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## PRELIMINARY SCIENCE REPORT ON THE DIRECTIONAL SOLIDIFICATION OF HYPEREUTECTIC CAST IRON DURING KC-135 LOW-G MANEUVERS

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16. ABSTRACT

An ADSS-P directional solidification furnace has been reconfigured for operation on the KC-135 low-g aircraft. The system offers many advantages over quench ingot methods for study of the effects of sedimentation and convection on alloy formation. The directional sodification furnace system was first flown during the September 1982 series of flights. The microstructure of the hypereutectic cast iron sample solidified on one of those flights suggests a low-g effect on graphite morphology. Further experiments are needed to ascertain that this effect is due to low-gravity and to deduce which of the possible mechanisms is responsible for it.

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TABLE OF CONTENTS

## LIST OF ILLUSTRATIONS

TECHNICAL MEMORANDUM

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## INTRODUCTION

Alloys solidified in a low-gravity environment can, due to the elimination of sedimentation and convection, form unique and often desirable microstructures [1-4]. One method of studying the effects of low-gravity on alloy solidification has been the use of the KC-135 aircraft flying repetitive low-g maneuvers. Each maneuver gives from 20 to 30 seconds of low-g which is between about 0.1 and 0.001 gravity. The frequency of KC-135 flights can provide an opportunity for a much faster learning curve than is available at the present time from either sounding rocket or orbital experiments. For experiments that require more low-g time then afforded by the droptube or droptower facilities [2,5] and which are precursorary to orbital processing experiments, the KC-135 provides a valuable research environment in which the experimenter can interact with his experiment in real time.

Previous alloy solidification experiments on the KC-135 have attempted to quench ingots of molten metal within the time span of a single low-g parabola [6-8]. Directional solidification, however, has many important advantages over the quenched ingot methods for alloy solidification experiments, especially in the KC-135. In directional solidification, the solidification interface can be slowly advanced through a rod of the sample. Controlled solidification can continue through a number of aircraft parabolas. The known solidification rate of the sample can then be correlated with accelerometer data to determine the gravity level during solidification for any location on the sample. The thermal gradient and solidification rate can be controlled independently; whereas, for the quench ingot method the complexity of the three dimensional cooling problem makes their control very difficult. Consequently, by directional solidification we can obtain fundamental scientific information not obtainable by the ingot quench techniques. Directional solidification also greatly mitigates the need to obtain one-g control samples (solidified under conditions that precisely match the flight conditions) in order to detect a gravitational effect. Directional solidification can also remove constraints on the sample cooling rates, since in directional solidification it is not necessary to completely solidify the sample in a single low-g parabola. In a collaborative effort with Dr. Mary H. Johnston, a G. E. built ADSS-P directional solidification furnace was modified to make it compatible for use on the KC-135.

Cast iron has been identified (9) as a candidate material of possible value in the study of the effects of sedimentation and convection on alloy solidification. NASA is presently involved in research efforts studying low-g solidification of cast iron for its scientific value [10] and also to obtain data of possible significance to commercial casting processes [11]. Off eutectic compositions of cast iron may be especially interesting for study in low-g. For example in hypereutectic cast iron melts, a lighter graphite phase is present in a heavier melt. Sedimentation effects are likely to be significant during the solidification process [9,12].

The directional solidification furnace was first integrated in the KC-135 during the September 82 series of flights. The two objectives were: first, to flight test the

furnace system, and second, to study any low-g effects on the solidification of hypereutectic cast iron and of a superalloy composition. This report deals with the results of the solidification of the hypereutectic cast iron sample.

## PROCEDURES

A G. E. prototype ADSS furnace [13] was modified for use in the KC-135 (Fig. 1) primarily by adding a portable quench block water circulation system. The system consists of a 10 liter reservoir, an expansion tank, and a peristaltic pump (Fig. 2). On top of the quench block in the furnace canister is an 11 cm long furnace element consisting of platinum rhodium wire double wound around an alumina core. The sample is placed in a 1/2 cm ID, approximately 40 cm long, alumina crucible. The furnace is then translated, using a motor and gearing system, and thus the sample is gradually solidified. Figure 3 shows the quench block temperature versus time with the closed cooling system and a maximum furnace temperature of 1500°C. The change in quench block temperature of approximately 25°C will have an insignificant effect on the thermal gradient in the sample. A thermal profile of an empty crucible, measured with the maximum furnace temperature of approximately 1500°C, is given in Figure 4. By varying the maximum or plateau temperature in the furnace cavity we can obtain various thermal gradients. Figure 5 shows the thermal gradients in an empty crucible obtained for various maximum furnace temperatures. The hypereutectic iron sample solidified during the flight was prepared by adding excess carbon to a commercial cast iron composition. The compositional analysis after alloying is given in Table 1.

To facilitate the analysis of any low-g effects, it is desirable to have at least 1/2 mm of solidification during the approximately 20 sec of low-g in each parabola. The commercial iron composition used could be solidified at 2 mm/min and still obtain grey iron, avoiding the metastable white phase, by employing a thermal gradient of approximately 400°C per cm. Figure 6 shows the graphite microstructure at 1 and 2 mm/min translation rates.

The procedure during the flight experiment was as follows: The furnace temperature was brought up to 1500°C within a period of about 20 min. The furnace was then translated vertically upwards at a rate of 1.2 mm/min for 13 min during which time 10 low-g parabolas were flown. The furnace was then shut off and the translation stopped.

The directionally solidified sample was sectioned longitudinally. One half was reserved for compositional analysis while the second half was polished for photometallography. For comparision a control sample was run in the lab simulating the thermal and temporal environment experienced by the flight sample.

## RESULTS

### Furnace Operation

The furnace operated nominally during the flight experiment except for two problems. The first was that the translation motor RPM during the flight tended to drift by a maximum of about 10 percent. This required frequent adjustment by the

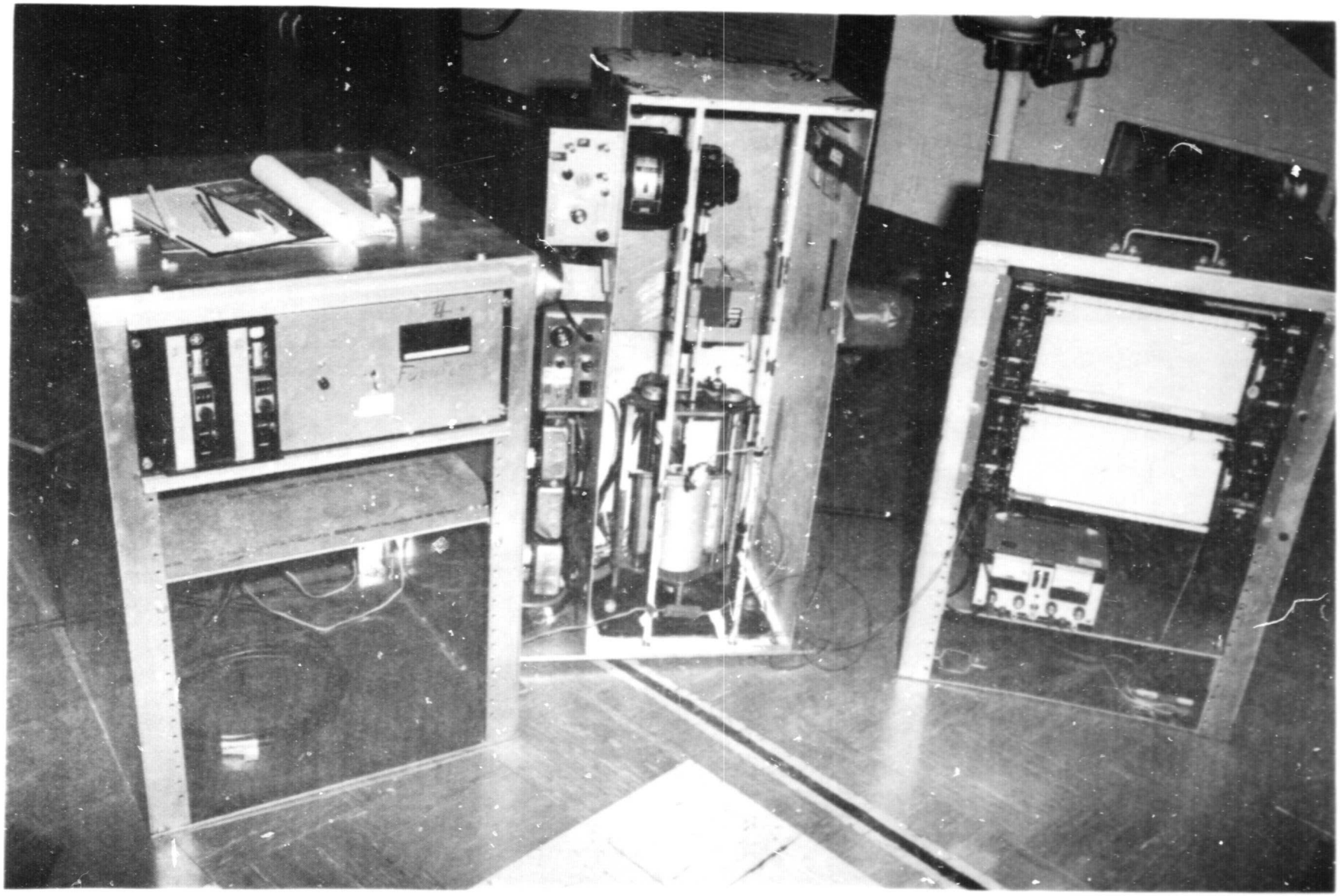

Figure 1. Hardware for the ADSS-P KC-135 experiment: temperature controller and phaser package (left), automatic directional solidification furnace (center), accelerometer and recorder package (right).

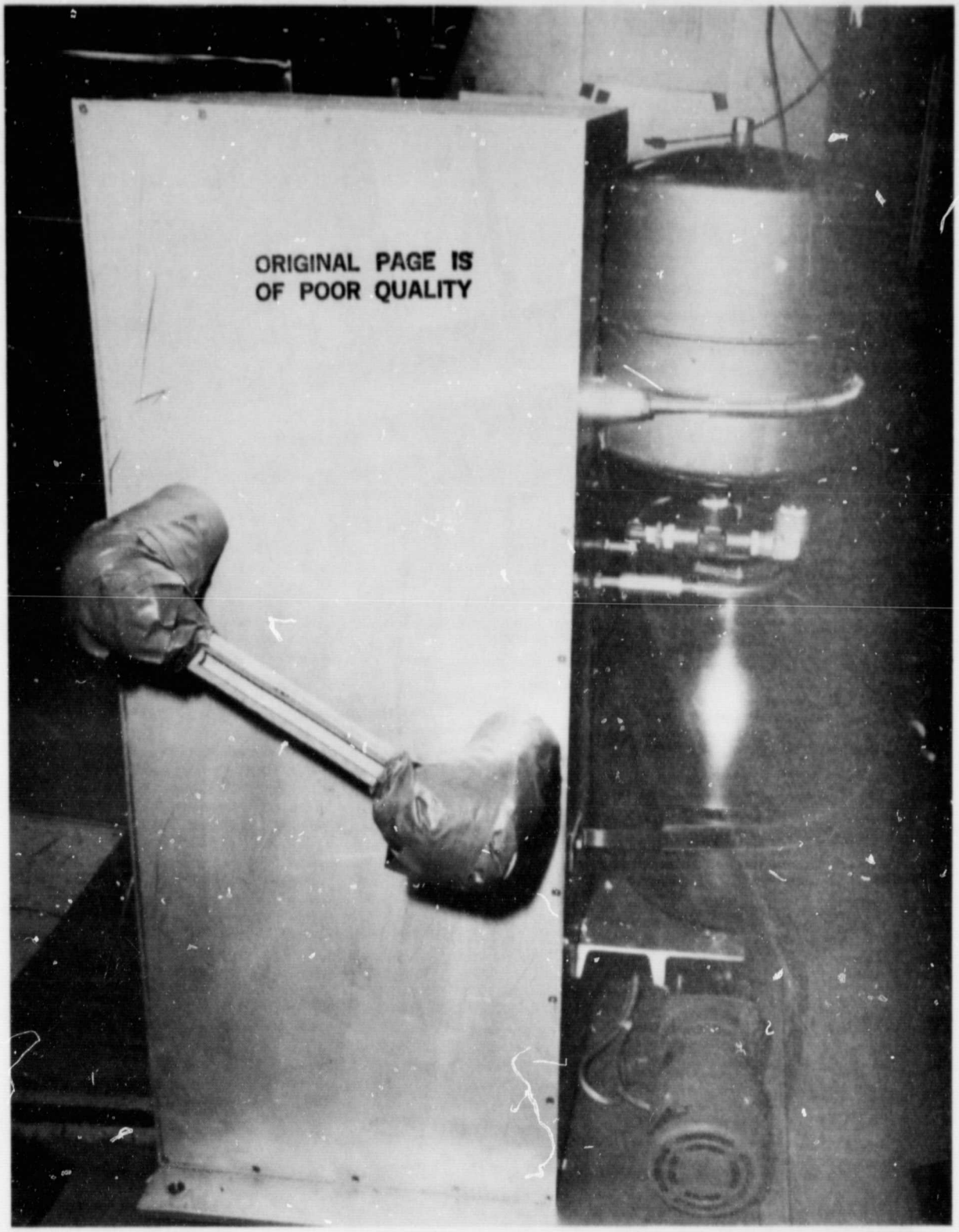


Figure 2. Directional solidification furnace showing the portable quench block water circulation system - expansion tank (top), 10 liter reservoir (center), and peristaltic pump (bottom).

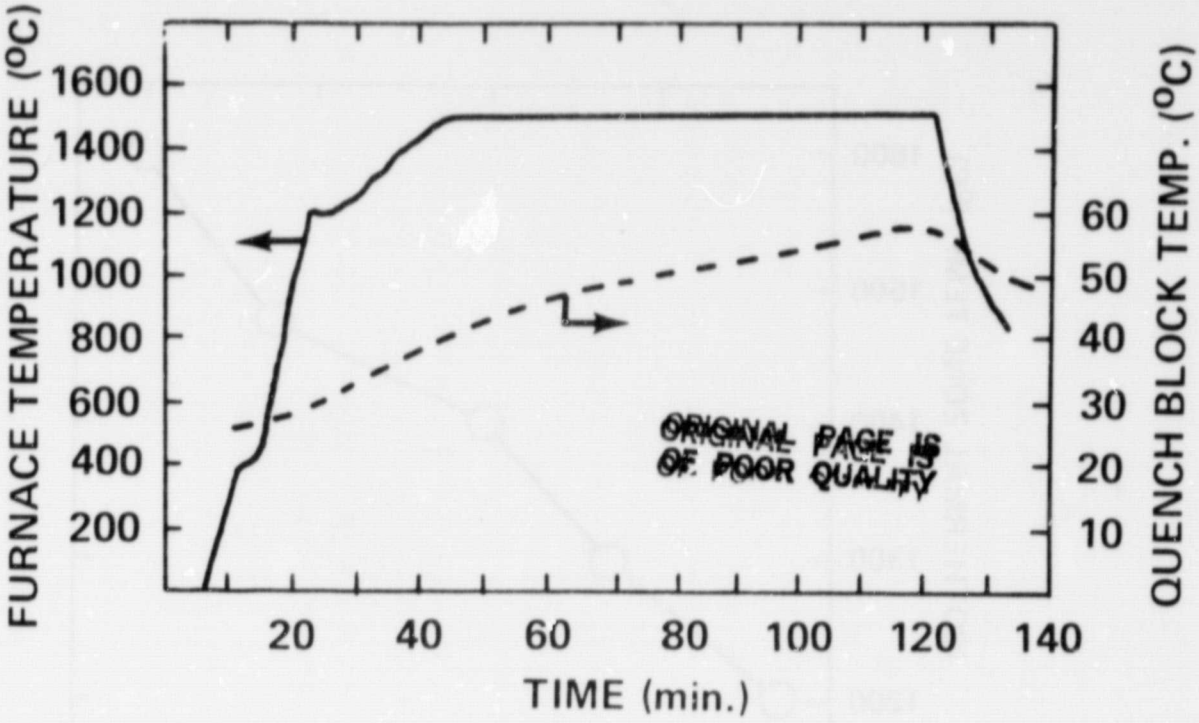


Figure 3. The change in quench block temperature with time for a furnace temperature of 1500°C.

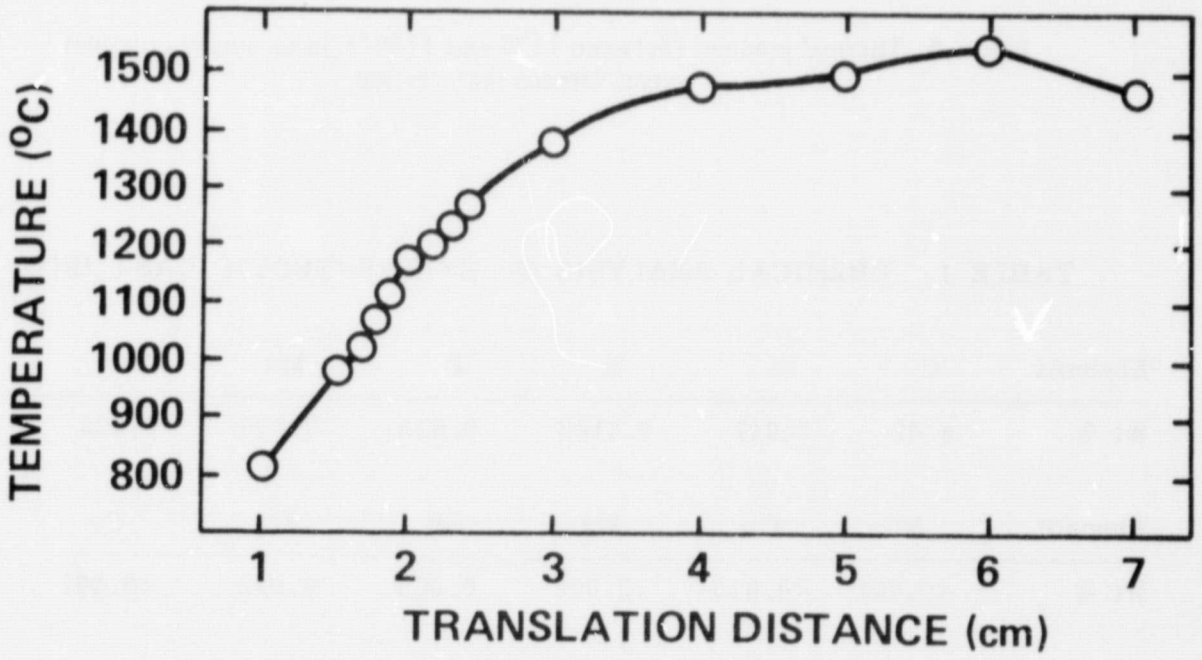


Figure 4. The thermal profile of an empty crucible for a furnace temperature of 1500°C.

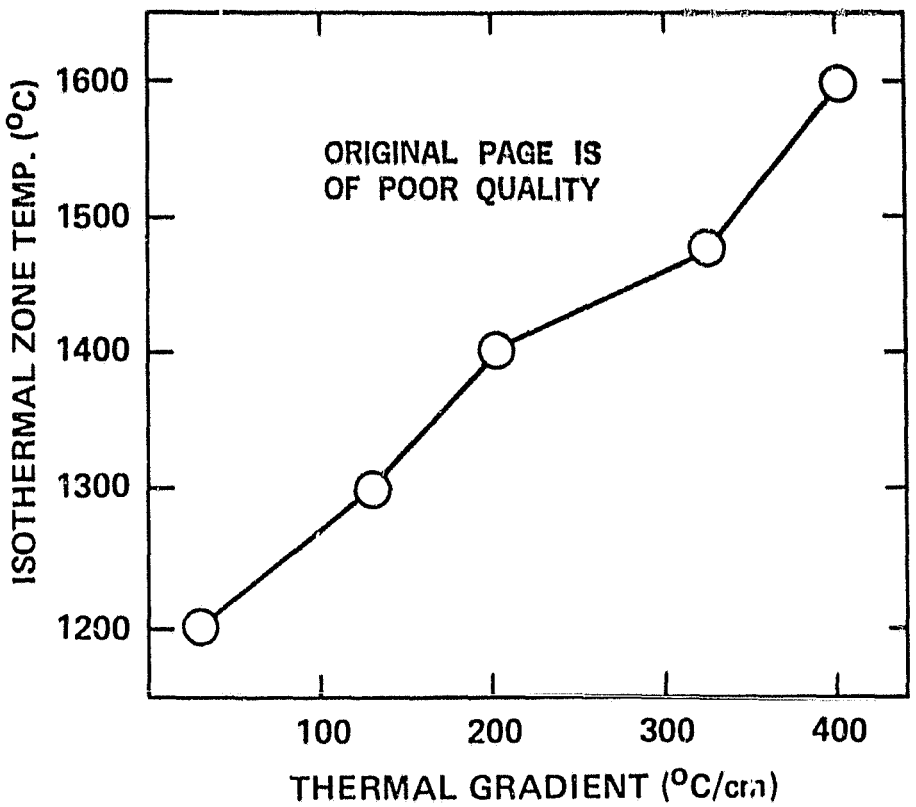


Figure 5. Thermal gradient (between 1120 and 1180°C in an empty crucible) versus furnace temperature.

TABLE 1. CHEMICAL ANALYSIS OF HYPEREUTECTIC CAST IRON

| Element | C | Si | S | P | Mn | Cr | Ni |
|---|---|---|---|---|---|---|---|
| Wt % | 4.52 | 2.047 | 0.0102 | 0.013 | 0.039 | 0.026 | 0.030 |

| Element | Mo | Cu | Mg | Sn | Al | Ce |
|---|---|---|---|---|---|---|
| Wt % | <0.001 | <0.010 | <0.002 | 0.005 | 0.002 | <0.001 |

| Element | Ti | Te | V |
|---|---|---|---|
| Wt % | 0.028 | <0.001 | 0.025 |

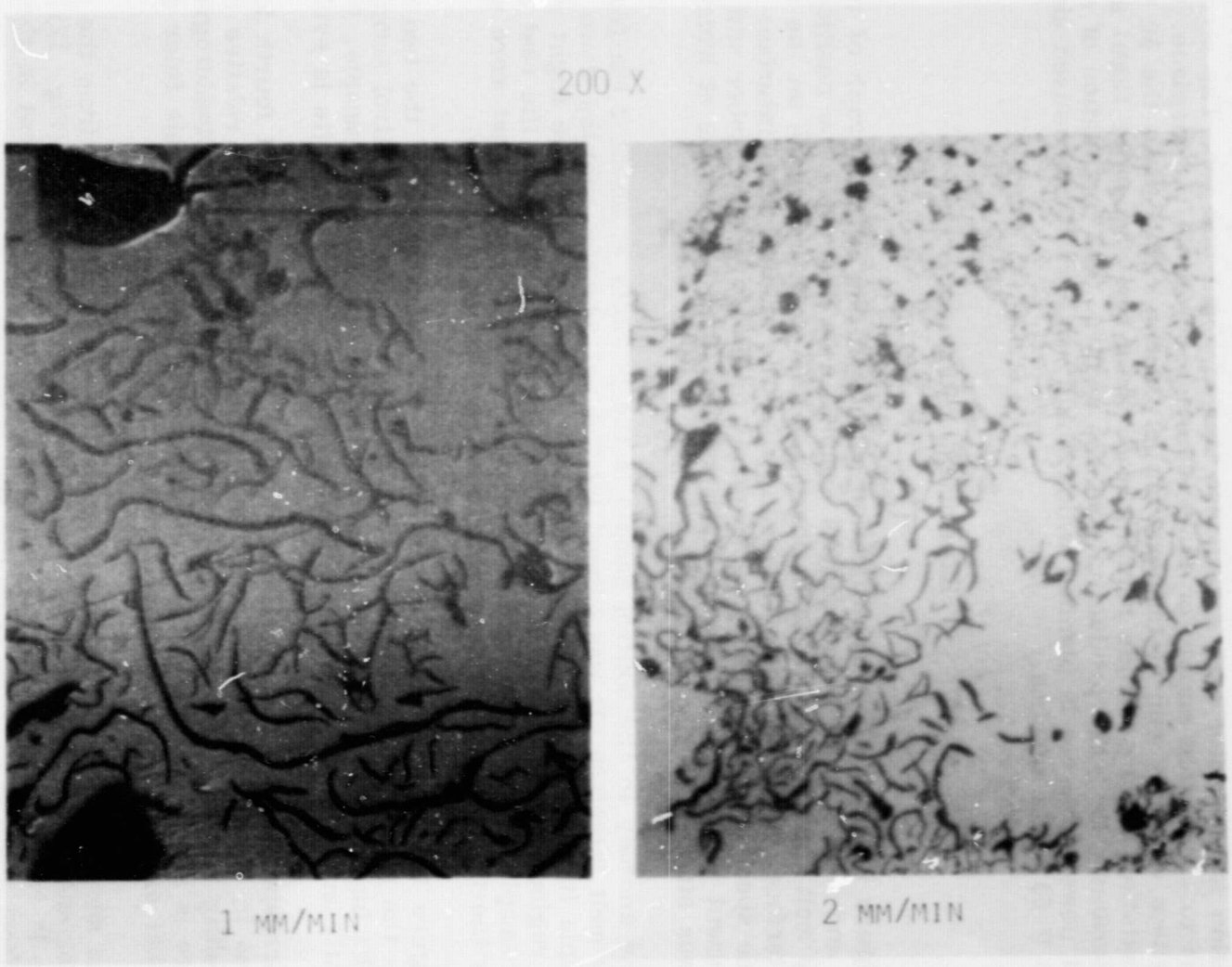


Figure 6. Microstructure of commercial grey iron solidified in a thermal gradient of 403°C/cm and at translation rates of 1 mm/min and 2 mm/min.

operator on board the airplane (Fig. 7). It is not known whether the RPM fluctuation was caused by the change in gravity level or by input power fluctuations. Since the flight, however, an electronic servo motor controller, employing a photosensitive tachometer, has been added to the furnace system to eliminate this difficulty. Figure 8 shows the motor control system prior to installation in the flight furnace. Tests of the system have shown it to be able to compensate for up to a 20 percent power fluctuation to the motor with a response time of about one sec. The second difficulty was due to the limited supply of 110 V 60 cycle power on the airplane. Consequently, the furnace was only run at temperature for 15 min rather than the 30 min planned. This limitation could preclude running more than one sample per flight and thus could seriously limit the productivity of the system. The installation of an additional aircraft power inverter is planned which would make available unused aircraft power for 110 V 60 cycle systems.

## Experimental Results

Figure 9 shows a low magnification composite photometallograph of the flight sample and of a ground based control sample. Also given are the position of the primary melt interface and a scale corresponding to the distance on the sample from that interface. A second scale (assuming that the solidification interface advanced at the same speed as the furnace) corresponds to the parabola number and the gravity environment for the flight sample. The position for both samples at which the translation was stopped is also indicated.

One anomaly in the control sample is the porosity at about 2 mm from the primary melt interface. It was caused by the melted metal running down the side of the sample in the crucible. It precludes any comparison with the flight sample in the porous region and could also have effected the solidification of the rest of the control sample. Work is continuing to obtain a control sample that more accurately reflects the thermal conditions encountered by the flight sample.

Figure 10 shows a higher magnification photomicrograph of the two samples between the primary melt interface, and the porosity in the control sample and to about the beginning of the second low-g parabola in the flight sample. Fine, apparently eutectic graphite, as well as irregular shaped graphite is evident.

Corresponding to the width and position of the second and fourth low-g parabolas in the flight sample, there is a pronounced increase, relative to the surrounding regions, of coarser graphite flakes. A 200 Xs photomicrograph of this structure is shown in Figure 11. The higher magnification reveals finer graphite flakes that exist between coarser flakes.

The microstructure between approximately 2 mm and 8 mm from the primary melt interface in the control sample remains relatively constant (Fig. 12). The graphite is roughly equivalent to the fine graphite that is present in the flight sample from about 3 to 8 mm from the melt interface.

From about 8 mm to about 13 mm both samples contain a mixture of both fine and coarse graphite flakes. A typical microstructure for each sample is shown in Figure 13.

In the flight sample at about 13 mm from the primary melt interface there occurs a more randomly orientated system of very large flakes in a matrix of finer

Figure 7. Directional solidification system being operated during the 9-22-82 KC-135 flight. (MSFC personnel Robert Shurney is measuring furnace RPM, and Wendy Alter is taking data.)



Figure 8. Servo motor control system before installation into the flight furnace.

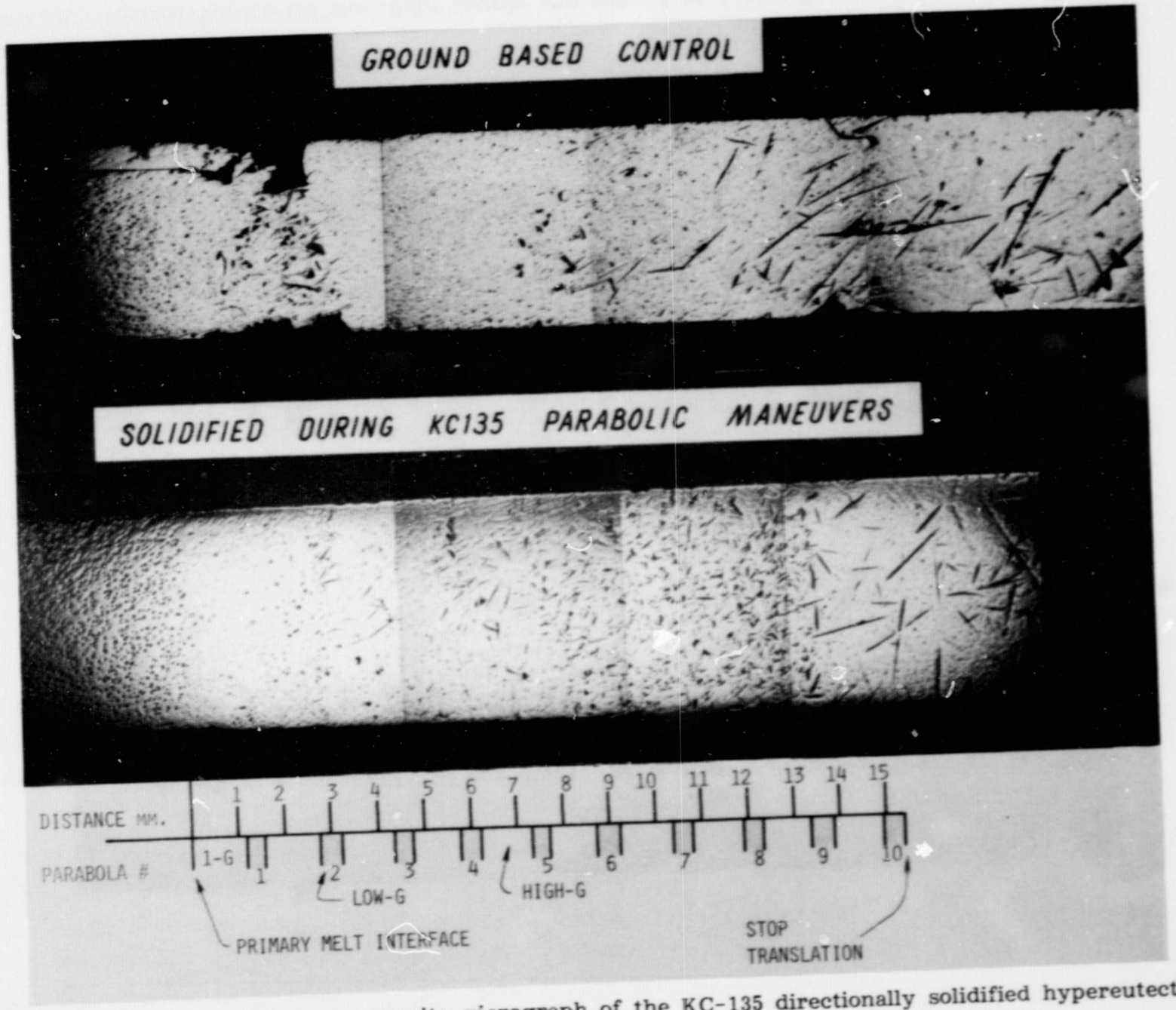


Figure 9. Low magnification composite micrograph of the KC-135 directionally solidified hypereutectic cast iron flight sample and a control sample.

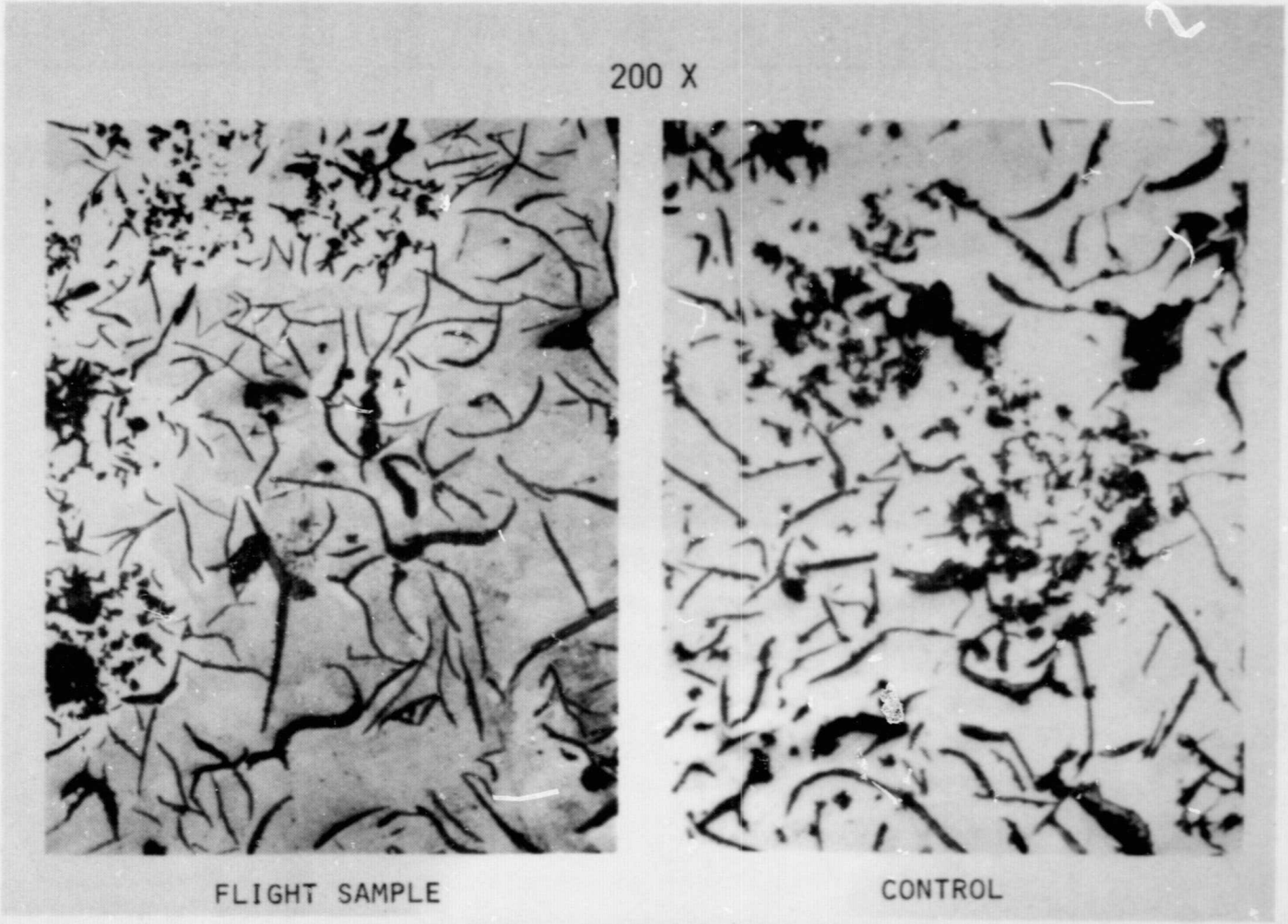




Figure 10. Microstructure for the flight sample and control at 2 mm after the primary melt interface.


Figure 11. Microstructure for the flight sample 2.5 mm from the primary melt interface.

flakes of graphite. This continues through the rest of the sample. In the control sample a similar microstructure is obtained but more gradually.

## DISCUSSION

A promotion of coarser graphite during solidification in low-g, relative to that in high-g, is suggested by the microstructure of that part of the flight sample that was solidified between the first and fifth parabolas. Coarsening of the graphite during low-g solidification could be explained by either of the following hypothesises:

A) Lack of convection lessens the tendency for the graphite leading the solidification front to break up.

B) Lack of flotation allows primary graphite to be incorporated into the solidification front.

The observation that microstructure in the low-g "bands" contains a matrix of fine -

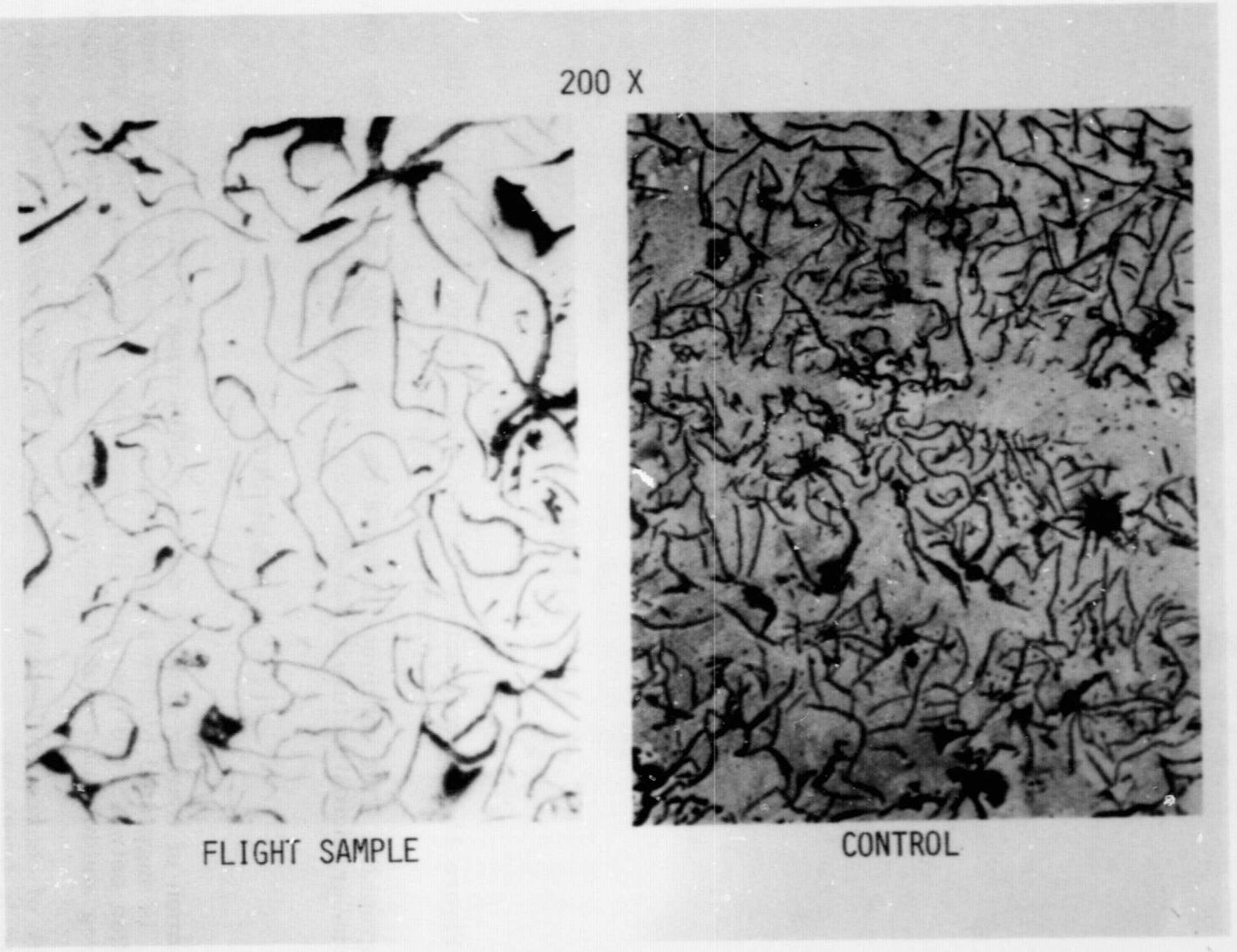




Figure 12. Microstructure for the flight sample and control at 4 mm from the primary melt interface.

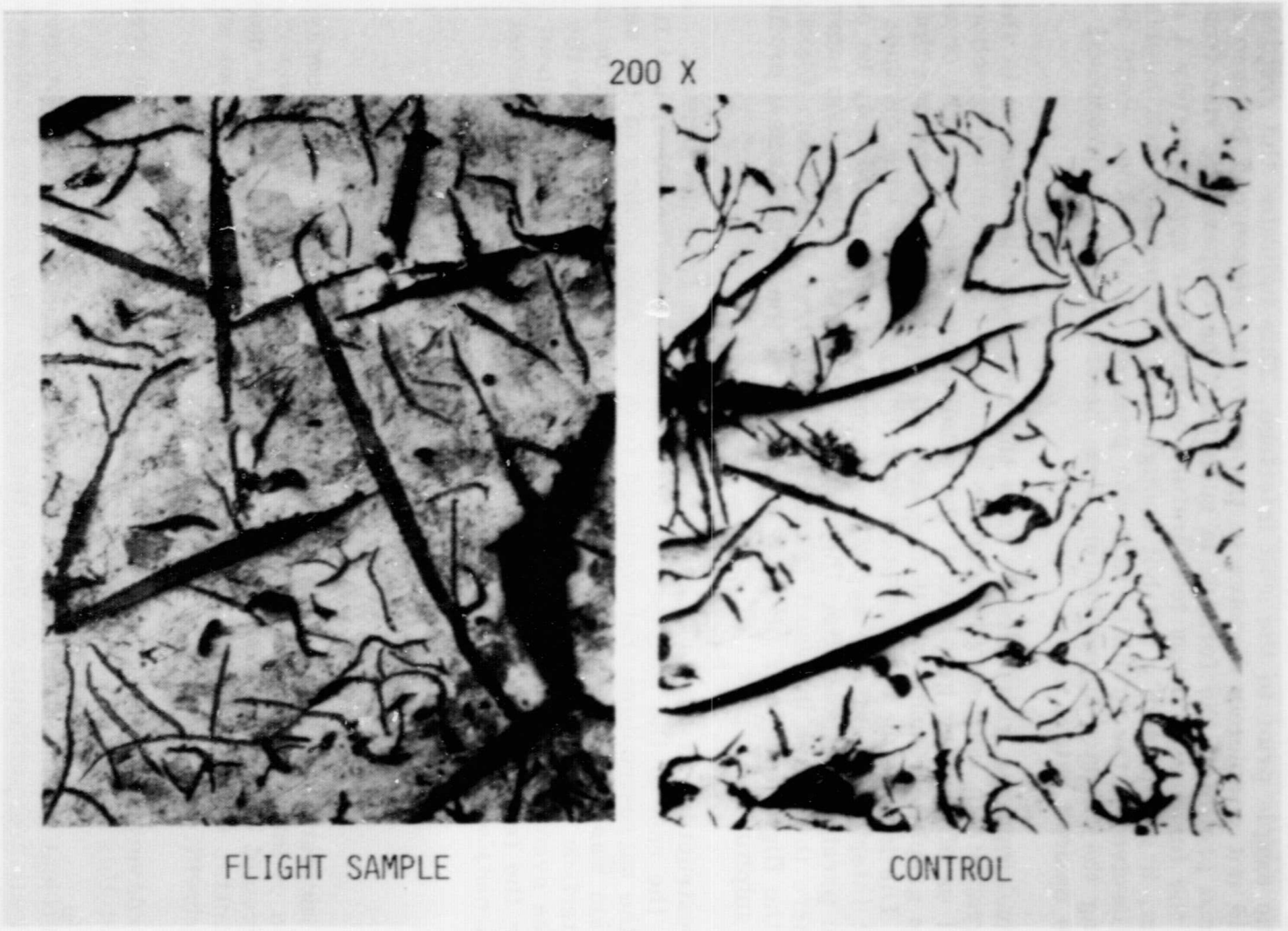




Figure 13. Microstructure for the flight sample and control at 10 mm from the primary melt interface.

probably eutectic - graphite along with the coarse flakes (Fig. 11) tends to favor hypothesis B.

The morphology (Fig. 10) of the irregular graphite between the primary melt interface and approximately 2 mm from it is similar to the graphite in the unmelted portion of the sample prior to the melt interface. This indicates the graphite up to that point did not completely dissolve in the melt; the reason can be seen in Figure 4. The thermal profile of the furnace is such that the temperature rise from the melt interface to the furnace maximum temperature occurs over approximately 1 cm of sample. Thus, it was probably not hot enough near the primary melt interface to completely dissolve all the graphite that was present. This problem could be remedied by translating the furnace approximately 1 cm before beginning directional solidification at the desired rate.

The gradual increase with distance from the primary melt interface in the amount of coarser graphite in both samples can be explained by either the rejection of sulphur [14] or of primary graphite at the solidification interface. The observation that the fine graphite remains about the same size throughout supports the latter hypothesis. The increasing concentration of primary graphite with distance from the primary melt interface could also explain why coarse bands would only be observed in the initial parabolas, since after the primary graphite concentration increases to the level where the flakes intersect flotation is no longer significant. Chemical analysis of the flight sample is planned to determine the compositional profiles of carbon and sulphur.

The microstructure in the samples from about 13 mm from the primary melt interface though the rest of the samples remains approximately the same, and it is essentially the microstructure that occurred from the slow cooling of the sample after the translation was stopped and the furnace was turned off. It should be noticed that this interface (in the flight sample) occurs at about 13.5 mm from the primary melt interface even though the furnace translation had been stopped about 2 mm later - after the tenth parabola. This indicates that 2 mm of sample melted back after the furnace was turned off.

## CONCLUSIONS

1) The microstructure of the flight sample suggests that low-g promotes the incorporation of large graphite flakes into the solidification front. However, further flight and ground experiments and more detailed analysis are needed to determine with a reasonable certainty that this is a gravity induced effect and not an artifact of the experiment.

2) The conversion of additional aircraft power to 110 V 60 cycle is needed to fully support this series of experiments.

3) Directional solidification can greatly enhance the information obtained from alloy solidification experiments on board the KC-135. The method promises to be a valuable tool for the study of low-g effects on alloy solidification.

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## APPROVAL

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

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Director, Space Science Laboratory