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CONTAINERLESS PROCESSING OF HYPERMONOTECTIC AND GLASS FORMING ALLOYS  
USING THE MARSHALL SPACE FLIGHT CENTER  
100 METER DROP TUBE FACILITY

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CONTAINERLESS PROCESSING OF HYPERMONOTECTIC AND GLASS FORMING  
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DROP TUBE FACILITY

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**ABSTRACT**

Two separate projects were carried out to study alloys whose solidification structures can be strongly influenced by the presence of a container during melting and solidification.

One project involved containerless solidification of hypermonotectic  $Au_{35}Rh_{65}$  alloys. This alloy exhibits liquid immiscibility over a temperature range. In previous studies on similar alloy systems, attempts to obtain dispersions of the immiscible liquid phases by using microgravity processing to prevent sedimentation have often failed. Surface tension driven flows occur when the minority liquid phase completely wets the walls of the container, resulting in massive segregation even under microgravity conditions. It has been suggested that containerless melting might be one solution to this problem. However, surface tension driven flows could also lead to accumulation of the minority liquid phase at the external surface of a containerlessly melted alloy. The research underway is a first step in determining the influence of containerless, microgravity processing on immiscible alloys.

Nickel-niobium alloys are also being studied using the drop tube facility. One alloy in this system, a  $Ni_{60}Nb_{40}$  alloy, is a good candidate for the formation of a bulk metallic glass. Amorphous alloys of this composition have been produced using thin film and mechanical alloying techniques. However, theory indicates that if heterogeneous nucleation can be avoided, it should be possible to produce an amorphous structure in this system using a moderate cooling rate from the melt. The containerless melting and solidification capabilities of the drop tube facility provide ideal conditions for a study of this type. To date, several  $Ni_{60}Nb_{40}$  samples have been levitated, melted and cooled during 4.6 seconds of free fall in the 100 meter drop tube. The structures obtained are discussed in the following report.

## ACKNOWLEDDgements

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## FORWARD

The possibility of experimentation involving low gravity, containerless processing provides new opportunities for research in many intriguing areas. Among these areas are studies concerning immiscible alloys (hypermonotectic alloys) and metallic glasses. This report will discuss research in these two areas.

A convenient way to carry out experiments requiring relatively short duration containerless, microgravity conditions is through the use of a drop tube [1-4]. The following report describes two projects carried out using the 100 meter Drop Tube Facility located at the Marshall Space Flight Center in Huntsville, Alabama. This facility permits containerless melting followed by containerless solidification during 4.6 seconds of free fall [5]. The report is divided into two parts in order to maintain continuity in the discussion of each project.

## PART ONE - GLASSY METALS

### INTRODUCTION

Amorphous metallic alloys were first produced by quenching materials from a melt in 1960 [6]. Very high cooling rates were utilized in this original work to rapidly "solidify" the molten alloy and quench in the amorphous, liquid like structure. Of course, in order to produce an amorphous metal it is necessary to avoid nucleation of the crystalline phase during cooling from the melt. If a high enough cooling rate is utilized ( $10^6$  °C/sec in most systems) the glass transition temperature,  $T_g$ , is reached before nucleation can take place [7]. Once the sample is below the glass transition temperature, atomic movement is restricted and nucleation does not readily occur. The amorphous structures that result are obviously somewhat unstable and in several systems can decompose at room temperature.

After the initial break through in 1960, it was soon found that amorphous metals could be formed from many alloy systems. However, it was also apparent that production was limited to very thin films (approximately 40um) in order to allow rapid heat extraction. Commercialization of amorphous alloys eventually took place in the late 1970's with the production of thin ribbons of ferromagnetic "glassy metals". These alloys had no magnetic anisotropy since they had no crystal structure. This feature, along with other attractive magnetic properties, made the alloys preferable to their crystalline counterparts for applications where soft magnetic materials were needed, such as transformers.

To date, the majority of the work carried out in the production of amorphous metals has been involved with thin films. Very little has been done with bulk samples because the moderate cooling rates attainable are insufficient to suppress heterogeneous nucleation. However, if heterogeneous nucleation can be prevented in bulk samples, it appears that the achievable cooling rates are sufficient to suppress homogeneous nucleation in at least a few alloy systems.

Since the main heterogeneous nucleation site in most experiments is the container wall, a process is needed where this contact can be eliminated. The Marshall Space Flight Center 100m Drop Tube Facility provides the containerless melting and solidification capabilities that are needed to avoid this contact. As a result, it is reasonable to assume that if high purity, inclusion free samples can be produced and then containerlessly melted and solidified using the drop tube, heterogeneous nucleation can be avoided.

Once the problem of heterogeneous nucleation is overcome, the next difficulty is that of homogeneous nucleation. There are several alloy systems where it may be possible to avoid homogeneous nucleation using

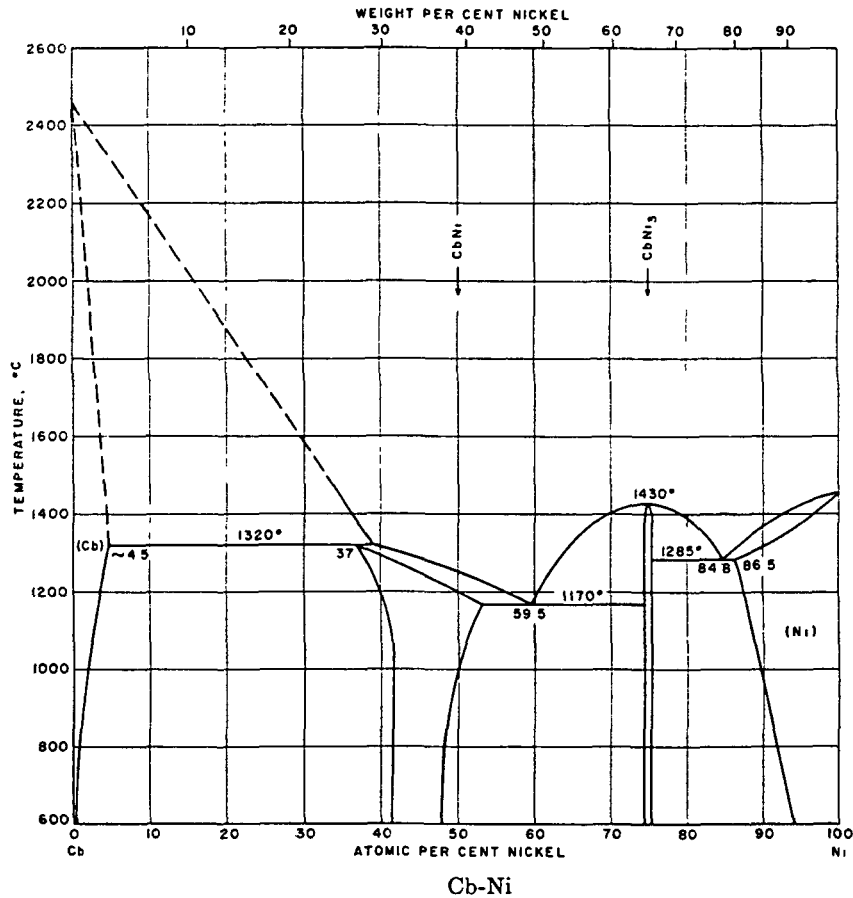


Figure 1. Phase diagram of the nickel-niobium system.

only moderate cooling rates. What is necessary is an alloy that has a high glass transition temperature,  $T_g$ , in relation to its liquidus temperature,  $T_m$ . Alloys in this category would be expected to have a relatively low homogeneous nucleation rate. The ratio  $T_{rg} = T_g/T_m$  is used to quantify this relationship and is called the reduced glass transition temperature. The highest reduced glass transition temperature found in the literature is 0.67 and is for a  $Ni_{60}Nb_{40}$  alloy [8]. The phase diagram for this system is shown in Figure 1. Note that the  $Ni_{60}Nb_{40}$  alloy has the lowest liquidus temperature in this system.

Work by Drehman, Greer and Turnbull [9] has shown that for  $Pd_{40}Ni_{40}P_{20}$  alloys ( $T_{rg}=0.66$ ), if heterogeneous nucleation can be avoided,

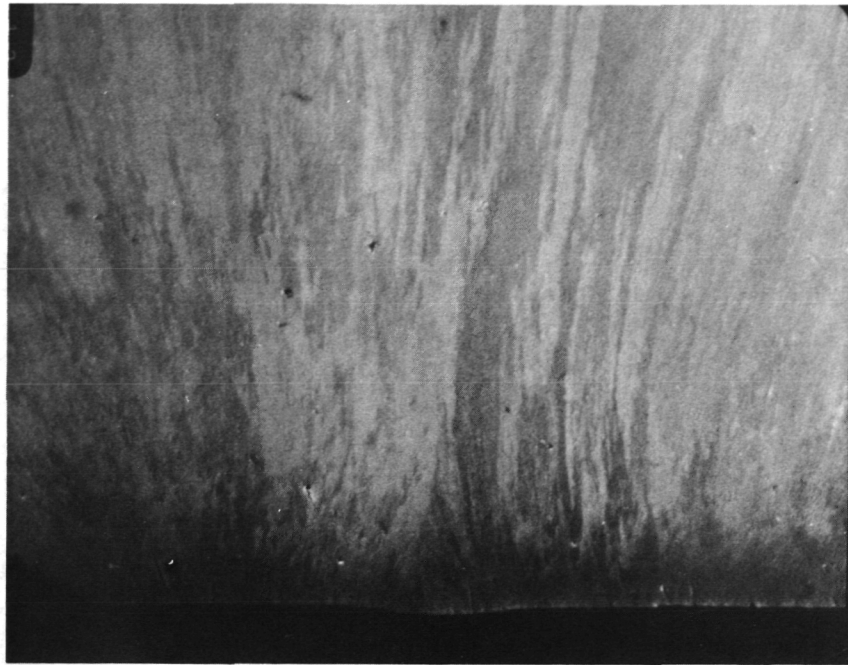


Figure 2. Microstructure of an arc melted Ni<sub>60</sub>Nb<sub>40</sub> alloy. (60X)

cooling rates of only several degrees per second are adequate for the formation of an amorphous structure. Because of the similar  $T_{rg}$  for Ni<sub>60</sub>Nb<sub>40</sub> alloys, it should also be possible to form a glassy metal using moderate cooling rates, if heterogeneous nucleation is avoided. The containerless processing capability of the Drop Tube Facility should be most useful in this experimentation.

#### EXPERIMENTAL PROCEDURE

Ni<sub>60</sub>Nb<sub>40</sub> alloys were prepared from Marz grade nickel and niobium. Each of the constituents was etched for 60 seconds in concentrated HF and washed with acetone before alloying. Melting and alloying were carried out using a small tungsten electrode arc melting unit with a water cooled copper hearth. Melting was carried out under an argon atmosphere.

One of the arc melted samples was sectioned and prepared for microscopic analysis. Analysis revealed a uniform structure consisting of columnar grains aligned parallel to the direction of heat flow during solidification. See Figure 2.



Figure 3. Microstructure of a Ni<sub>60</sub>Nb<sub>40</sub> sample melted and solidified under containerless conditions. (60X)

After arc melting, samples were etched for one minute in concentrated HF in order to remove any oxides or other contaminants from the surface. A slight change in surface color (from silvery to a bluish tint) was noted as a result of the etching treatment. The etched samples were then utilized for drop tube experiments.

An absolute pressure of 500 Torr of argon was used in the belljar assembly of the drop tube which houses the electromagnetic levitator. 500 Torr of helium was used in the remainder of the tube in order to maximize the cooling rate of the sample.

#### RESULTS AND DISCUSSION

Samples levitated easily and were dropped at temperatures ranging from 2420K to 2700K. The dropped samples had a relatively dark appearance indicative of an oxidized surface.

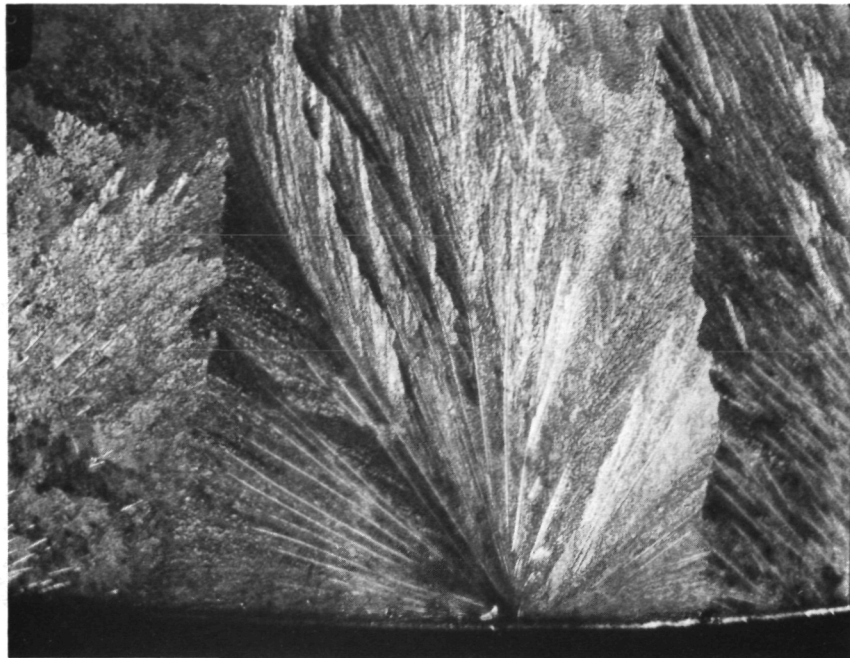


Figure 4. Needle like structure radiating from nucleation site on a dropped Ni<sub>60</sub>Nb<sub>40</sub> sample. (240X)

Two of the samples were sectioned, mounted and polished for metallographic examination. Etching with a solution of 4 parts H<sub>2</sub>O, 3 parts HNO<sub>3</sub> and 1 part HF for ten seconds provided microstructural contrast under sensitive tint. Analysis indicated that nucleation of the crystalline phases had occurred at relatively few sites on the surfaces of the samples and had led to the formation of several columnar grains. See Figure 3. Unusual, needle like structures were evident which appeared to radiate out from the nucleation sites. See Figure 4. Energy dispersive x-ray analysis is planned in order to determine the composition of these needles.

A second series of samples of the same composition was produced using a similar procedure. However, in this case the arc cast samples were not subjected to etching in HF before being dropped. This was because of concern over the discoloration experienced in the first series of alloys. A helium - 6% hydrogen gas mixture at an absolute pressure of 500 Torr was utilized in the bell jar assembly of the drop tube when levitating and melting these samples. The reducing gas mixture was used to minimize the chances of the formation of oxides which could serve as heterogeneous nucleation sites.

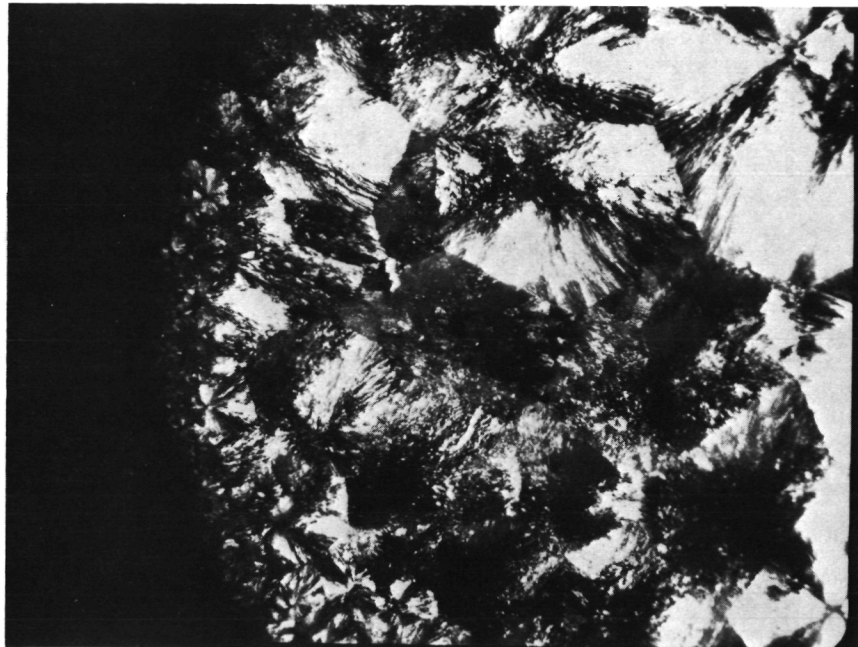


Figure 5. Equiaxed grain structure obtained from a Ni<sub>60</sub>Nb<sub>40</sub> sample dropped without prior surface etching. (37.5X)

The samples were again easily levitated and melted. An attempt was made to drop these samples at temperatures closer to their liquidus than those dropped previously. Drop temperatures ranged from 1850K to 2340K. All of the dropped samples appeared to have some surface oxidation. Metallographic analysis again revealed that nucleation of the crystalline phase had occurred. However, in this case a much larger number of nucleation sites was apparent. The grains were smaller and more equiaxed, with the grain size ranging from a minimum near the surface to a maximum near the center of the samples. See Figure 5.

While amorphous structures have not yet been obtained in these bulk Ni-Nb samples, there is good reason to believe this will eventually be accomplished in the drop tube. Current plans are to experiment with various surface etchant and bell jar atmosphere combinations as a first step. Based on the results, additional experiments may be carried out using an amorphous flux coating on the samples in order to minimize surface oxidation and remove any impurities. If the flux maintains an amorphous structure it should not contribute significantly to nucleation of the crystalline phase.



## CONCLUSIONS

Additional work is needed in order to determine the feasibility of producing bulk amorphous samples using the containerless melting and solidification capabilities of the drop tube. At this time, one difficulty appears to be surface oxidation of the samples, either during melting in the levitator or during free fall down the length of the tube. The use of a flux is being considered for future experiments.

Results indicated that samples which were etched in HF, melted in an argon atmosphere and dropped through helium, contained a columnar microstructure resulting from nucleation occurring at relatively few sites on the surface. Samples melted in the unetched condition, but in a reducing helium-hydrogen gas mixture, and then dropped through a helium atmosphere, contained a much more equiaxed grain structure with a relatively fine grain size at the surface varying to a much larger grain size near the center of the sample. Obviously, nucleation events were not limited to the surface of the samples under the latter conditions. Continued work on this project is planned.

## PART TWO - IMMISCIBLE ALLOYS

### INTRODUCTION

In immiscible alloy systems, an example being systems that contain a monotectic reaction, two liquids are found over some temperature and composition range which will not mix. When immiscible alloys are solidified under lg conditions, segregation usually occurs due to sedimentation of the more dense liquid phase. Alloys in these systems are interesting to study using microgravity conditions since solidification in the absence of a gravitational field removes the driving force for sedimentation. As a result, microgravity processing should permit the formation of a dispersion of the two liquid phases instead of the gross segregation that normally occurs during solidification of immiscible alloys [10-13].

Because of their grossly segregated structures, most immiscible alloys have been only of academic interest. However, if fine dispersions of the immiscible phases can be obtained in some of these alloy systems, it is quite possible that materials with unique properties can be produced. Applications could include electrical contact materials, catalytic materials, and even fine particle magnets and superconductors [14].

For these reasons, there is considerable interest in processing immiscible materials under microgravity conditions. The low gravity, containerless solidification capability of the drop tube can be most useful in this area. Therefore, the objective of this work was to determine the effect of solidification under the microgravity conditions obtained during free fall of a sample in the drop tube. Of particular interest was the distribution of phases in an immiscible alloy.

Several factors had to be considered in selecting an appropriate alloy system for study. It was desirable to use a system with a known phase diagram and with sufficient differences in the densities of the immiscible phases to permit them to easily segregate when solidified under lg conditions. A system was also needed that possessed a relatively high monotectic temperature. This was needed for two reasons. First, a high monotectic temperature would result in fairly rapid cooling rates and help ensure solidification during free fall. Second, high temperatures would result in sufficient radiation to permit tracking of the samples during free fall using the infrared detectors in the tube. It was also desirable to use an alloy system in which oxidation would not pose a major problem.

One system that met all of these requirements was the gold-rhodium system. See Figure 6. This system provided an additional benefit since the color differences between the phases would greatly facilitate their identification during analysis.

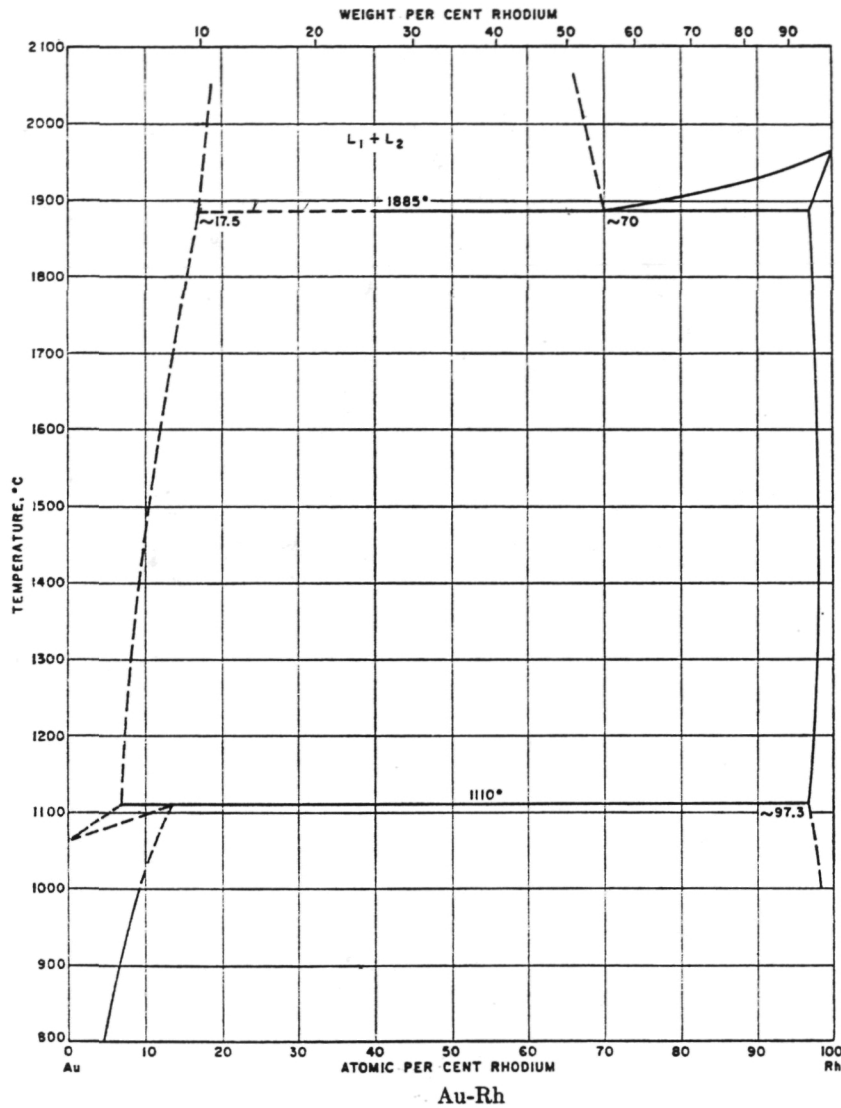


Figure 6. Phase diagram of the gold-rhodium alloy system.

#### EXPERIMENTAL PROCEDURE

Hypermonotectic gold-rhodium alloys ( $Au_{35}Rh_{65}$ ) were produced by melting/alloying Marz purity elements together in a small arc melter. A tungsten electrode was utilized and melting and alloying were carried out under an argon atmosphere. The constituents were first melted together and then allowed to solidify. The solidified samples were then inverted and remelted. This was done several times. The structures obtained were roughly as expected; the more dense, gold rich immiscible phase tended to settle to the bottom of the sample. This was apparent from a simple analysis of the exterior of the samples. See Figure 7.

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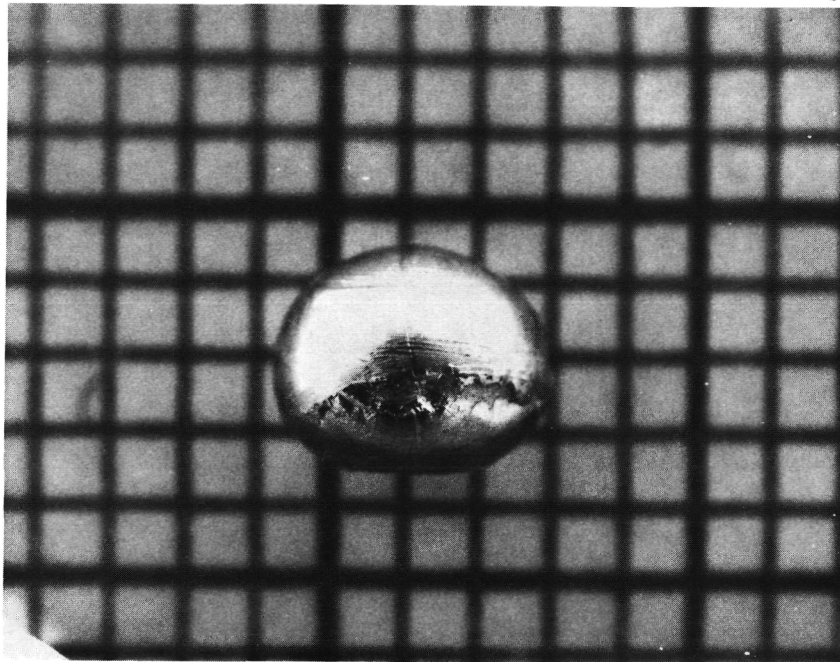


Figure 7. General appearance of an arc melted  $Au_{35}Rh_{65}$  alloy. (10X)

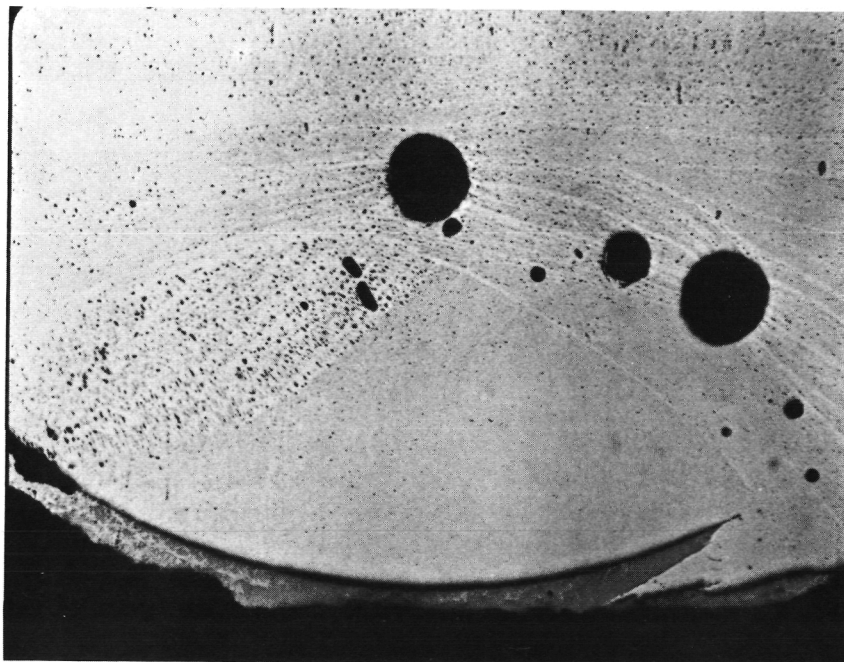


Figure 8. Section through an arc melted  $Au_{35}Rh_{65}$  alloy. Note segregation. Grey areas are gold rich. (60X)

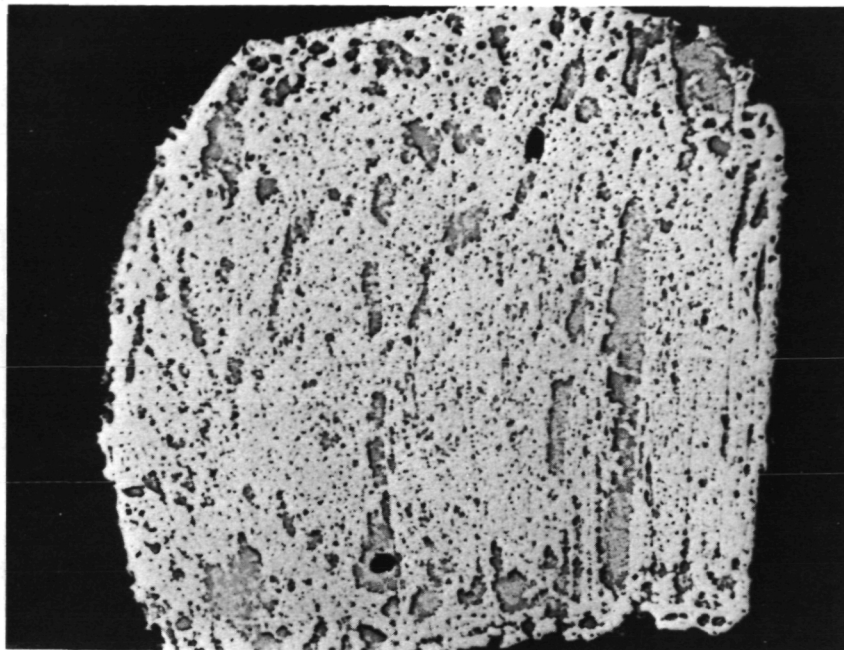


Figure 9. Fragment from dropped  $Au_{35}Rh_{65}$  sample showing partially dispersed structure. Grey areas represent location of the gold rich minority liquid phase. (60X)

An arc melted sample was sectioned revealing a gold rich region near the bottom with a rhodium rich region of roughly monotectic composition being present near the top. See Figure 8.

Samples were then taken to the drop tube for levitation and melting using the electromagnetic levitator/furnace. An atmosphere of 500 Torr of argon was used in the bell jar assembly while 500 Torr of helium was utilized in the tube. Helium was used in order to aid in cooling during free fall.

#### RESULTS AND DISCUSSION

When taken to the drop tube for containerless, low gravity processing, the  $Au_{35}Rh_{65}$  samples levitated nicely when power was initially applied to the levitator coil, but then fell unexpectedly from the coil after reaching a temperature of approximately 2200K. As a result, the first sample processed hit the tube isolation valve which is normally left closed until just before the samples are dropped. The fragmented sample was retrieved and later sectioned and polished using normal metallographic procedures. Etching was not required. Microscopic examination revealed the partially dispersed structure shown in Figure 9. From this micrograph it appears as if some melting of the sample occurred before it fell from the coil. However, it is difficult

to tell if the sample reached a sufficient temperature to get into the single phase liquid region. Optical pyrometer data was obtained, but is somewhat suspect due to uncertainty in the emissivity of the samples.

It is not known at this time why these samples fell from the levitation coil during heating. It is speculated that they fell due to a change in their electrical characteristics which occurred either at the monotectic temperature or at the temperature where a single phase liquid would form. Significant changes in the compositions and amounts of the phases will take place at both of these temperatures. As mentioned previously, due to the uncertainty in the emissivity of the alloy it is difficult to determine the actual temperature at which the samples fell from the coil. However, data obtained in subsequent tests indicated only a 25°C spread between the temperatures at which four samples fell.

Bench tests were carried out to determine if the levitation coil power supply could be tuned to better levitate Au<sub>35</sub>Rh<sub>65</sub> samples. These tests were inconclusive but indicated that levitation of even the solid samples was considerably more difficult than for most alloys.

An additional set of Au<sub>35</sub>Rh<sub>65</sub> alloys was prepared using the same procedure discussed previously. Four 300mg samples were prepared and one 650mg sample. One of the 300mg samples was saved for sectioning without dropping.

It was decided to attempt additional drops by opening the tube isolation valve before power was applied to the levitation coil. With this approach, if the samples fell from the coil prematurely, they would fall the entire length of the tube instead of hitting the tube isolation valve. An atmosphere of 500 Torr of helium was used in the drop tube, while a mixture of helium with 6% hydrogen was used in the bell jar assembly. The samples again levitated nicely, at first, but then fell from the coil after reaching an elevated temperature. The samples fell the length of the tube and, with one exception, were intact when retrieved from the catch pan.

All of the dropped samples had a gold colored surface as opposed to the partially gold colored, partially silver colored surfaces on the arc melted samples. This was not totally unexpected. In several of the flight experiments that have been carried out on hypermonotectic alloys, surface tension driven segregation has been encountered due to one phase wetting the walls of the container [10]. It has been observed that if the minority liquid phase wets the container wall, flows occur in these alloys, even under microgravity conditions, that can lead to almost complete segregation. The resulting structure shows a central region which consists of the majority liquid phase, with the minority liquid being in the outer regions, in contact with the container. Similarly, it is possible for the minority liquid phase to wet the outer surface of a containerlessly melted and solidified sample. The gold colored outer surface is an indication that this may be taking place in Au<sub>35</sub>Rh<sub>65</sub> alloys.

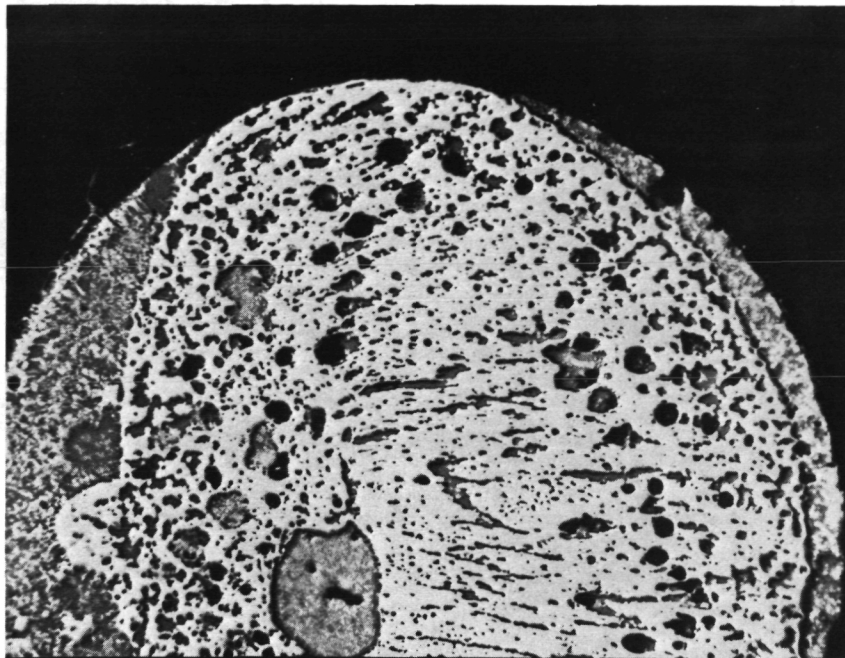


Figure 10. Dropped  $Au_{35}Rh_{65}$  sample. Note wetting tendency of the gold rich minority liquid phase. (37.5X)

A section through one of the dropped  $Au_{35}Rh_{65}$  samples is shown in Figure 10. The darker colored regions on each side of the spherical sample and dispersed throughout represent the location of the gold rich minority liquid phase when solidification took place. The tendency for this minority liquid to wet the outer surface is evident from the low contact angle the interface makes with the surface.

Several additional  $Au_{35}Rh_{65}$  samples were levitated and dropped. The structures that resulted from two of these samples are shown in Figures 11 and 12. Note that in both figures, regions of unalloyed pure rhodium (white areas) are present. More segregation is present in the sample shown in Figure 11 than in Figure 10. However, there are regions where relatively fine dispersions of the phases are present. Figure 12 shows a more uniform dispersion of phases than in either of the two previous samples. While not easily discernible from the photomicrographs, the surfaces of the latter two samples were also coated by the gold rich, minority liquid phase.

Obviously, for samples of hypermonotectic composition, primary rhodium regions should not be present. The rhodium regions present in



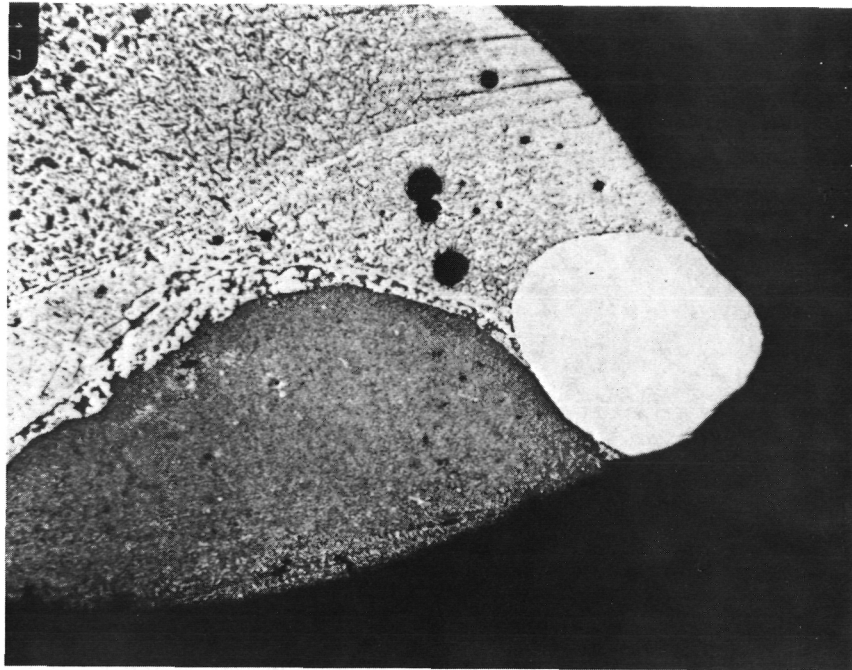


Figure 11. Dropped  $Au_{35}Rh_{65}$  sample. The white area is unalloyed rhodium. (37.5X)

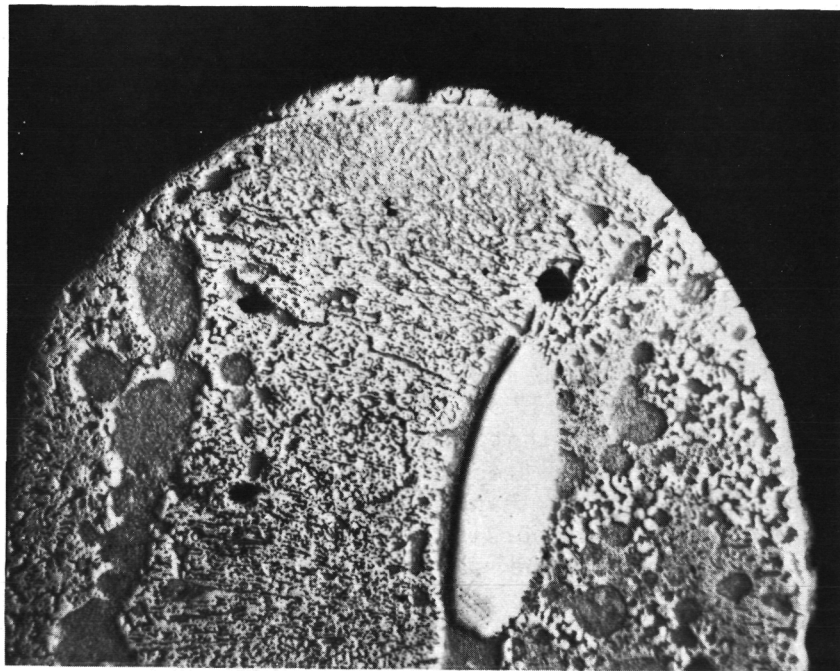


Figure 12. Dropped  $Au_{35}Rh_{65}$  sample. The white area is unalloyed rhodium. (37.5X)



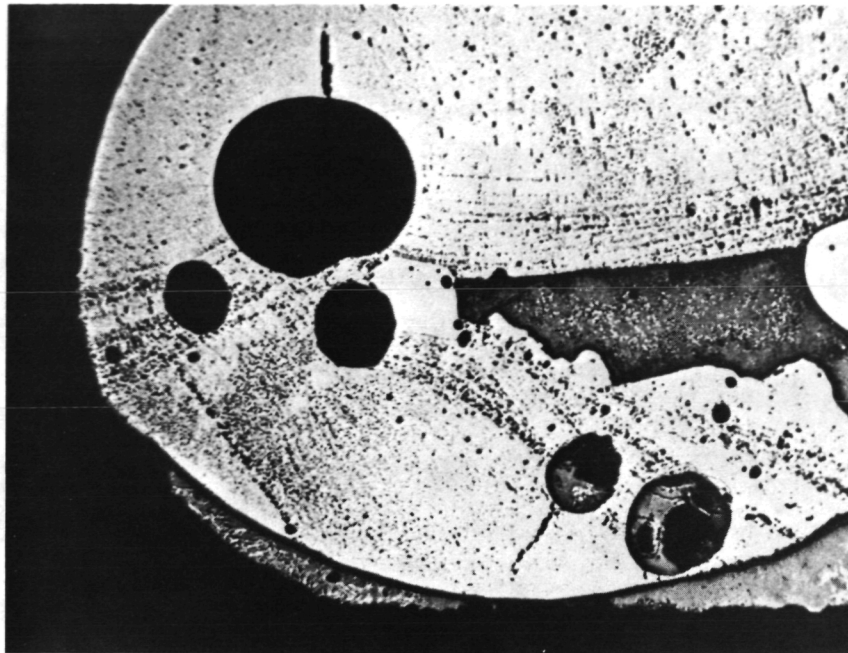


Figure 13. Arc melted  $Au_{35}Rh_{65}$  sample. The white areas indicate unalloyed rhodium was present after arc melting. (37.5X)

Figures 11 and 12 were essentially pure, indicating they probably resulted from incomplete alloying in the initial preparation of the samples in the arc melter. To test this, an additional arc melted sample was sectioned and prepared for metallographic analysis. Analysis revealed areas of pure rhodium (See Figure 13) indicating greater care is needed in preparation of these alloys. The normal procedure was to repeatedly invert and remelt samples until an approximately spherically shaped sample was obtained (four to five attempts were usually required.) After obtaining a spherical shape, samples were usually inverted and remelted one or two additional times. This should be expanded to at least five times to ensure complete alloying of the high melting rhodium component.

While the previous samples had a mass of 300 mg each, one much larger sample of approximately 650mg mass was also produced. This sample was prepared to determine what effect, if any, sample size would have on the tendency of the samples to fall out of the levitation coil when heated.

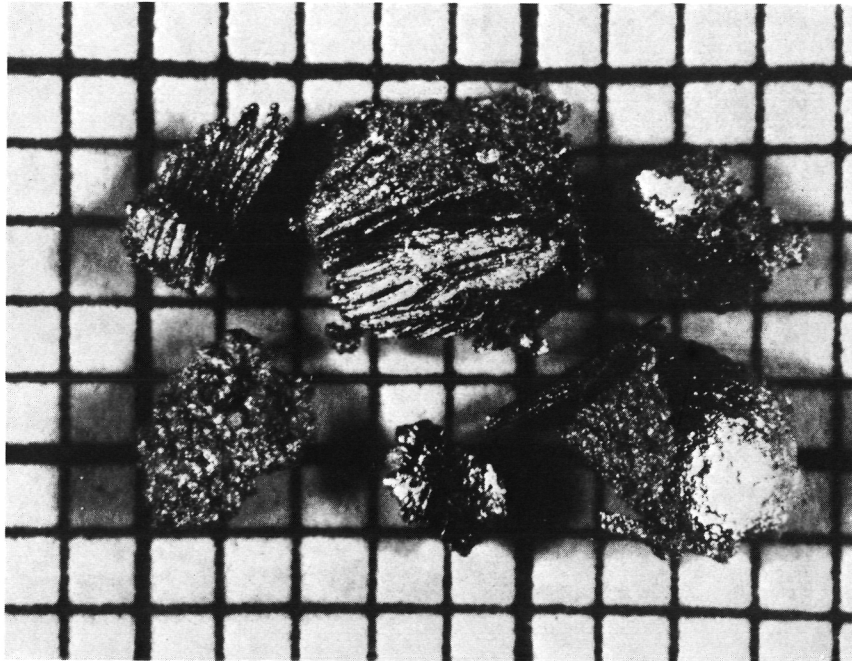


Figure 14. Fragmented 650mg Au<sub>35</sub>Rh<sub>65</sub> sample. Note the fibrous appearance of several of the fragments. (10X)

In previous attempts, 300 mg Au<sub>35</sub>Rh<sub>65</sub> samples had fallen from the levitator after approximately five seconds of full power. In the case of the 650mg sample, levitation took place for at least 15 seconds. In addition, a definite shape change was noted in the sample before it fell. When retrieved, the sample was found to have fractured upon impact at the bottom of the tube. Several of the fragments had a rather unusual fibrous appearance. See Figure 14. This structure was not expected.

Portions of the sample were sectioned perpendicular and parallel to the fiber axis and the resulting surfaces prepared for metallographic examination. Figure 15 shows the microstructure revealed by the section parallel to the fiber axis. (Note the higher magnification.) Filaments of the gold rich phase are apparent in the structure. A transverse view is shown in Figure 16. It can be seen from Figure 16 that the gold rich filaments are not rod like. This micrograph indicates there may be a relatively high connectivity in the structure.

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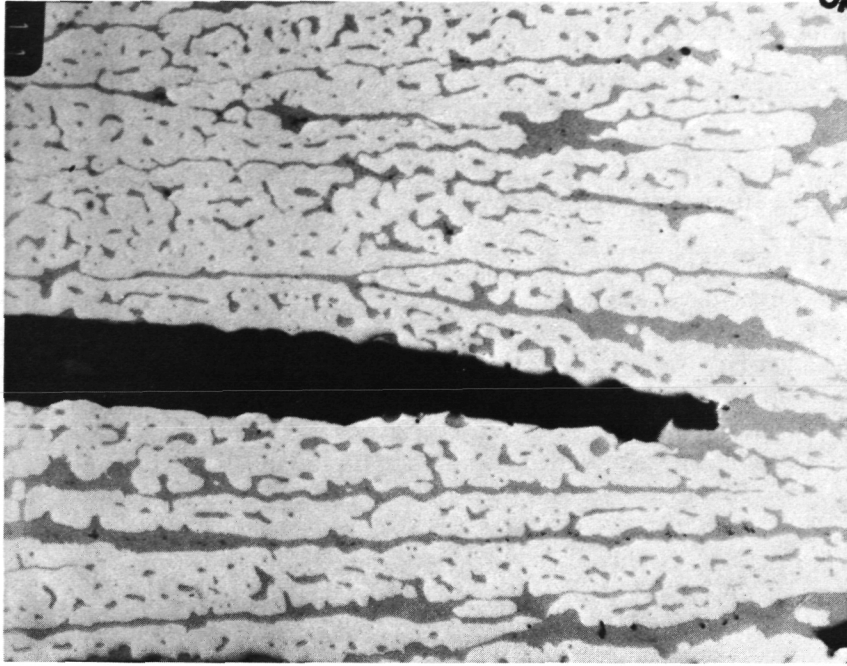


Figure 15. Section parallel to the fiber axis in an aligned structure obtained in a  $\text{Au}_{35}\text{Rh}_{65}$  drop tube sample. (240X)

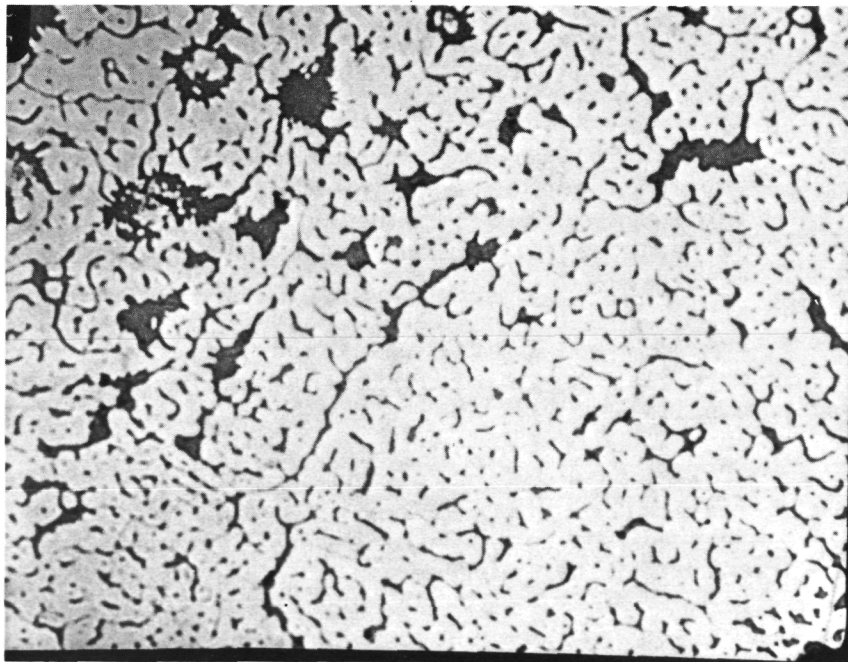


Figure 16. Section perpendicular to the fiber axis in an aligned structure obtained in a  $\text{Au}_{35}\text{Rh}_{65}$  drop tube sample. (240X)

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To date, the cause of the aligned structure is uncertain. Aligned microstructures are normally found in directionally solidified samples. It is possible that some degree of directionality of heat flow occurred in the dropped samples due to convective heat transfer. But this is purely speculative. Additional testing is required to determine the cause of these structures.

Additional research is planned in this area. The structures obtained to date are quite unusual and warrant further investigation. As a first step, it will be necessary to improve the ability to levitate these samples in order to make certain they are heated into the single phase liquid region before they are dropped. It is also desired to melt and drop Au-Rh samples of varying compositions in a vacuum instead of an inert atmosphere in order to eliminate any influence of aerodynamic drag on the solidification process. Many exciting and informative experiments are possible.

### CONCLUSIONS

It appears it may be possible to obtain disperisons of immiscible phases in metallic systems through use of the drop tube. Preliminary tests indicate a tendency towards the formation of a dispersion in Au<sub>35</sub>Rh<sub>65</sub> samples. However, experimental difficulties have been encountered in levitating samples for a sufficient length of time to ensure the attainment of a single phase liquid before samples are dropped.

From research carried out to date, the following conclusions can be drawn.

1. Hypermonotectic Au<sub>35</sub>Rh<sub>65</sub> samples processed under 1 g conditions exhibit gross segregation due to sedimentation of the more dense, gold rich, minority liquid phase. This segregation can be easily seen by eye due to the color differences between the upper and lower regions in arc melted samples.
2. The majority of the levitated and dropped samples contain a more dispersed microstructure than samples processed under 1g conditions.
3. There is a definite tendency for the gold rich, minority liquid phase in Au<sub>35</sub>Rh<sub>65</sub> alloys to wet the outer surface of dropped samples. This is readily apparent due to their gold colored outer surface.

Additional tests are necessary to explain some of the unusual microstructures obtained in this alloy system. Plans are currently being made to continue this investigation.

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