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Interim Report No. 1
Part A
Contract No. NAS8-24612

INVESTIGATION OF THE EFFECT OF GRAVITY
ON THE GROWTH OF CRYSTAL WHISKERS

by

Jack H. Davis

Submitted by

RESEARCH INSTITUTE
THE UNIVERSITY OF ALABAMA IN HUNTSVILLE
P. O. Box 1247
Huntsville, Alabama

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FOREWORD

This report summarizes the results of work done from June 17, 1969, through April 17, 1970, on NASA contract NAS8-24612, Part A. (Part B is reported under separate cover.) The objective of Part A is:

- To determine the effects of gravity on crystal whiskers.

This study is sponsored by George C. Marshall Space Flight Center, National Aeronautics and Space Administration, Huntsville, Alabama. Mr. T. C. Bannister of the Space Sciences Laboratory is the contracting officer. Dr. J. H. Davis, Associate Professor of Physics, and Dr. Upendra Roy, Assistant Professor of Engineering, are the Principal and Co-Principal Investigators. The assistance of graduate student Paul Bisenius and of undergraduate students Rajinder Mehta and Howard Camp is gratefully acknowledged. We very much appreciate the use of NASA's scanning electron microscope which was operated by Mr. Dave Nichols in Astrionics Laboratory of Marshall Space Flight Center.

SUMMARY

The effects of gravity on crystal whisker growth are being studied. This interim report is only the "first chapter" since new ideas are being generated each month.

The results thus far are:

1. High aspect-ratio $\left(\frac{\text{length}}{\text{diameter}}\right)$ whiskers are best suited for composite strengthening.
2. High aspect-ratio whiskers are most easily destroyed or broken off by direct gravity force and by gravity-induced convective flow drag.
3. Hydrodynamic calculations show that drag forces due to gas velocity in gravity-induced convection in a typical vapor-deposition growth are large enough to knock down or break off growing whiskers. This is especially true with thin whiskers since the drag force is practically independent of whisker diameter and the whisker rigidity drops inversely proportional to the diameter cubed.
4. Air drag forces are so large that they have obscured the effect of gravity on whiskers in the past.
5. The whisker base has been found to be the weakest point on the whisker.
6. An electron microscope photograph shows that the base diameter of some iron whiskers is a factor of 5 thinner than the rest of the whisker. This makes the whisker 125 times more vulnerable to gravity forces.

7. Our scanning-electron-microscope photographs reveal a weak tin whisker base and some odd U-shaped whisker cross sections.

8. Cd whiskers grew at 5-g with some reduction in whisker quality believed due to increased convection.

9. We hypothesized that stress induced by gravity is responsible for growth stoppage in "squeeze" grown whiskers.

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Part A

INVESTIGATION OF THE EFFECT OF GRAVITY

ON THE GROWTH OF CRYSTAL WHISKERS

INTRODUCTION TO PART A

The purpose of the Part A investigation is, by analysis, by experimentation, and by literature search, to determine the value of a zero-gravity environment to the whisker growth process. This is part of NASA's Space Processing Program.

Whiskers are high strength, filamentary single crystals. They are of interest mainly because of their high tensile strength which approaches the theoretical limiting strength of a material. Bulk crystal tensile strengths are from two to three orders of magnitude below the predicted theoretical value.¹ As the crystal's cross section area becomes smaller in the neighborhood of 1 square micron, the tensile strength increases by about a factor of 100. The smaller the area, the greater the tensile strength with the tensile strength being roughly inversely proportional to the diameter.²

So far, technology has been unable to fully utilize the high strength potential of whiskers.³ This is partly because no rapid mass production method of growing good quality whiskers is available in an earth environment. Whiskers now available are either too short, have multiple branches, or are too scarce to be used in the reinforcement of high strength composites. In a zero-gravity space environment whiskers may grow longer, thinner, and stronger.

Whiskers used to strengthen composites should have a high aspect ratio.⁴ The aspect ratio is the ratio of whisker length to thickness (or diameter). The long thin (high aspect ratio) whiskers have more surface area per unit whisker tension; thus they do not pull free from the plastic matrix when the composite is under tension.

For a good technical review of the field of whisker growth, the reader is referred to references 4 and 9. However, the objective of this report is not to review the field of whisker growth, but to pinpoint the accomplishments of the past nine months.

I. EVIDENCE COLLECTED THAT INDICATES AN ADVERSE GRAVITATIONAL EFFECT ON WHISKERS

a. Whisker Destruction Due to Gravity Bending

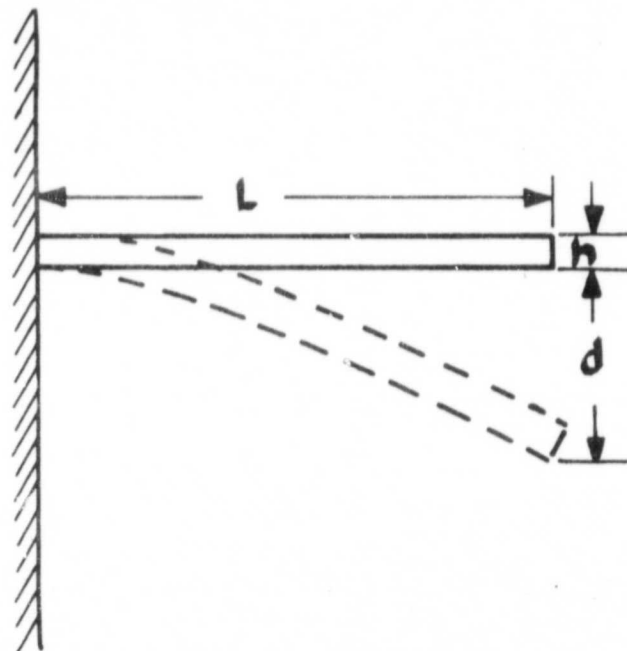
Whiskers may be destroyed by a gravity-field as they grow. In this section we are not interested in the atomic growth process, but in preserving the whisker from mechanical destruction as it grows.

The thinnest whiskers have the highest aspect ratios and tensile strengths. However, the full potential of these small fibers has not been realized because they are not rigid enough to support their own weight so they fall due to gravity to a nearby surface where they adhere. This surface molecular adhesive force is quite large; probably 100 to 1000 times larger than gravity. Whiskers stick to a surface like adhesive tape (Figs. 5 and 6) and they may be peeled away under a microscope. A whisker sticking to the substrate obviously cannot grow by vapor condensation since nucleation could occur near the point of contact of the whisker and the substrate. Such nucleation would result in a distortion of the whisker shape. Just how long and thin must a whisker be before it bends under a 1 g force and is captured by an even stronger adhesive force?

The gravitational effect on a long thin whisker may be analyzed using the equation, presented in the October '69 MSFC Space Processing Meeting, for the deflection of horizontal cantilever beams shown in Fig. 1.

$$d = \frac{WL^4}{8EJ} \quad (1)$$

CANTILEVER WHISKER BEAM BENDING DUE TO GRAVITY



$$d = \frac{wL^4}{8EJ} \quad J = \frac{bh^3}{12}$$

Where

w = Weight per unit length

L = Length

h = Vertical thickness

E = Youngs Modulus = 4.5×10^{10} dynes/cm²

J = Area Moment about neutral axis

b = Whisker thickness

$$.2 = \frac{d}{L} = \frac{wL^3}{8EJ} = 2.3 \times 10^{-10} \frac{L^3}{h^2}$$

$$L_{MAX} = 216 h^{2/3}$$

Good Aspect Ratio is $L/h = 1000$

Fig. 1. The horizontal whisker is bending under the influence of gravity and is governed by the above equation.

W is the weight per unit length of the whisker, L is the length, E is Young's modulus and J is the area moment about the neutral axis. $J = \frac{bh^3}{12}$ for a rectangular cross section of thickness h, and width b.

For tin the following equations:

$$d = 2.3 \times 10^{-8} \frac{L^4}{h^2}$$

or

$$\frac{d}{L} = 2.3 \times 10^{-8} \frac{L^3}{h^2} \quad (2)$$

result from using $E = 4.5 \times 10^{11}$ dynes/cm²

$$\rho = 7.1 \text{ gr/cm}^3$$

$$g = 980 \text{ cm/sec}^2$$

Let's say that if a whisker deflects 2% of its length, it is endangered.

Letting $d/L = 0.2$ in the above equation, we have:

$$L_{\max} = 216 h^{2/3} \quad (3)$$

L_{\max} represents the maximum length that a horizontal tin whisker of thickness h may have without being deflected by more than 20% of its length.

Fig. 2 shows L_{\max} as a function of h. Whiskers having L and h coordinates falling in region 3 are not affected by gravity. If the L and h coordinates

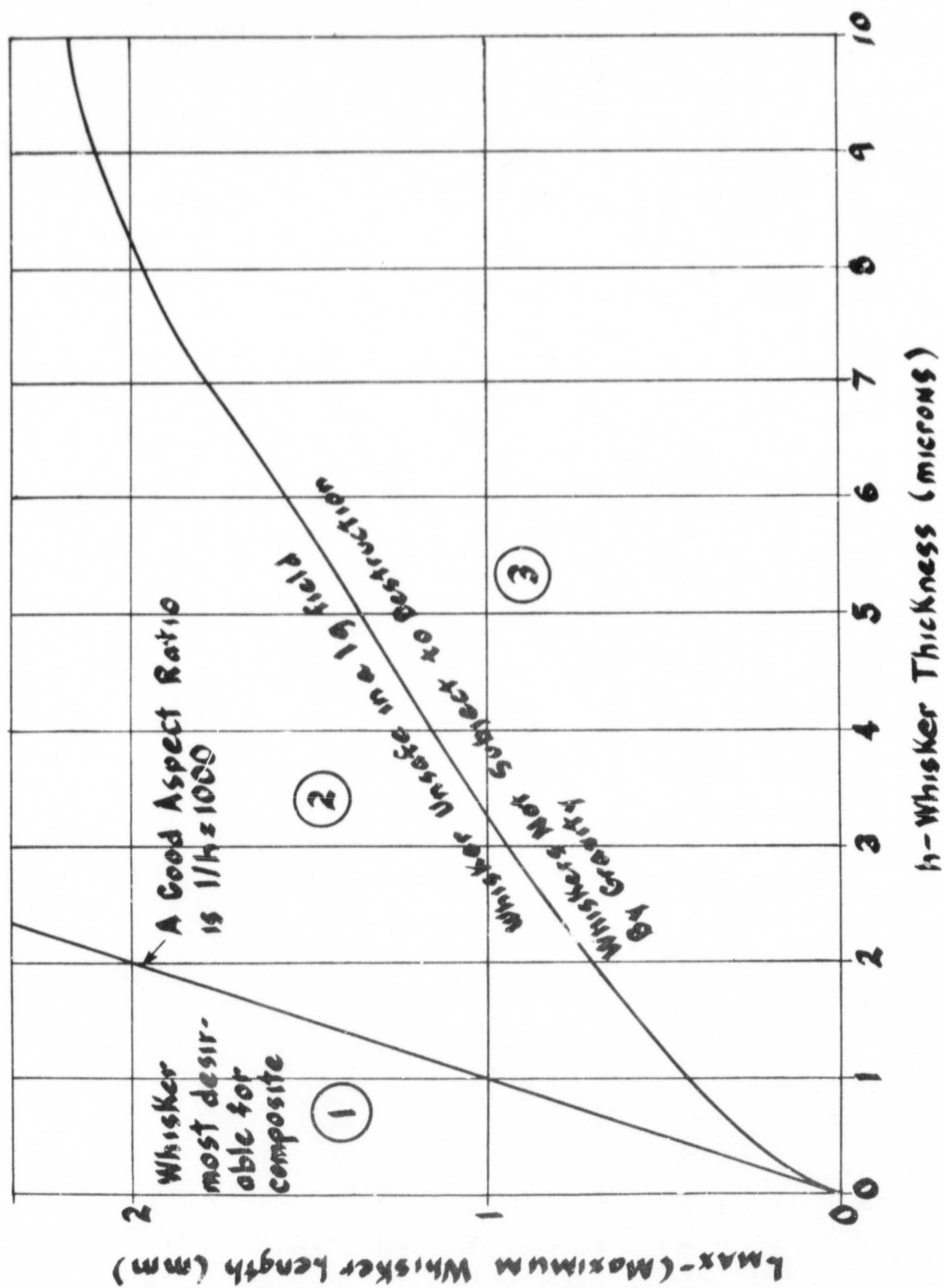


Fig. 2. Whisker length and thickness are plotted as coordinates to show three regions: (1) most desirable aspect ratio, (2) intermediate region, (3) coordinates of whiskers not affected by gravity. Whiskers in regions (1) and (2) are subject to destruction by gravity in our model.

fall in region 1, the aspect ratio is greater than 1000 indicating that the whisker has good bonding properties in a matrix. However, there are no coordinates common to both regions 1 and 3 indicating that all whiskers having desirable aspect ratios are endangered by gravity.

Are there any flaws in this cantilever beam model used here? First, the g-field is not perpendicular to all whiskers and the deflection-length ratio of 0.2 is rather arbitrary. However, the whiskers always break off at the base indicating that the base is the weakest point. This suggests that gravity is even more important than predicted.

b. Thin Whiskers Should Be Longest

In compression-induced whisker growth, experimenters have found that thick whiskers have a terminal length which is much less than the terminal length of thin whiskers. This relation has not been precisely determined nor is it fully understood. It seems that there may be a limited volume of material available for a squeeze-grown whisker and that the whisker length may be increased by a reduction in the whisker cross section area. Just how far does this tendency go? We have pointed out that the thinner whiskers are destroyed by a gravity field so we do not know. Possibly the length would increase several orders of magnitude as the diameter decreases. That is, $1\ \mu$ thick whiskers which are now limited to a length of 1 mm may become as long as 100 mm if their area is reduced to $0.1\ \mu$.

c. Whisker Base Is Weakest

The whisker base is most important in supporting the whisker. The

recently-published electron microscope photographs (Fig. 3) by Gaber and Blocker⁶ show the base of an iron whisker which is several times thinner than the rest of the whisker. This could easily reduce the rigidity of the whisker by a factor of 100 since the deflection of the cantilever beam in Eq. 1 is inversely proportional to h^3 .

d. Whisker Observations

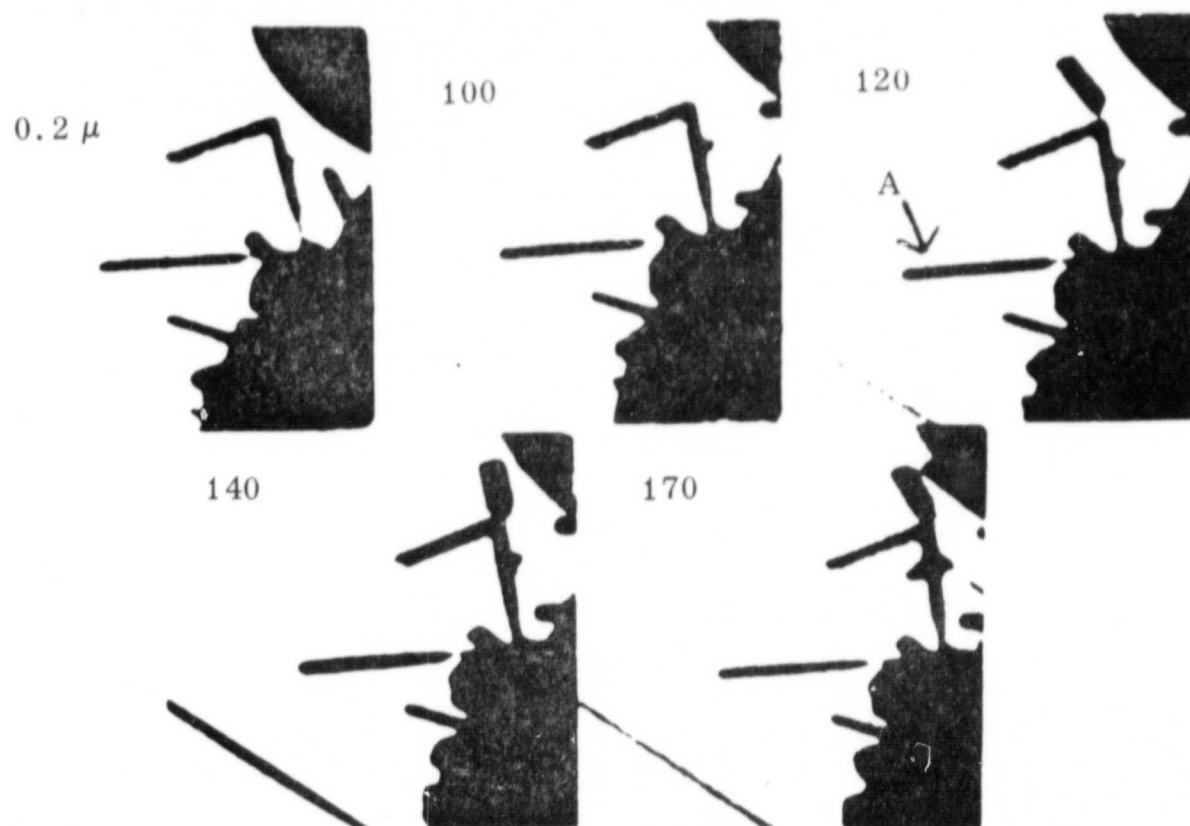
Whisker growth has often been studied by microscopic observations. One interesting observation is the sudden disappearance of whiskers (Hack and Neumann⁷). It is believed that these whiskers broke off at the root, possibly due to gravity.

In our lab, whiskers of Ca, Sn and Tl have been observed lying flat on the substrate surface, probably due to gravity and/or vibration. They were sealed under vacuum away from air currents.

e. A simple aerodynamic calculation may explain why so few effects have been related to gravity in the past. This calculation compares air flow pressure, P_a , to gravitational pressure, P_g , on whiskers.

$$\text{Pressure}^8 \text{ due to air} = P_a = 2\rho_a v^2 = \frac{.3 \text{ dynes}}{\text{cm}^2} \quad \dagger \quad (4)$$

† This equation requires modification to take into account the fact that the Reynolds number $R = VD/\nu$ becomes extremely large for whiskers. D is the whisker diameter and ν is the kinematic viscosity. Preliminary calculations using small cylindrical rod theory indicate the air velocity of 0.1 cm/sec imparts a force equivalent to gravity to a 1 micron diameter tin rod. (H. Lamb, Hydrodynamics, p. 616, Dover, N. Y., 1945.) This topic will be covered in detail in the next monthly report.



IRON WHISKERS

From Gabor and Blocher. J. Appl. Phy., Vol. 40, No. 7, 1969, p. 2703.

Fig. 3. Whiskers seen growing in an electron microscope have diameter of only .01 microns at the base. They would fall easily in a gravity field as they grow longer.

for air at 10 cm/sec velocity.

$$\text{Pressure due to gravity} = P_g = \frac{F}{LW} = \rho_w gh = \frac{.5 \text{ dynes}}{\text{cm}} \quad (5)$$

for a 1 micron thick whisker of density 5 gm/cm^3 .

where

ρ_a - air density

v - velocity of air

ρ_w - whisker density

g - acceleration of gravity

$L W h$ - whisker length, width and height

So an air flow of 10 cm/sec is about equivalent to 1 g in the bending of a whisker. The shielding effects of the substrate has been neglected. Thus in looking at the gravitational effects, it is quite important to assume that air velocity is no more than a few cm/second. This rules out breathing, forced air room ventilation, or even walking across the room with the exposed whiskers. Normal breathing is found to cause strong fluttering of whiskers when they are observed under a microscope. So it is concluded that gravitational effects on whiskers have been lost in the larger air flow forces.

The above calculation answers the question "do convection cycle fluid forces destroy the whiskers mechanically?" A typical convection velocity is only 1 cm/sec, so the whisker would be disturbed directly by gravity before being disturbed by a typical convective flow.

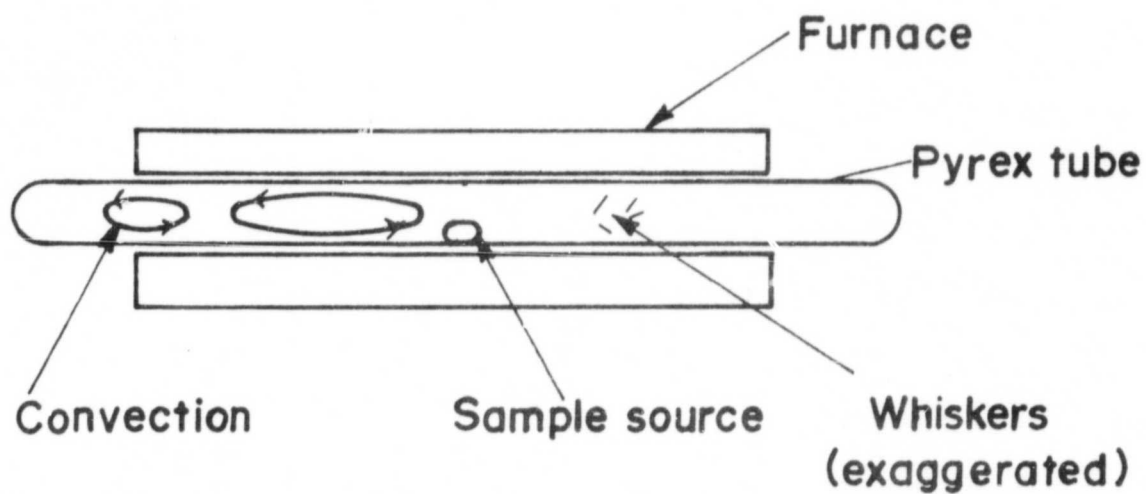
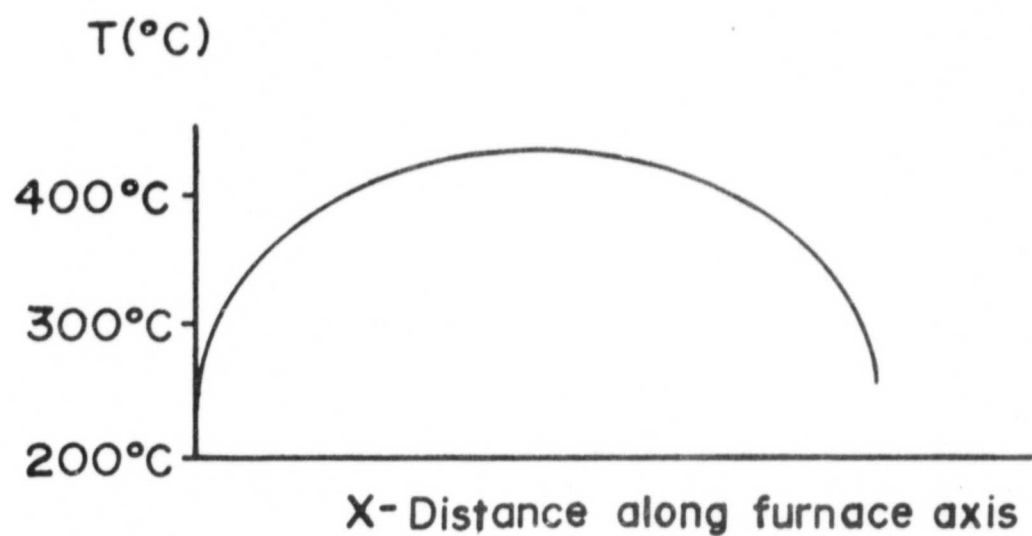
II. EXPERIMENTAL PROGRESS TOWARD MEASURING EFFECTS OF GRAVITY ON WHISKER GROWTH

a. The Effect of Increased Gravity on Whiskers Grown by Vapor Deposition

A gravitational field could affect the atomic whisker growth mechanism. This would be much more likely in the vapor deposition growths which involve the gaseous state. In the vapor deposition process,⁹ a source material is slowly vaporized in the hot portion of the furnace and as the vapor moves to the cool region, it becomes supersaturated and condenses metal atoms to make a solid which often grows in whisker form. This is done in an inert atmosphere as shown in Fig. 10. The diffusion should be very slow. In a temperature gradient, convection currents are set up which add to the diffusion in transporting the atoms to the lattice. These turbulent circulating currents (due to gravity) are usually unstable; thus, they have an adverse effect on whisker growth. That is, excessive supercooling occurs in various regions that results in massive nucleation which produces polycrystals instead of a single crystal. The convection, due to warm and cool gas having a different density in the presence of a gravitational field, is very pronounced in gases. A more uniform stable vapor for better crystal growth can be produced in zero-gravity.

An experiment is underway to study the role of gravity using a centrifuge. If 1-g produces an adverse effect on whisker growth, then 2-g should produce an even more adverse effect, etc. By applying several g's of acceleration to the whiskers during the growth by vapor deposition, and by comparing this growth to 1-g growth, one would hopefully then predict the effects of 0-g.

TEMPERATURE PROFILE



THE VAPOR DEPOSITION METHOD OF WHISKER GROWTH.

Fig. 10. A diagram of a vapor deposition whisker growth furnace. Convection plays a role in moving the atoms from the source to the growth site.

Vapor deposition growth is suited to materials having a high vapor pressure at their melting point. Cadmium with a melting point of 321°C and a vapor pressure of 0.1 mm (at 321°C) is being used.

Preliminary results of the first Cd whisker growth test at 5-g are given below. A cadmium growth tube as pictured in Fig. 10 was prepared by doubly distilling Cd into a 25 mm o.d. Pyrex tube at a pressure of 10^{-7} Torr. The $2/3$ atmosphere of argon was released into the tube and the tube was then sealed by a torch. The Pyrex tube was placed in the furnace as shown in Fig. 10 for 23 hours with a center temperature of 360°C . Whiskers grew at both ends of the Pyrex tube; however, some unusual results were noted: (1) All the Cd source was completely removed from the center of the tube and had deposited in the cooler tube ends. This is not too surprising at 5-g since convection is expected to increase by a factor of 5. (2) Whiskers at one end of the tube were of fair quality, whereas, those at the other end were of extremely poor quality. This is strange since both ends of the tube are symmetrical. However, one tube end was closer to the outside of the furnace and hence was cooler, thus producing more convection on the cooler end. Inspection of the tube shows that the high-convection end had practically no whiskers, whereas, the low-convection (hotter) end had fair quality whiskers. It is too soon to be certain, but this is a good indication that convection has an adverse effect on whisker growth.

The source material (cadmium) is placed in the warmest portion of a furnace as shown in Fig. 10. The Cd slowly vaporizes and condenses in the

cooler regions of the furnace. If the temperature gradient along the furnace is carefully controlled, the vapor will condense in the form of single crystal whiskers.

The experimental arrangement is shown in Fig. 11. The gradient furnace was placed on a 1 m arm in the 0-75 G centrifuge. The centrifuge was equipped with an optics system, slip rings and gas lines, to monitor and control the furnace during crystal growth. A typical growth requires about 12 hours to 24 hours.

At this time whiskers are being grown in the furnace shown in Figs. 10 and 11. The furnace is mounted in the centrifuge for increased g growth. During the first centrifuge growth, temperature oscillations of about 10°C were set up. However, by adjusting the furnace feedback control, this problem has been solved. The furnace gradient has been checked with a fan running to show that the air flow at a velocity of 15 miles per hour will not affect the temperature.

An accelerometer has been mounted in the centrifuge to monitor the vibration during experimentation. An adjustment screw for fine balancing of the centrifuge in both the horizontal and vertical directions has been installed. This device allows dynamic balance to minimize vibrations.

The thermocouple voltage and furnace power passes through slip rings into the furnace and control box that are mounted on the centrifuge. Slip ring noise was found to be negligible (only a few microvolts).

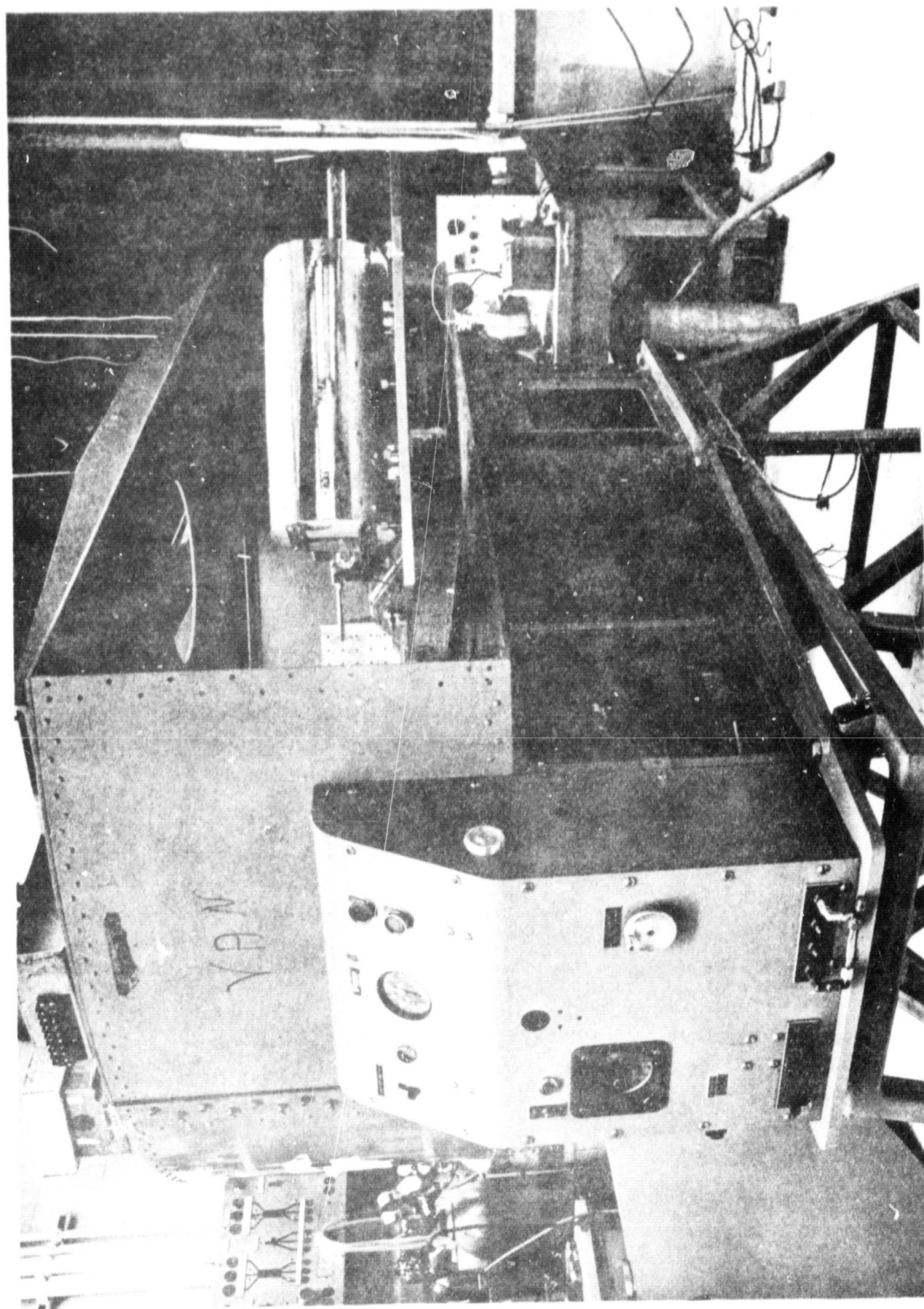


Fig. 11. Crystal growth furnace is seen mounted on the centrifuge arm. The radius is 1 m. This 0 to 75 g centrifuge was obtained from salvage at no cost, thus saving about \$10,000.00.

b. Scanning Electron Microscope (SEM) Reveals Weak Base in Tin Whiskers
and Shows New Detail of Whisker Cross Section

Tin whiskers are grown in our lab by what is known as the "squeeze" technique. A thin film of tin (a few microns thick) is compressed between 2 steel disks. The stress induces atoms of tin to migrate to the unstressed edge of the disc. At the edge they fall into a crystal lattice; the result is a whisker growing from its base at the point of extrusion.

Very little is known about the size distribution and surface features of the very small whiskers since they are too small to measure in an optical microscope. The optical microscope is limited both in resolution and depth of field; however, both of these limitations have been removed by the use of a scanning electron microscope (SEM).

From photographs taken with the SEM, we observe that some tin whiskers have very weak bases. The tin whisker shown in Fig. 4 first grew in the regular whisker form and then grew in an extruded form. This extrusion is too weak to support the crystal in a gravitational field.

Figs. 6 and 7 show tin whiskers which have fallen to the substrate. They fall most likely because of gravity and/or air motion. In the absence of gravity and air, these whiskers would still be in a growing position. There are still other factors besides falling to the substrate which cause growth to stop which are not understood.¹⁰ However, maintaining the whisker in a growing position is important in producing longer, more useful whiskers. Thus, a zero-g environment would

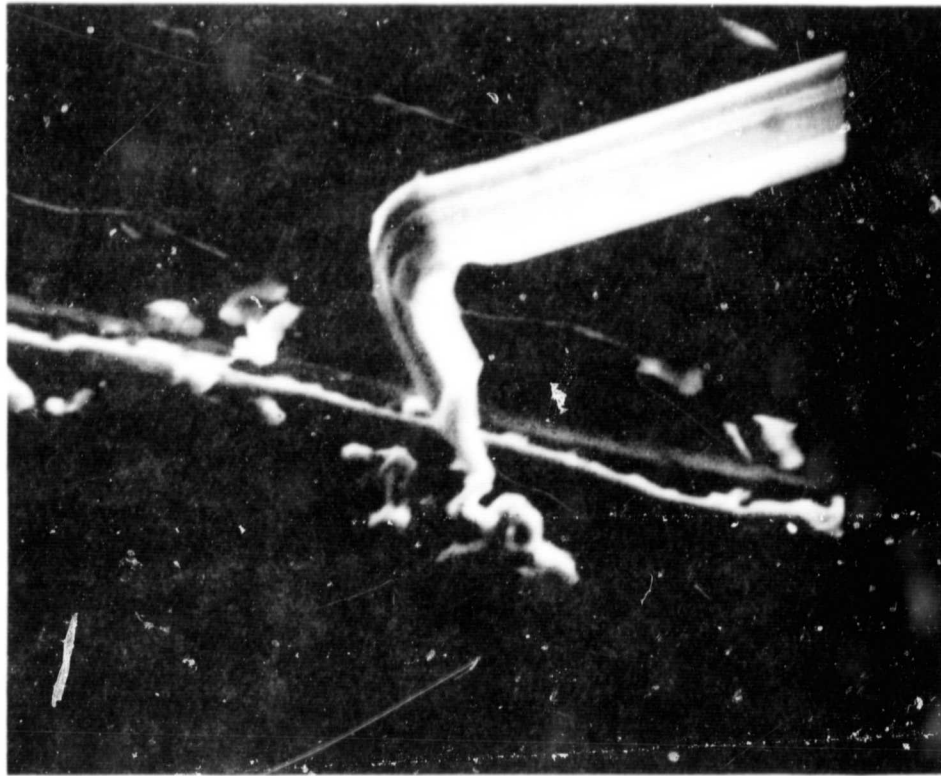


Fig. 4. Tin whisker base seen with scanning electron microscope. Note the weak extruded base which makes it unstable in a gravity field. (Photo by Dave Nichols, MSFC.)

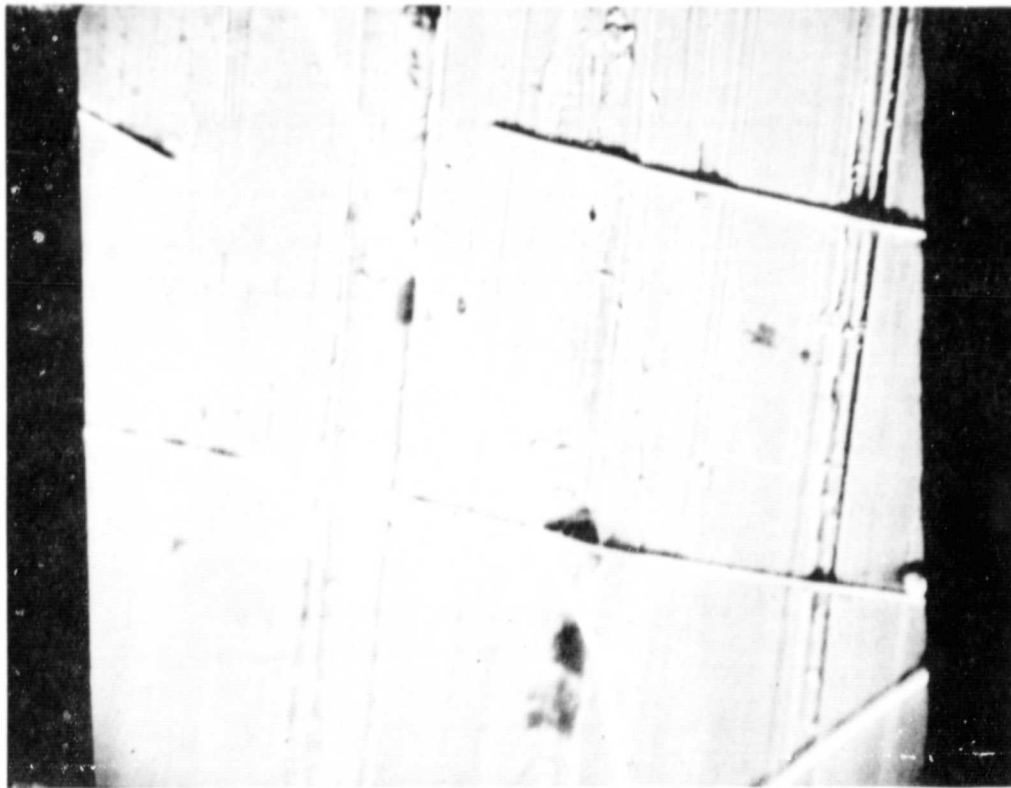


Fig. 5.

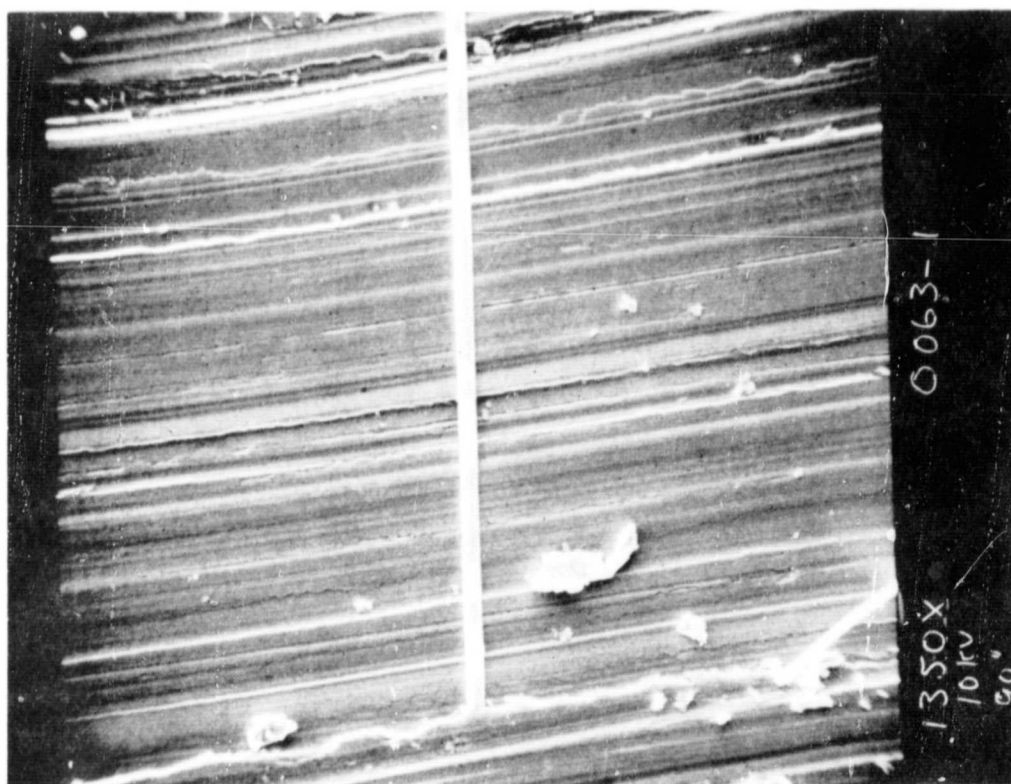


Fig. 6.

Fig. 5 This tin whisker has fallen to the substrate.

Fig. 6. More fallen tin whiskers (1350x). Note that whiskers are strongly attracted to substrate. Whiskers probably fell due to some combination of gravity, vibrations and air motion.

(Photos by Dave Nichols.)

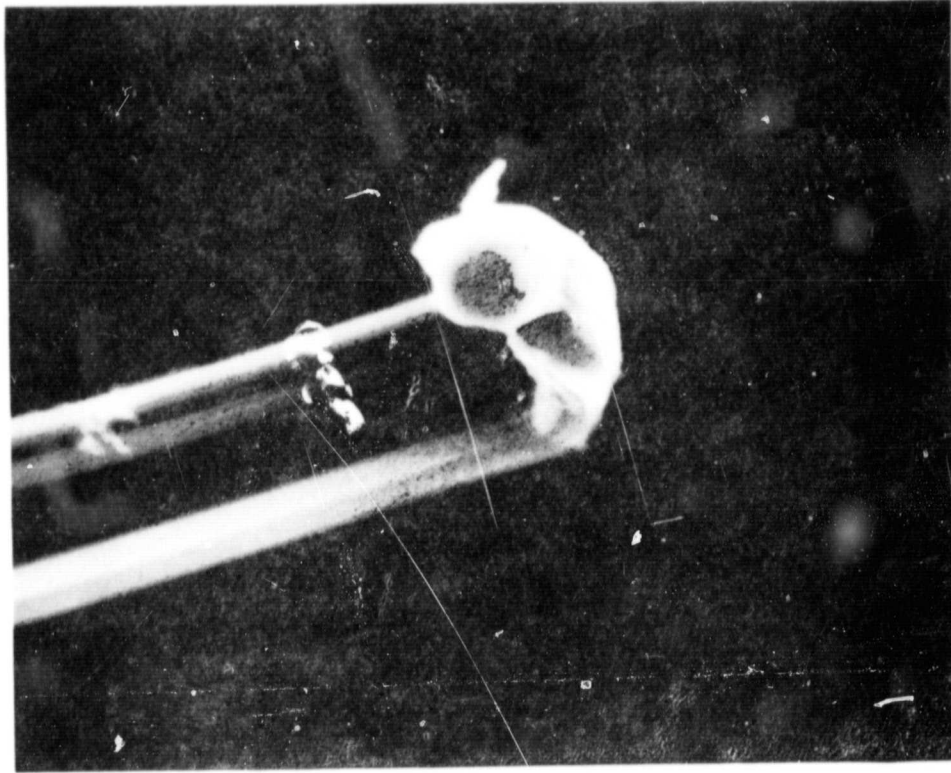


Fig. 7. Note the cross section of this whisker. (Photo by Dave Nichols.)

help in producing better whiskers.

The SEM brings out surface features of whiskers which have not been observed directly. Previous nonscanning (transmission) electron microscopes showed only a shadow of a whisker or, at best, replica impression had been made to determine surface features. Now, with the SEM, surface features are observed directly. (The improved picture detail may be appreciated by comparing Fig. 3, which was from a transmission electron microscope, to Figs. 4 through 9, which are from a SEM.) The most noted features are the longitudinal "fluted" markings along the length of the whisker seen in Figs. 4, 7, 8, and 9.

Observers have reported that whiskers were hollow since they split lengthwise easily. However, this splitting is now easily understood in view of our whisker in Fig. 8 which looks like two parallel whiskers connected by a thin web. So we conclude that some of the whiskers reported to be hollow actually had the shape shown in Fig. 8.

Figs. 7 and 9 show some interesting growth shapes. The helix, although not understood, is not uncommon. Perhaps further study of the helix will yield data which will improve whisker growth theory in general.

c. Reduced Air Motion and Vibration Found Beneficial to Whisker Growth

A whisker growth bolt enclosed in a glass container was bolted to a wall plate for about 6 months. These whiskers were subject to a gravity force acting in a fixed direction.

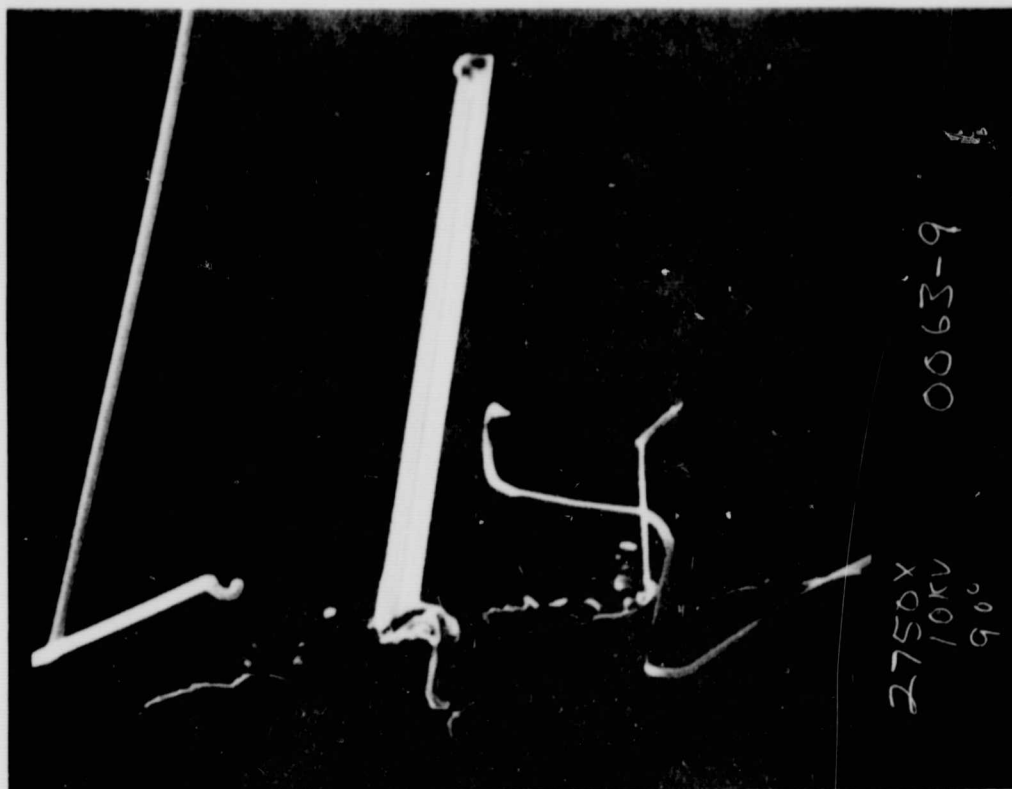


Fig. 8.



Fig. 9.

Fig. 8. Odd shape tin whiskers. Note that magnification is 2750x.

Fig. 9. Helix shape whisker. Note the good depth of field which would not be obtainable with an optical microscope.

(Photos by Dave Nichols.)

The whisker quality was noticeably improved under this condition of low vibration and reduced air flow. The whisker lengths were up to 3 mm long, about twice as long as whiskers which are unshielded.

d. No Long Whiskers Were Detected Using Electrical Probe in Shielded Vibration-Free Environment

Whisker growth bolts were mounted on a heavy wall plate and enclosed with a metal electrode to detect long thin whiskers which might not be observed with a microscope. Whiskers which grew to a length of 1 cm would contact the electrode, thus shorting it to ground as shown in Fig. 12. An ohmmeter was used to test for shorted electrodes. No shorted electrodes were found after about six months, indicating that no whiskers had grown to 1 cm in perpendicular length.

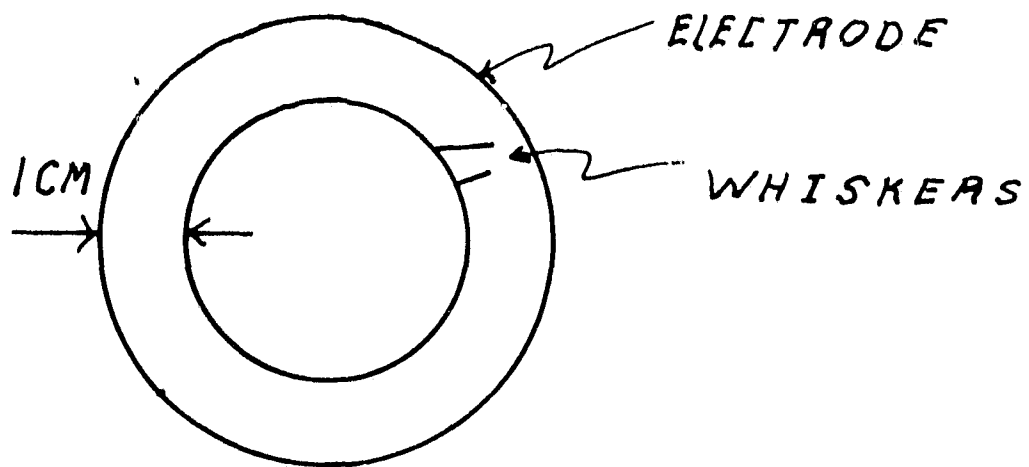


Fig. 12. An electrode was placed around the whisker growth bolt in an effort to detect ultra thin long whiskers of lengths up to 1 cm. None were detected.

III. NEW TECHNIQUES GENERATED FOR STUDYING THE EFFECTS OF GRAVITY ON WHISKER GROWTH

a. Rotation of Growth Tube May Reduce Convection Transport by an Order of Magnitude

It appears that reduced convection experiments for crystal whisker growth may be performed in the laboratory. A rough calculation shows that the convection cycles (Fig. 10) move at a velocity of about 1 cm/sec. If the Pyrex tube in Fig. 10 were rotated about its axis at a rate of about 4 revolutions per second, then the element of gas would be rotated from bottom to top, thus reversing the convection force before the mass element could move more than 0.25 cm. As the tube rotates, the convective force on an element of gas would oscillate back and forth producing minimal net flow of gas.

This experiment should represent the nearest approximation to zero gravity on whisker growth by the vapor deposition technique. By the same arrangement, one could predict the effect of reduced convection on bulk single crystal vapor growth. This would also be of great benefit to R.P.I. in defining an optimum crystal growth flight package.

This rotating tube could be used to study vapor transport in a temperature gradient both with suppressed and unsuppressed convection, i.e., by stopping the rotation, the transport with unsuppressed convection could be measured.

This experiment would be carried out by increasing the furnace temperature in Fig. 10 until a thin film began to plate out on the portion of the tube exterior to the furnace. Then, the plating thickness would be measured after an

equal time interval, both with rotation and without.

Of course, the direct gravity force mentioned in Section I.a is still present in the rotating tube and is of more importance since the g-vector is rotating with respect to the whisker.

b. New Method of Determining Whisker Diameter

Whisker diameters are obviously important since they are related to the aspect ratios. However, precise data on diameters of important thin whiskers such as Cd are not easily obtained. Whisker diameter may possibly be calculated from whisker bending measurements in a known air flow velocity. Eq. 2 involves the whisker thickness h and the displacement of the tip d so h could be calculated. With an air flow, a new constant factor is required. (Refer to Section I.e of this report where it is shown that a velocity of 10 cm/sec is equivalent to 1 g.)

c. Stress May Stop Whisker Growth

Observers have reported that "squeeze" whiskers grow to a length of 1 mm and then stop growing. But by pulling away the 1 mm whisker, another is found to grow in its place. The stress due to gravity and air motion concentrated at the growth site of a 1 mm whisker is suspected to be large enough to alter the dislocation associated with whisker growth. This hypothesis explains our observations (mentioned in II.d) that whiskers grow to longer lengths when shielded.

d. Test on Old Growth of Tin Whiskers to See if Regrowth Will Occur After Five Years of Dormancy

Some of the tin whiskers in our lab were "squeeze" grown five years ago.

By using a rubber tip such as a pencil eraser, one may clear all whiskers from a portion of the substrate. It would be very interesting to determine the possibility that regrowth could occur in this "cleared" area after five years of no growth. This would strengthen the argument that some external stress at the whisker base, rather than a lack of whisker material, is preventing growth.*

e. Reduced Convection Cell

A reduced-convection cell is proposed here on earth to approximate 0-g environment in space. Most whisker growth takes place in a temperature gradient with convection. However, if the growth-region temperature could be held constant by a good conducting material, crystal whisker growth would take place in an almost zero-gradient zero-convection region. This would represent a good approximation to a 0-g environment from the standpoint of convection.

The apparatus would be similar to Fig. 10 except that to assure an isothermal growth area, a copper cylinder would be placed around the tube over the whisker growth area. Some baffles would be added at each end of the cylinder inside the tube to assure that the convection cycles from the nonisothermal regions do not carry over into the isothermal region.

This cell was conceived by Mr. Bisenius.

*In some cases this stress could be due to gravity.

IV. ORBITAL FLIGHT EXPERIMENTS

The knowledge and experience gained in this study is being used to recommend flight experiments and to propose changes in existing flight packages to accommodate whisker growth experiments in orbit.

Westinghouse has designed a furnace to grow GaAs single crystals from solution. A brief proposal has been submitted to reduce the furnace temperature to 390° and 250° on the hot and cool end respectively. A drawing was submitted for modifying the furnace to accommodate a larger growth tube to make the growth more sensitive to gravity.

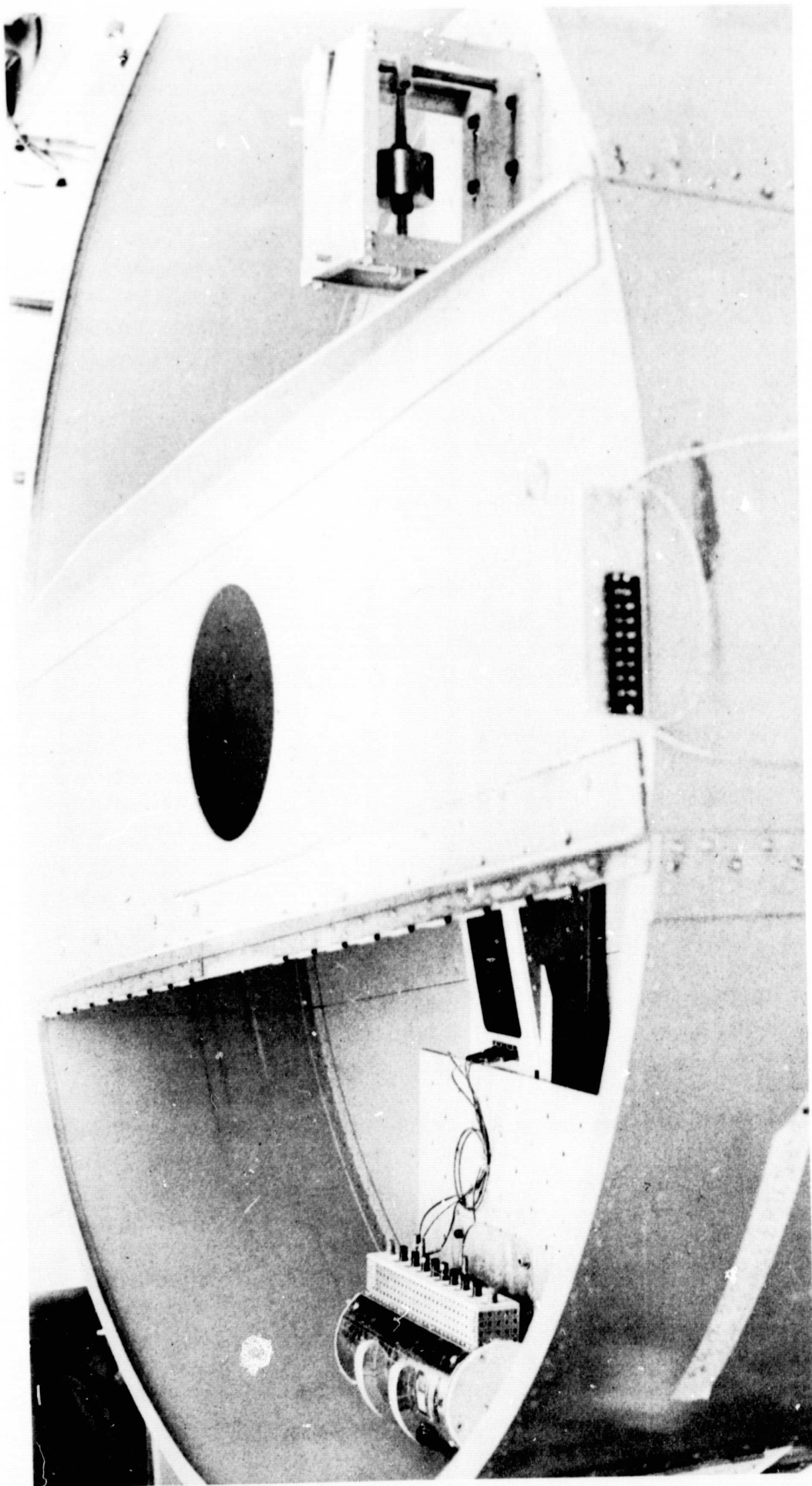


Fig. 13. This shows a top view of the centrifuge with counterbalance. Note that the cylindrical weight may be moved vertically as well as radially.

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