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FINAL REPORT

TRANSPARENT MATERIALS PROCESSING SYSTEM

(NASA-CR-150411) TRANSPARENT MATERIALS PROCESSING SYSTEM Final Report (Hetherington (J. S.), Inc., Menlo Park, Calif.) 28 p HC A03/MF A01 CSCL 13H

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Prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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

Contract Number

NAS 8-31417

by

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ABSTRACT

A low temperature (30-400°C) and a high temperature (400-1000°C) working model of a Transparent Materials Processing System has been built and used for the acquisition of data and practical experience. Because of technical practical problems that demand expensive development effort, it is recommended that the development be postponed until the Space Shuttle era. The reduced requirements of the Space Shuttle over the Black Brant Rocket regarding shock, vibration, power, space, time and other parameters might reduce the development task to a cost-effective level.

FINAL REPORT

TRANSPARENT MATERIALS PROCESSING SYSTEM

NAS 8-31417

INTRODUCTION

This contract embodies two tasks:

A. Design, fabricate, test and deliver to MSFC a test model of a transparent materials processing furnace system.

B. Make engineering projections of a fully operational transparent materials processing furnace system.

Both tasks support the common objective of designing a zero-gravity processing facility that will allow acquisition of photographic or other visual information while the sample is being processed.

ORGANIZATION OF THE STUDY

During the early part of 1976 a meeting was held at MSFC and the dominant interest was seen to be processing between 50°C and 400°C. The original contract had specified a maximum temperature of 1000°C, but no minimum temperature. It was decided that the project should encompass two test models:

A. A low temperature test model with a flat specimen heated by quartz-halide lamps.

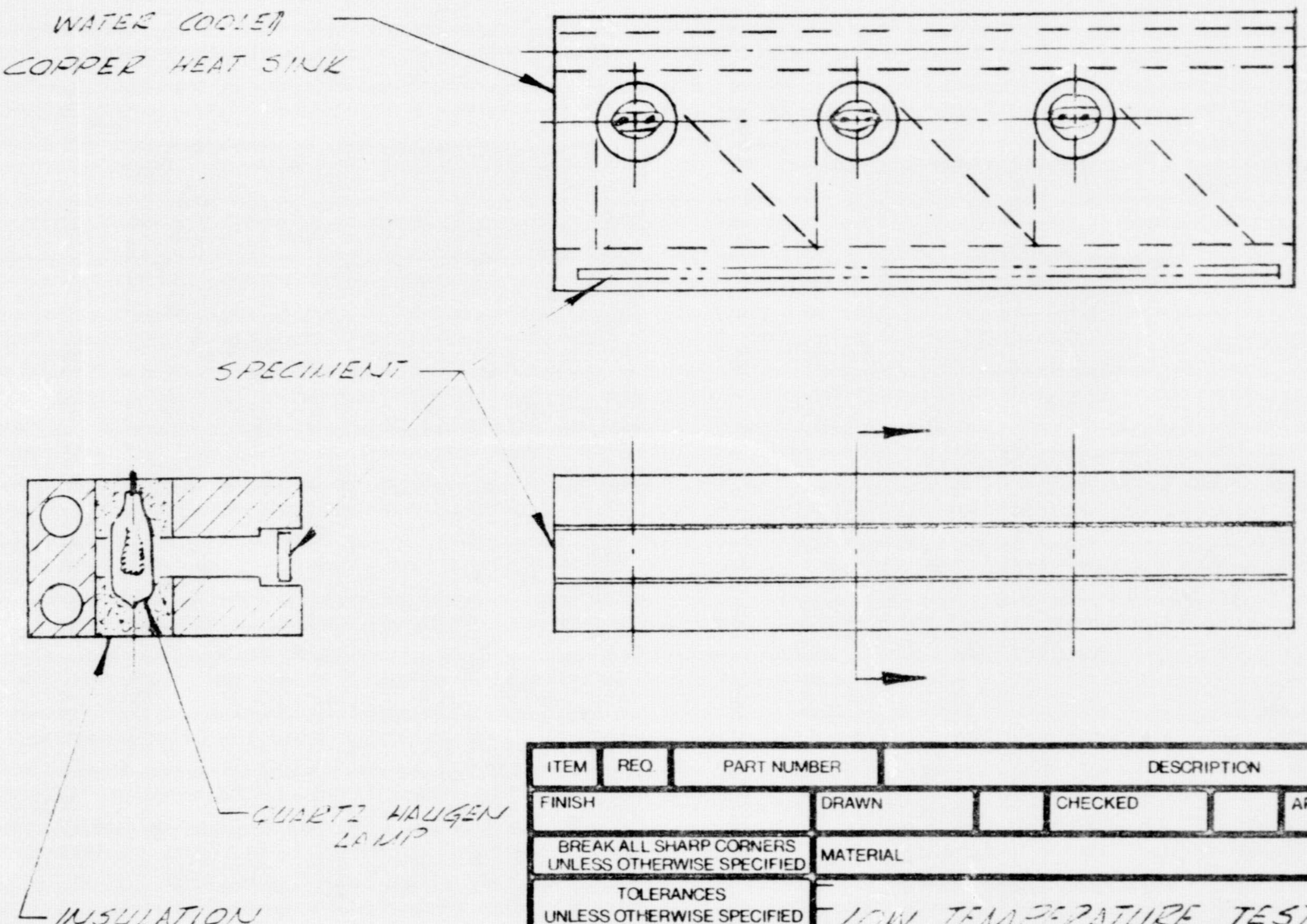
B. A high temperature best model heated by resistance heaters, utilizing a cylindrical specimen and optics.

Each of these two test models were designed and built and are discussed below.

LOW TEMPERATURE TEST MODEL

The low temperature test model was designed for temperatures up to 400°C. It is shown in Sketch 1 and Photo 1, LOW TEMPERATURE TEST MODEL. The following comments are pertinent.

A. The requirement for 1000°C maximum temperature places restraints on specimen size (via limiting the power available) that are unnecessary at low temperatures.



ITEM	REQ.	PART NUMBER	DESCRIPTION

FINISH | DRAWN | CHECKED | APPROVED

BREAK ALL SHARP CORNERS UNLESS OTHERWISE SPECIFIED | MATERIAL

TOLERANCES UNLESS OTHERWISE SPECIFIED
.X = ± ______
.XX = ± ______
.XXX = ± ______
ANGULAR ± 0°30'
SURFACE ROUGHNESS √

LOW TEMPERATURE TEST MODEL

ELECTROFUSION CORP.
MENLO PARK
CALIFORNIA

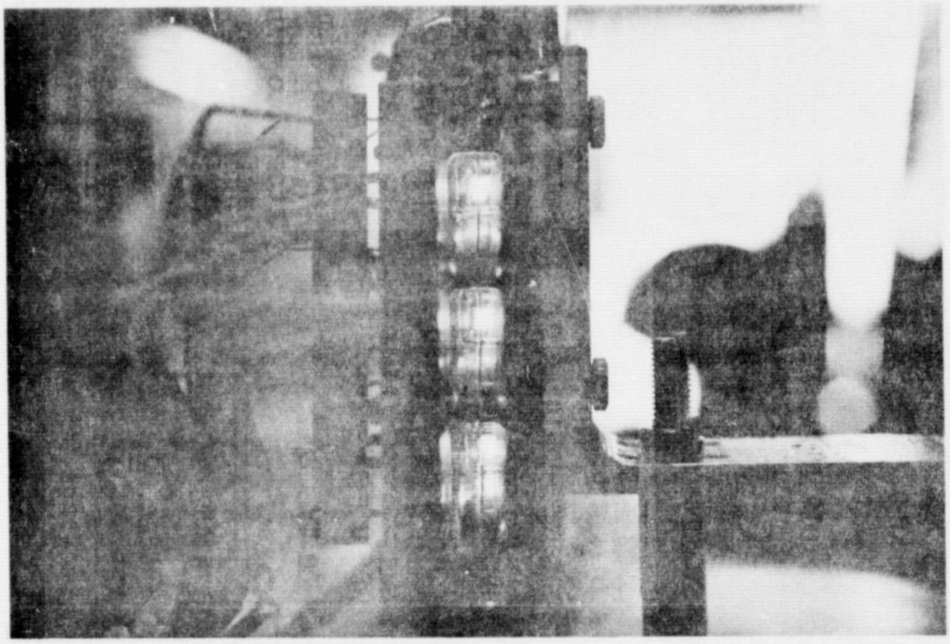
DO NOT SCALE DRAWING

SK-1

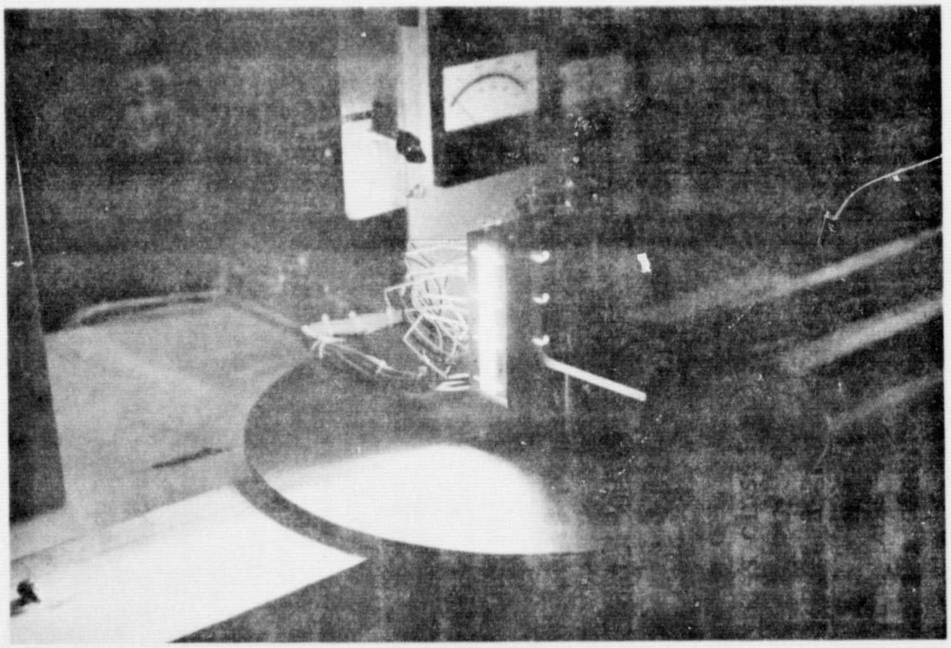
JOB NUMBER | FINAL ASSEMBLY | NEXT ASSEMBLY | SCALE

PHOTO 1

LOW TEMPERATURE TEST MODEL



1A With specimen removed to show quartz Halogen lamps.

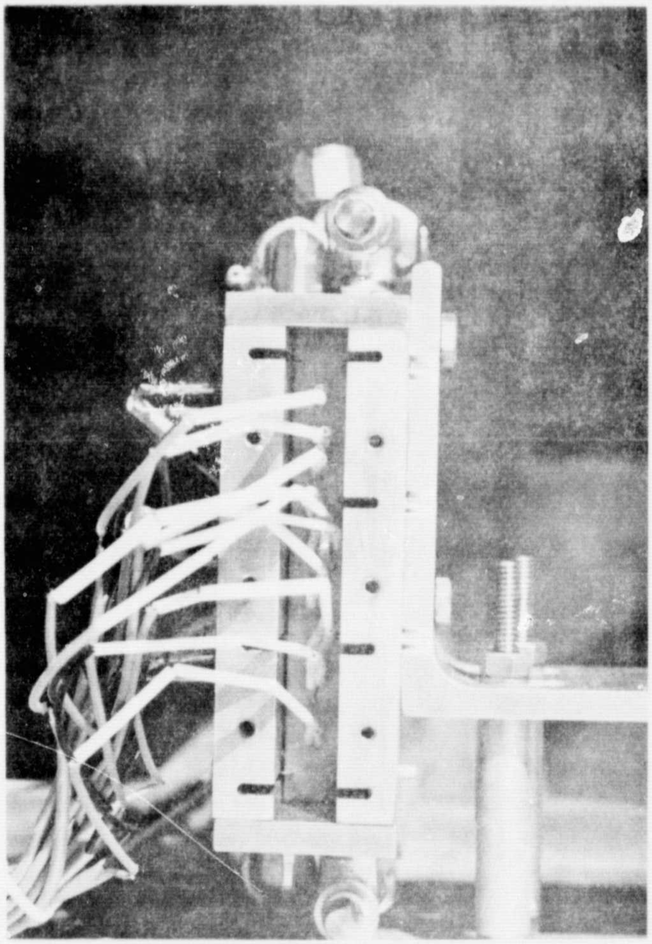


1B With specimen removed, showing lamps at full brightness.

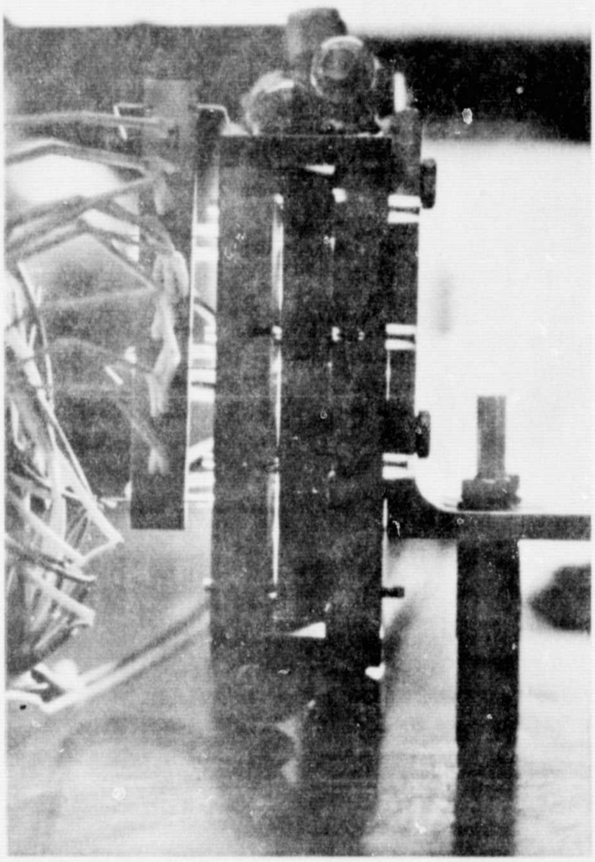
REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR

PHOTO 1

LOW TEMPERATURE TEST MODEL



1C Low Temperature Test Model with instrumented specimen installed.



1D Low Temperature Test Model with specimen installed, installed, instrumented specimen aside.

REPRODUCIBILITY OF THE ORIGINAL PAGE IS POOR

The low temperature specimen size was 1.5mm thick, 10mm wide and 100mm long.

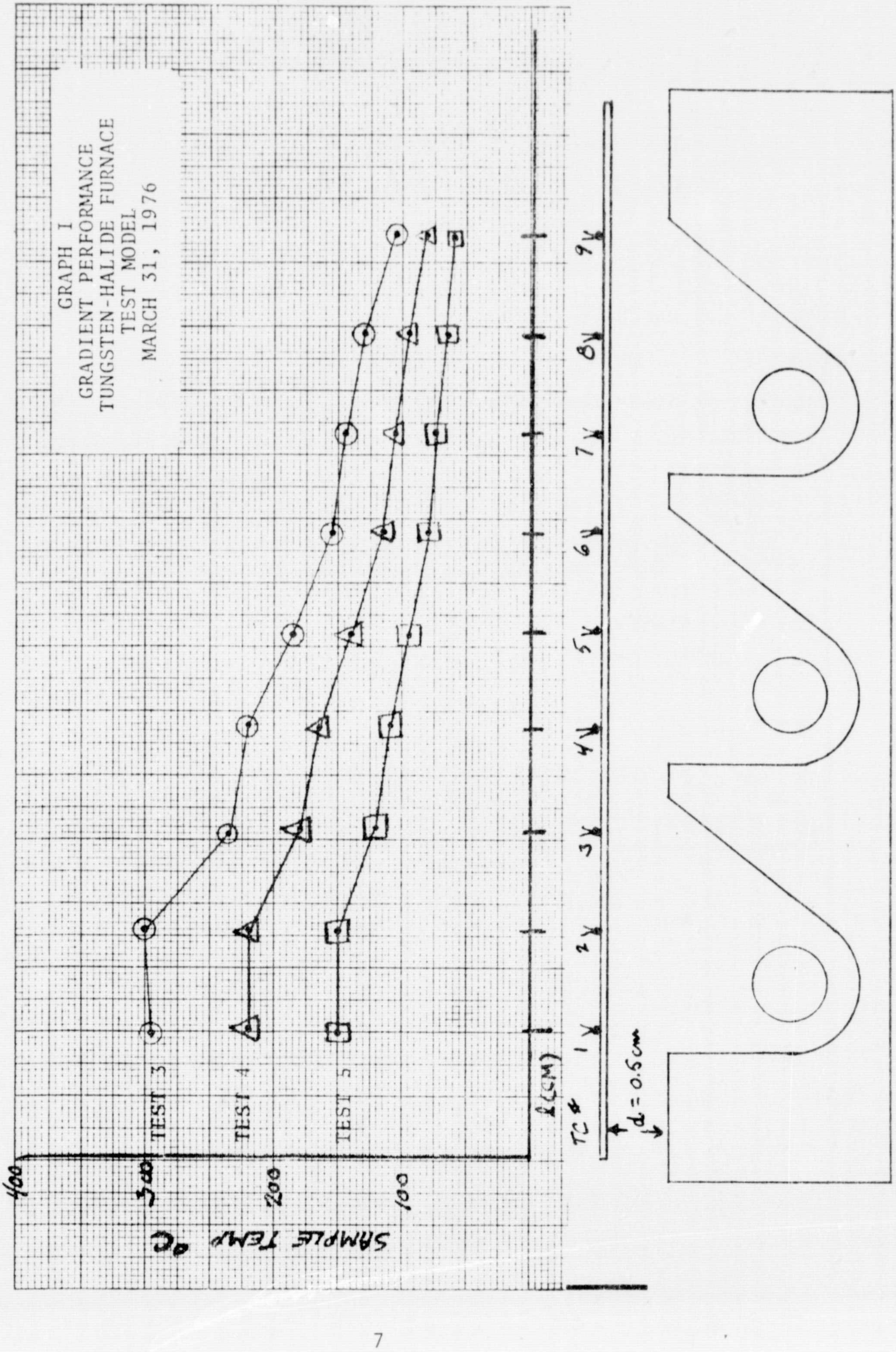
B. At low temperatures it is a simple matter to heat a rather large, flat specimen which has the following design features:

1. It can be heated indirectly from one side and photographed from the other, making the optical and photographic system comparatively simple. Although the specimen is not incandescant, it can be illuminated if opaque. Transparent specimens present a difficult photographic problem because the temperatures of the three quartz-iodine lamps are different from each other and changing with time if gradient techniques are involved. It is the conclusion of this study that the low temperature system is useful for opaque specimens or transparent specimens at uniform temperature, in which case the specimen can be heated indirectly by an opaque receptor.

2. Since the flat specimen can be made quite thin, the thermal conductivity from end-to-end could be made quite high and it was comparatively easy to attain large, uniform temperature gradients in metals such as stainless steel. Graph I shows data exemplary of this mode of operation using a stainless steel specimen located 0.5 cm from the reference face and no water or gas cooling at the end of the specimen.

3. Since the source of radiant energy does not surround the specimen the distance from the source to the specimen can be changed easily. Tables I and II show data for the bulbs at the same power and at three different power levels to establish a gradient. For a specimen of given material it is most convenient to adjust the specimen dimensions and location to obtain the desired uniformity or gradient.

4. Because of the low thermal mass and high operating temperature of the quartz-halide lamps and the high surface area to volume ratio of the specimen in this configuration, heat up and cooldown rates are quite acceptable. Graph II shows heat up and cooldown curves for a stainless steel specimen in air, without water cooling.



GRAPH I
GRADIENT PERFORMANCE
TUNGSTEN-HALIDE FURNACE
TEST MODEL
MARCH 31, 1976
TEST 3
TEST 4
TEST 5
SAMPLE TEMP °C
400
300
200
100
TC #
l (CM)
1
2
3
4
5
6
7
8
9
d = 0.5cm

TABLE I

LOW TEMPERATURE TEST FURNACE

SPECIMEN TEMPERATURE DISTRIBUTION
vs.
REFERENCE SPACING

DATE: March 29, 1976

OPERATOR: G. Geis

OBJECTIVE: Study temperature uniformity as a function of spacing.

CONDITIONS: Uniform bulb temperature-stainless steel specimen.

BULB	VOLTAGE	CURRENT
TOP	24	5.65
CENTER	24	5.70
BOTTOM	24	5.50

TEMPERATURE DISTRIBUTION

°C	TC#	0	5 MM	10 MM	15 MM
	1	285	280	250	200
	2	320	320	290	235
	3	310	335	305	260
	4	330	355	320	265
	5	335	355	315	275
	6	290	330	290	260
	7	310	340	290	265
	8	280	310	260	250
	9	240	250	240	240

TABLE II

LOW TEMPERATURE TEST FURNACE

SPECIMEN TEMPERATURE
vs.
REFERENCE SPACING

DATE: March 29, 1976

OPERATOR: G. Geis

OBJECTIVE: Study gradient linearity & magnitude as a function of spacing.

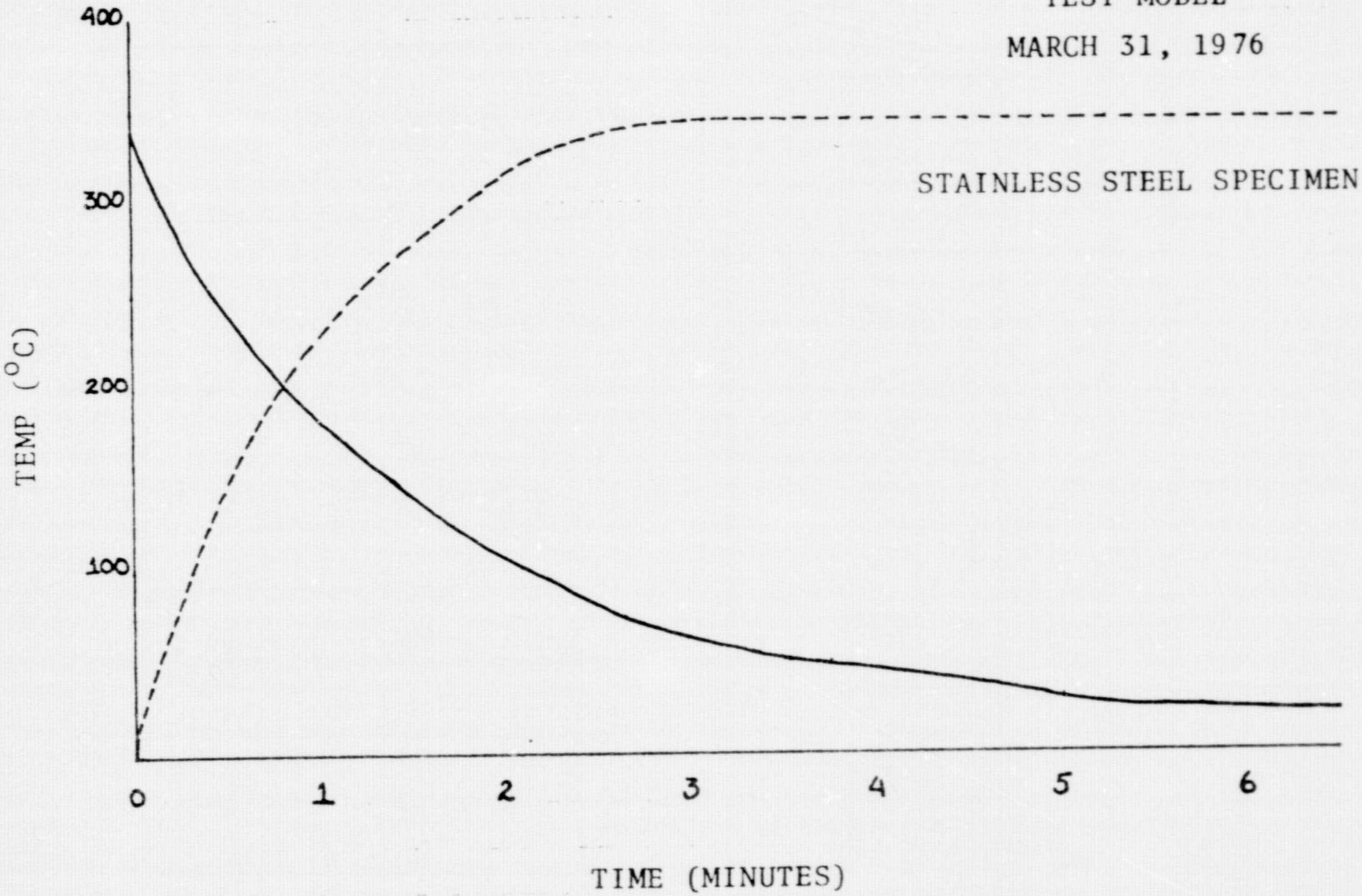
CONDITIONS: Gradient bulb temperatures-stainless steel specimen.

BULB	VOLTAGE	CURRENT	POWER
TOP	24	5.60	134.4
CENTER	20	5.20	104
BOTTOM	16	4.60	73.6

TEMPERATURE DISTRIBUTION

TC#	0	5 MM	10 MM	15 MM
1	260	265	230	190
2	300	300	260	220
3	280	300	270	230
4	280	305	270	235
5	270	290	260	230
6	225	255	230	205
7	220	240	220	200
8	195	215	200	190
9	140	165	160	155

GRAPH II
HEATUP/COOLDOWN CHARACTERISTICS
TUNGSTEN-HALIDE FURNACE
TEST MODEL
MARCH 31, 1976



T/C #2
ALL BULBS 134.8 WATTS

The low temperature model was built and tested and sufficient experience acquired to project the probable success and characteristcs of a fully-developed system of this type.

HIGH TEMPERATURE TEST MODEL

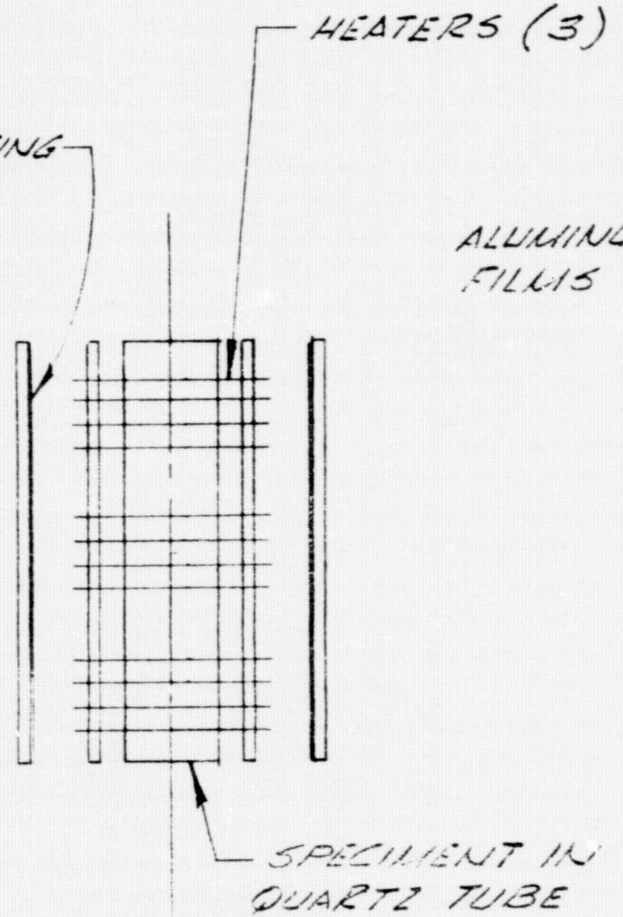
Anticipating that the performance below 400°C for the high temperature system would be unsatisfactory and that performance below 400°C could be obtained by another furnace, a high temperature test model was designed, built and tested for the purpose of acquiring experience and data concerning various aspects of the feasability of such a furnace. The high temperature test furnace is shown in SKETCH 2 and PHOTO 2. The high temperatue test model that was built and operated revealed several profound difficulties that would require extensive development effort before such a furnace could become operational in a Black Brant Rocket. They are:

A. OPTICAL SYSTEM. The desirability for photographing as much of the surface of the sample as possible is very, very high. Pursuit of this goal within the constaints of space and weight allowed tend to force the specimen to being very small. The best choice appears to be simple, conical mirrors (after study of spherical and other mirrors) fabricated from a single piece of plastic. If necessary the plastic reflecting surfaces can be backed up with a heat sink material if the aluminum film is covered with a protective coating of magnisium dioxide or silica.

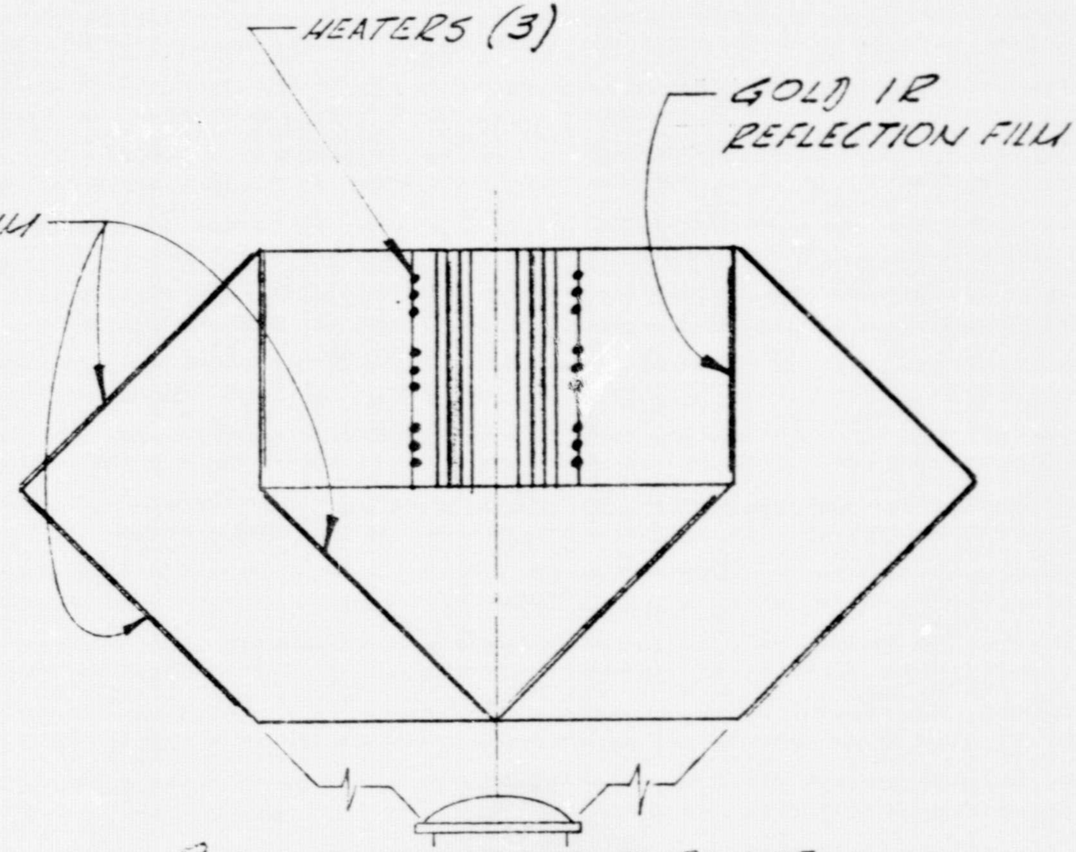
The advantages of the plastic optical system are numerous. Some of them are:

1. One-piece construction completely eluminates alignment problems arising from shock and vibration or even the need to disassemble the optical components for refurbishing or loading and unloading the furnace.

2. Weight. The support frame to hold the components (of a conventional optical system) rigidly enough to withstand the shock and vibration of the Black Brant Rocket are prohibitively heavy. The one possibility studied, beryllium, is technically possible but prohibitively expensive. Since plastic is less dense than quartz or glass, both of those materials are eliminated because the optical system for a reasonably sized sample exceeds the weight allocation for the entire furnace.



2A - BASIC TEST FURNACE (SCHEMATIC)



2B - BASIC TEST FURNACE WITH PLASTIC OPTICS

ITEM	REQ.	PART NUMBER	DESCRIPTION

FINISH | DRAWN | CHECKED | APPROVED

BREAK ALL SHARP CORNERS UNLESS OTHERWISE SPECIFIED | MATERIAL

TOLERANCES UNLESS OTHERWISE SPECIFIED
X = ±
XX = ±
XXX = ±
ANGULAR ± 0° 30′
SURFACE ROUGHNESS √

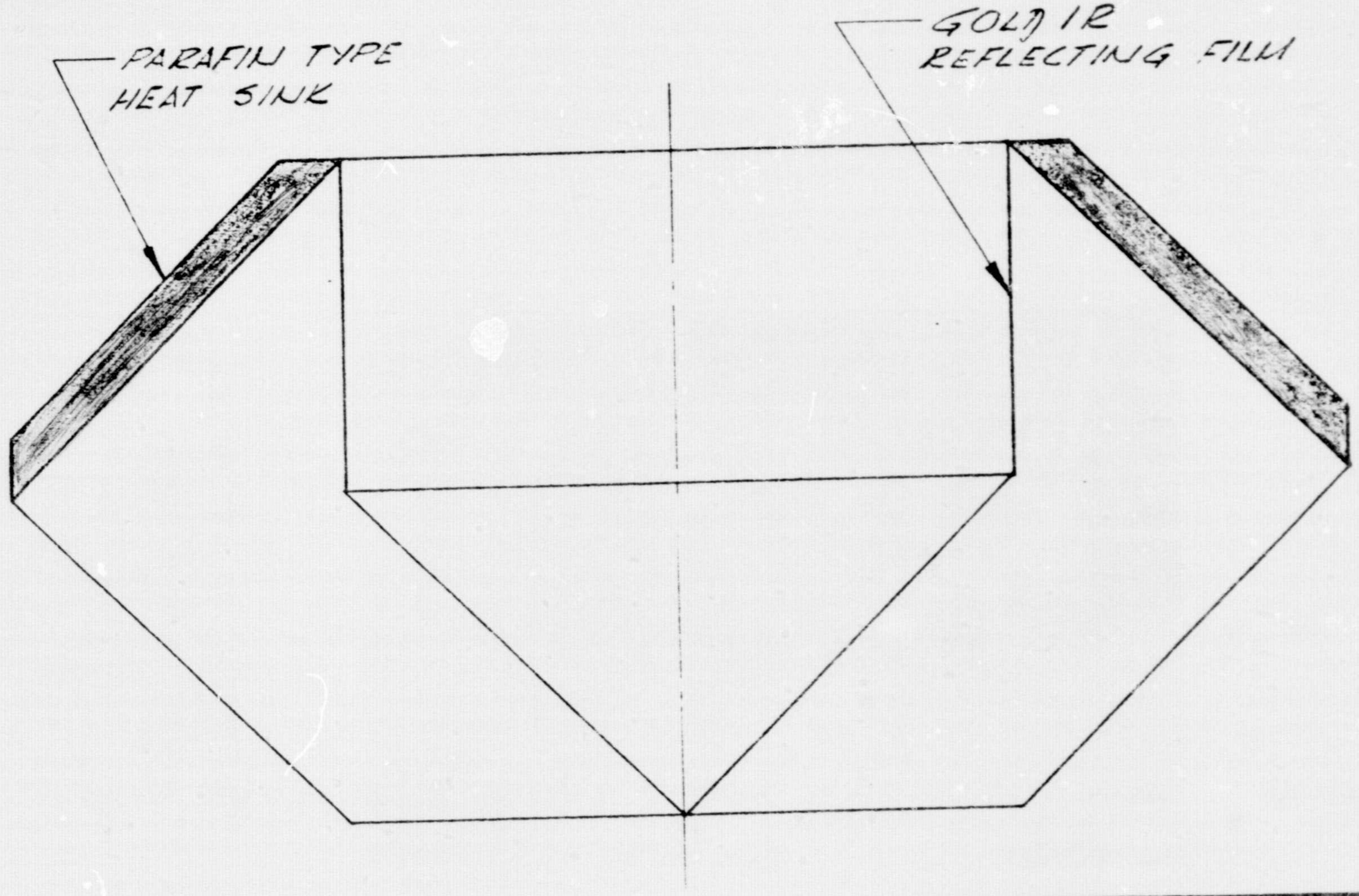
HIGH TEMPERATURE TEST FURNACE

ELECTROFUSION CORP.
MENLO PARK
CALIFORNIA

DO NOT SCALE DRAWING

SK-2A&B

JOB NUMBER | FINAL ASSEMBLY | NEXT ASSEMBLY | SCALE



ITEM	REQ.	PART NUMBER	DESCRIPTION		
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BREAK ALL SHARP CORNERS UNLESS OTHERWISE SPECIFIED		MATERIAL			
TOLERANCES UNLESS OTHERWISE SPECIFIED X = ± XX = ± XXX = ± ANGULAR ± 0°30' SURFACE ROUGHNESS √		CONICAL MIRROR WITH HEAT SINK			
		ELECTROFUSION CORP. MENLO PARK CALIFORNIA		DO NOT SCALE DRAWING SK-2c	

JOB NUMBER	FINAL ASSEMBLY	NEXT ASSEMBLY	SCALE

PHOTO 2

HIGH TEMPERATURE TEST FURNACE

WITHOUT PLASTIC OPTICS

REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR

2. As the heating element surface area decreases and the temperature difference between the heating element and the specimen increases, the specimen will exhibit "toaster burns", or hot spots, that conform to the location of the heating element. In much experimental work this will be intolerable.

The heating element must also make compromises with several other realities, such as:

a. Within the physical size constraints it must have the correct electrical resistance. This can be done with alloys such as Chromel-A and Hoskins Alloy 875, but platinum wire (for example) cannot be made to "fit" mechanically and electrically simultaneously.

b. While matching electrically and mechnically, the heating element must withstand the shock and vibration of the rocket firing, sometimes while the heating element is at a high temperature.

c. The thermal mass of the heating element and its supporting frame must be very small so that the element can change temperature quickly. This leads to the problem of the heating element resonating during the vibration of firing.

Such a heating element appears to be feasable if a coil of resistance wire is mounted on an Alumina rope then covered with a braid made of the same material. This could then be mounted in a groove in a quartz tube to prevent chafing. This design was attempted, but preliminary tests were not completed. All tests were done with a nickle-chromium wire supported on a helical spiral of quartz.

C. CENTRAL QUARTZ CYLINDER. The central quartz tube that holds the heating element must have a spiral groove on the outside and be optically polished both inside and outside. The first attempt to make two pieces resulted in one broken piece and one optically unacceptable piece.

3. Specimen Coverage/Focus. Using radial symmetry places the entire surface of the specimen in focus without depth of field problems inherent in a cylindrical specimen being photographed from one radial position. The entire specimen surface, minus the amount obscured by the heating element, is photographed. This feature is particularly attractive in experiments involving a nucleation site that occures at a random place and then grows.

In the course of building the high temperature test furnace an optical consultant was retained to design and supervise the fabrication of the plastic optical piece. Unanticipated difficulties were encountered in the following areas:

1. The gear-driven lathe used for grinding caused "chatter", gauging, and scoring of the optical surfaces to an extent that could not be polished away.

2. For various reasons tolerances could not be held.

3. The variation of polishing speed with radius gave a much larger effect than envisioned.

For these and other technical reasons, it was decided to abandon the effort rather than invest additional funds in a more elaborate procedure.

B. HEATING ELEMENT. There is a very basic problem with the heating element because it impedes the transparency that is so desirable. If the heating elements are made very small to avoid hiding the element, the gains in visibility are made at the expense of two other difficulties.

1. In order to obtain a given specimen temperature, the heating element temperature must increase with decreasing heating element surface area. This increased temperature difference makes photography very difficult, as photographic films generally have a very limited dynamic range (particularly color films) and cannot accommodate large differences in color temperature.

D. THERMAL RESPONSE. The limitation of approximately 300 watts (or 500 for short periods) and the limitation of the heating element material to about 1200°C makes it almost impossible to heat more than a very small (2mm dia x 8 mm long, est.) specimen to 1000°C or to cause a specimen temperature excursion from, approx. 800°C to 900°C in time periods short campared to the zero-g period of four minutes. Natural cooling periods with large specimens were prohibitively long, but forced cooling with water or helium yielded cooling times of practical magnitude with large (5mm x 100 mm) samples.

E. AMPOULE DESIGN. While it is beyond the scope of this contract to design an appropriate ampoule to hold the sample, a few words in this report are appropriate. Since the ampoule must be transparent, the only candidate for a material is quartz. In developing an ampoule the following obvious problems would require solutions.

1. The quartz has a very low coefficient of expansion (7×10^{-7} in/in/°C) whereas most metals and other materials are comparatively large (15×10^{-6} in/in/°C). This difference dictates that the sample must fit loosely in the ampoule at the outset, otherwise, it will expand and crack the ampoule as it heats the first time. When the rocket is fired the loose sample is likely to "rattle" and break the ampoule. Melting the sample prior to firing might complicate the vortexing problem during spin/despin.

2. Inherently, it is expected that the sample will freeze solid before the end of the zero-g period. Presumably, it will again contract and be "loose" in the ampoule. After the end of the zero-g period the rocket can "tumble", causing forces as high as 50 g's on the sample. It is easy to envision the loose sample bursting through the ampoule and causing considerable damage if, indeed, the sample is not lost altogether.

3. A more remote, but more subtle, problem is posed during freezing, particularly in freezing from one end to the other. There is a large probability that voids left by a contracting solid will be replaced by liquid, causing a "rippled" surface.

4. Materials, such as silicon, which have negative coefficients of expansion will expand when frozen, cracking the quartz ampoule.

5. Differential expansion also implies that there will always be a void (probably vacuum) somewhere in the ampoule. The presence of the void, under zero-g conditions might result in problems of two kinds.

   a. The void comes to rest at the cooled end of the ampoule, disturbing the pattern of freezing.

   b. The void freezes elsewhere at an undesirable place.

6. The ampoules will have to accomodate a great variety of materials and be filled and sealed by laboratory workers in various parts of the world. The equipment and skills needed for filling and sealing must be inexpensive and easily acquired.

Undoubtedly MSFC has gained a great deal of experience with quartz during past programs, and if necessary, this experience could be brought to bear on these problems.

F. PHOTOGRAPHY. The one major problem that will have to be given a great deal of attention and development work for acceptable performance is the photography. Some problems and potential problems are commented on below:

1. The temperature difference between heating element and photographed specimen. The only practical design concepts have the heating element in view when the specimen is photographed. An exposure value for the specimen surface will cause "blooming" at the over exposure of the heating element. There are two ways around this problem:

a. Illuminating the specimen by a very blue flash lamp, filtering out all of the red/yellow light reaching the camera. This is very inefficient because the gold insulating film is a poor transmitter of blue light, and all other surfaces imply multiple losses.

b. If photo requirements were for one photo every minute, the power could be electronically switched off once a minute, then the flash "triggered" electronically when the heating element and specimen temperatures match. Since the specimen is a large thermal mass compared to the heating element it would change in temperature very little by comparison. While such a system might be acceptable in some experiments, this might be unacceptable in other experiments.

2. The ability of the film to accommodate a range of brightness will limit the ability to photograph a specimen with a gradient. But present experience indicates that the thermal conductivity of the specimen will dominate and limit the magnitude of the gradient.

3. Color Distortion. One can imagine that there will be experiments in which the color of the surface during the zero-g time is of great interest. If so, there are some formidable barriers to excellent color reproduction. Namely;

   a. The presence of the gold film for insulation. One could envision an "equal and opposite" filter to be used during enlarging the prints as a correction; however, the gold films in the reflectors might require frequent changes because they probably "age" very quickly and soon would not match the correction filter.

   b. Color distortion resulting from using blue flash/red filters. These two colors added to the gold film compound the difficulty of obtaining excellent temperature rendition.

c. Temperature Gradients. Without the blue flash/red filter technique the severe change in color temperature of the heating element will always result in color distortion.

d. Temperature Changes. In many cases a series of photos will be desired showing the surface as it cools from one end to the other. It is likely that color distortion will be introduced here and that a scheme will have to be developed to introduce color correction filters into the film processing sequence.

e. Spurious reflections from the multiple quartz surfaces interfere with the "view" of the surface.

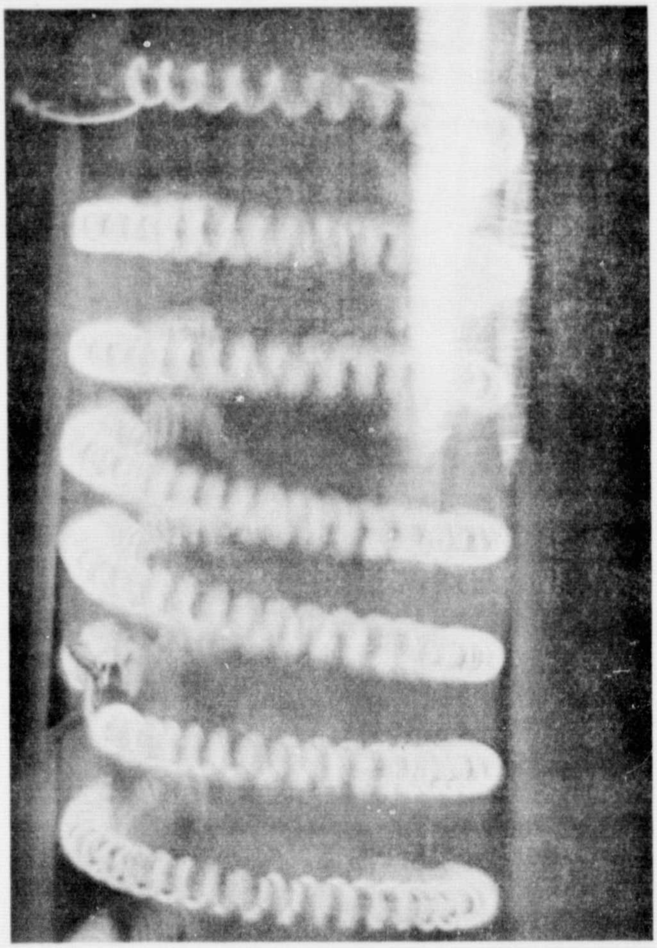
f. Ampoule coating. In some cases it was apparent that a reaction between the quartz ampoule and the liquid metal created a film that obscured the "true" surface of the molten metal.

Examples of some photographs taken in the course of work with the high temperature test furnace are shown in Photos 3, a, b, and 4.

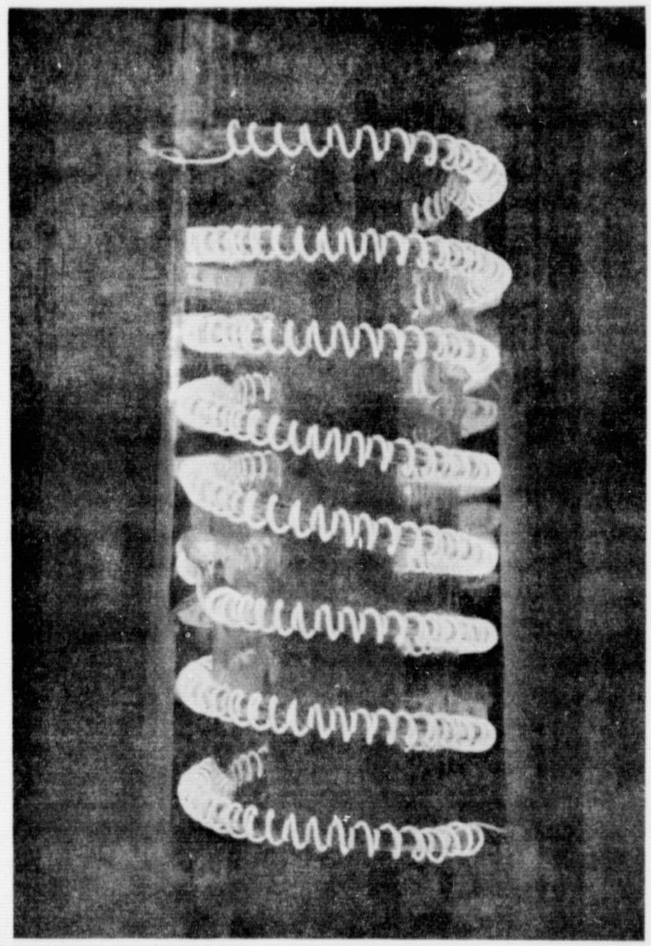
In view of these rather formidable practical problems, it is the final conclusion of this report that development of a transparent materials processing system for use on the Black Brant Rocket will be the result of a very expensive development. Only an experimental objective with unusually high importance and absolute need to acquire photographic information concerning the surface of the sample would justify such a development. Because the shock and vibration, weight, power and time constraints on the Space Shuttle are far less demanding, the development of such equipment for use on the Space Shuttle would be far less expensive and much more productive.

PHOTO 3

(Illustrating the difficulty of photographing the sample surface in the presence of the heating element.



a. High Temperature Test Furnace. Uniform temperature 680°C. Sample temperature.



b. High Temperature Test Furnace. Uniform temperature 840°C. Sample temperature.

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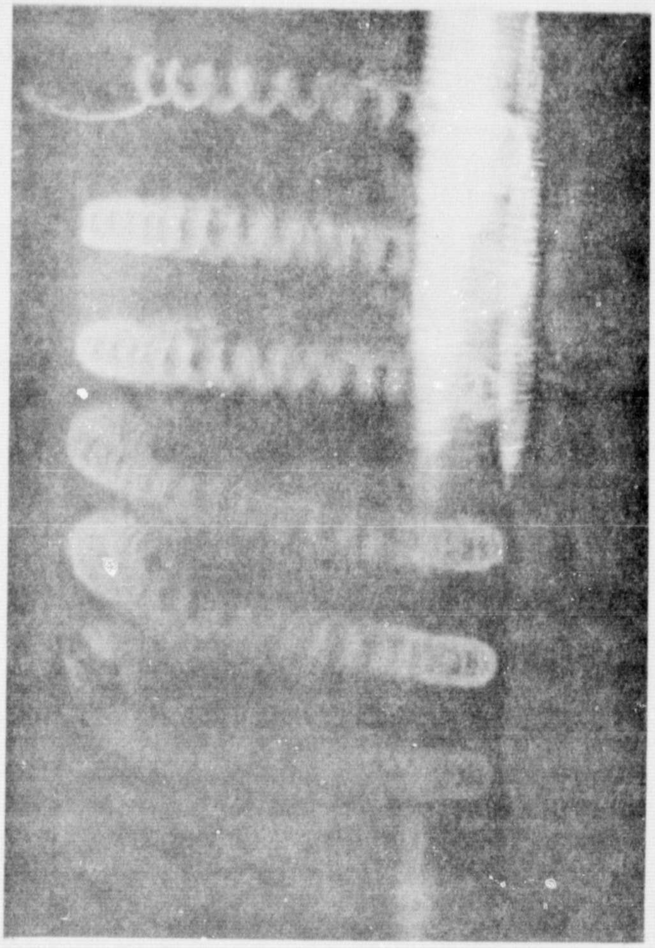
ORIGINAL PAGE IS POOR

PHOTO 4

(Showing the difficulty of photographing a gradient).



ENGINEERING PROJECTIONS OF A FULLY OPERATIONAL TRANSPARENT MATERIALS PROCESSING SYSTEM.

Looking forward to the availability of the Space Shuttle as a vehicle, one can project from the test data acquired on the test models the following characteristics of a low temperature furnace.

A. Configuration (Sketch 3). It appears that more than three quartz-iodine lamps are necessary to maintain either excellent isothermal conditions or to establish uniform gradients. Therefore, it is likely that the system will utilize a quartz ampoule 1 cm wide x 20 cm long with ten quartz-iodine lamps behind it in an arc-shaped housing. This will bring the end bulbs closer to the sample ends to overcome end effects and provide more increments of adjustment.

B. Power. Each bulb operated at 24 volts, 300 watts. Three KW will be needed to produce full temperature in all ten bulbs at once.

C. Cooling. Water will be required to cool one end of the sample for steep-gradient work.

D. Quench. In reduced gravity condition it will be most convenient to use helium as a quench medium.

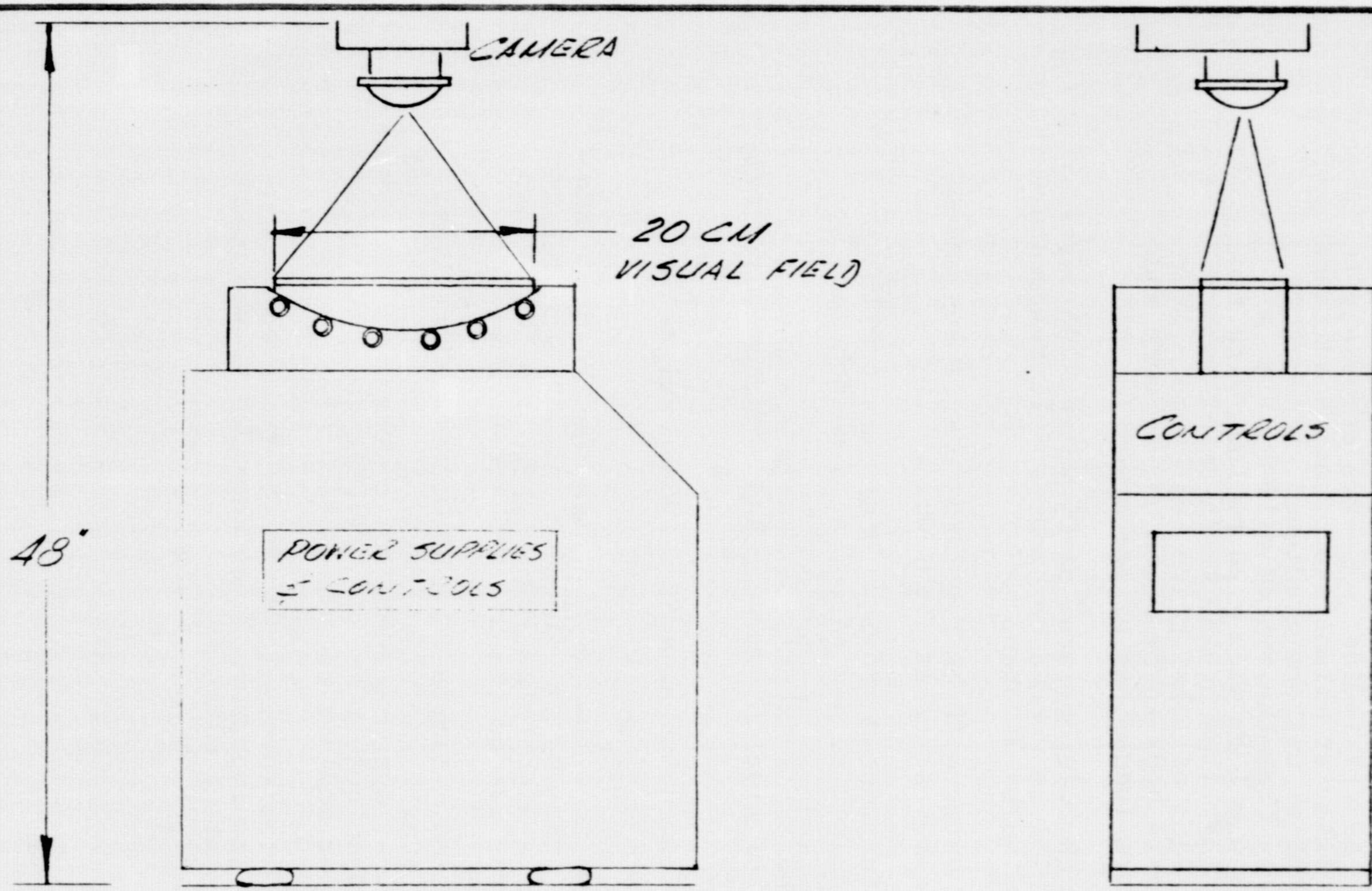
E. Space. The entire apparatus should fit onto a pallet 41 cm wide, 61 cm deep and stand approximately 122 cm high.

F. Weight. The entire apparatus should weight less than 77 kg's.

Experience has shown that the low and high temperature operations are best performed by two different pieces of apparatus. The projections for a high temperature transparent materials processing system for use on the Space Shuttle yield.

A. Configuration. Sample 1 cm diameter x 10 cm long, sealed in quartz ampoule. Heating element; platinum rhodium wire, approximately 30% coverage of the sample. Wire wound on quartz helix. Plastic conical optical system water and air cooled (Sketch 4).

B. Power. Approximately 1 KW will be required, transformed to the impedance level demanded by the platinum wire heating elements.



ITEM	REQ.	PART NUMBER	DESCRIPTION

FINISH

DRAWN | CHECKED | APPROVED

BREAK ALL SHARP CORNERS UNLESS OTHERWISE SPECIFIED

MATERIAL

TOLERANCES UNLESS OTHERWISE SPECIFIED

X = ±

XX = ±

XXX = ±

ANGULAR ± 0°30'

SURFACE ROUGHNESS √

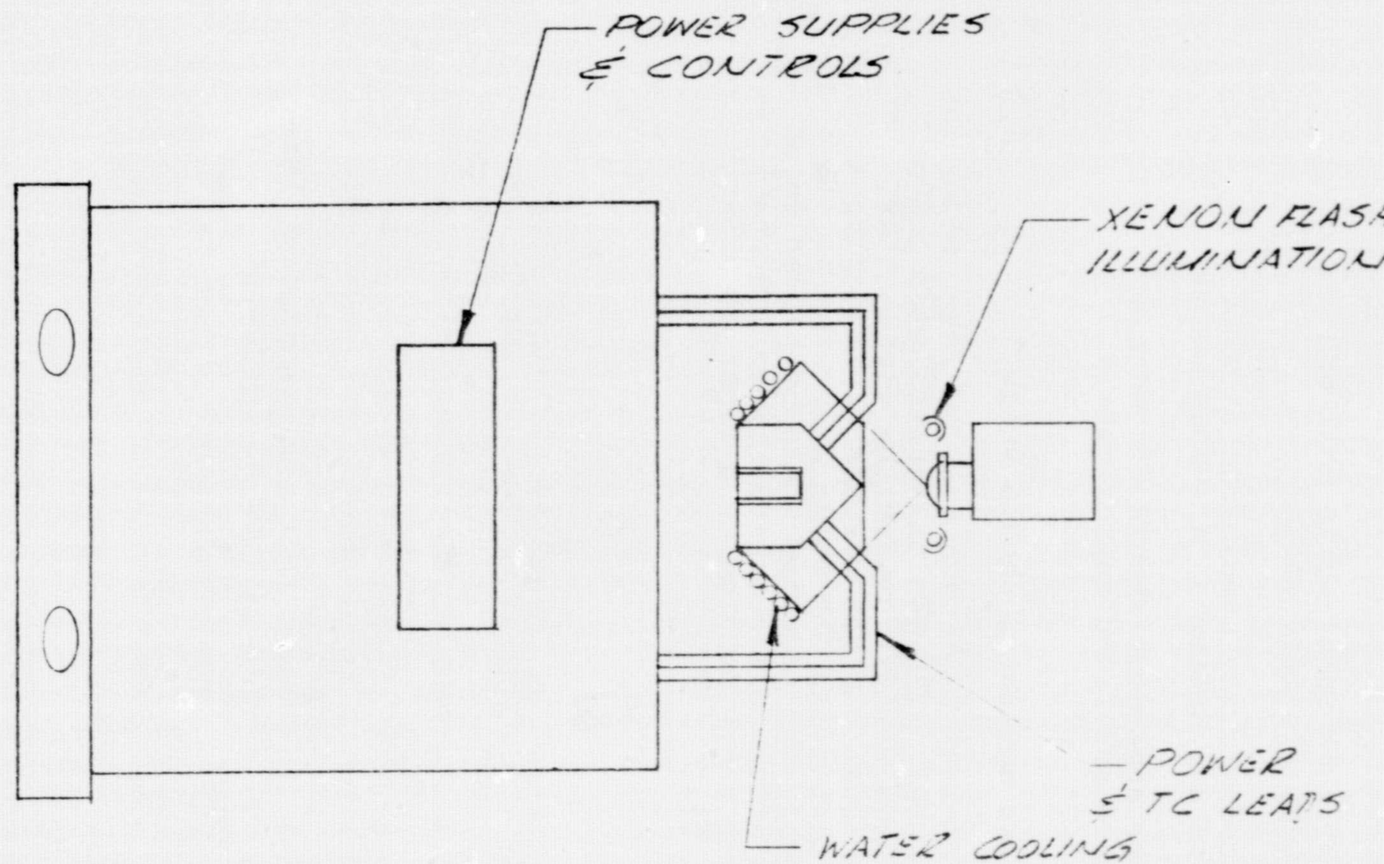
LOW TEMPERATURE TRANSPARENT FURNACE. CONFIGURATION FOR SPACE SHUTTLE

ELECTROFUSION CORP.
MENLO PARK
CALIFORNIA

DO NOT SCALE DRAWING

SK-3

JOB NUMBER | FINAL ASSEMBLY | NEXT ASSEMBLY | SCALE



ITEM	REQ.	PART NUMBER	DESCRIPTION
FINISH		DRAWN	CHECKED	APPROVED

BREAK ALL SHARP CORNERS UNLESS OTHERWISE SPECIFIED

MATERIAL

TOLERANCES UNLESS OTHERWISE SPECIFIED
X = ±
XX = ±
XXX = ±
ANGULAR ± 0°30'
SURFACE ROUGHNESS

HIGH TEMP. TRANSPARENT FURNACE FACILITY. CONFIGURATION FOR SPACE SHUTTLE

ELECTROFUSION CORP.
MENLO PARK
CALIFORNIA

DO NOT SCALE DRAWING

SK-4

JOB NUMBER | FINAL ASSEMBLY | NEXT ASSEMBLY | SCALE

C. Cooling. Water will be required to cool one end of the sample for steep-gradient work.

D. Quench. In reduced gravity conditions it will be most convenient to use helium as a quench medium.

E. Space. The entire apparatus should fit onto a pallet 61 cm square and stand approximately 122 cm high.

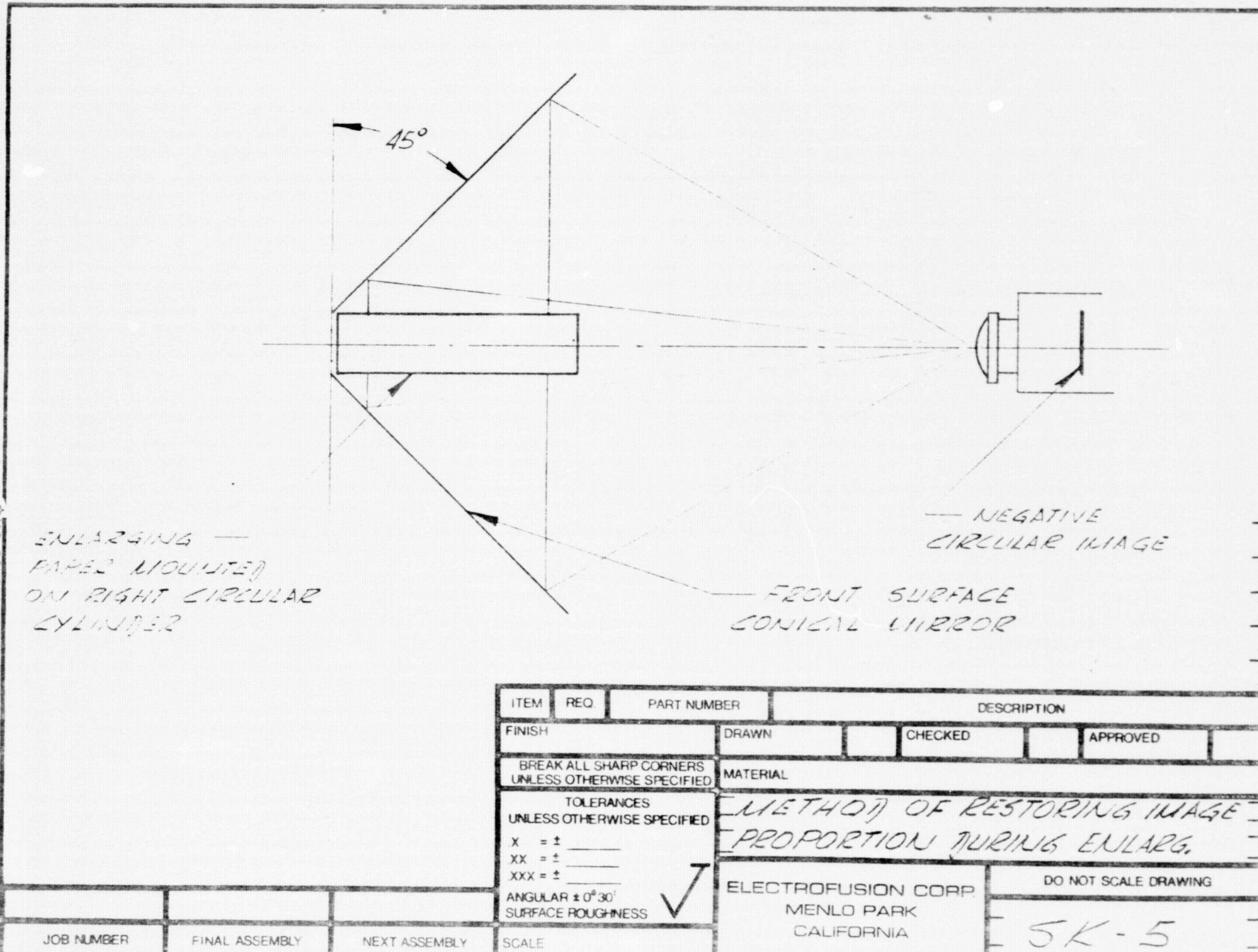
F. Weight. The entire apparatus should weigh less than 86 kg's.

G. Information Retrieval. The conical optics surrounding the cylindrical sample will convert the rectangular sheet that would cover a right circular cylinder to a circular sheet, distorting the image accordingly. To reverse this effect, the negative would be placed in an enlarger and projected into a conical mirror onto a sheet of enlarging paper mounted on a right circular cylinder (Sketch 5).

CONCLUSION

The practical problems presented by flight on the Black Brant Rocket make development of the transparent furnace justifiable only by the most rewarding project in which a photographic record of events on the surface of the sample is of primary importance.

Availability of a human operator, longer time spans, reduced shock, vibration and acceleration requirements, more available power, water, inert gases, working space, and other assets reduce the development task to a level that the transparent furnace might be justifiable for investigations of longrange interest and fundamental value.


45°
ENLARGING PAPER MOUNTED ON RIGHT CIRCULAR CYLINDER
NEGATIVE CIRCULAR IMAGE
FRONT SURFACE CONICAL MIRROR
ITEM
REQ.
PART NUMBER
DESCRIPTION
FINISH
DRAWN
CHECKED
APPROVED
BREAK ALL SHARP CORNERS UNLESS OTHERWISE SPECIFIED
MATERIAL
TOLERANCES UNLESS OTHERWISE SPECIFIED
X = ±
XX = ±
XXX = ±
ANGULAR ± 0° 30'
SURFACE ROUGHNESS
METHOD OF RESTORING IMAGE PROPORTION DURING ENLARG.
ELECTROFUSION CORP.
MENLO PARK
CALIFORNIA
DO NOT SCALE DRAWING
SK-5
JOB NUMBER
FINAL ASSEMBLY
NEXT ASSEMBLY
SCALE