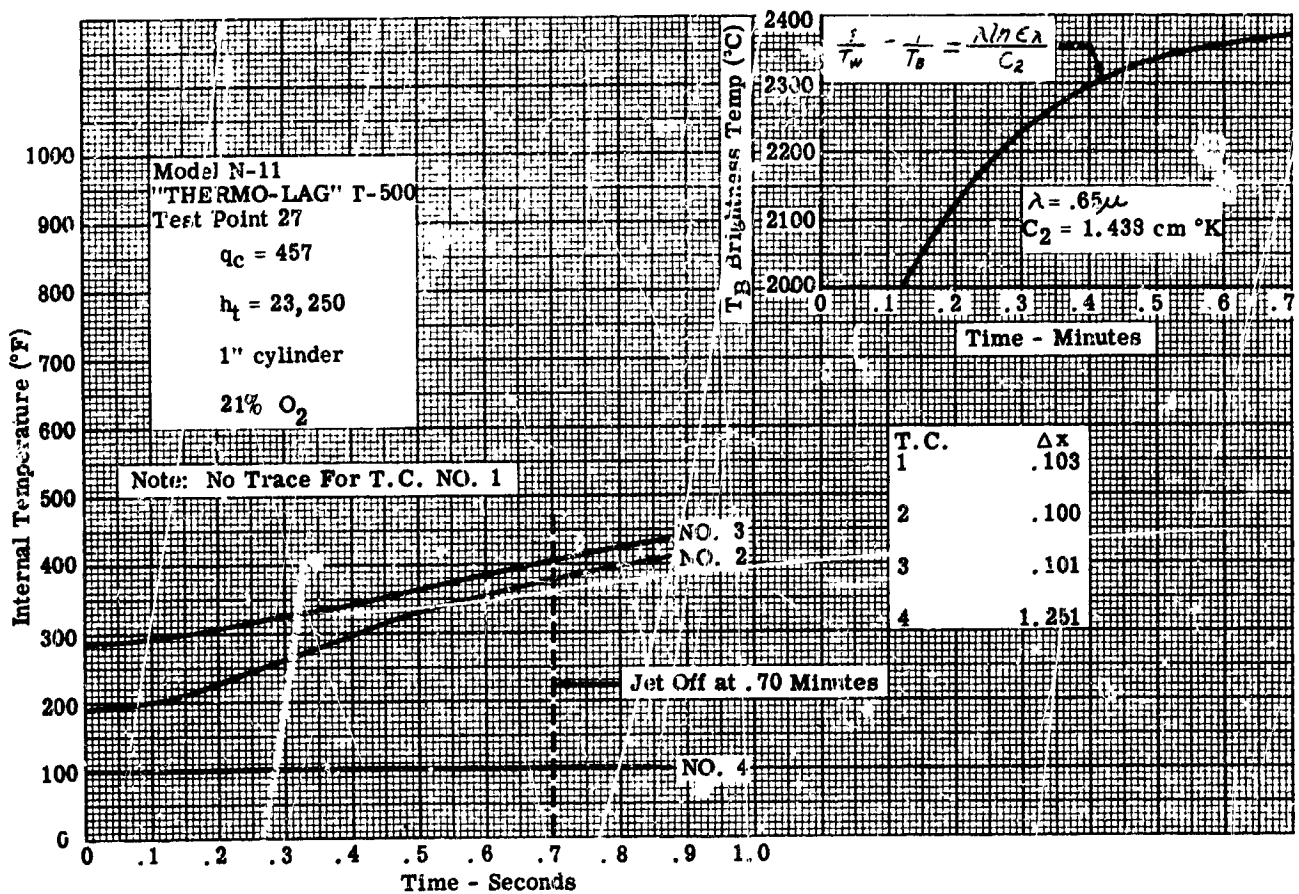
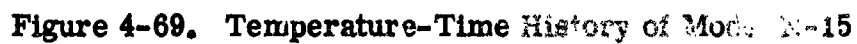


Figure 4-68. Temperature-Time History of Model N-11



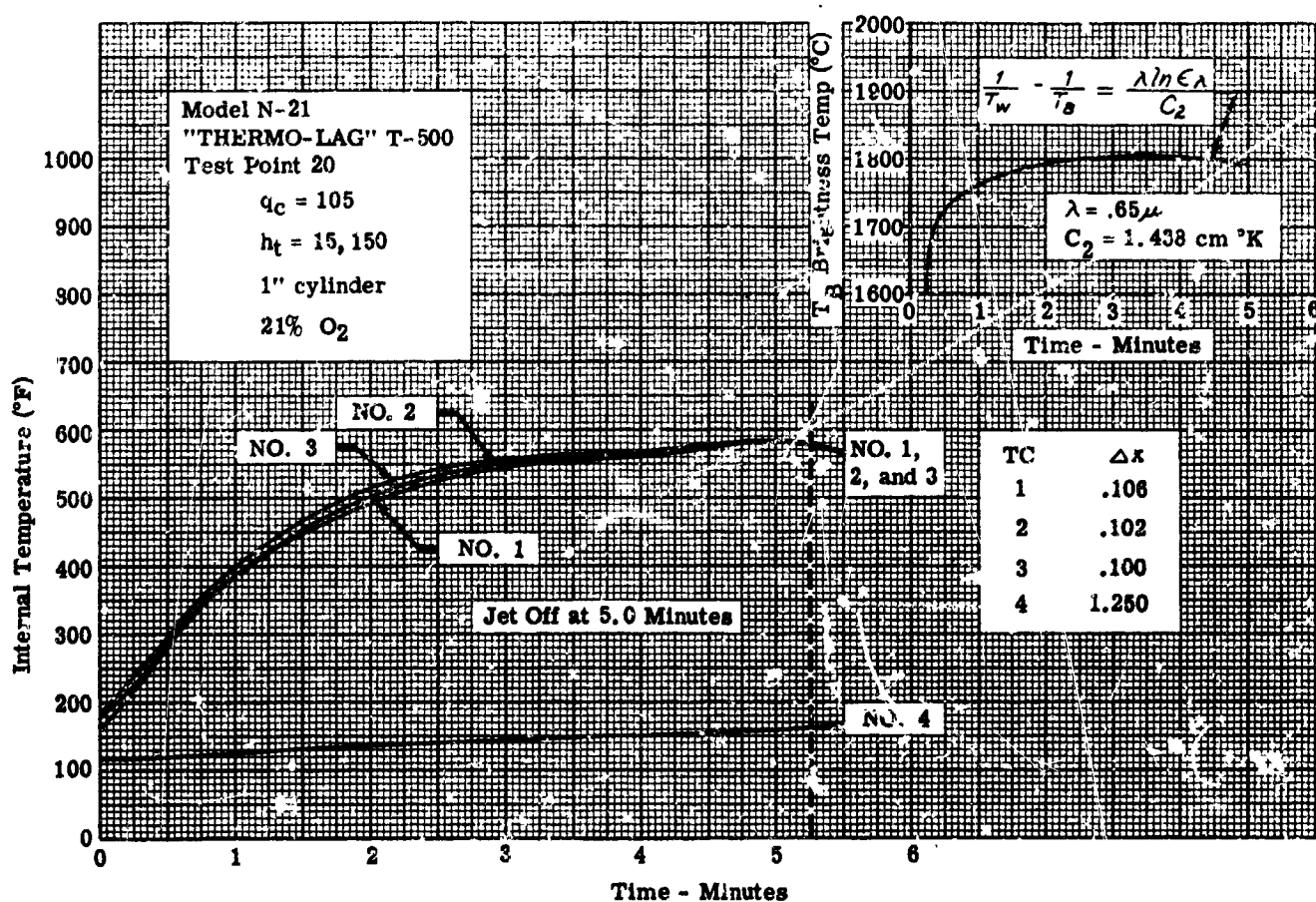


Figure 4-70. Temperature-Time History of Model N-21

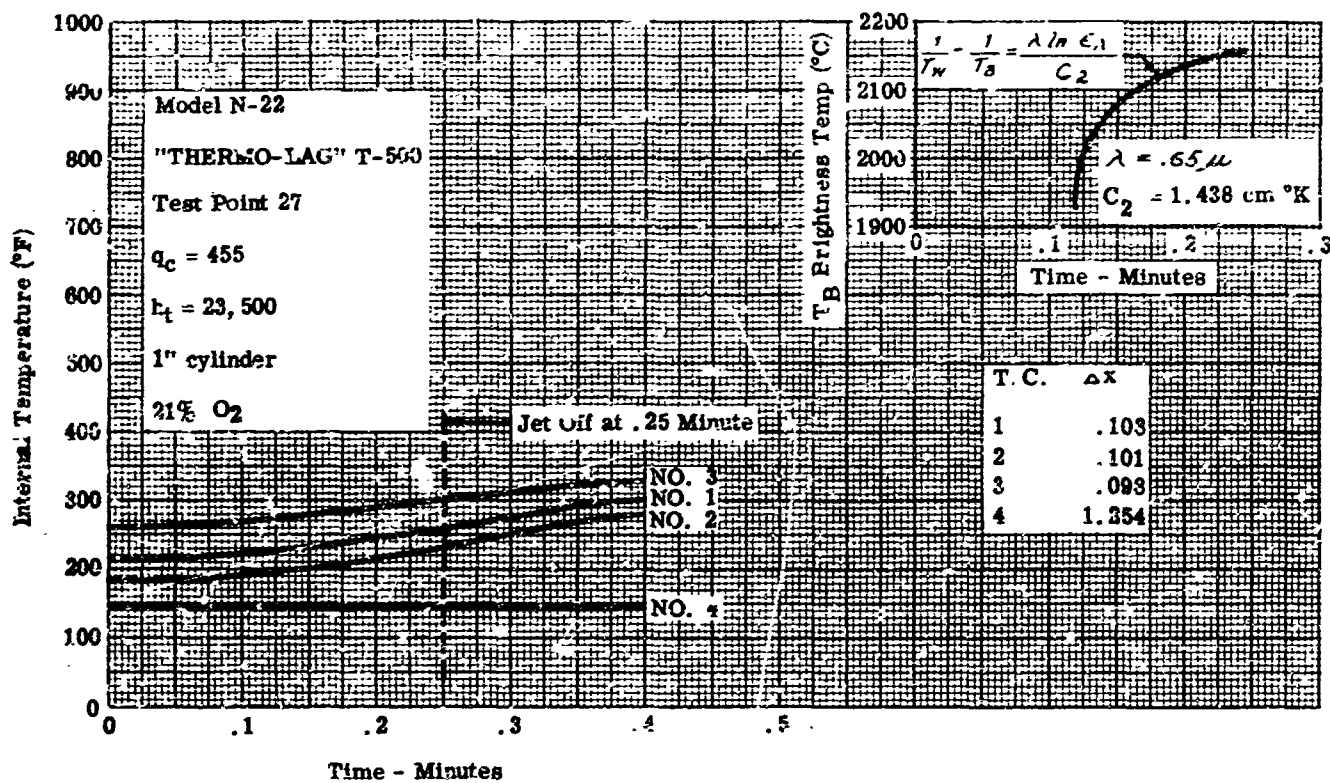


Figure 4-71. Temperature-Time History of Model N-22

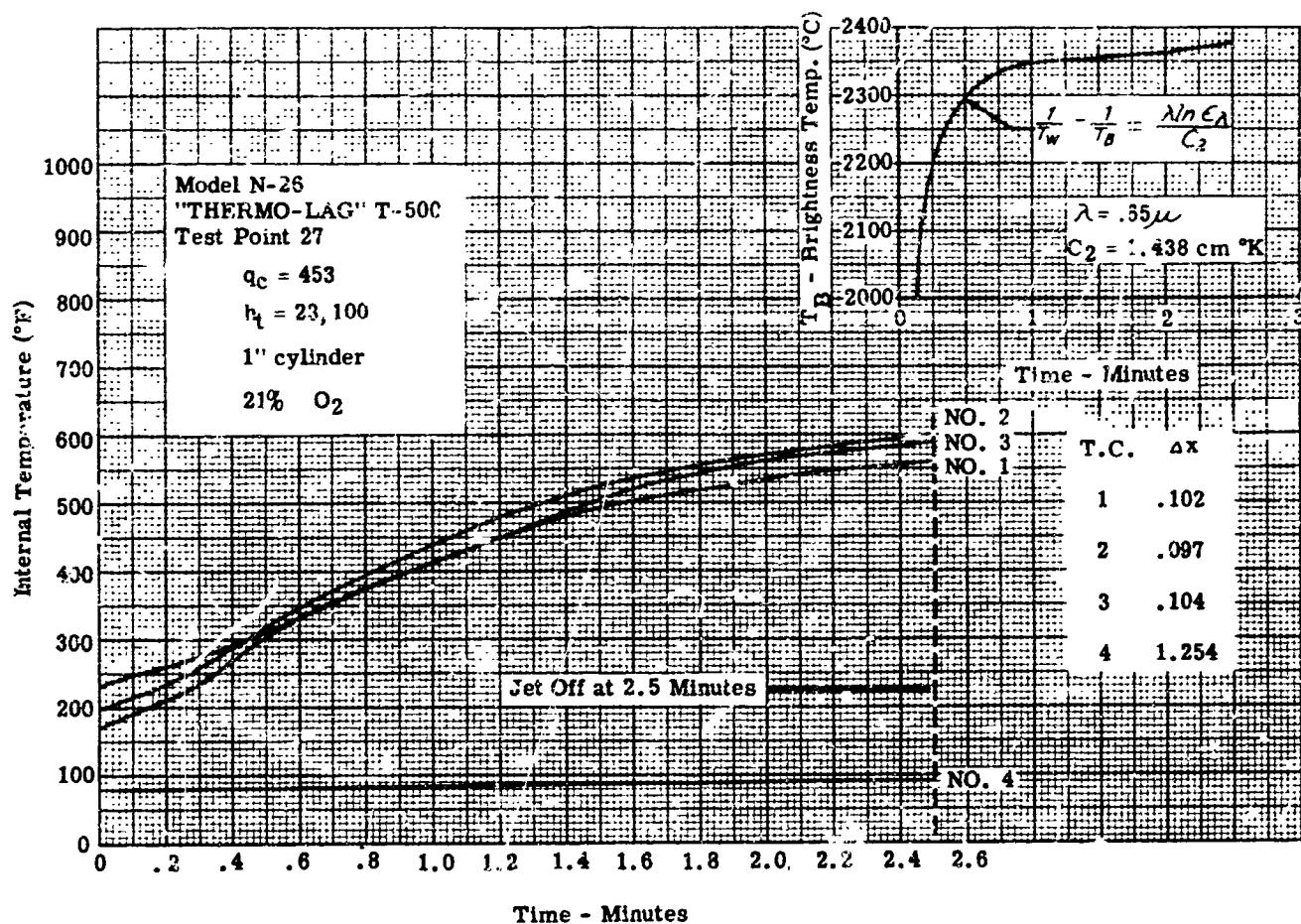


Figure 4-72. Temperature-Time History of Model N-26

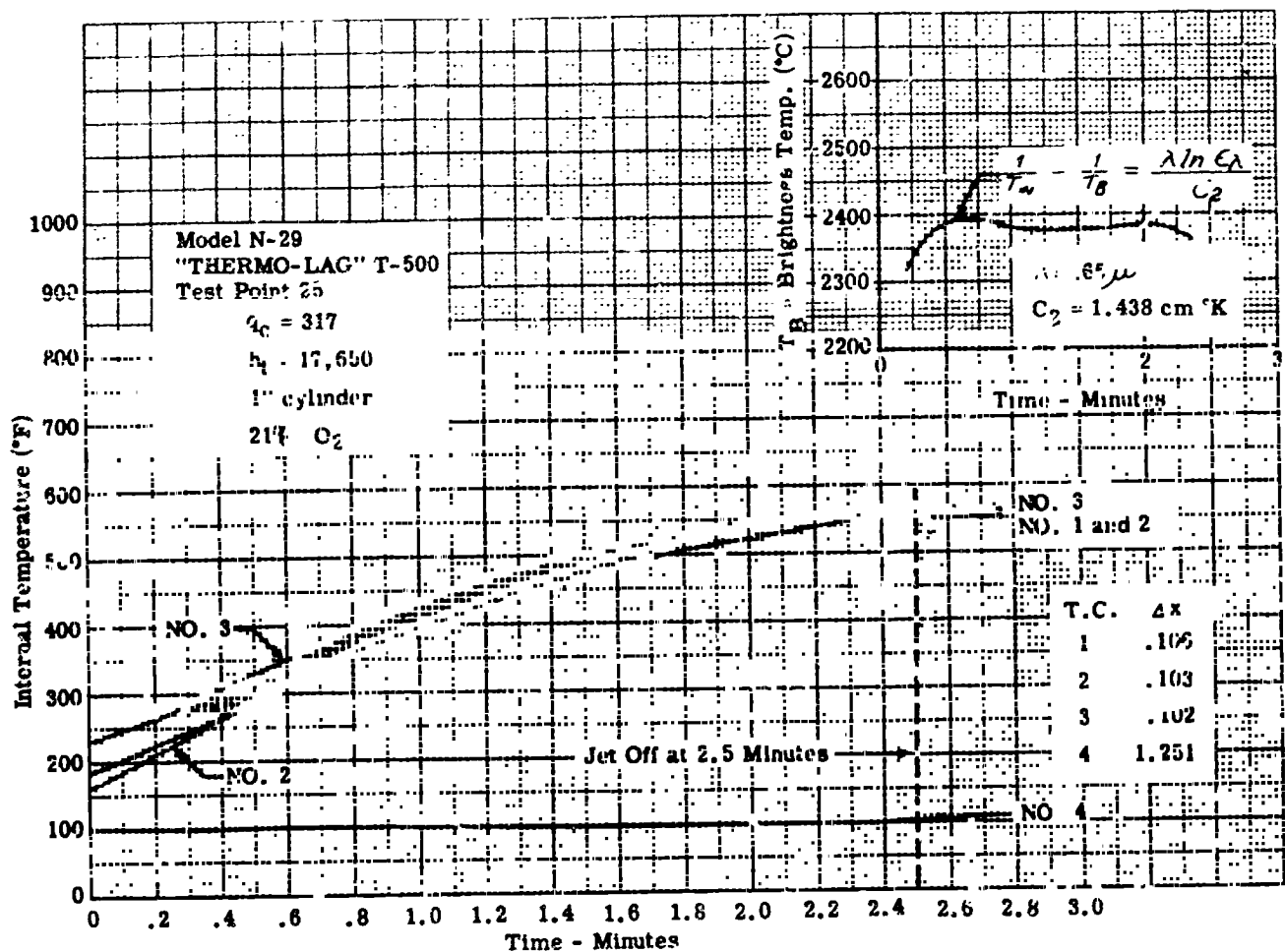


Figure 4-73. Temperature-Time History of Model N-29

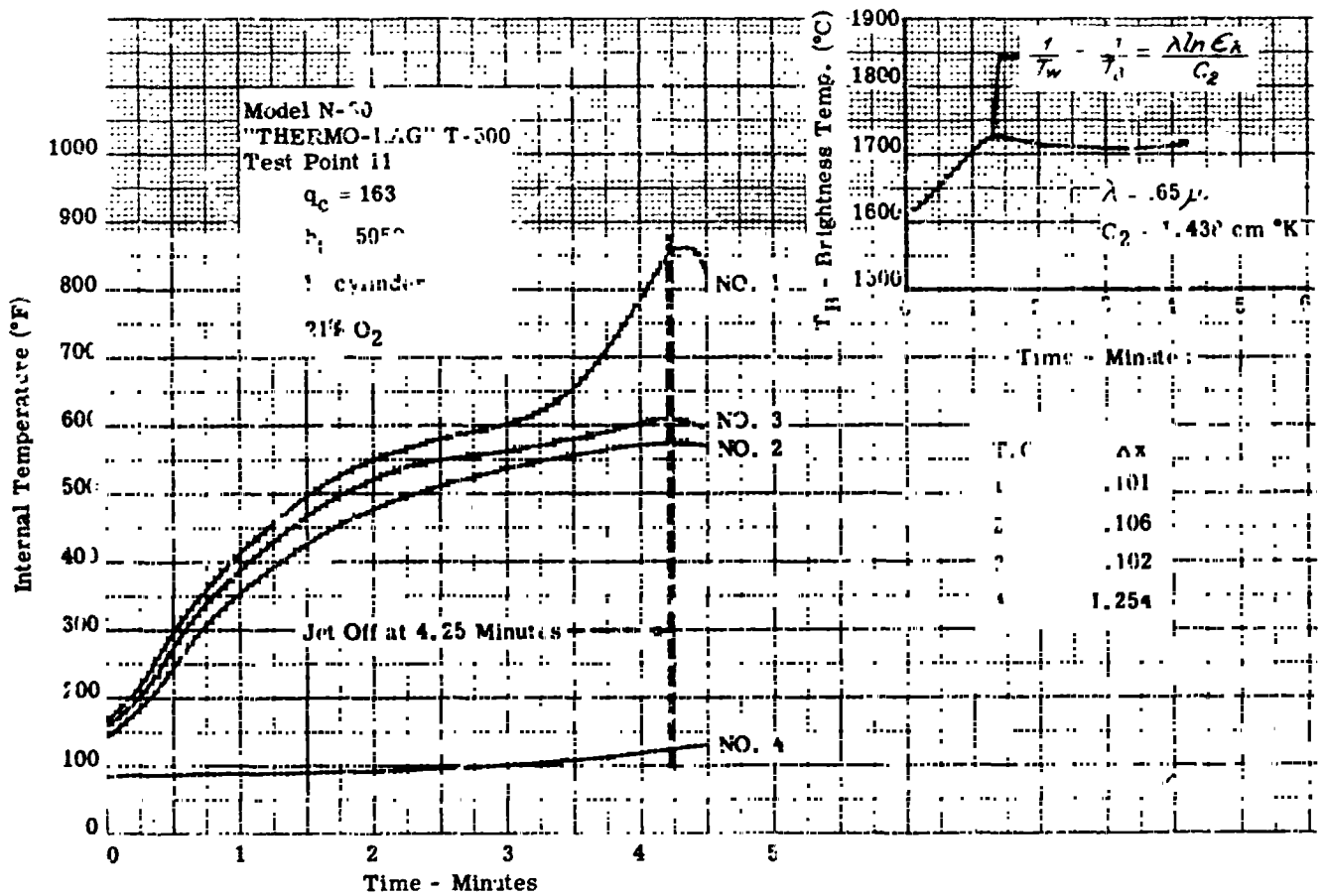


Figure 4-74. Temperature-Time History of Model N-30

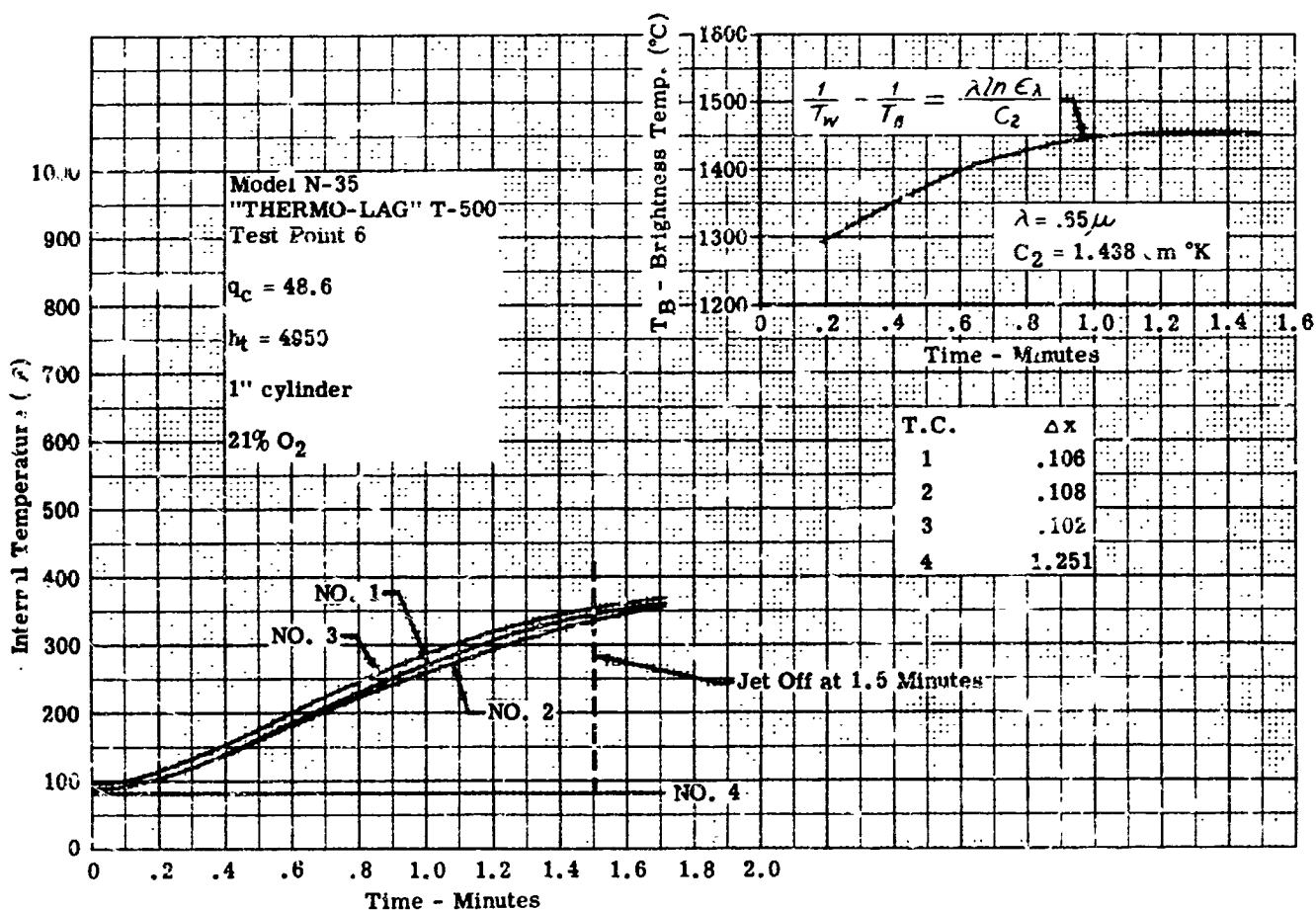


Figure 4-75. Temperature-Time History of Model N-35

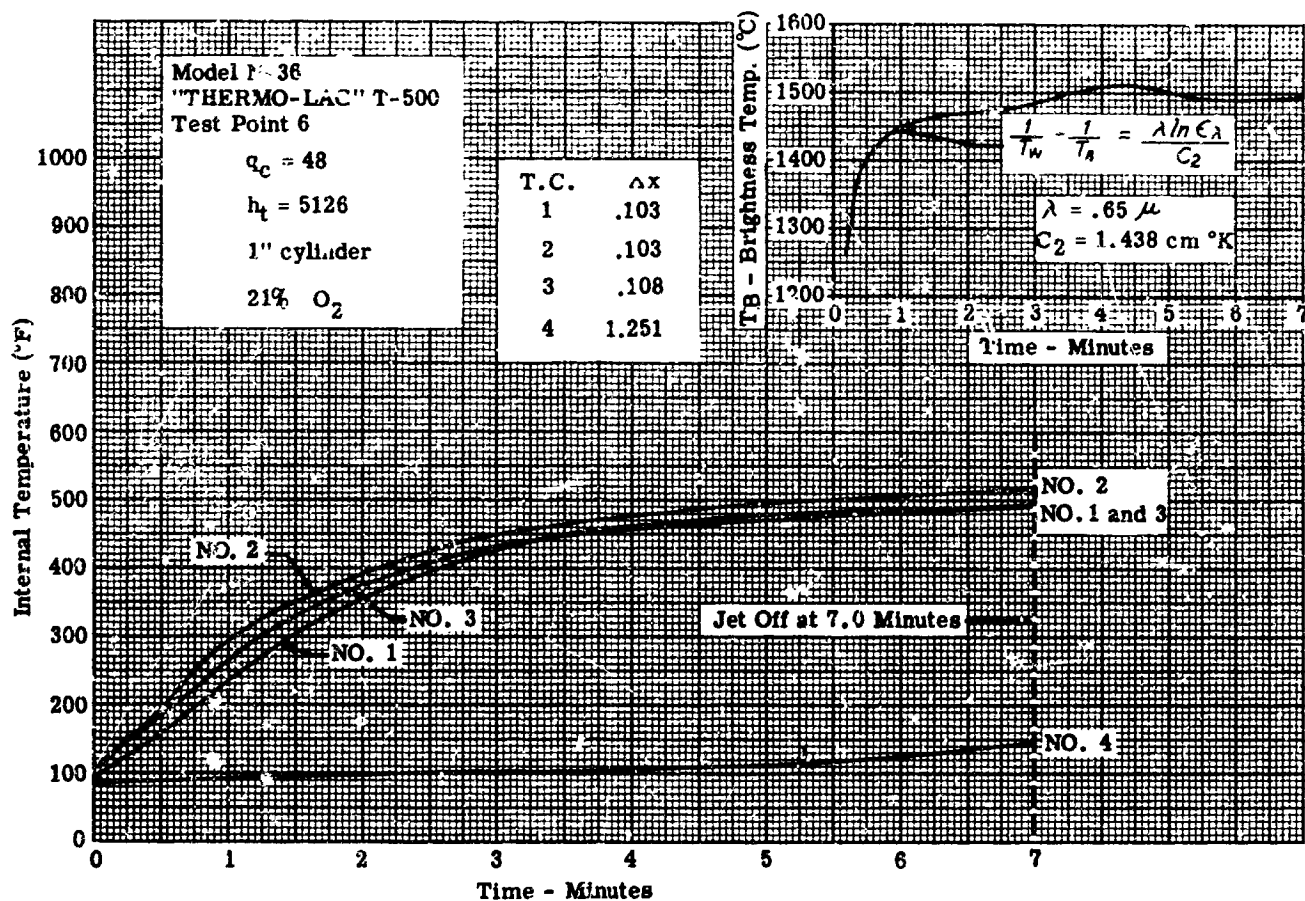


Figure 4-76. Temperature-Time History of Model N-36

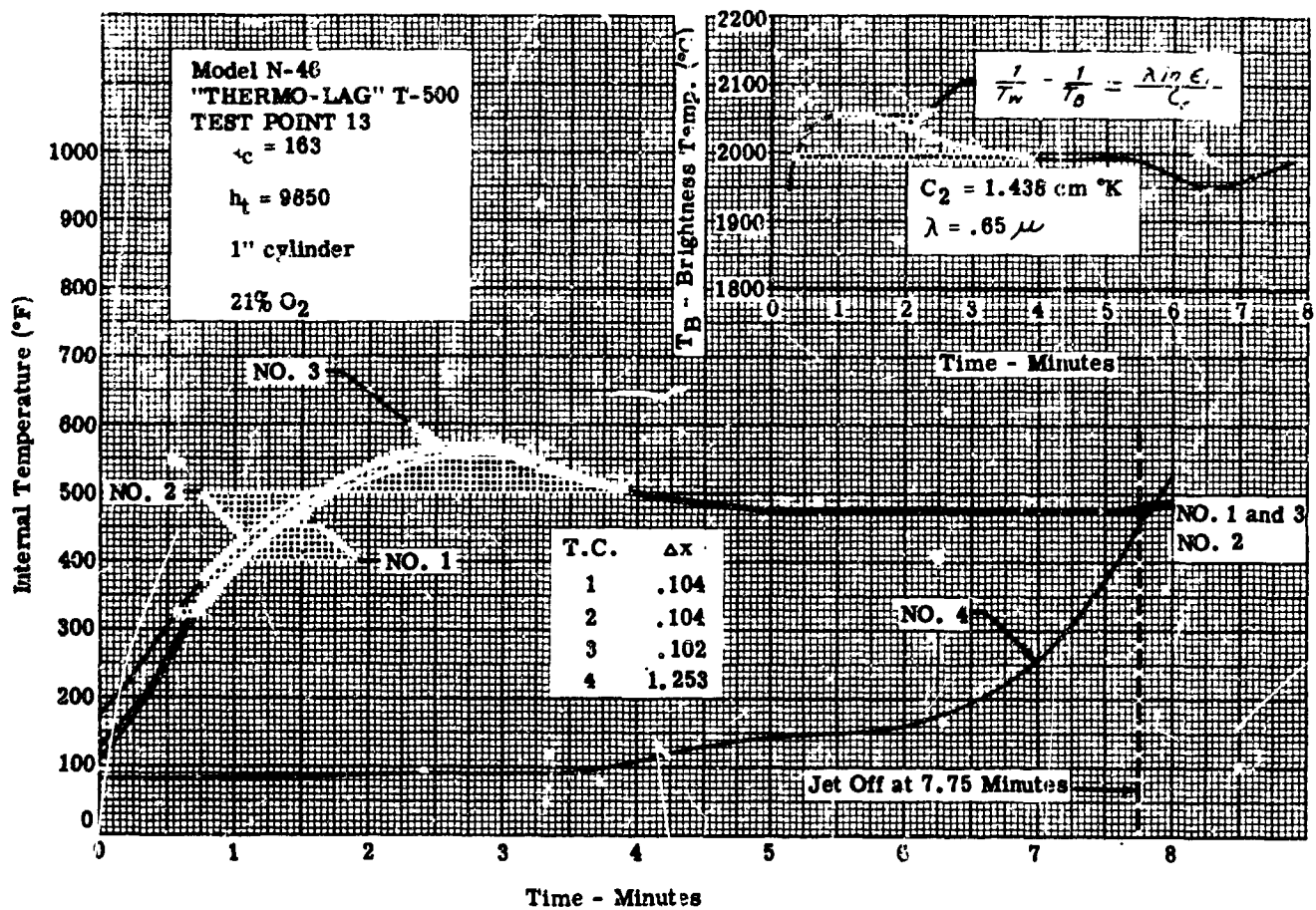


Figure 4-77. Temperature-Time History of Model N-46

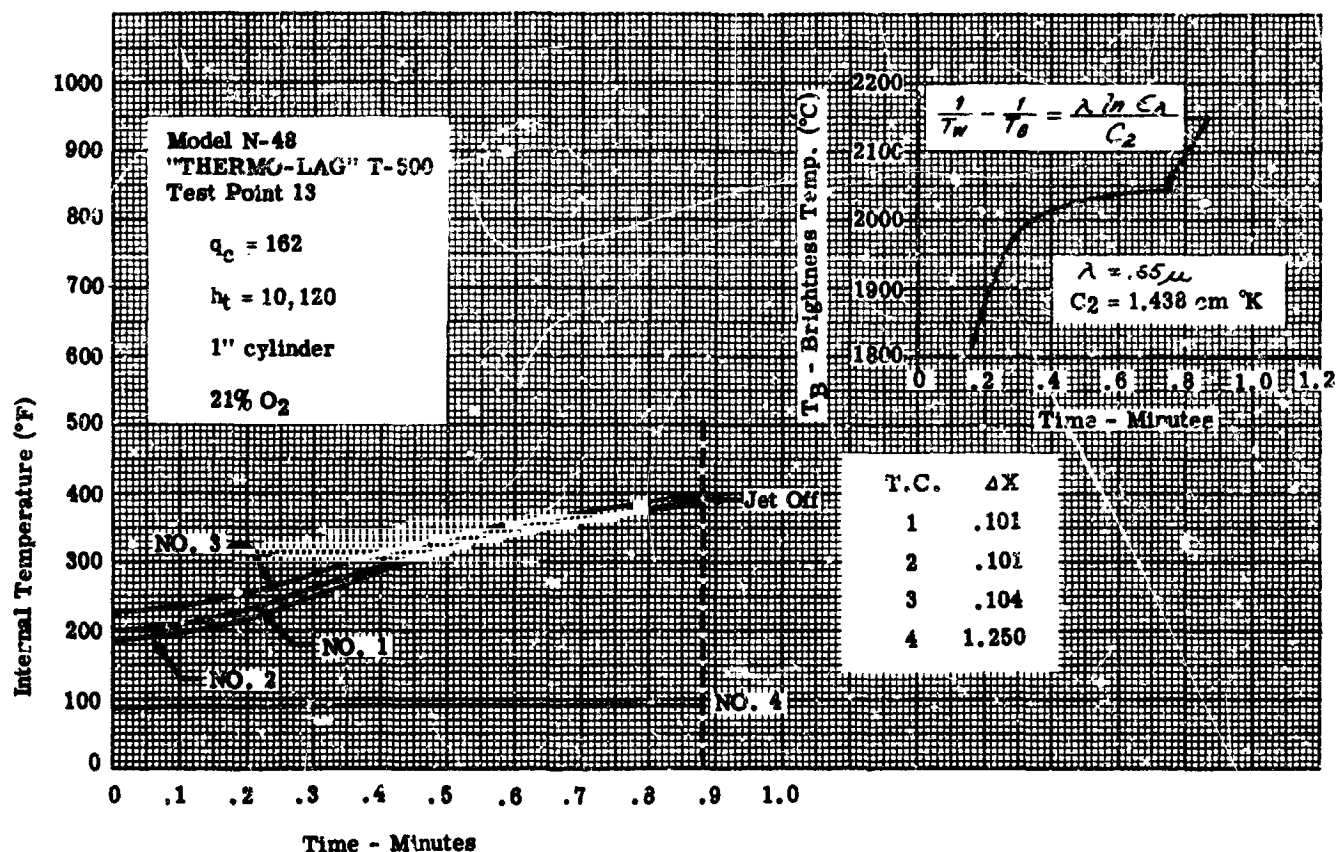


Figure 4-78. Temperature-Time History of Model N-48

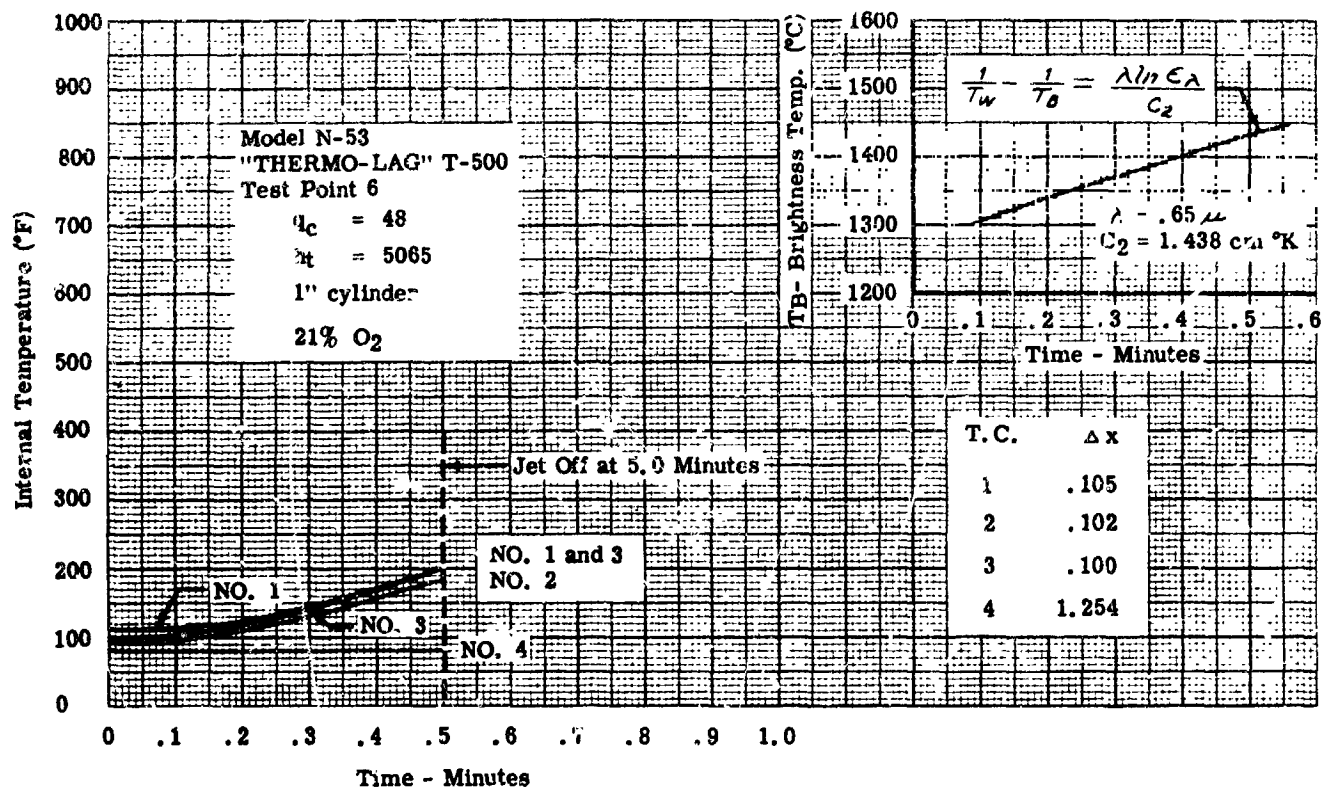


Figure 4-79. Temperature-Time History of Model N-53

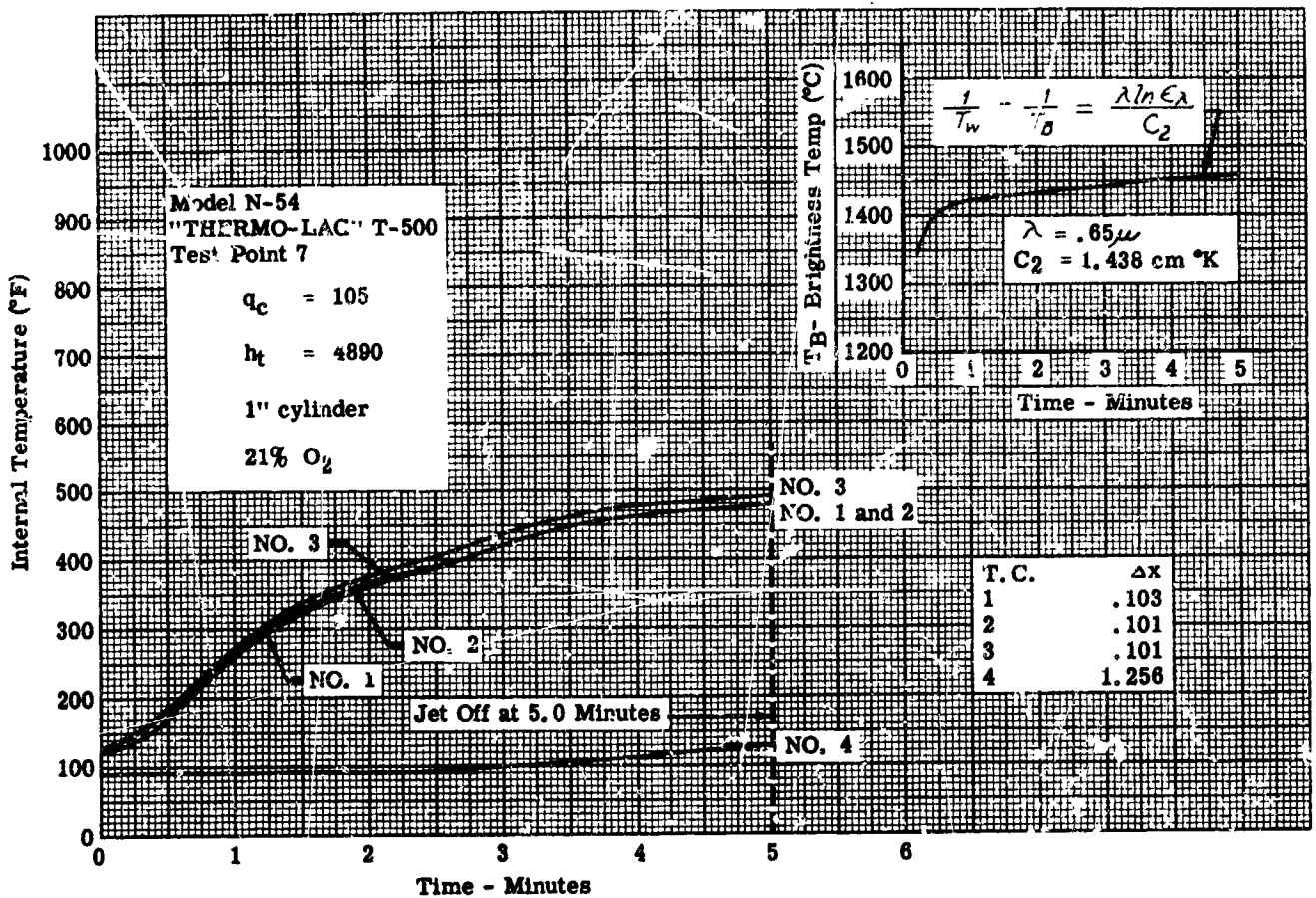


Figure 4-80. Temperature-Time History of Model N-54

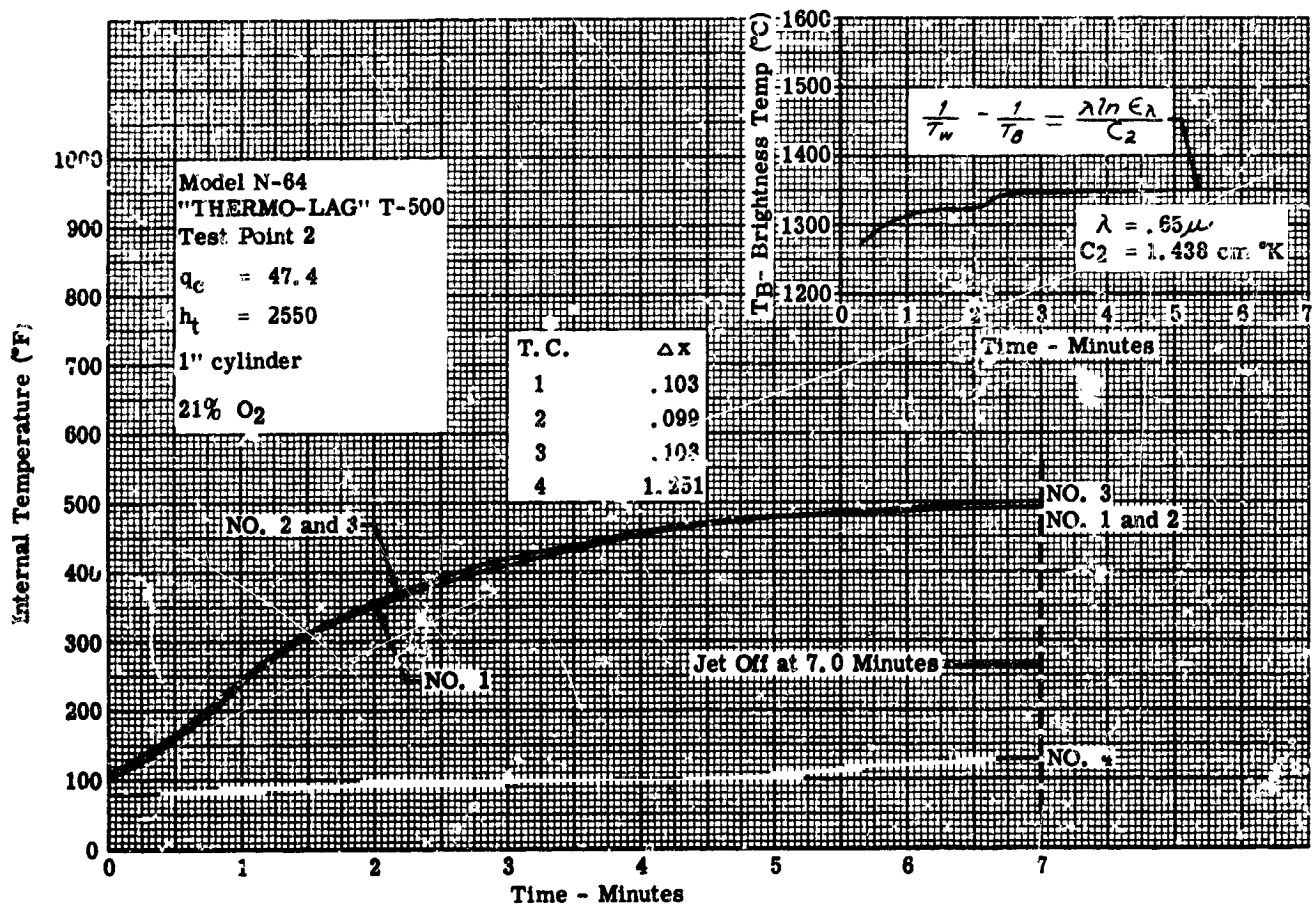


Figure 4-31. Temperature-Time History of Model N-64

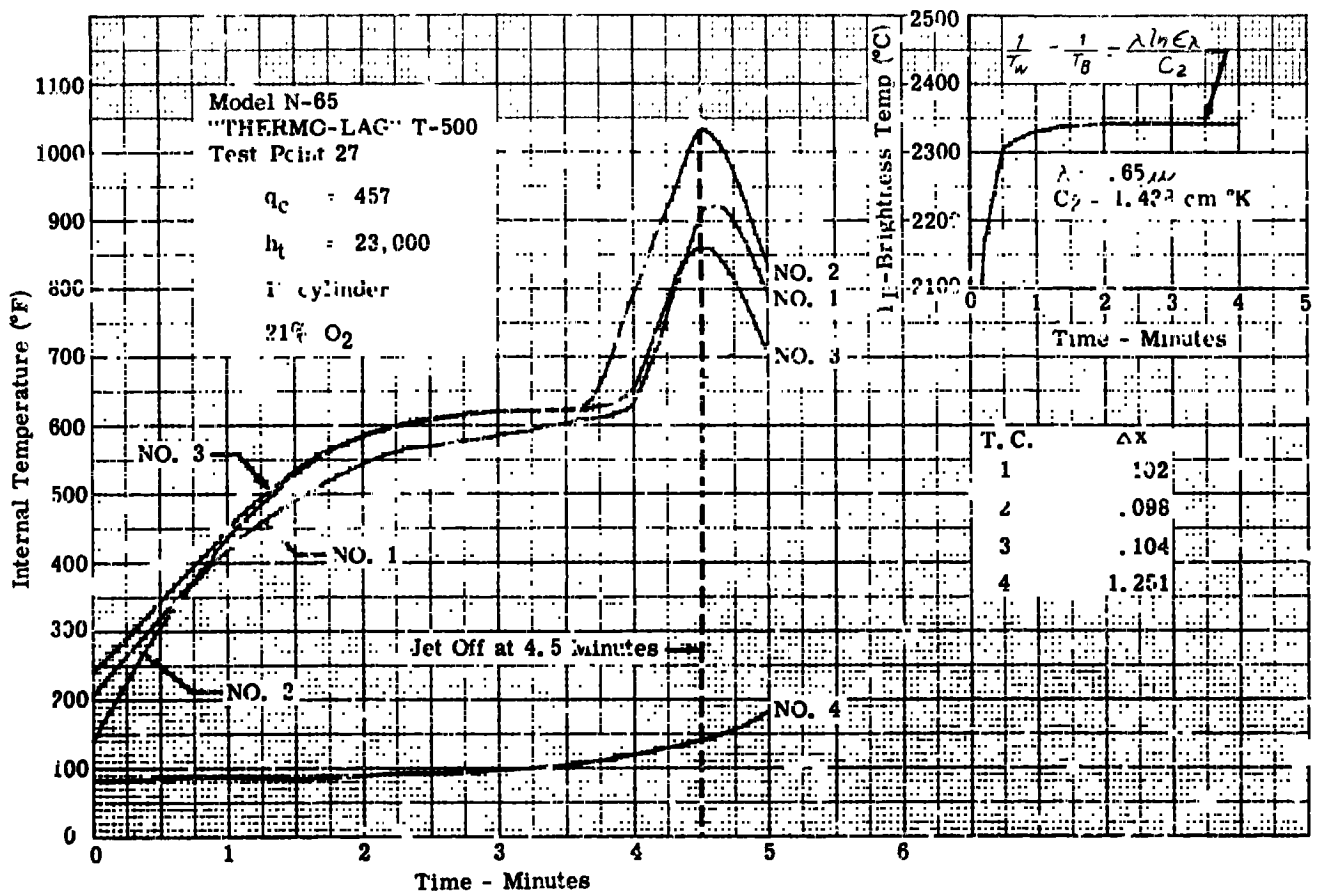


Figure 4-82. Temperature-Time History of Model N-65

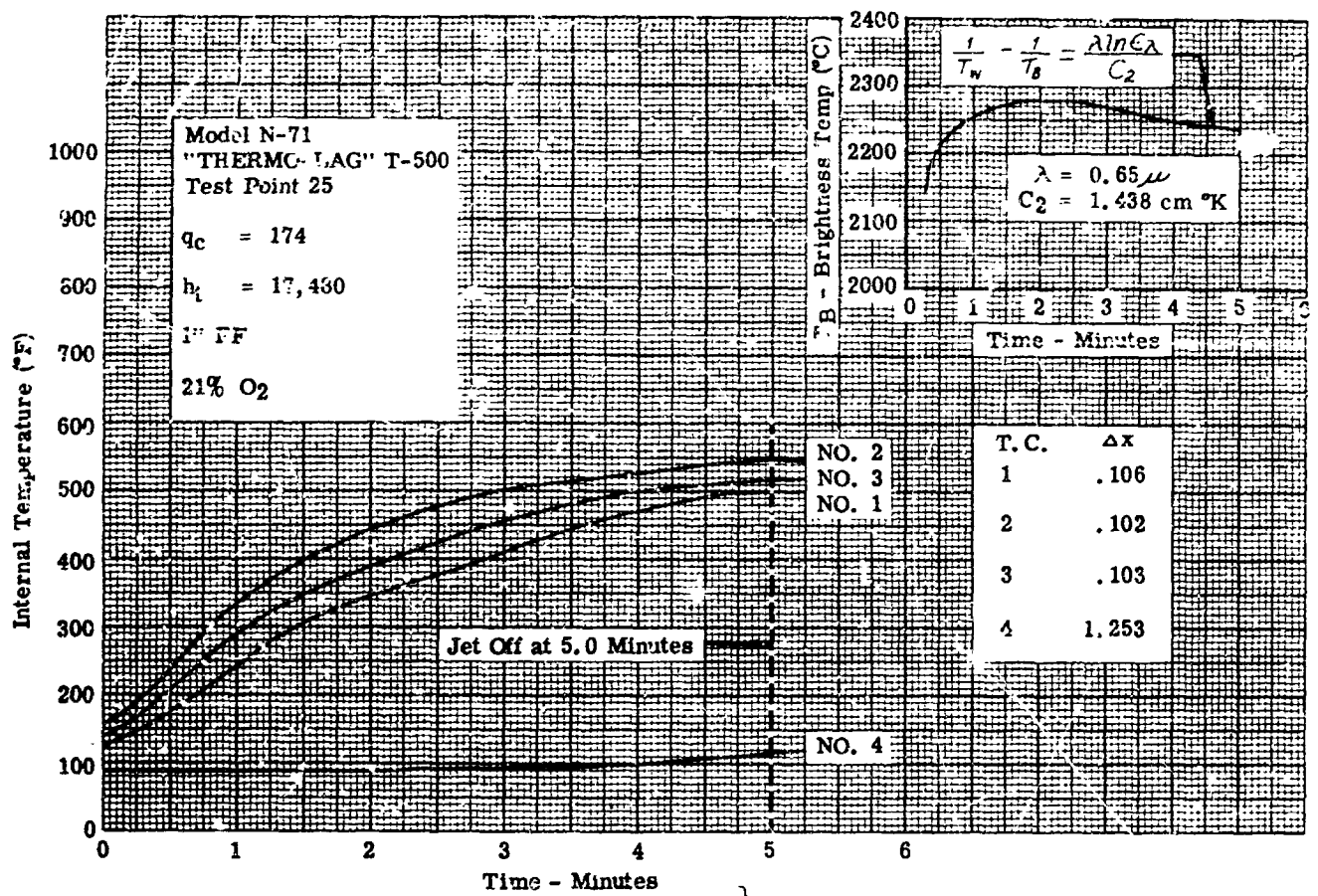


Figure 4-83. Temperature-Time History of Model N-71

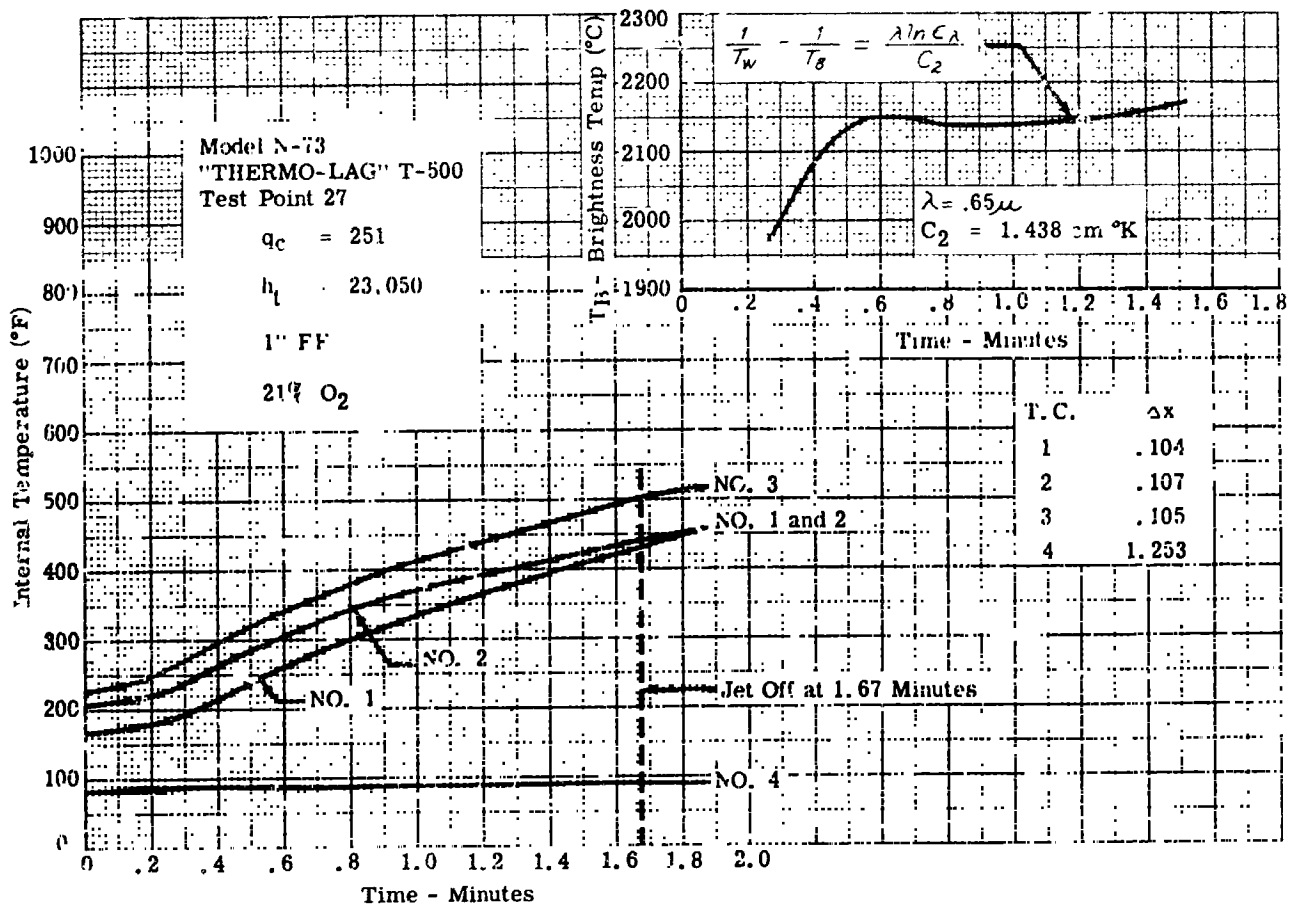


Figure 4-84. Temperature-Time History of Model N-73

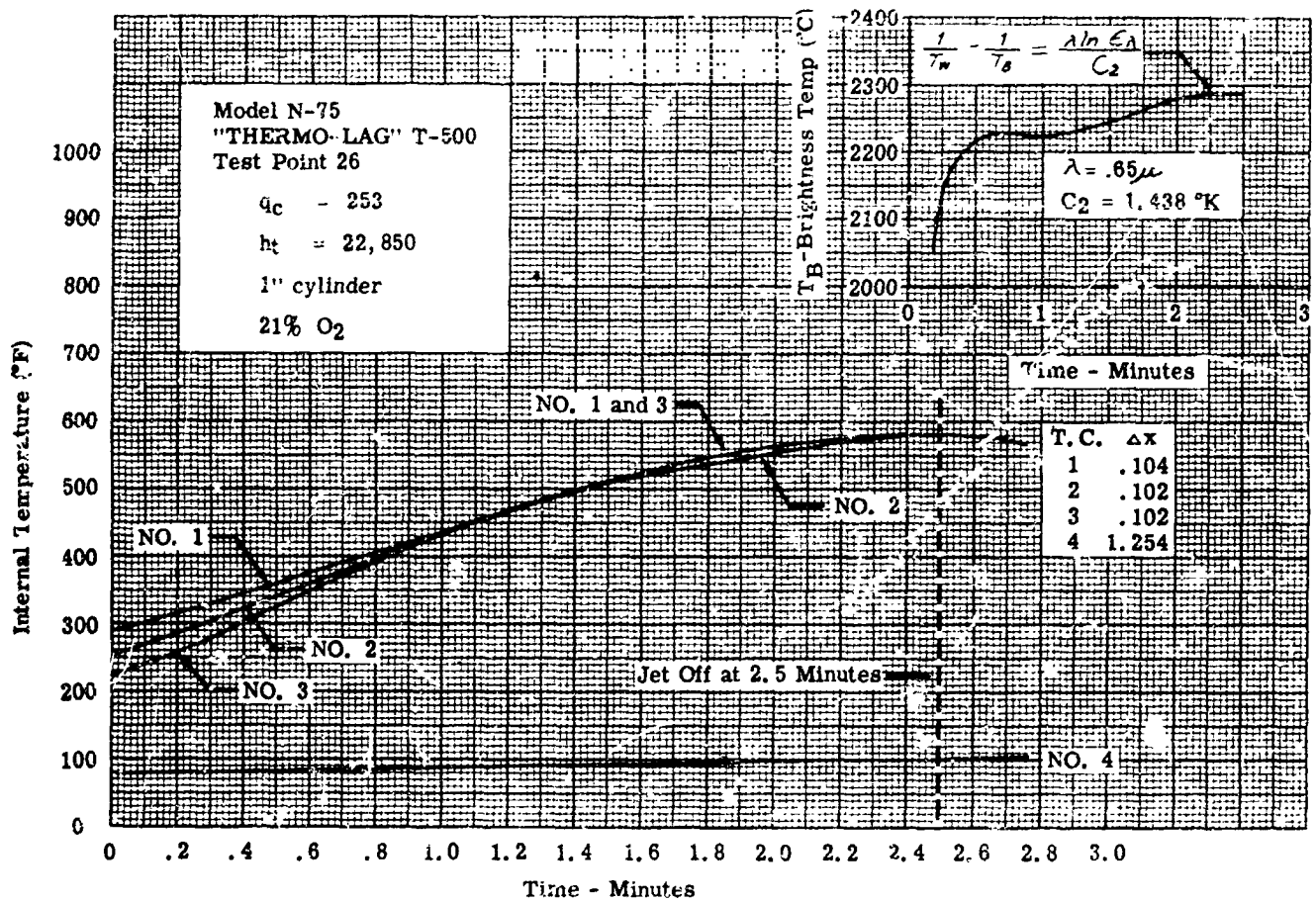


Figure 4-85. Temperature-Time History of Model N-75

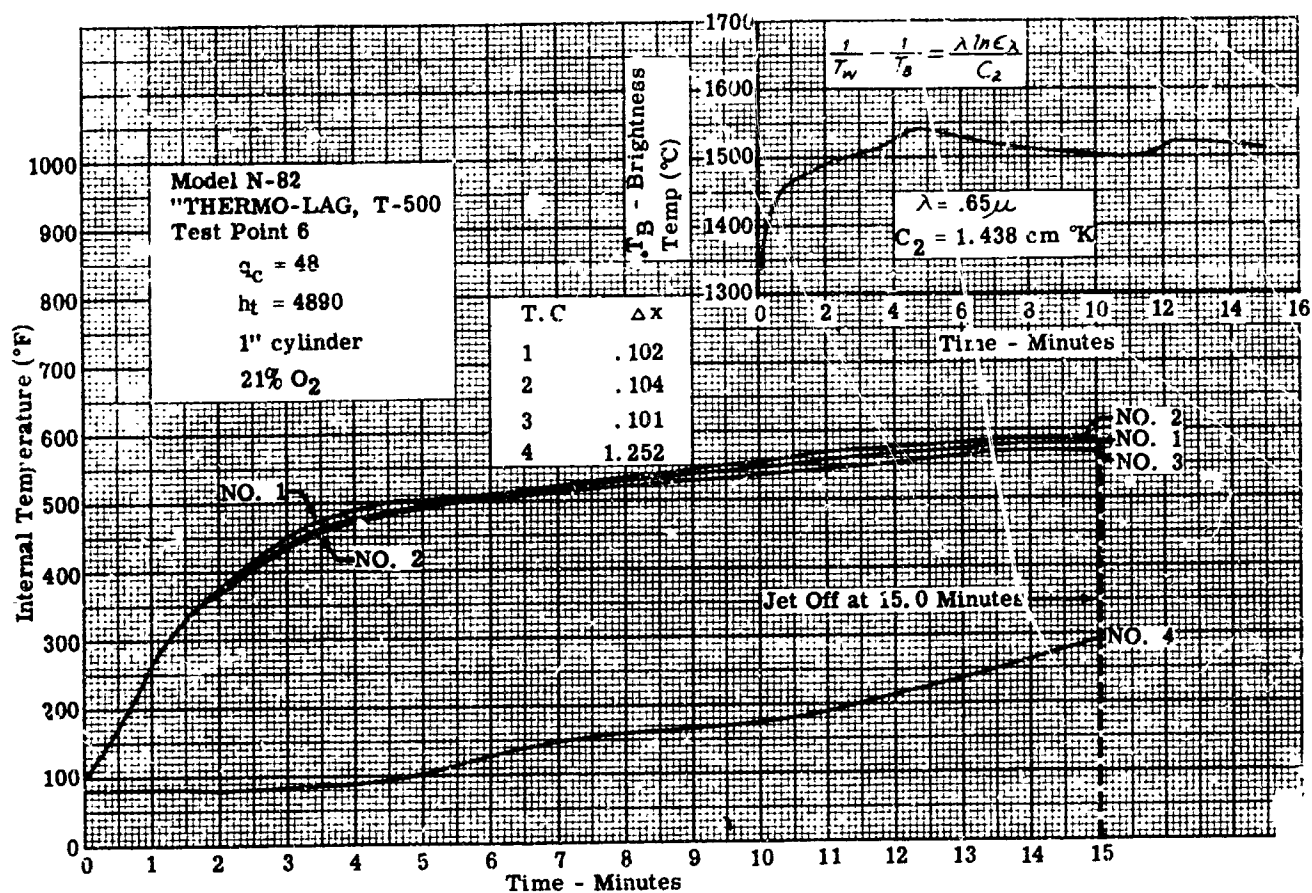


Figure 4-86. Temperature-Time History of Model N-82

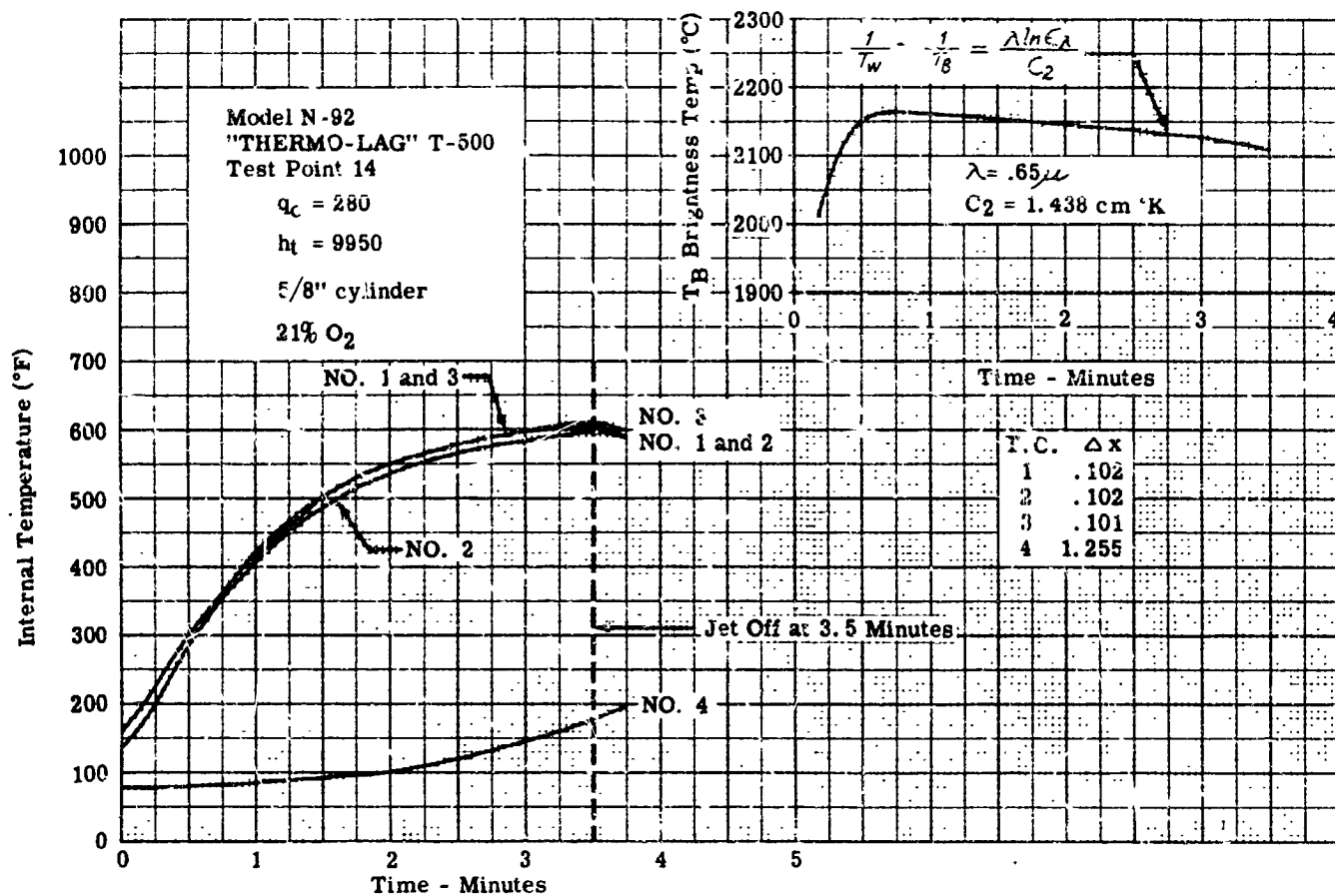


Figure 4-87. Temperature-Time History of Model N-92

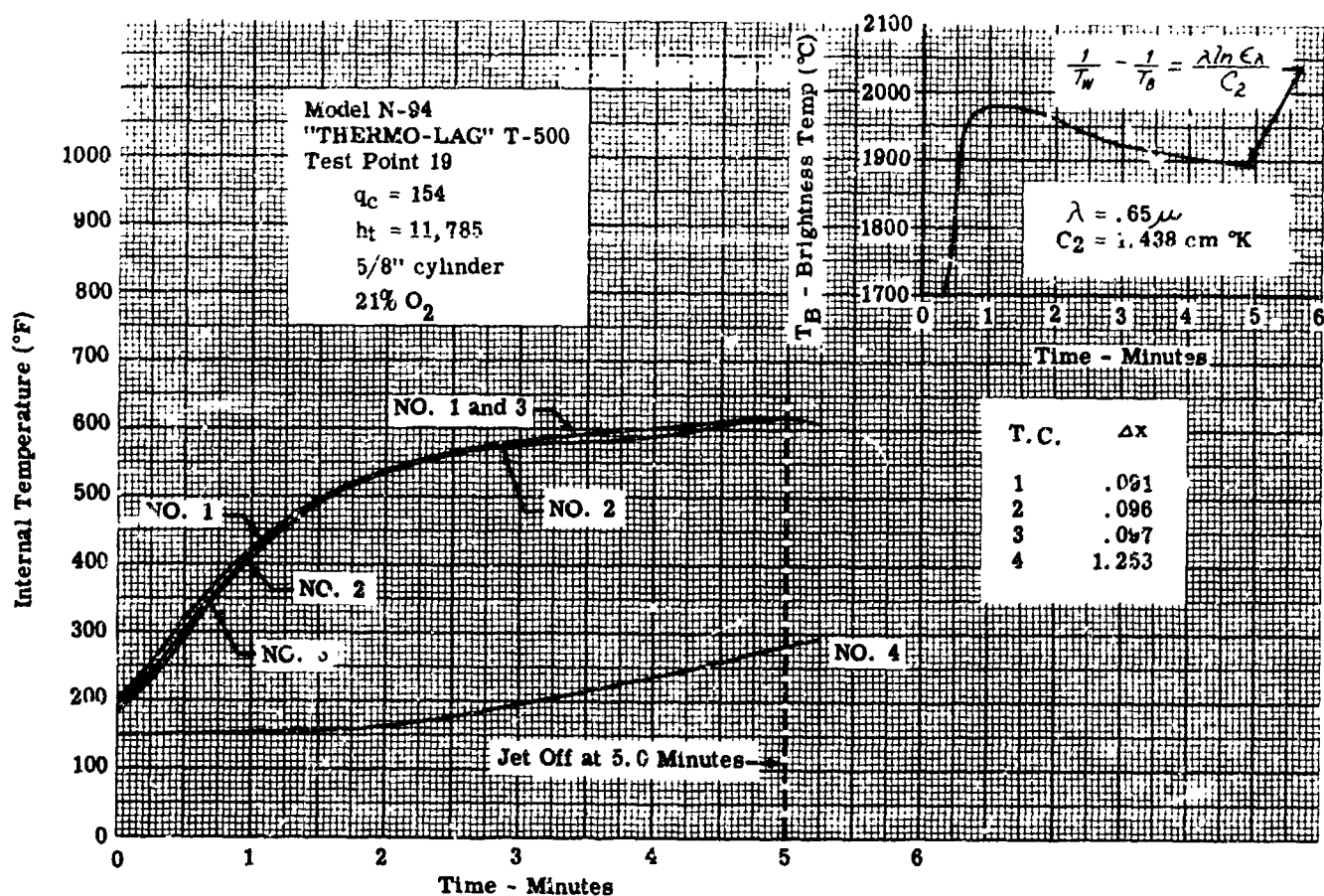


Figure 4-88. Temperature-Time History of Model N-94

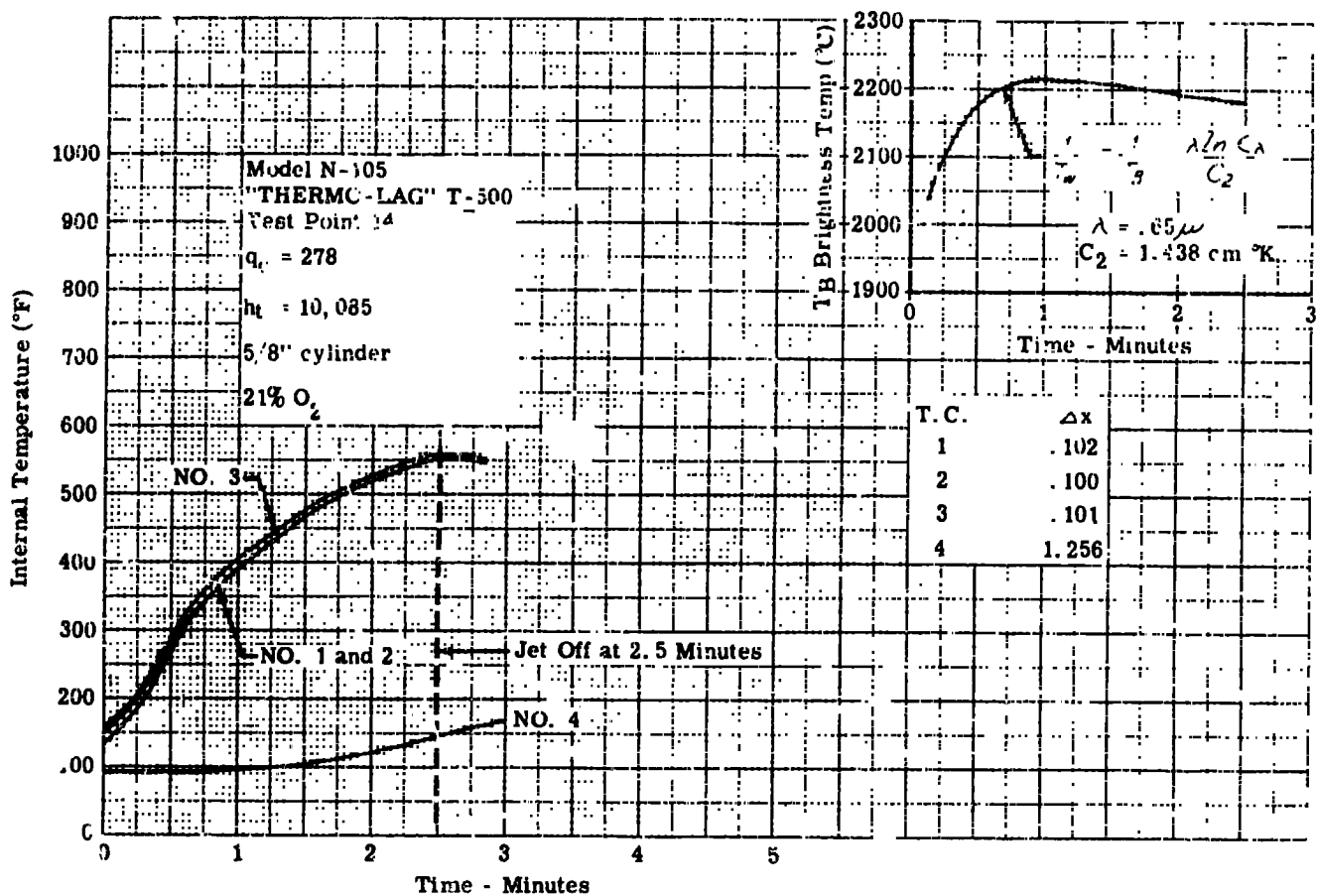


Figure 4-89. Temperature-Time History of Model N-105

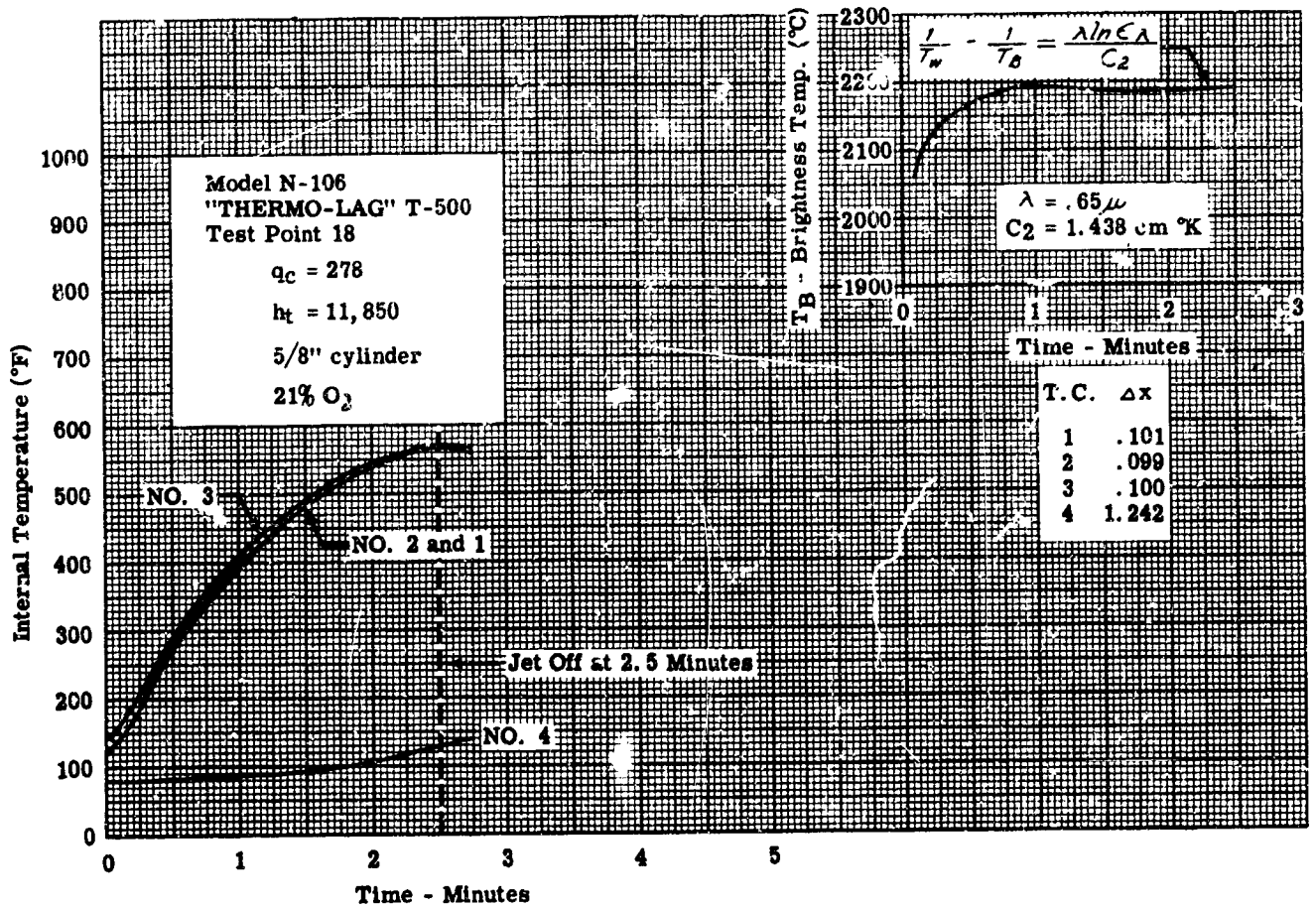


Figure 4-90. Temperature-Time History of Model N-106

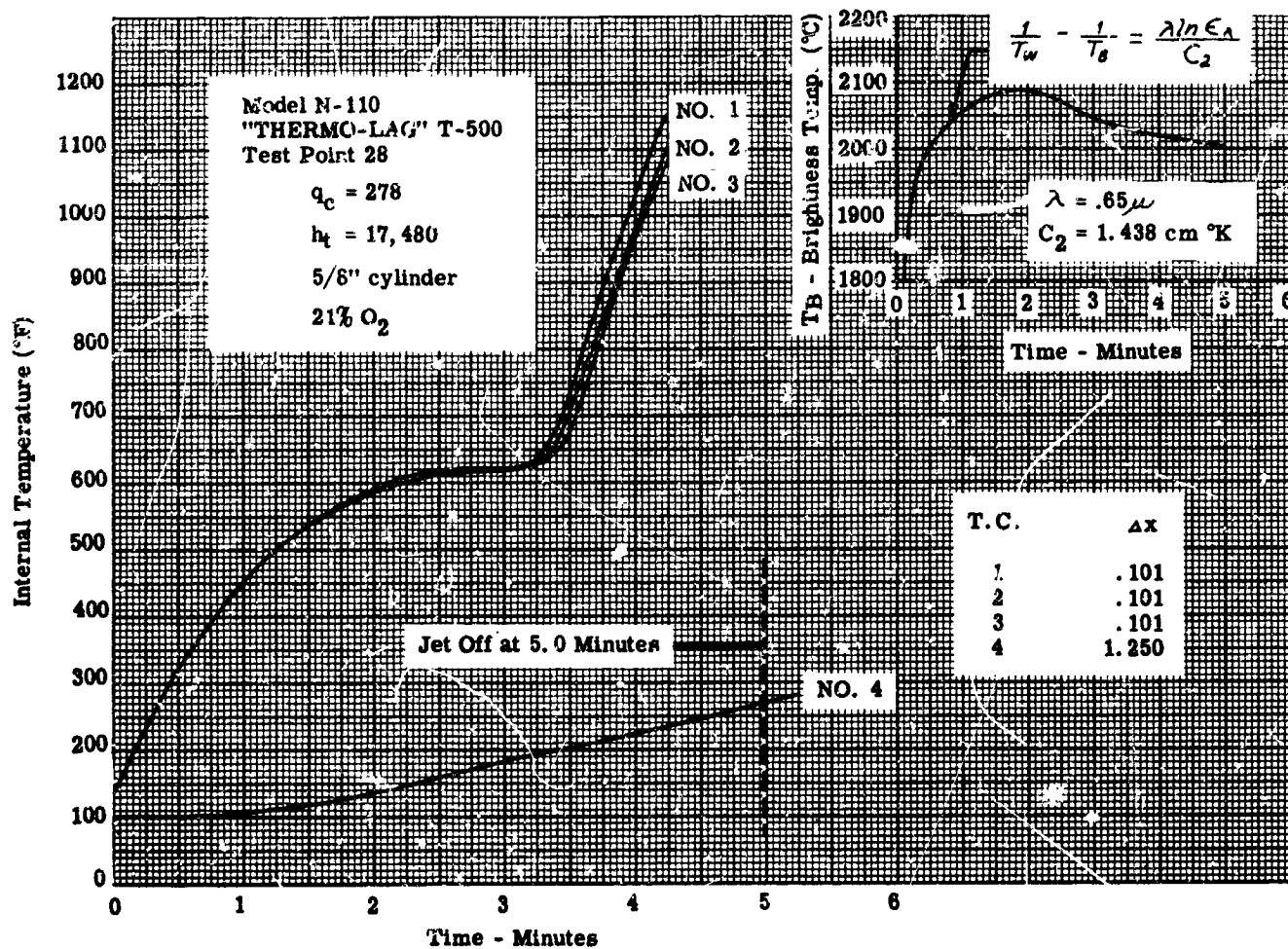


Figure 4-91. Temperature-Time History of Model N-110

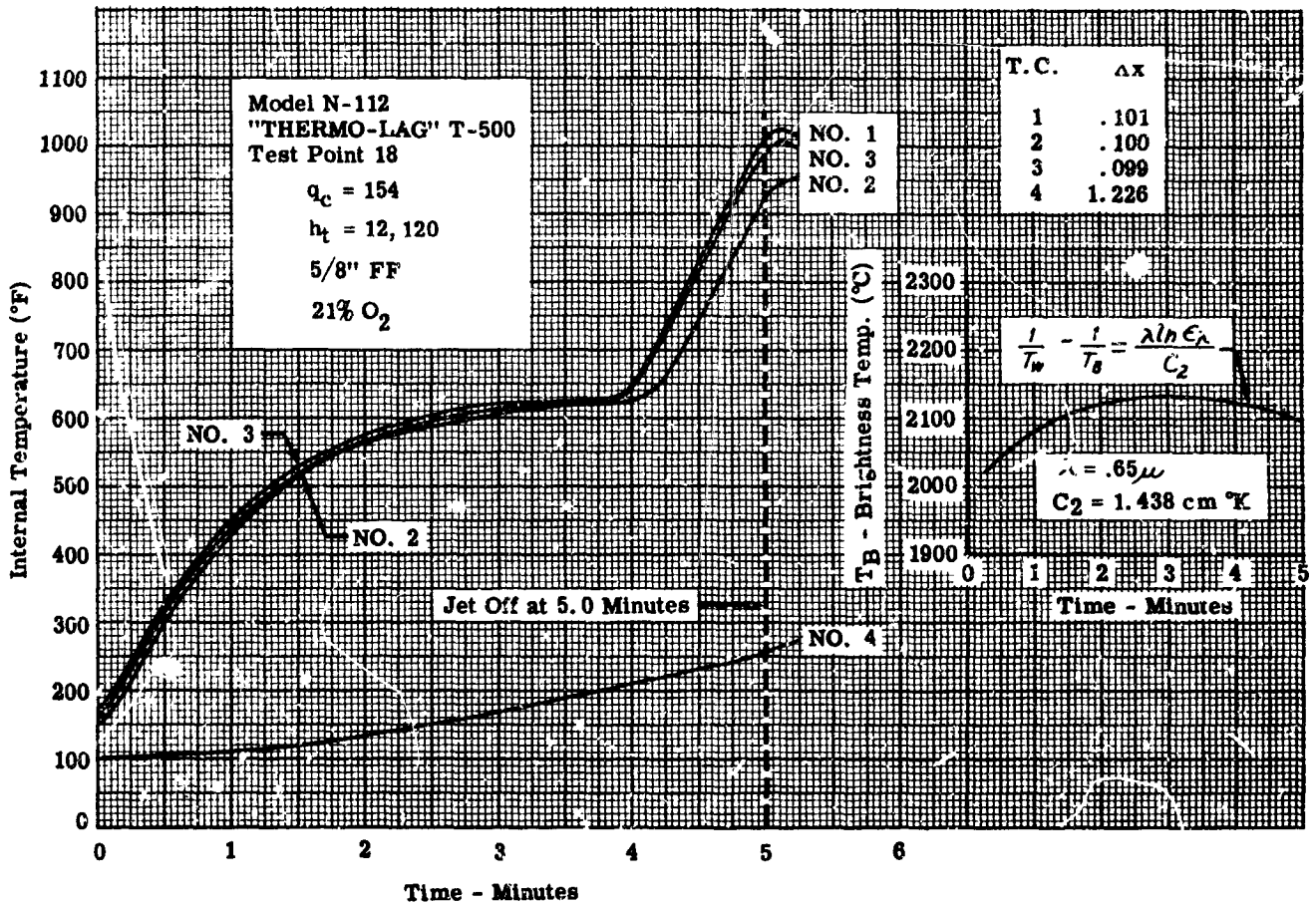


Figure 4-92. Temperature-Time History of Model N-112

Model No.	Nose Geometry (IN.)	Plasimadyne Test Point	Percent Oxygen	Run Duration (SEC)	Stagnation Enthalpy (BTU/LB) h_T
M-6	1 cone	20	21	30	14890
M-9	1 cone	12	21	150	9865
M-10	1 cone	12	21	300	9900
M-18	5/8 cone	13	21	420	10100
M-16	1 cone	24	21	420	17576
M-19	5/8 cone	18	21	300	11980
M-21	5/8 cone	28	21	45	17658
M-22	5/8 cone	28	21	90	17680
M-23	5/8 cone	28	21	150	17500
O-1	5/8 hc	15	15	150	9897
O-2	5/8 hc	16	7	150	10055
O-3	5/8 hc	17	0	150	10165
O-6	1 hc	21	15	300	14988
O-7	1 hc	22	7	300	15185
O-8	1 hc	23	0	300	15052
O-9	1 hc	8	15	300	4965
O-11	1 hc	9	7	300	5000
O-13	1 hc	10	0	300	5110
O-14	1 hc	3	15	420	2488
O-15	1 hc	4	7	420	2510
O-16	1 hc	5	0	420	2505
D-1-2	1 hc	13	21	300	9526
D-2-2	1 hc	13	21	420	9810
E-1-2	1 hc	13	21	420	10433
E-2-2	1 hc	13	21	300	9870
GG-1	1 hc	13	21	165	9486
GG-3	1 hc	13	21	420	9489
HH-1	1 hc	13	21	420	9899
HH-2	1 hc	13	21	175	9211
D-1	1 hc	12	21	75	9855
D-2	1 hc	12	21	80	9826
D-9	1 hc	12	21	320	9926
D-10	1 hc	12	21	385	9871
D-11	1 hc	12	21	855	10109
D-12	1 hc	12	21	300	9875
E-15	1 hc	12	21	75	9301
E-16	1 hc	12	21	75	10024
E-17	1 hc	12	21	50	10125
E-18	1 hc	12	21	50	10035
GG-1A	1 hc	12	21	140	10001
GG-2	1 hc	12	21	75	10179
GG-4	1 hc	12	21	150	10101

Table 4-9. Summarized Data of Conducted Flight Simulation Tests

Stagnation Cold Wall Heat Flux q_c (BTU/FT ² -SEC)	Stagnation Pressure (ATM) P_T	Average Surface Temperature (°F) ($\xi = .9$) T_w	Enthalpy Difference (BTU/LB) ΔH
110	.00267	3660	13840
110	.0064	3840	8705
110	.0064	3840	8740
215	.016	4290	8750
170	.005	3970	16365
300	.023	4480	10530
300	.01	4160	15358
300	.01	4290	16330
300	.01	4230	16150
297	.0291	3970	8737
295	.0289	3760	9005
301	.0291	2840	9415
103	.00267	3640	13968
105	.00265	3570	14185
107	.00266	3510	14087
109	.026	3370	4055
107	.0258	3240	4130
109	.026	3080	4285
48.7	.0255	2470	1858
4	.0258	2380	1780
47.9	.0256	2240	1935
168	.016	3770	8461
168	.016	3730	8770
168	.016	3730	9393
168	.016	3730	9830
168	.016	3700	8451
168	.016	3750	8439
168	.016	3770	8834
168	.016	3750	8161
109	.0064	3930	8335
109	.0064	3890	8811
109.6	.0064	3930	8906
109	.0064	3930	8851
110	.0064	3930	9089
109	.0064	3960	3853
109	.0084	3930	8781
109.6	.0064	3910	9006
110	.0064	3910	9107
109.6	.0064	3960	9013
109.6	.0064	4000	8976
109.6	.0064	3960	9157
109.6	.0064	3960	9079

Stagnation Hot Wall Q_h Heat Flux (BTU/FT ² -SEC)	Recession to Surface (IN.)	Debris Layer Thickness (IN.)	Total Recession (IN.)	Cold Wall Gross Heat Blockage H_{ef} (BTU/LB)
103.2	.039	.047	.086	5750
93.5	.141	.110	.251	9860
97.5	.346	.125	.471	10480
189	1.112	.094	1.206	11220
159.5	.461	.149	.610	17580
267	.902	.094	.996	13560
280	.078	.070	.148	13680
279.5	.169	.102	.271	14920
279	.259	.110	.369	18280
268	.280	.080	.360	18525
267.8	.117	.170	.287	23100
282	.022	.200	.222	30500
102.25	.268	.170	.438	11090
98.8	.156	.250	.406	11610
101.5	.063	.270	.333	14475
91.6	.068	.300	.368	13310
90.8	.042	.300	.342	14030
93.8	.026	.310	.336	14590
38.5	.035	.190	.225	13620
35.15	.023	.190	.215	13690
39.15	.009	.170	.179	16805
151.8	.370	.118	.488	15490
152.25	.490	.118	.608	17390
153.5	.458	.138	.596	17725
152.5	.433	.116	.549	13780
152	.211	.083	.294	8890
151.75	.511	.112	.623	16990
152	.527	.142	.669	15800
151.2	.186	.135	.321	13720
99	.113	.063	.176	6960
99.4	.124	.068	.192	6805
99.9	.436	.142	.578	9100
99.2	.531	.118	.649	8790
99.45	1.331			
99.25	.505	.091	.596	8210
98.95	.128	.077	.205	5975
101.7	.128	.079	.207	5950
100.3	.061	.073	.134	6150
99.8	.116	.078	.194	4235
99.6	.206	.093	.299	7690
99.85	.095	.079	.174	7075
99	.238	.087	.325	7580



Hot Wall Gross Heat Blockage (BTU/LB)	Sidewall Cold Wall Q_{sw} Heat Flux (BTU/FT ² -SEC)	Sidewall Average Thickness (IN.)	Time for Sidewall Substrate To Reach 500°F (SEC)
5395	17.31	.036 & .100	25.20
8840	17.31	.0325 & .0975	35.85 & 106.5
9290	17.31	.035 & .096	28.5 & 79.95
9870	17.38	.050 & .0995	60.0 & 96.0
16480	17.08	.0415 & .100	48 & 53.8
12050	17.71	.0505 & .1005	49.2 & 78
12767	22.71	.051 & .0945	36.6
13900	22.71	.052 & .100	58.8 & 97.5
17000	22.71	.050 & .100	45 & 84
16720		.110	
20950		.109	
28580		.110	
10500		.1055	300
10925		.106	
13170		.1085	
11190		.109	
11925		.1025	
12560		.1075	
10780		.1035	
10290		.1075	
13780		.105	
13990			
15780			
16200			
12500			
8045	22.93	.10167	48.0
15320	22.93		132.0
14300			
12375			
6325	Note: hc = Hemispherical cylinder nose body		
6205	FF = Flat face nose body		
8300			
8000			
7475			
5425			
5510			
5610			
3855	17.25	.10167	96.0
6955	17.25	.029	19.398
6445	17.25	.10567	94.2
6850	17.32	.0320	21.0

Table 4-9. Summarized Data of Conducted Flight Simulation Tests (Continued)

Model No.	Nose Geometry (IN.)	Plasmadyne Test Point	Percent Oxygen	I
GG-5	1 hc	12	21	
GG-7	1 hc	12	21	
GG-8	1 hc	12	21	
GG-9	1 hc	12	21	
GG-11	1 hc	12	21	
D-2-4	1 hc	12	21	
D-2-5	1 hc	12	21	
N-2	1 hc	27	21	
N-6	1 hc	27	21	
N-9	1 hc	7	21	
N-11	1 hc	27	21	
N-15	1 hc	1	21	
N-21	1 hc	20	21	
N-22	1 hc	27	21	
N-26	1 hc	27	21	
N-29	1 hc	25	21	
N-30	1 hc	11	21	
N-35	1 hc	6	21	
N-36	1 hc	6	21	
N-46	1 hc	13	21	
N-48	1 hc	13	21	
N-53	1 hc	6	21	
N-54	1 hc	7	21	
N-64	1 hc	2	21	
N-65	1 hc	27	21	
N-71	1 Flat	25	21	
N-73	1 Flat	27	21	
N-75	1 hc	26	21	
N-82	1 hc	6	21	
N-92	5/8 hc	14	21	
N-94	5/8 hc	19	21	
N-105	5/8 hc	14	21	
N-106	5/8 hc	18	21	
N-110	5/8 hc	28	21	
N-112	5/8 FF	18	21	

Run ation SEC)	Stagnation Enthalpy (BTU/LB) h_T	Stagnation Cold Wall q_c Heat Flux (BTU/FT ² -SEC)	Stagnation Pressure (ATM) P_T	Average Surface Temperature (°F) ($\xi = .9$) T_w	Enthalpy Difference (BTU/LB) ΔH
90	9872	109	.0064	3960	8850
780	9865	109	.0064	3960	8843
35	9987	109	.0064	3960	8965
40	9900	109	.0064	3930	8880
390	10291	109.6	.0064	3930	9271
345	10334	109.6	.0064	3980	9310.5
420	10230	109.6	.0064	3940	9209
90	23100	456	.0270	4720	21520
30	22900	457	.0270	4640	21380
280	4950	104	.0260	3120	4105
45	23250	457	.0270	4590	21780
420	3560	48.5	.0150	3030	2750
300	15150	105	.0027	3700	14080
15	23500	455	.0270	4090	22320
50	23330	453	.0270	4690	21780
150	17650	317	.0200	4770	16030
265	5050	163	.0590	3570	4080
90	4950	48.5	.0076	3030	4140
420	5125	48	.0076	3170	4265
405	9850	163	.0160	4090	8650
45	10120	162	.0160	4090	8920
30	5065	48	.0076	2960	4275
300	5100	105	.0260	3060	4280
420	2550	47.4	.0210	2900	1785
270	23000	457	.0270	4660	21460
300	17480	174	.0200	4490	16070
00	23050	251	.0270	4330	21730
50	22850	253	.0088	4540	21290
00	4890	48	.0076	3230	4020
210	9950	280	.0290	4330	8630
300	11785	154	.0058	3960	10595
50	10085	279	.0290	4440	8725
50	11850	280	.023	4420	10470
300	17480	280	.016	4180	16240
00	12120	154	.023	4310	10790

Table 4-9. Summarized Data of Conducted Flight Simulation Tests (Continued)

Stagnation Hot Wall q_h Heat Flux (BTU/FT ² -SEC)	Recession to Surface (IN.)	Debris Layer Thickness (IN.)	Total Recession (IN.)	Cold Wall Gross Heat Blockage H_{effc} (BTU/LB)
99.2	.123	.083	.206	7130
99.1	.246			
99.4	.048	.049	.097	5895
99.05	.048	.049	.097	6745
100.05	.582	.096	.678	9450
100.1	.540	.108	.648	8745
100.05	.676			
428	.210	.061	.271	21950
430	.063	.055	.118	17410
88.5	.383	.553	.936	37950
431	.096	.067	.163	18920
39	.055	.289	.344	8875
98.4	.289	.164	.453	10420
435	.019	.049	.068	14000
425	.435	.061	.496	20500
290.25	.514	.068	.582	12250
134.75	.478	.100	.578	11200
41.65	.013	.093	.106	5175
41.1	.139	.205	.344	8945
145.1	1.173	.144	1.317	8620
144.9	.097	.048	.145	7540
41.6	.001	.052	.053	4075
90.45	.047	.238	.285	16500
35.1	.042	.244	.286	10420
429.5	.785	.090	.875	21100
161	.675	.094	.769	10200
238	.055	.115	.170	22100
237.25	.342	.092	.434	13100
40.6	.326	.353	.679	9545
246.25	.733	.058	.791	11125
140	.430	.153	.583	11890
244.5	.584	.062	.646	9705
250	.514	.072	.586	10720
262.25	.587	.095	.682	18450
138.8	.755	.074	.829	8375



Hot Wall Gross Heat Blockage (BTU/LB) H_{eff}	Sidewall Cold Wall q_{sw} Heat Flux (BTU/FT ² -SEC)	Sidewall Average Thickness (IN.)	Time for Sidewall Substrate To Reach 500°F (SEC)
6495	17.32	.10333	102.0
	17.32	.029	22.4
5370	17.32	.03333	24.0
6125	17.32	.1025	103.5
8640			
7995			
20575	25.42	.101	78.0
16390	25.41	.10167	
32350	16.61	.10433	429
17880	25.41	.10133	
7140	8.97	.102	366
9760	16.71	.10267	118.5
13390	25.48	.10067	
19225	25.481	.101	87.0
11205	27.66	.10367	98.6
9260	22.51	.103	112.5
5305	8.97	.10533	
7525	8.90	.10467	367.5
7680	22.51	.10333	100.1
6740	22.44	.102	
3530	8.9	.10233	
14280	16.71	.10167	
7720	9.03	.10167	420
19850	25.41	.10133	82.4
7625	23.34	.10367	243
20975	27.11	.10533	100.2
12280	27.2	.10267	85.8
8085	8.9	.10233	307.5
9800	27.58	.10167	93.3
10795	21.79	.09467	93.0
8540	27.6	.101	106.5
9590	27.58	.100	96
17290	27.58	.101	76.5
7540	21.79	.100	79.8

Table 4-9. Summarized Data of Conducted Flight Simulation Tests (Continued)



SECTION V

ATTACHMENT DEVELOPMENT

ATTACHMENT TECHNIQUES.

Species selection studies were performed to determine the bonding technique most suitable for primary and secondary bonding over the 0°F to 300°F temperature range. For primary bonding, 13 general categories of surface treatments for annealed PH 15-7 molybdenum steel were studied. This steel was used as it is one of the higher strength space age steels. The treatments with preliminary results are given in Table 5-1.

From the screening study, five techniques warranted further investigation and are recorded in Table 5-2. Of the categories, three were mineral acids, reverse etch treated. Although a previous test using a nitric or sulphuric concentrate gave better adhesion, such treatment involved the risk of excessive hydrogen and the embrittlement of the steel. The treatments were tested at temperatures of 77°F, 150°F, and 300°F. The results of bond shear strength tests for the five treatments are recorded in Table 5-3 and are illustrated in Figure 5-1.

Results of bond shear and tensile tests performed on sprayed "THERMO-LAG" T-500, using the mineral acid reverse etch, sandblast, and clean treatment techniques are presented in Figures 5-2 through 5-6 for this reporting period.

SECONDARY BONDING.

Studies to determine the adhesive most suitable to meet the conditions imposed by the temperature environment from -150°F to 300°F were conducted on five general categories of adhesives. The entities tested were flexible materials of a rubber nature such as polysulfides and silicones, semiflexible materials such as urethanes, and rigid almost glassy materials such as epoxies and phenolics.

These materials were subjected to preliminary screening tests using aluminum "T"'s scratched on 1-7/8 x 2 inch surface specimens. The adhesives were then plentifully applied to both faces of the "T" by brush. The initial cure was at 300°F for one-half hour. Some specimens required higher curing temperatures, as recorded. Preliminary screening test data are recorded in Tables 5-4 through 5-6.

The selection process eliminated Epoxylite 810, requiring the choice of another rubber adhesive. During this process three phenolics, one silicone-phenolic, one epoxy-phenolic, two epoxies, and one polysulfide were checked. The epoxies appear to be of questionable value at temperatures below 0°F.

Screening efforts will continue during the next reporting period on the cementing of molybdenum steel, PH-15-7, to the aluminum "T"'s and also on the cementing of the preformed "THERMO-LAG" material to the steel specimens for the bond shear and tensile tests.

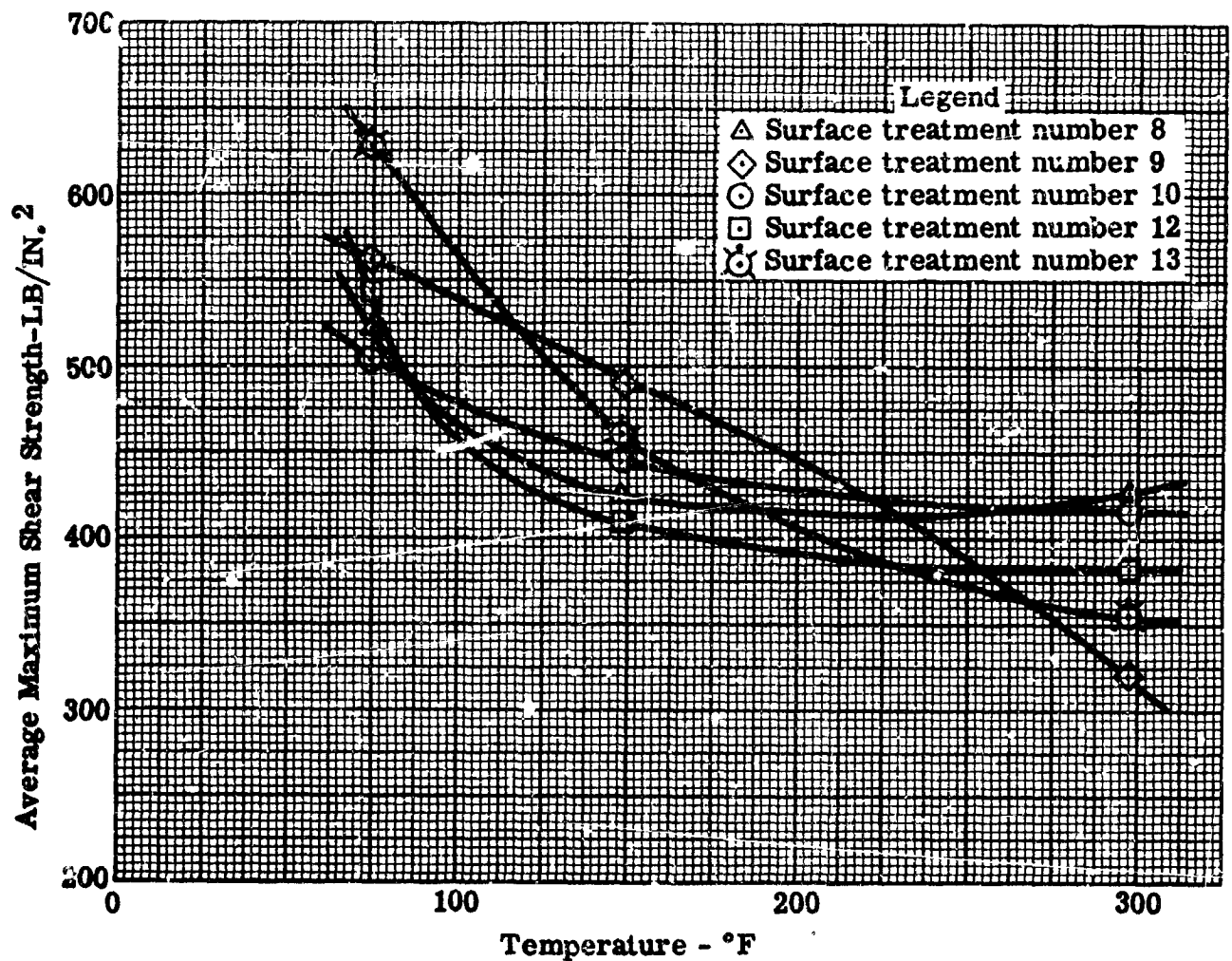


Figure 5-1. Effect of Surface Treatment on Bond Shear Strength

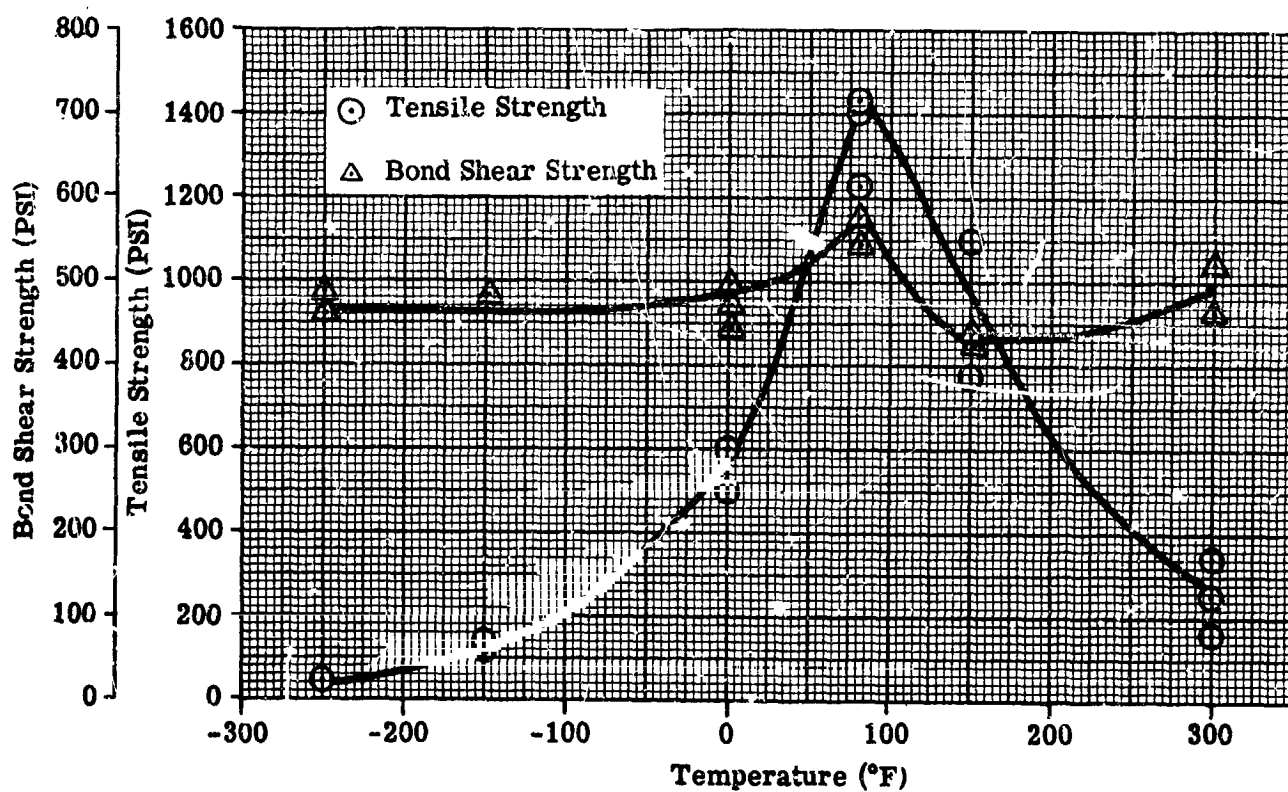


Figure 5-2. Bond Shear and Tensile Strength for Reverse Etch Treatment No. 8

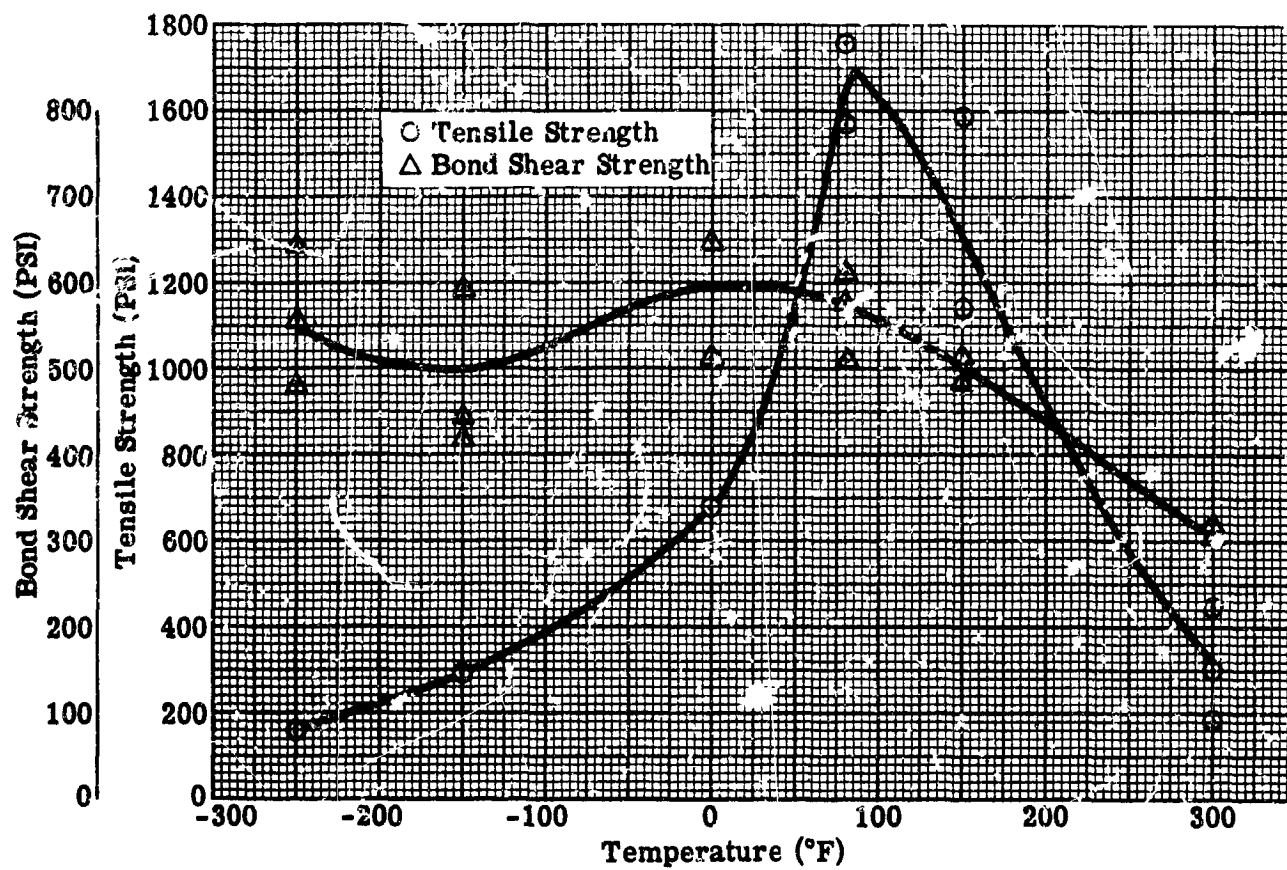


Figure 5-3. Bond Shear and Tensile Strength for Reverse Etch Treatment No. 9

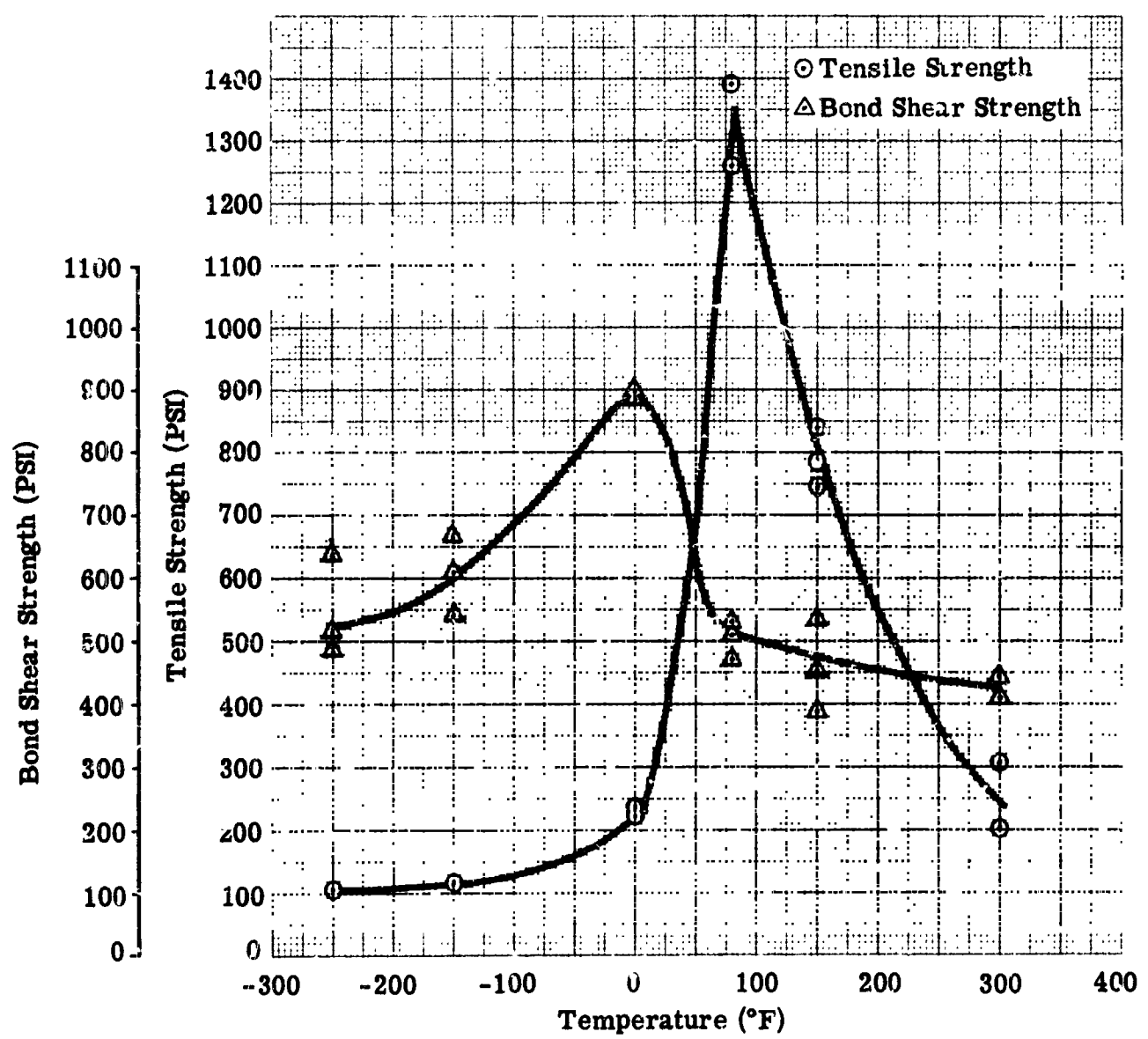


Figure 5-4. Bond Shear and Tensile Strength for Reverse Etch Treatment No. 10

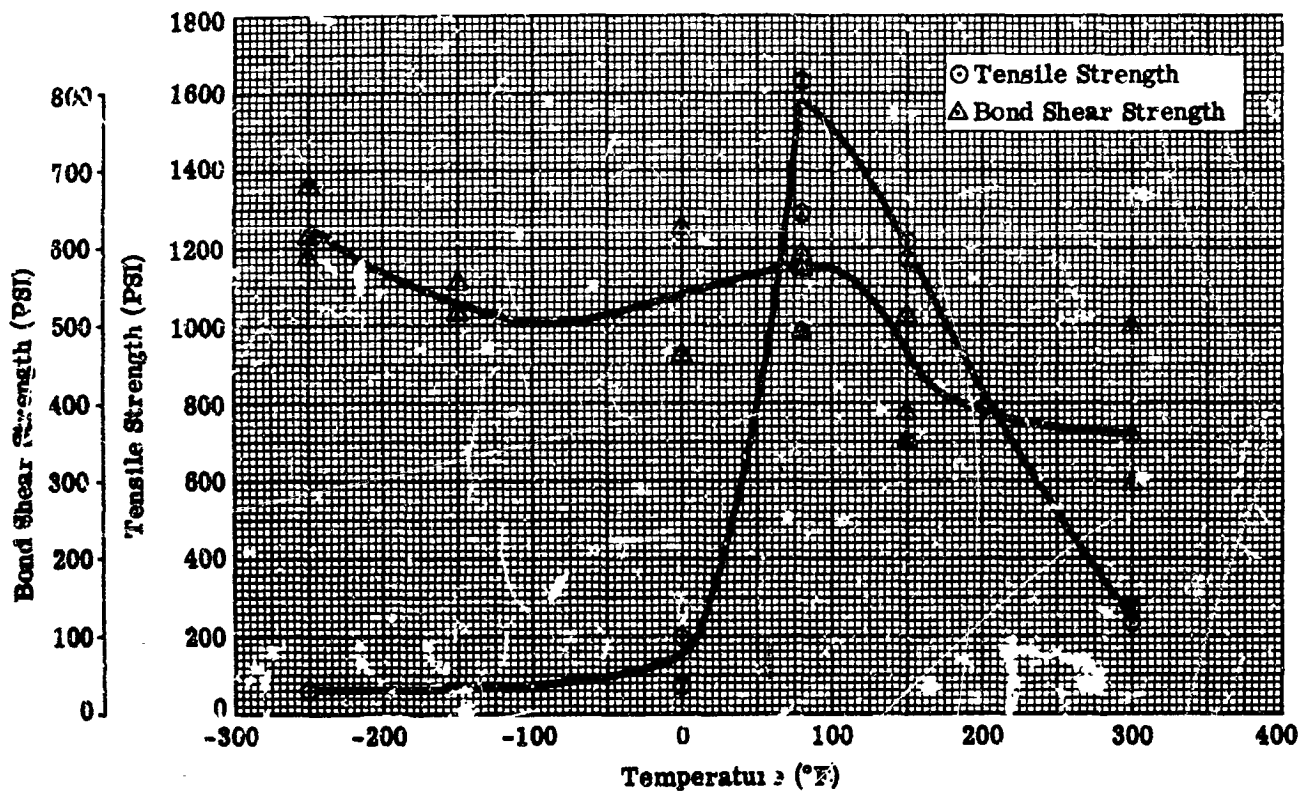


Figure 5-5. Bond Shear and Tensile Strength for Chemically Cleaned Treatment No. 12

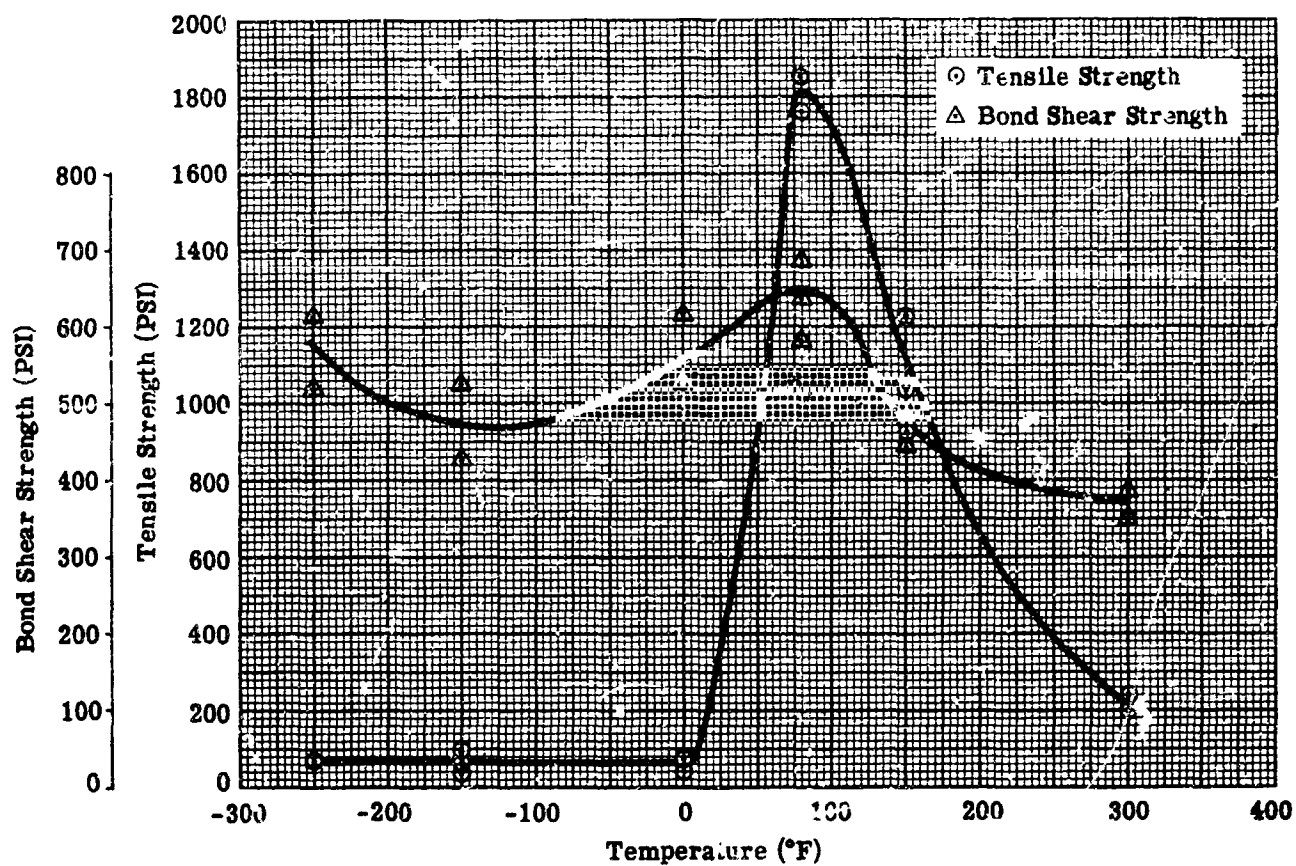


Figure 5-6. Bond Shear and Tensile Strength for Sandblast Surface Treatment No. 13

Treatment Number	Solution Composition	Results
1	40% Phosphoric + 40% Acetic + 20% Nitric	Little or no observable results cold, nor when heated to 180°F
2	15% Phosphoric Acid in Water	Some slight gassing and slight lightening of the matte mill scale
3	10cc Iron Nitrate	No results, hot or cold
4	Epcostrip	No results
5	Oxalic Acid	No appreciable results
6	CS 105 Descale	Overnight, green scum, etched surface and some cleaning
7	Sulfuric Acid conc.	Removal of mill scale and some cleaning
8	75% Sulfuric acid reverse etch 9 volts 150-180°F 1-2 MIN	Definite cleaning and etch to surface. Looks good
9	63% Phosphoric acid 15% Sulfuric acid reverse etch 9 volts 80-180°F 1-2 MIN	Definite cleaning and polishing of the surface of the steel
10	CR 110 Chromic acid 53% Sulfuric acid .53% Reverse etch 9 volts 180°F 1-2 MIN	Definite cleaning and etching of the surface similar to that obtained with reverse etch using sulfuric acid
11	Degrease only	Oil and grease removed to some extent, dirt remains and a streaked appearance results

Table 5-1. Surface Treatments for Annealed PH 15-7 Mo Steel



Treatment Number	Solution Composition	Results
12	Chemically clean Vapor degrease Anodic Alkaline Clean Cold water rinse Cold water rinse HCl Neutralize Cold water rinse Sulfuric-Nitric Etch Cold water rinse Sulfuric-Nitric-HF Etch Cold water rinse Cold water rinse	Clean surface. Water break free
13	Chemically clean (Treatment 12) Sandblast Chemically clean (Treatment 12)	Definite cleaning. Porous surface

Table 5-1. Surface Treatments for Annealed PF 15-7 Mo Steel (Continued)

Treatment No. 8

Sulfuric acid reverse etch: 50-75% in tap water

Voltage:	9 Volts
Time of Etch:	1-2 MIN
Temperature:	150-180°F

Treatment No. 9

Sulfuric acid (15%), phosphoric acid (63%) reverse etch

Voltage:	9 Volts
Time of etch:	1-2 MIN
Temperature:	80-180°F

Treatment No. 10

CR110 (Unichrome Inc.) (33 LB), CrO_3 (0.33 LB), H_2SO_4 reverse etch

Voltage:	9 Volts
Time of etch:	1-2 MIN
Temperature:	160-180°F

Treatment No. 12

Vapor Degreasers

Anodic alkaline clean, cold water rinse (twice)
HCl neutralize, cold water rinse
Sulfuric-nitric etch, cold water rinse
Sulfuric-nitric-HF etch, cold water rinse (twice)

Treatment No. 13

Anodic alkaline clean, cold water rinse (twice)
HCl neutralize, cold water rinse
Sulfuric-nitric etch, cold water rinse
Sulfuric-nitric-HF etch, cold water rinse (twice)
Sandblast prior to the above

Table 5-2. Surface Treatment Techniques



Temperature Specimen Number	77°F		150°F		300°F	
	Shear Strength psi	% A. F. *	Shear Strength psi	% A. F. *	Shear Strength psi	% A. F. *
10-1	510	50	451	50	409	60
10-2	471	30	535	40		
10-3	530	50	388	40	448	50
Avg.	504		456		428	
8-1	548	30	425	75	463	50
8-2	580	90	430		332	75
8-3	449	25	439	75	520	25
Avg.	526		431		436	
9-1	580	75	480	10	305	10
9-2	612	75	513	25	315	25
9-3	510	25				
Avg.	567		497		310	
13-1	688	80			388	10
13-2	577	50	479	50	349	10
13-3	637	60	444	70	349	10
Avg.	634		462		362	
12-1	493	45	509	50	354	60
12-2	573	75	350	50	295	40
12-3	590	85	383	20	498	20
Avg.	552		414		382	
*% A. F. - Percent Adhesive Failure						

Table 5-3. Surface Treatment Bond Shear Strength

Category	Materials	Manufacturer	Preliminary Screening Tests			
			Bond Tensile (PSI)		Bond Shear (PSI)	
			300°F	-250°F	300°F	-250°F
Silicones	R. T. V. 30	General Electric	230	1000+	20	1000+
	R. T. V. 60	General Electric	480	1000+	20	1000+
	R. T. V. 325	Dow Corning Corp.		1000+		492
	Silastic 140	Dow Corning Corp.				
	Sylgard 182	Dow Corning Corp.	610	1000+	96	1000+
Phenolics	Plyophen 23-973	Reichold Chemical Co.		1190		
	R-84029	Raybestos Manhattan	145	1275	120	583
	SC 1013	Monsanto Chemical Co.	372	786 1000+	574	564
	HT 424	Brunswick Corp.	1000+	1000+ 786	844	1000+
	605-4	B. F. Goodrich	442	755	95	740
Urethanes	1538	Product Research	...	763	...	492
	Adiprene L	DuPont				
	Daycollan 80	Dayton Rubber				
Polysulfides	Genthane S	General Tire and Rubber				
	Poly-Therm 40SA	American Poly-Therm Co.				
Epoxies	727	Coast Pro Seal Mfg.				
	Epoxylite 810	Epoxylite Corp.				
	Plyophen 23-983	Reichold Chemical Co.	110	950		
	Epon 828	Shell Chemical Co.				
	Epoxylite 5403	Epoxylite Corp.				

Table 5-4. Categories of Adhesive Materials Investigated



Temperature (°F)	Cleaned Only		Chromic Acid Reverse Etch	
	Bond Tensile	Bond Shear	Bond Tensile	Bond Shear
300		26	13	22
	31		28	23
	40	25		29
150		69	25	51
	104	76	26	52
	112	77	37	72
77		194		219
	390	200	200	220
	400	245	267	252
0		497		520
	2035	590	490	559
	2270	603	1730	618
-150		327		
		390		437
		467		498
-250		405		439
	120	433	119	492
		466		465

Temperature (°F)	Sulfuric Acid Reverse Etch		Cleaned-Sandblast-Cleaned	
	Bond Tensile	Bond Shear	Bond Tensile	Bond Shear
300		6		
	28	7		3
	38	5		4
150		30		27
	94	34		28
	127	43		30

Table 5-5. Piastiloc 605-4 Adhesive Bond Shear and Tensile Strength

Temperature (°F)	Sulfuric Acid Reverse Etch		Cleaned-Sandblast-Cleaned	
	Bond Tensile	Bond Shear	Bond Tensile	Bond Shear
77	160	157		116
	208	168		125
		179		140
0		573		476
	1850	608		572
	2055	623		579
-150		410		416
		418		424
		423		440
-250		308		
		405		457
		420		498

Table 5-5. Plastiloc 605-4 Adhesive Bond Shear and Tensile Strength (Continued)

Temperature (°F)	Cleaned Only		Chromic Acid Reverse Etch	
	Bond Tensile	Bond Shear	Bond Tensile	Bond Shear
300			44	
	14		54	
	22		90	
150	155			
	173		266	
	186		293	
77	567		900	
	690		915	
	715		925	

Table 5-6. HT 424 Phenolic Adhesive Bond Shear and Tensile Strength



Temperature (°F)	Cleaned Only		Chromic Acid Reverse Etch	
	Bond Tensile	Bond Shear	Bond Tensile	Bond Shear
0	405	448	1525	
	440	544	1810	271
			2200	248
-150		419	1335	
	25	444	1445	291
	36	461	1715	295

Temperature (°F)	Sulfuric Acid Reverse Etch		Cleaned-Sandblast-Cleaned	
	Bond Tensile	Bond Shear	Bond Tensile	Bond Shear
300				19
				23
				22
150				174
				167
				116
77				256
				273
				338
0		334		445
		353		502
		466		526
-150		428		445
		430		449
	245	474		466

Table 5-6. HT 424 Phenolic Adhesive Bond Shear and Tensile Strength (Continued)



SECTION VI

STRUCTURAL APPLICATIONS

DEVELOPMENT OF "THERMO-LAG" T-500 PUTTY FOR JOINT, GAP, AND EDGE FILLERS.

Development of techniques for the utilization of "THERMO-LAG" T-500 in the roles of joint, gap, and edge fillers were investigated to determine the optimum conditions for artificial curing. Successful application of "THERMO-LAG" for these purposes would qualify it for use in sealing gaps around the windows, hatches, and other orifices of a super-orbital vehicle. Techniques considered were those having utility under field conditions.

The T-500 used for the tests was compounded in the normal manner, and reduced to a doughlike or putty consistency by the addition of methyl ethyl ketone solvent. The test specimens were cylindrical patties, hand-shaped to a diameter of approximately two inches, and 5/8-inch thick.

Attainment of the desired degree of cure was determined by extracting unpolymerized solids from a vertical cross-section of the cured specimen through a predetermined procedure using methyl ethyl ketone. Evaporation of the solvent from the test specimen, induced by a thermionic heating device, left a residue of soluble solids. The percentage of solubles was calculated by determining differences in weight and the weight of the unextracted sample. The measured value was used as an index in determining the degree of cure attained by the "THERMO-LAG" T-500 putty. An inverse relationship existed between the percentage of solubles present and the degree of polymerization of the organic bonding agent, caused by the artificial cure technique.

The techniques which have undergone experimentation as sources of artificial cure for the T-500 putty include electrical infrared and conductive heat, and chemical acceleration.

INFRARED CURING TECHNIQUE. This technique utilized a bank of four 375-watt, 115/125-volt Westinghouse infrared heat lamps. The uncured patties were exposed to these lamps for varying periods of up to eight hours. The specimens were placed in a horizontal position directly beneath the face of the lamps, thus assuring uniform exposure.

After exposure the degree of cure was determined by the method of extraction. The data is recorded in Table 6-1 and illustrated in Figure 6-1. The presented data indicates that a more rapid cure was obtained at a 6-inch exposure distance. However, two disadvantages occurred at this exposure level, in that salt sublimation was observed, and some blistering occurred on the top surface of a few of the patties. At a 12-inch exposure distance, neither

of these two phenomena were observed. The exposure distance, as a criteria, will be investigated if the infrared technique warrants continued study.

Patties cured for periods exceeding three hours displayed a small amount of salt in the evaporate. The formation caused an increase in the above calculated percentage values. For the 12-inch exposure distance, the tabulated data indicates a four hour period as adequate to cure the T-500 putty patties.

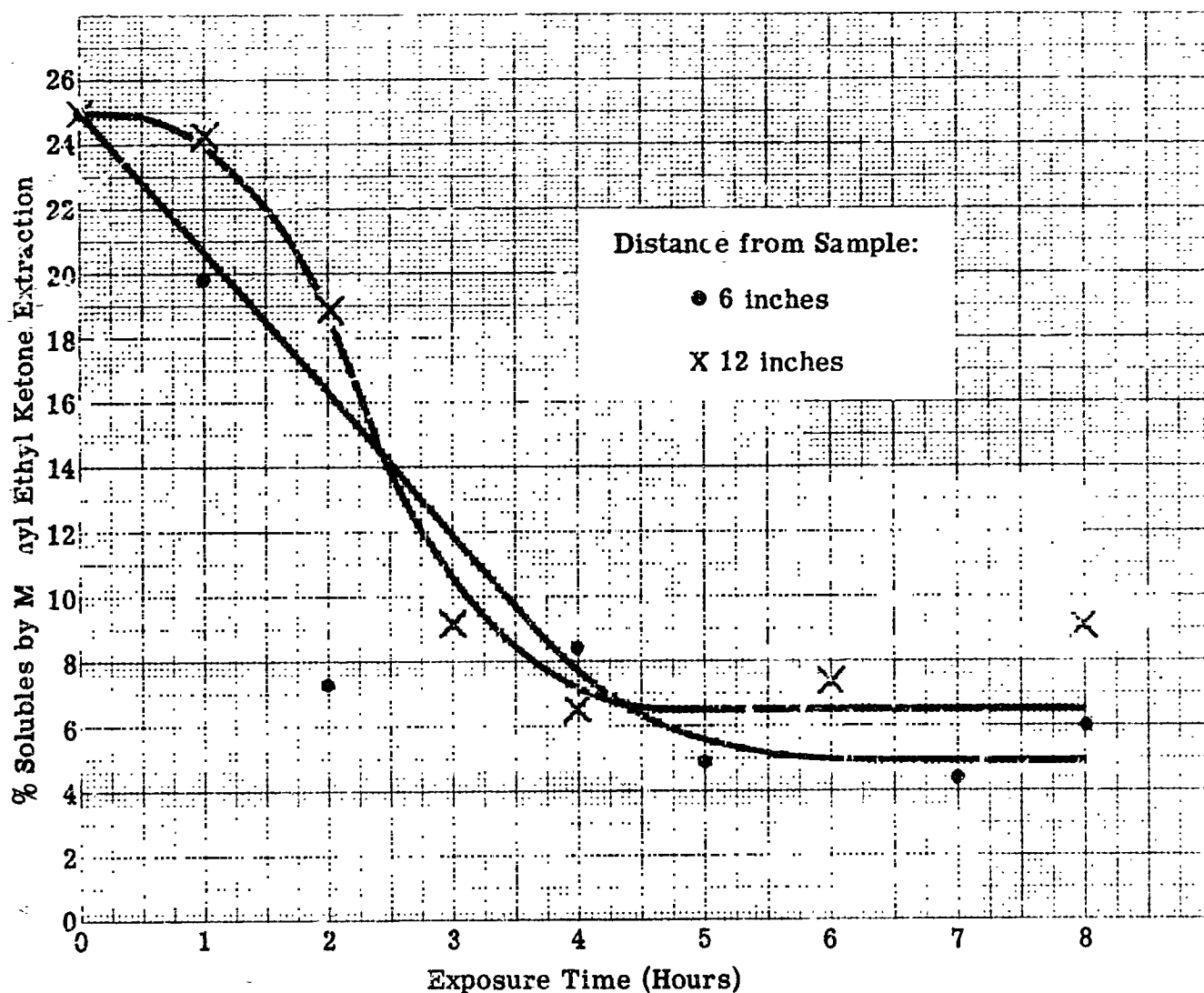


Figure 6-1. "THERMO-LAG" T-500 System Infrared Curing



Exposure Time (HR)	Percent Solubles at 6-Inch Exposure Distance	Percent Solubles at 12-Inch Exposure Distance
0	24.9	24.9
1	19.8	24.1
2	7.3	18.9
3	9.2	9.2
4	8.2	6.6
5	4.9	-
6	-	7.5
7	4.4	-
8	5.9	9.1

Table 6-1. Degree of Artificially Induced Cure by Infrared Source

ELECTRICAL HEAT INDUCED CURING. A resistance heating method to cure T-500 putty patties was investigated in the screening process. The conductive element was a graphite string obtained from a carbon fabric, 24 grams per square foot, manufactured by National Carbon Company, Union Carbide Corporation. The T-500 putty patty was sliced in half in a plane parallel to the two inch diameter dimension. The graphite string was sandwiched in between the putty slices in an "S" shape to allow for a large heating area. The putty sandwich was kneaded together to form the original patty size. The graphite string was equidistant from both surfaces of the patty and in a plane parallel to the two inch diameter dimension. The ends of the "S" shaped graphite string were connected to a variable AC transformer with small battery clips. A given current was applied over a one hour period. Both current and voltage readings were recorded. The degree of cure was determined by the previously described extraction method. The data is presented in Table 6-2.

The above percentage solubles values indicate that little curing occurred. Application of current in excess of 0.5 amperes caused the graphite string to burn through. An examination of the interior of each patty indicated the presence of voids adjacent to the position occupied by the graphite string. White salt accumulated on the surface of the voids at a heating current of 0.5 amperes. Some salt sublimation was observed at the point of entrance of the graphite string into the patty during the course of the experiments. The above phenomena can be contributed to high hot-spot temperatures.

An attempt to minimize the above disadvantages was made by embedding two graphite strings in a T-500 putty patty. For the test the patty was cut into thirds. Each graphite string was sandwiched between freshly cut surfaces in the shape of an "S". The "S" shapes were arranged so that one laid cross-ways on top of the other with a one-third section of putty in between. The ends of the graphite strings were connected together to form an "S" shaped parallel circuit. Current was applied in the same manner as described above. Voltage and current readings were taken and the degree of cure determined by the selected method. The data is presented in Table 6-3.

The tabulated solubility values indicate that little curing took place. In general, the same observations were noted as described for the single graphite string system. However, the size of the voids was observed to have decreased and no salt accumulation was visible. The conductive element technique does not warrant continued study as a method to cure T-500 putty.

Exposure Time (HR)	Amperes (AC)	Voltage (AC)	Percent Solubles
0	0.0	0.0	24.9
1	0.3	29.6	21.3
1	0.5	32.6	16.2

Table 6-2. Degree of Electrical Heat Induced Cure (Single Graphite String)

Exposure Time (HR)	Amperes (AC)	Voltage (AC)	Percent Solubles
0	0.0	0.0	24.9
4	0.3	12.0	19.8
4	0.4	17.0	17.7

Table 6-3. Degree of Electrical Heat Induced Cure (Double Graphite String)



CHEMICALLY ACCELERATED CURING. The choice of a chemical accelerator was investigated as a method of curing T-500 putty. The technique involves the addition of chemical accelerators to the uncured T-500 putty. The accelerator catalyzes and/or reacts with the organic bonding agent in the putty to cause polymerization or curing.

To date, three accelerators were tested in T-500 putty. The quantities used were obtained from published formulations on Hycar Polymers. Those used were:

1. Accelerator 808, a condensation product of butyraldehyde and aniline.
2. Thiuram M, tetramethylthiuram disulfide
3. Accelerator 833, a condensation product of butyraldehyde and butylamine.

The above three are manufactured by E.I. DuPont DeNemours and Company, Elastomer Chemicals Department, Wilmington, Delaware.

The accelerators were mixed in the putty with a pestle until a uniform, homogeneous mix occurred. The mix was shaped by hand into cylindrical patties of the previously described dimensions. The shaped patties were allowed to age in the laboratory at room temperature for definite time intervals. After aging, vertical cross-sections of each patty were extracted with methyl ethyl ketone to determine the degree of cure. The data is presented in Table 6-4.

The tabulated results indicate that Accelerator 808 and Thiuram M had no effect on curing. The first row of data, for no additive, is given for comparison. Accelerator 833 appeared to aid curing, but only after aging for 79.5 hours.

The process of selecting an optimum artificial curing technique for T-500 putty, facilitating its intended application, will continue into the next quarterly reporting period.

Additive	Quantity Present*	Aging Time (Hours)	Percent Solubles
0	0	0	24.9
Accelerator 808	2	20.5	20.8
		80.0	20.6
Thiuram M	3.5	20.0	20.5
		79.5	19.9
Accelerator 833	2	20.5	19.6
		79.5	12.6

*Parts/100 parts rubber (Based on rubber in organic binder used in T-560 putty formulation).

Table 6-4. Degree of Chemically Accelerated Cure



SECTION VII

PROGRAM PLANNING

During the first six months of the program, work progressed simultaneously on all phases for performance evaluation of "THERMO-LAG" T-500 material. Some phases of the evaluation and processing of the material yielded readily to original planning; however, other phases required replanning based on current assessments and re-evaluations.

Current program schedules are shown in Figures 7-1 through 7-6. All material evaluation and processing phases are presented and individual tasks within a phase are noted.

Summary Task Completion Schedule

Week Beginning	Oct			Nov		Dec		Jan		Feb		Mar		Apr		May		Jun		Jul		Aug		Sep		
	1	15	29	12	26	10	24	7	21	4	18	4	18	1	15	29	13	27	10	24	8	22	5	19	9	23
Phase I: Thermal Properties (Performance in Space Environment)																										
Phase II: Mechanical Properties																										
Phase III: Thermal Performance Analysis																										
Phase IV: Attachment Development																										
Phase V: Structural Schemes and Analysis																										
Phase VI: Fabrication of Test Articles																										
Phase VII: Recommendations																										
Progress Reports	X			X		X		X		X		X		X		X		X		X		X		X		X
Final Report																										

Figure 7-1. Phase Schedules

Phase I: Thermal Properties (Performance in Space Environment)

Week Beginning	Oct			Nov		Dec		Jan		Feb		Mar		Apr			May		Jun		Jul		Aug		Sep	
	1	15	29	12	26	10	24	7	21	4	18	4	18	1	15	29	13	27	10	24	8	22	5	19	9	23
1. Thermal Conductivity																										
2. Specific Heat																										
3. Emittance																										
4. Coefficient of Expansion																										
5. DTA																										
6. TGA																										
7. Density																										
8. Vacuum																										
9. Chromotographic Studies																										

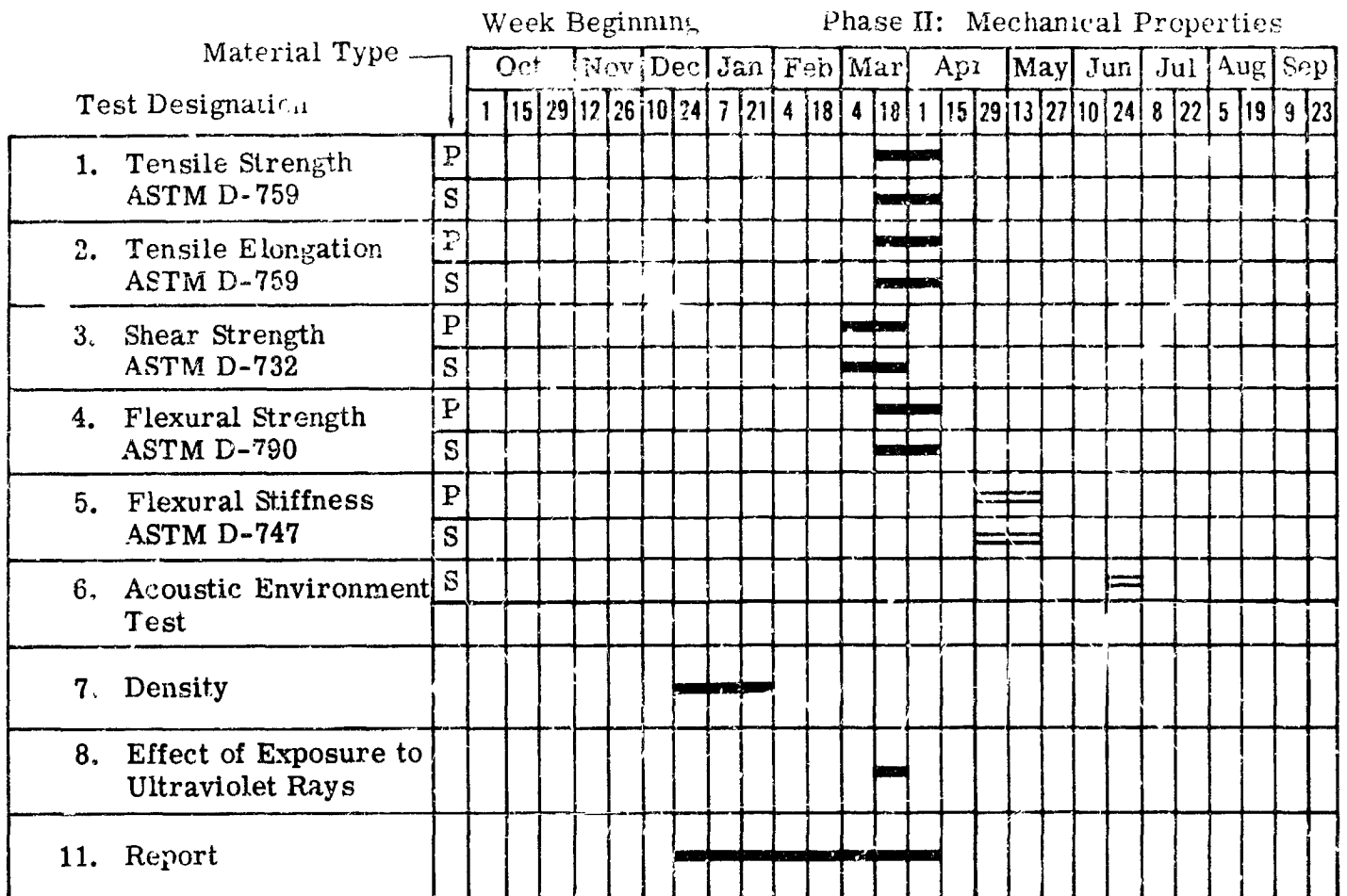
Figure 7-2. Phase I Schedules (Sheet 1 of 2)



Phase I (Continued)

Week Beginning	Oct			Nov			Dec			Jan			Feb			Mar			Apr			May			Jun			Jul			Aug			Sep		
	1	15	29	12	26	10	24	7	21	4	18	4	18	1	15	29	13	27	10	24	8	22	5	19	9	23	6	20	13	27	10	24	7	21	4	18
10. General Flight Simulation Tests																																				
a. Low Shear																																				
d. Oxidation Variable																																				
11. Transient Cases																																				
12. Gap Condition Tests																																				
13. Radiation Simulation																																				
14. Trajectory Simulation																																				
15. Report Preparation																																				
16. Model and Calorimeter Design																																				
17. Model and Calorimeter Fabrication																																				

Figure 7-2. Phase I Schedule (Sheet 2 of 2)



S = Sprayed

P = Precast

Figure 7-3. Phase II Schedule



P = Precast B = Bond M = Mech. Attach.

A

Phase IV: Attachment Development

Week Beginning		Phase IV: Attachment Development																																			
Material Type —		Oct			Nov			Dec			Jan			Feb			Mar			Apr			May			Jun			Jul			Aug			Sep		
		1	15	29	12	26	10	24	7	21	4	18	4	18	1	15	29	13	27	10	24	8	22	5	19	9	23	16	30	13	27	20	3	17	31	14	28
Test Designation																																					
1. Investigation of Bonding Materials	P																																				
a. Bond Tensile Strength ASTM D-952																																					
b. Bond Shear Strength ASTM D-1002																																					
2. Investigation of Mechanical Attachment Techniques	P																																				
a. Bond Tension																																					
b. Bond Shear																																					
3. Acoustic Environment	B																																				
	M																																				
	A																																				
4. Equipment Design and Fabrication																																					
5. Specimen Preparation																																					
6. Report Preparation																																					

Figure 7-4. Phase IV Schedule

Week Beginning

Figure 7-5. Phase V Schedule

Week Beginning

Figure 7-6. Phase VI Schedule



APPENDIX A

MATERIAL TEST SPECIMEN MODELS

NOTE

Dimensions shown in Figures A-1 through A-9 are in inches.

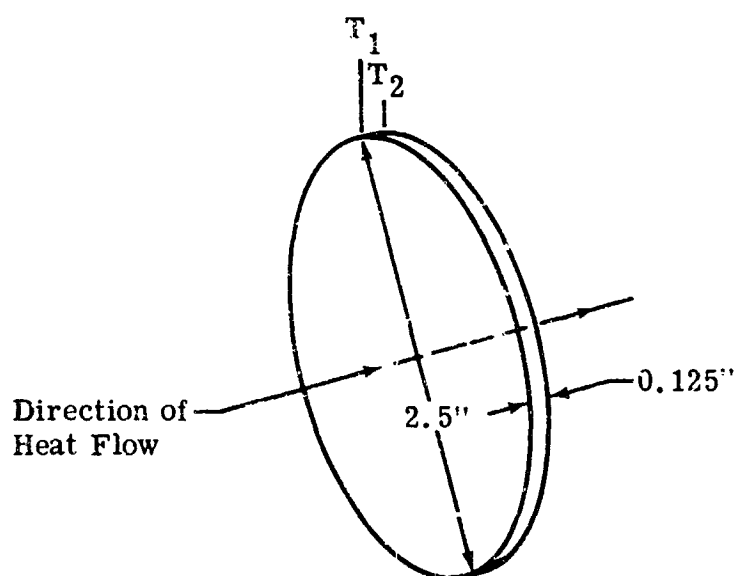


Figure A-1. Thermal Conductivity Test Specimen NA 1020

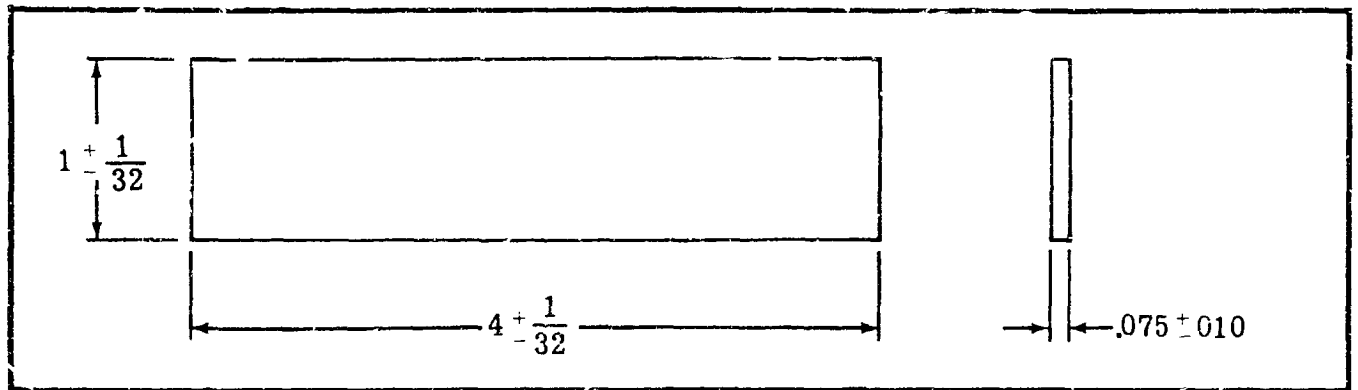


Figure A-2. Specific Heat NA 1021

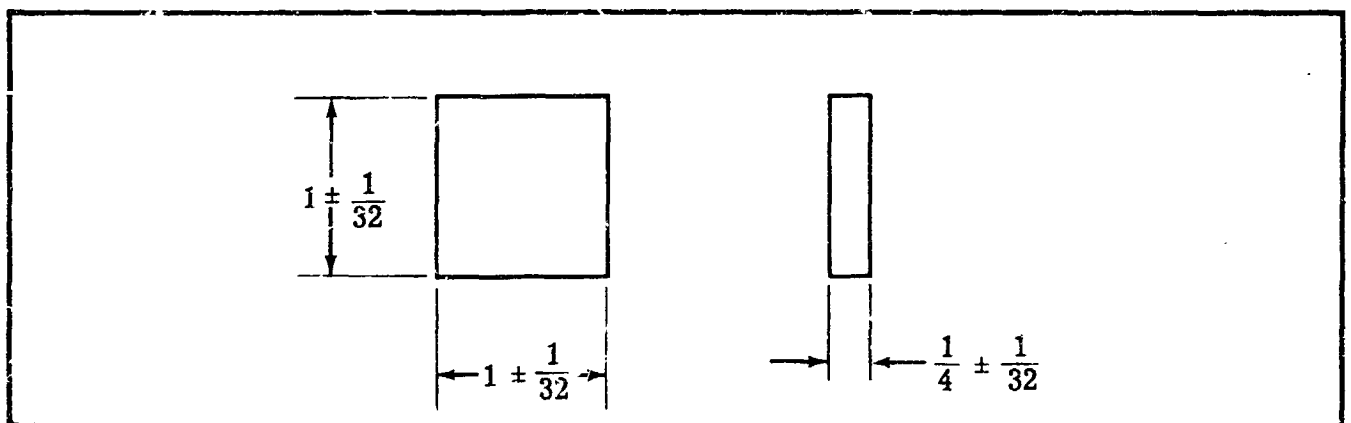


Figure A-3. Density NA 1016

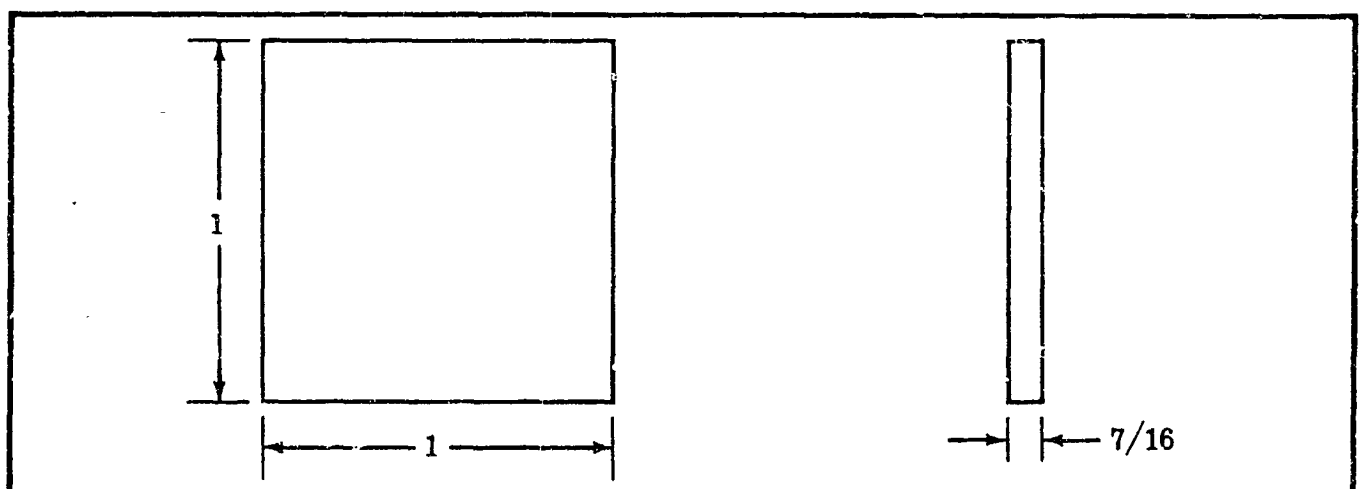


Figure A-4. Effect of Vacuum Environment Model NA 1019

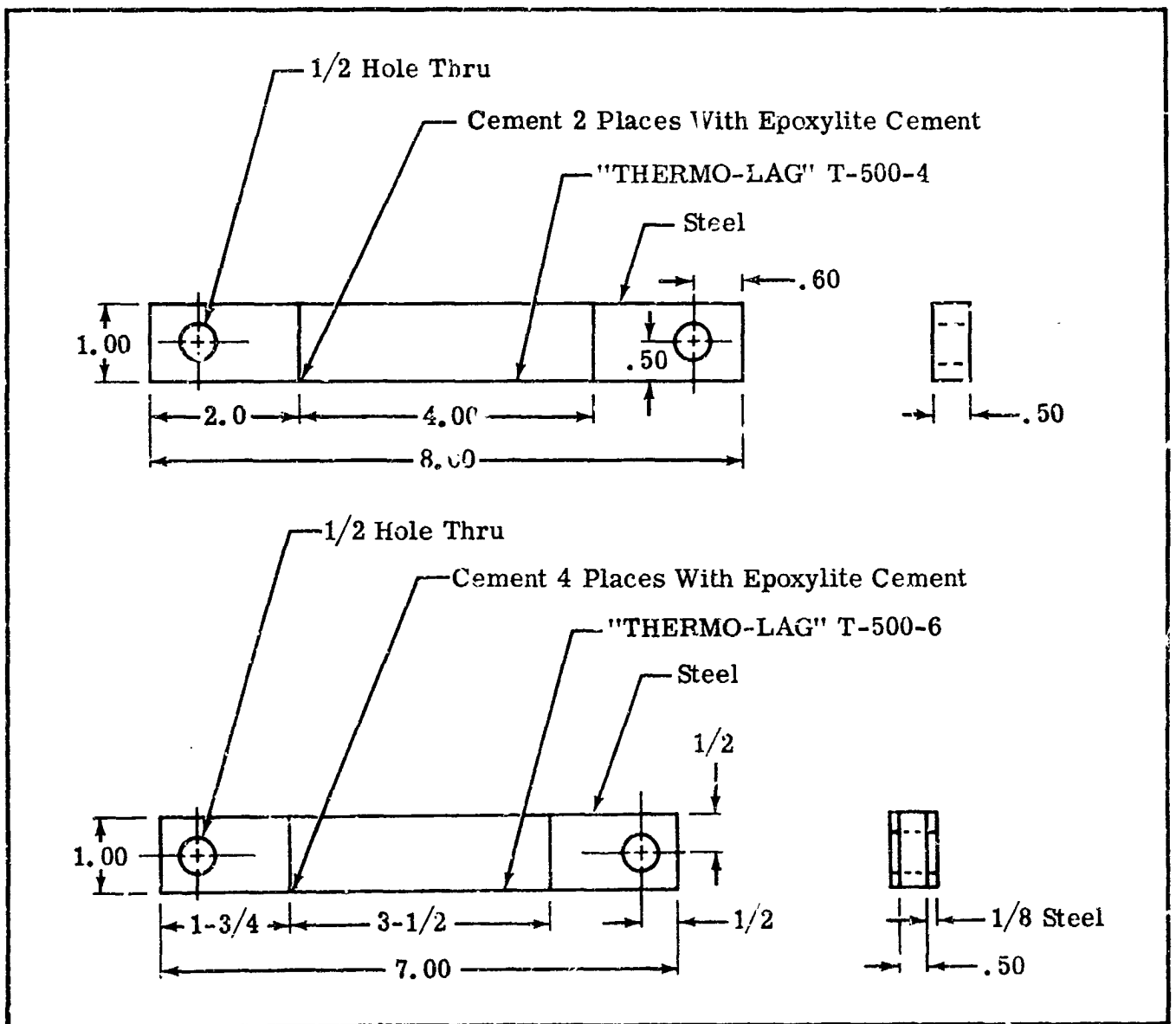


Figure A-5. Tensile Strength and Elongation Models

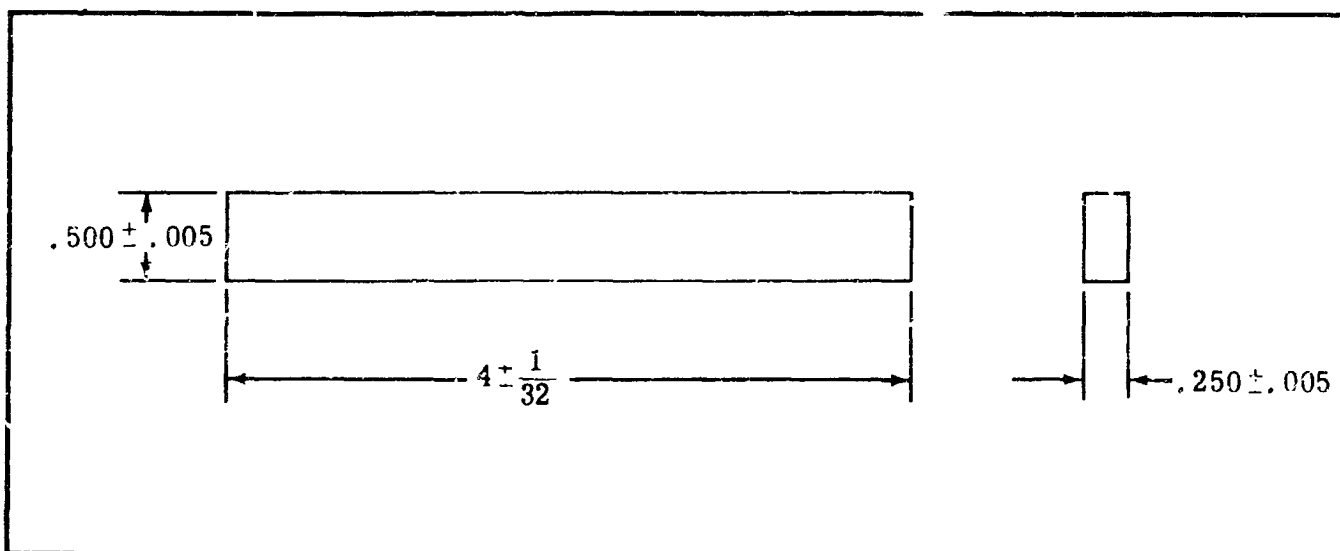


Figure A-6. Flexural Strength NA 1012

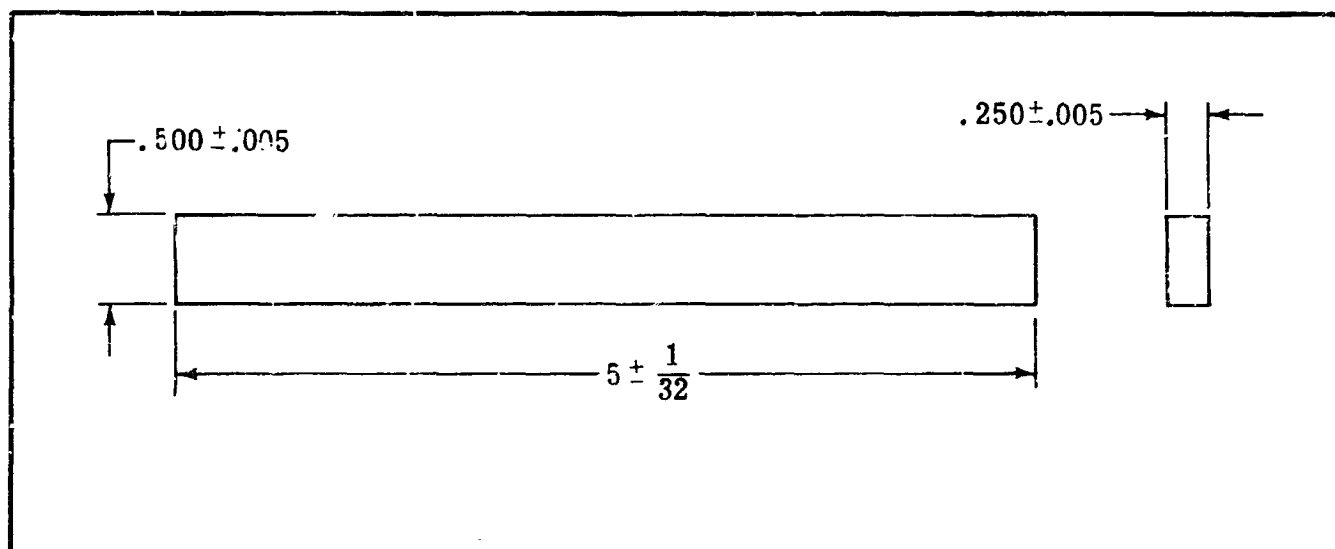


Figure A-7. Ultraviolet Exposure NA 1017

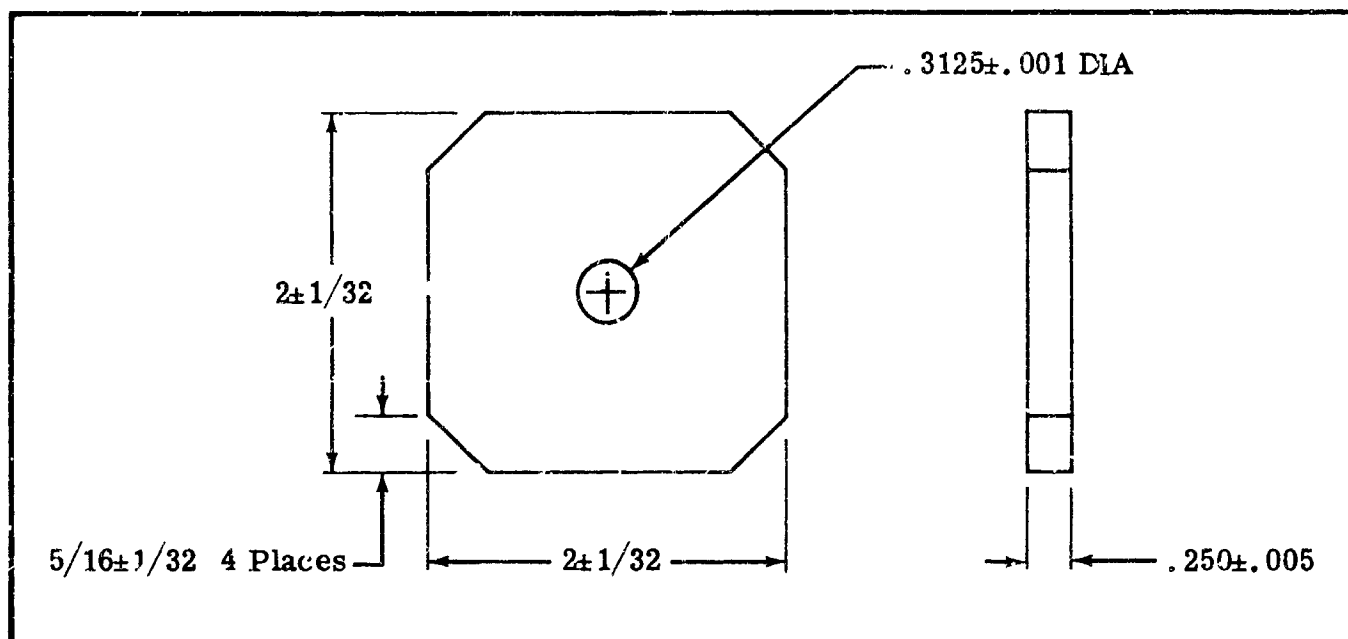


Figure A-8. Shear Strength Model NA 1011

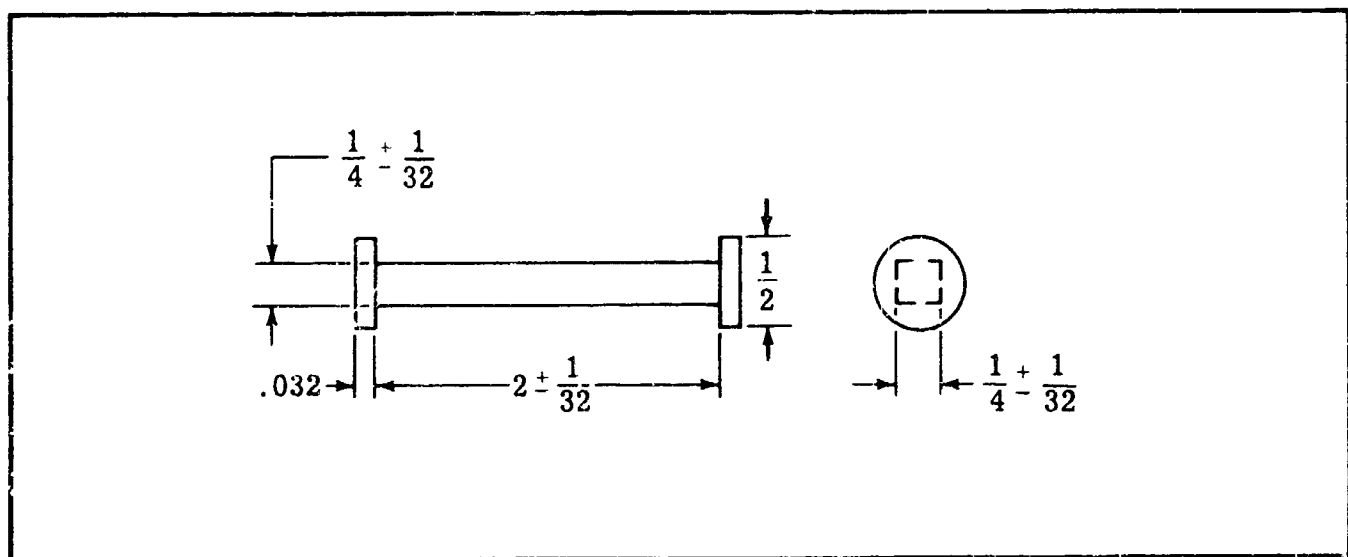


Figure A-9. Coefficient of Thermal Expansion NA 1023