### Department of Metallurgy Massachusetts Institute of Technology Cambridge, Massachusetts

#### RESEARCH ON MECHANISMS OF ALLOY STRENGTHENING

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#### INTRODUCTION

There are two aims to this program, namely:

- a) Studies of oxide dispersion strengthened alloys, to establish strengthening mechanisms, to examine deformation and fracture behavior, and generally to improve thereby the structure and properties of such alloys;
- b) Studies to determine the potential of rapidly quenched liquid metal droplets as a basis for alloy development (splat cooling). The very large changes in solute solubility; the extreme refinement of grain size, dendrite size, and size of excess phases; the minimizing of segregation and the refinement of inclusion particle size; the generation of near-amorphous (homogeneous) structures; the ability to produce a variety of metastable intermetallic compounds; all these suggest a vast potential for structure modifications and property improvements. The first aim is to achieve rapid quenching (greater than about 10<sup>3</sup> or 10<sup>4</sup>°C sec<sup>-1</sup>) with pound lots of alloys to provide sufficient material for mechanical, chemical, and physical testing. The second aim is to select promising alloy systems.

The results of these' studies over the recent six month period are described below

#### I. ALLOY STRENGTHENING BY FINE PARTICLE-DISPERSION

## A. Oxide Dispersion Strengthened Iron-BeO Alloys

All four of the iron-BeO compositions are now known to have been successfully extruded, yielding sound material in all cases. The powder compacts underwent an area reduction ratio of 17:1 on conversion into bar stock. With one exception, the extrusions were performed in the temperature range 1550 to 1570°F; one was deliberately extruded in the austenitic range at 1800°F. The lower temperature extrusions were intended to produce a cold worked alpha phase which retains the stored energy at temperatures up to the transformation temperature; the extrusion at 1800°F would result in a recrystallized structure, free of residual cold work.

Density measurements showed all of the materials to be essentially 100% of theoretical density. Metallographic and chemical analysis checks have indicated that the alloys are clean, of the intended composition, and of excellent metallurgical quality. While further checks will be made, it does appear that the iron oxide content is very low, probably less than a few tenths of a weight percent. It appears that the extensive preventive measures which were taken during processing were largely successful in avoiding contamination by iron oxide. This is considered to be vital in maintaining a stable, sound structure during long-time high temperature exposure.

The microstructures of all of the alloys were similar except for the oxide content. The grain structure can be delineated by means of a dilute nital etch. Assomewhat elongated grain structure is observed, with occasional small regions of what appears to be an equi-axed grain structure. It is intended to determine whether a texture was developed as a result of the extrusion, and to establish whether the observed grain structure plays an important role in determining the mechanical properties of the alloys. Using a light microscope at 1000X, it is not possible to delineate the BeO dispersion, but it is quite evident that clustering was avoided, and a totally satisfactory distribution has been achieved.

Cursory Laue X-ray back-reflection studies of the as-extruded alloys indicate a finely recrystallized matrix. Exposure at 1725°F, in the austenitic range, does not appear to change the pattern significantly. It would appear from this that there may have been sufficient heating during extrusion to raise the temperature of the billets up to or above the transformation temperature. These studies are continuing.

Among the extruded alloys are the following compositions and structural variables:

- a) 0.75 v/o BeO
- b) 3 v/o BeO
- c) 6 v/o BeO
- d) approximately 2.5 v/o BeO in a matrix of iron containing approximately 0.5 wt. % Be remaining in solid solution

Composition (d) was attained by diffusing the naturally formed iron oxide on attrited, submicron powders into the bulk of the powder to form a partially, internally oxidized structure. The central portion of the submicron flake material remains unoxidized, leaving beryllium in solid solution. Composition (c) was achieved with two different powder sizes. One was -44 micron material; the other was attrited near-micron flakes. In both cases internal oxidation was complete, however, the diffusion distance for the near-micron flake powder is exceedingly short, and internal oxidation was readily achieved.

Room temperature hardness measurements, after exposure of the as-extruded material to progressively higher temperatures, serve as a prime measure of the stability of the resultant structures. As examples of the stability of these alloys in terms of hardness, the following results were achieved:

- a) The .75 and the 3 v/o BeO alloys in the as-extruded forms showed values of approximately Rockwell G 60, maintaining these values on annealing up to temperatures of 1625°F. On heating into the austenitic range, the .75 BeO alloy dropped to Rockwell G 40, and the three percent BeO alloy dropped to Rockwell G 55.
- b) The 6% BeO alloy showed hardness values of Rockwell G 74 76 in the asextruded condition, and also on annealing as high as 1625°F. The hardness values dropped only to Rockwell G 70 on annealing at 1725°F in the austenitic range.
- c) The Fe-Be solid solution matrix containing 2.5% BeO showed a Rockwell G hardness of 94, which did not decrease on heating to 1625°F, and in fact, did not soften on annealing at 1725°F (it may still have been in the ferritic range at 1725°F because Be closes the gamma loop).

Current creep and creep rupture tests confirm the hoped for stability of these alloys. The creep rupture results will be reported as soon as the tests are somewhat more complete.

Tension test results are reported in Table I for the several alloys described above; results are reported for the as-extruded condition and after annealing at

high temperatures. Additional data are being compiled and discussion is deferred until such time as these tests are complete. In particular, since thermal-mechanical treatments will also be utilized to further strengthen these materials, it appears appropriate to defer discussion of the tension data.

It will be noted in Table I that annealing in the austenitic temperature range results in a loss of yield and ultimate tensile strength, with little or no effect on the ductility. Of considerable importance is the general high level of the observed ductility values of these internally oxidized iron alloys.

Preliminary studies of the effects of additional cold work, with or without intermediate annealing treatments, look quite promising. Some results are reported for the 3% BeO alloy:

- a) The hardness approached a maximum after about 25% reduction of area, whether intermediate annealing treatments were utilized or not. The hardness increased from Rockwell G 60 to about 74 after 25% cold work.
- b) Annealing of the cold worked structure at 1625°F resulted in a slight hardness drop, from Rockwell G 74 to Rockwell G 71. After annealing in the austenitic range at 1725°F, the hardness dropped significantly, to Rockwell G 53.
- c) Tension tests showed that after annealing at temperatures as high as 1625°F, the swaged material continued to show higher strength values than were achieved for the as-extruded condition. After swaging to 35% reduction of area, the yield and ultimate strength values increased to 90,000 and 95,000 psi, respectively. Annealing of this cold worked structure at 1200°F reduced these values only to 82,000 and 88,000 psi. Annealing at 1600°F further reduced these values to 70,000 and 80,000 psi, respectively.

Laue X-ray back-reflection patterns showed broad continuous rings for the swaged conditions, and after annealing treatments to 1625°F; annealing at 1725°F resulted in reversion to the spotty ring pattern.

TABLE I

Tension Tests at 80°F for Fe-BeO Alloys

Alloy Comp. vol. % BeO	Condition	Upper Y. S. in 1000 psi	Lower Y. S. in 1000 psi		Elong.	Red. Area
0.75 BeO	As-ext.	76	62	72	25	75
•	1625° ann.	. 56	52	73	27	75
	1725° ann.	53	46	61	27	74
3.0 BeO	As-ext.	82	61	74	19	52
	1725° ann.	60	50	68	19	50
6.0 BeO	As-ext.	84-91	73-80	98-97	9-11	21-32
	1725° ann.	71-72	63-67	82-87	9-13	20-33
2.5 BeO	As-ext.	130	124	148	10	11
(in Fe-Be matrix)						

Merely to give some idea of the levels of strength values being achieved in the creep and stress rupture tests, and stresses for lives of 20 hours and 100 hours at 1200°F have been in the range of 16,000 to 20,000 psi, and 12,000 to 17,000 psi, respectively, for the weaker and stronger compositions. The slopes of the curves on a log stress versus log rupture time plot are extremely flat. It is interesting to observe that the highest strength values reported here are about the same as those which had been achieved by iron alloys dispersion-strengthened with  ${\rm Al}_2{\rm O}_3$ , derived through mechanical blending of fine powders. In the latter case, it was necessary to utilize about 10 volume percent of oxide, compared to about 3 volume percent BeO in the current studies. The use of only 3% BeO/in a significantly more ductile and workable structure due to the lower oxide content.

We are extremely pleased with the results being obtained currently, and feel that these alloys will undoubtedly be the strongest ferritic iron base alloys reported in modern metallurgy for use at temperatures up to 1200 to 1400°F. Additional creep rupture tests are planned for 1400°F.

The first of the series of surface oxidized, copper-aluminum alloys has been produced in extruded form. This alloy, 92.5% Cu-7.2% Al - 0.3%  ${\rm Al}_2{\rm O}_3$ , was produced in the following manner:

- a) -325 mesh atomized powder of copper-7.5% aluminum was attrited for 6 hours in ethanol to produce a flake product of thickness less than 0.1 micron and of diameter
   3 5 microns.
- b) The material was allowed to air oxidize naturally during comminution, forming both copper oxide and  ${\rm Al}_2{\rm O}_3$  on the surface.
- c) The powder was reduced in hydrogen for 3 hours at  $450^{\circ}\text{C}$  to eliminate the surface copper oxide, leaving only the  $\text{Al}_2\text{O}_3$ .
- d) The flake powders were loaded and compacted into a copper can under an argon atmosphere. Extreme care was taken to avoid oxygen contamination. A packing density of about 60% of theoretical was achieved in this step.
- e) The canned billet was sealed in a mild steel jacket and Heliarc welded to produce a vacuum tight system. The weld was carefully checked and found to be vacuum-tight.
- f) The canned billet was heated to a temperature of 740°C, and extruded at a reduction of area ratio of 20:1 to yield bar stock of approximately 1/2" diameter. The maximum extrusion pressure was 720 tons, and the average was 640 tons force.

Density measurements showed that the alloy had a density of at least 99.7% of theoretical. The structure in a preliminary analysis appeared satisfactory; accordingly, tests have been initiated to check out this produce before extruding the remaining billets. Hardness measurements at room temperature after one hour annealing treatments at progressively higher temperatures are shown in Table II. The hardness results indicate an extremely stable alloy structure. Accordingly, tension tests with increasing temperature, and stress rupture tests at 450° and 650°, are under way.

The following alloys have now been processed through step (c) above: 99 Cu - 1 Al; 97 Cu - 3 Al; 79 Cu - 20 Ni - 1 Al. Extrusions of all of these materials should be completed within the next three to four weeks.

The second portion of this same investigation, namely, the production of internally oxidized powders in submicron flake form, is under way. These powders, as noted in earlier reports, will be reacted to utilize the surface copper oxide to convert more Al to Al<sub>2</sub>O<sub>3</sub>. The alloys will contain a higher percentage of aluminum oxide and a smaller amount of aluminum in solid solution. This will permit a comparison of the relative strengthening effects of oxide content versus solid solution strengthening when both strengthening mechanisms are used simultaneously.

TABLE II

Room Temperature Hardness of the Cu - 7.2 Al - 0.3 Al $_2$ 0 $_3$  Alloy After 1 Hour Annealing

Condition	Hardness, R <sub>B</sub>
As-extruded	99
400°C (792°F) anneal	95
600°C (1112°F) anneal	93
800°C (1472°F) anneal	93
900°C (1652°F) anneal	92
1000°C (1832°F) anneal	91

### C. Chromium Oxide Control in Oxidation Resistant-Oxide Dispersion Strengthened Alloys

In addition to the 10 micron diameter, type 316 stainless steel powders, which were available to this program and which were used to produce two stainless steel extrusions each containing 5 v/o thoria, additional compacts of two oxidation resistant alloys, prepared by halide transport, have been obtained and are being processed. It is anticipated that these compacts, produced by the halide process, will be unusually low in chromium oxide and should serve as reference materials for comparison with higher chromium oxide alloys.

As recounted in the previous progress report, two type 316 stainless steel compacts were prepared, each containing about 5 v/o thoria (introduced by mechanical blending), and were then subjected to a hydrogen reduction treatment to try to control the chromium oxide content. After hydrogen reduction, alloy 1 was found to contain 1.23 wt. % chromium oxide, and alloy 2 was found to contain 0.9 wt. % chromium oxide. These alloys were compacted in mild steel cans, sealed, heated to 2000°F, and extruded at a reduction of area ratio of 16:1.

Hardness tests at room temperature were made to get a preliminary measure of the stability of these alloys as a function of increasing annealing temperature for one hour. The results are shown in Table III.

Both alloys appear to show good stability to 2500°F, however, alloy 1 appears to be the harder and more stable of the two materials. It is not expected that the observed hardness difference between the two alloys is accountable by the small difference in chromium oxide content.

Laue back-reflection X-ray patterns indicate that the first signs of recrystallization appear in these alloys after one hour exposure at 2000°F. The effect of such recrystallization on mechanical properties has not yet been determined.

Tension specimens were evaluated at a strain rate of 0.03/minute, and the results are shown in Table IV. In line with the observed higher hardness of alloy 1, this alloy is significantly stronger than alloy 2. In spite of the significant difference in

TABLE III

Room Temperature Hardness of Type 316 Stainless - 5 Volume Percent  $^{2}$  Alloys as a Function of Annealing Temperature

Treatment °F	Alloy 1 (1.23 % Cr <sub>2</sub> 0 <sub>3</sub> )	Alloy 2 (0.9 % Cr <sub>2</sub> 0 <sub>3</sub> )		
As-extruded	100 R <sub>B</sub>	88 R <sub>B</sub>		
1600 - 1 hour	98 .	82		
1800 - 1 hour	94	80		
2000 - 1 hour	94	78		
2300 - 1 hour	92	78		
2500 - 1 hour	90	75		

Room Temperature Tension Properties of SS-ThO<sub>2</sub> Alloys

Alloy	Condition	0.2% Y.S.,psi	U.T.S., psi	Elongation, %
1	as-extruded	92,000	123,500	17.2
.1	ann. 1 hour-2000°F	87,000	128,000	20.7
2	as-extruded	43,600	82,400	21.7
2	ann. 1 hour-2000°F	41,800	81,000	25.3

strain values, the ductilities of the two alloys are quite similar. Annealing for one hour at 2000°F did not change significantly either the strength or the ductility of these two alloys, suggesting that the alloys are relatively stable, and might give excellent high temperature creep rupture properties. On the basis of these tests, creep rupture tests are planned for 1800 and 2000°F, preliminary to tests at still higher temperatures if the results look good at the two lower temperatures.

Metallographically, alloy 2 appears to have a significantly poorer dispersion of thoria, even though both materials were prepared in a similar fashion.

Both materials will be given additional cold work at room temperature, after which tension tests will again be made to establish changes in properties and changes in stability of the resultant structure.

Two additional oxidation resistant alloys have been prepared by the halide transport process, namely, an 80 Ni - 20 Cr alloy containing about 3 v/o thoria, and an 80 Fe - 20 Cr alloy containing about 3 v/o thoria. It is expected that both alloys will have very low chromium oxide content, due to the nature of the halide transport process. The thoria dispersion itself is an unknown quantity as yet in these alloys. Because of the success with internal oxidation of Fe-Be alloys (see above), powders were again prepared by internal oxidation, yielding a powder product of Fe - 3 v/o BeO; these powders will be chromized by the halide transport process to produce an 80 Fe - 20 Cr - 3 v/o BeO alloy to compare against the Fe-Cr-ThO2 alloy described above. Again, it is expected that the chromium oxide content of this third alloy will be low, thus providing a comparison between thoria and beryllia as dispersoids. It is expected that the BeO will be uniformly and very finely dispersed, due to the internal oxidation process, whereas the thoria dispersion is not known as pointed out above. The compacts of the first two compositions are now available and are being prepared for extrusion.

#### D. Modes of Deformation and Fracture in Oxide Dispersed Metal Systems

There remains the problem of selecting the most suitable compositions for the deformation and fracture studies. Fortunately, portions of the over-all program are beginning to produce interesting materials which can serve as part of the materials stock for this phase of the program, also. In the meantime, creep rupture tests have been completed of the copper-aluminum solid solution alloys to serve as a base line for comparison with the internally oxidized, and with the internally oxidized plus cold worked structures. Based on creep rupture studies at 500 - 650°C, an activation energy for creep of 46,500 calories/mole has been found. This is equivalent to the activation energy for self-diffusion of copper, namely, 47,000 calories/mole. There has been reported to be a change in the creep mechanism of pure Cu in the temperature interval 500 - 600°C; no change was observed with the Cu-Al solid solution alloys. Further tests are planned.

It was observed that aluminum in solid solution appears to retard grain boundary migration significantly, and leads to a decided roughening of the grain boundaries. Grain boundary migration occur's at 600°C and higher, and seems to take place on transverse boundaries to a greater extent. Typically, the extent of grain boundary migration was greater near triple points than elsewhere.

Measurement of slip band spacing give values of the order of 1 micron. These values are similar to those reported for copper-zinc solid solutions. Slip band - twin intersections occasionally happen, causing some secondary slip to occur at the twin boundary.

Fracture in these creep studies is extensively intercrystalline, and is associated with copious void formation, primarily on boundaries which are nearly transverse to the tension axis. Void size, at any given strain, increases with increasing temperature. Crack initiation occurs primarily at triple points where void coalesence appears to be favored.

Efforts to avoid excessive oxidation of the copper during the internal oxidation treatments of the Cu-Al strip samples are now meeting with greater success. At internal oxidation temperatures of 800°C a dispersoid of fine platelets has been observed, with thickness values of 150 to 300 Å and plate dimensions of 1300 to 1600 Å. Occasional regular, triangular, or hexagonal platelets are also observed. As expected, the hardness values of these internally oxidized materials, which are free of cold work, are low. The hardness values do not change, however, on exposure to temperatures through 800°C. Unfortunately, the room temperature ductility appears to be low, and as little as 6% reduction in thickness by cold rolling has caused some surface cracking. These samples are being given a hydrogen reduction treatment to try to eliminate all the oxygen which may exist either in the surface or at grain boundaries near the surface.

In the meantime, specimens have become available from the internally oxidized iron-BeO samples, on which extensive mechanical test data are now available. This material will be included in the deformation and fracture studies. In addition, the copper-Al<sub>2</sub>O<sub>3</sub> alloys prepared by the SAP technique (see above) will also provide additional test samples for this study. Replication techniques are working well, and we do not anticipate any problems with the electron microscopic studies of the resultant structures.

#### Current and Future Activities

As indicated in the previous progress report, we are now entering a period of extensive mechanical testing, stability studies, and structure analysis, all of which should lead to a better understanding of the mechanisms of strengthening in these oxide dispersion strengthened systems.

The extreme precautions which were taken in the preparation and processing of these alloys appear to have paid off. In general, the structures are clean and apparently free of non-refractory oxides. As a result, it is expected that the stability of the alloys will show marked improvement over earlier alloys.

#### II. THE SPLAT COOLING PROCESS FOR ALLOY DEVELOPMENT

#### A. Development of Continuously Operating Atomization Units for Larger Scale Powder Production

The low melting Sn-8% Zn alloy has been utilized for continuing atomization studies to achieve refinements in the two major atomization units currently in use. The goal of these experimental trials was to establish or optimize the gun geometry in terms of the particle size and shape of the atomized metal droplets. Thus far, two of the guns have been tried, namely, one with an 0.035 inch orifice, and the other with an 0.050 inch orifice. The gas pressure utilized in both cases was the same, namely, 125 psi.

Collection of the solidified metal particles was done either against a metal substrate or from a water bath. Some of the results are summarized in Table V. The particle distribution of the collected powders is shown in Figure 1. Basically, two types of powders are present in the materials collected thus far, namely, a coarse, flake-like particle and a finer equi-axed particle. The shapes clearly suggest that the flake-like material is formed by impaction against a substrate while still in the molten form, whereas the finer, more spherical particles are typical of materials which solidified in flight.

Measurements of the grain size in these materials shows variations from about 10 - 70 microns, the 10 micron size being associated with the coarse, flattened platelets, and the 70 micron size being associated with the coarser equi-axed structures. This would indicate that the cooling rates for these two sets of conditions are quite different, and relatively slow (possibly 10<sup>4</sup>°C/second). Grain sizes near 10 microns are of interest to this program, and it is clear that variations in the atomization technique should be capable of producing grain sizes from less than about 1 to 10 microns. The aim is to produce pound lots of material which can then be compacted and prepared for testing. As has been indicated from more carefully controlled splat cooling studies (unfortunately involving 20 milligrams of material per shot), solidification of liquid metal droplets in flight must be avoided if very fine grain size is desired. The geometries involved in the present atomization studies suggest that this will not be difficult to overcome.

## TABLE V

# Characteristics of Atomized Powders

Atomization Run #	Orifice of Gun	Powder Characteristics Comments
1	.050 inch	atomized into water. Equi-axed. 10 micron grain size
2	.050 inch	atomized on metal substrate, 2 feet from gun. Flake. 10 micron grain size.
3	.050 inch	atomized on metal substrate, 5 feet from gun. Flake and equi-axed. Mixed grain size from 20 to 70 microns.
4	.035 inch	atomized on metal substrate, 5 feet from gun. Fine equi-axed, with few flakes. Mostly 70 microns.

#### B. Alloy Studies

- 1. <u>Decomposition of super-saturated aluminum-silicon alloys</u> Detailed electron microscopic studies of the precipitation of silicon particles from highly supersaturated aluminum-silicon solid solutions has resulted in a technical report which will be submitted for publication. This phase of the program is completed; the program has demonstrated one of the potential uses of rapidly quenched alloy powders, namely, the production of dispersion strengthened alloys (silicon in Al). This is possible in an alloy system which is normally plagued by very coarse, primary, silicon particles, the net result of which is poor strength and poor ductility.
- 2. <u>Dendrite spacing in splat-cooled aluminum alloys</u> The study of the dendrite cell size of aluminum alloys as a function of the cooling rate, involving cooling rates from 10<sup>Q</sup>C/sec (ingot solidification) to 10<sup>9</sup>°C/sec (splat cooling), has been completed and a technical report has been prepared for publication. A straight-line relationship exists between the log of the dendrite size and the log of the cooling rate over a distance of about 10 decades of cooling rate. This observed relationship is particularly important since it establishes for the first time the possibility to utilize microstructural features as a measure of the cooling rate. Direct measurements of cooling rate during splat-cooling, confirmed by calculations of the cooling rate from heat flow considerations, now find strong support in the observed relationship between dendrite size and cooling rate.
- 3. The determination of solid solubility in Al-Cu, Al-Ni, and other aluminum alloy systems Lattice parameter measurements of aluminum solid solutions in these systems, achieved through splat cooling, are being continued. The results essentially confirm and extend those described in the previous status report; this phase of the work will be concluded in the near future. The main effort at the moment has been shifted to electron microscopic studies of the precipitation processes which take place in these alloys on decomposition of the supersaturated solid solutions. These studies should establish the kinetics of the precipitation process, and will establish the potential among these alloys for production of high strength alloys through dispersion strengthening.

4. Transmission electron microscopy of Al, Al-Cu, Al-Ni, and Al-Pd splat-cooled materials - Because of the known increase in solubility of copper in aluminum under conditions of rapid quenching by splat-cooling, namely up to 11 percent copper, there is interest in studying these materials as potential alloys wherein large increases in strength can be achieved by doubling the amount of precipitate, and by bringing about the various precipitation steps at lower temperatures, resulting in finer precipitated particles. For this purpose, transmission electron microscopy will be extensively utilized. Attention is being directed to the determination of particle size, shape, and growth of the precipitate particles as a function of time and temperature. Studies thus far have been concentrated on the aluminum-copper and aluminum-palladium systems. Single-phase regions have been observed in the aluminum - 4% palladium and aluminum - 2% palladium alloys, indicating extended solid solubility ranges through rapid quenching. Electron transmission micrographs of these regions revealed only bend contours and occasional dislocations.

In quenched, solid aluminum and aluminum-copper alloys, the dislocation configurations are a function of the solution treatment temperatures and solute content. Dislocation loops form in quenched aluminum and aluminum-copper alloys at solute concentrations up to 2% copper. Helical dislocations are seen in the aluminum-copper alloys containing a higher solute concentration than this. Measurements of dislocation loop diameters and of the radius and pitch of the helices allow calculations of the vacancy concentration on quenching. Because splat-cooled materials can contain vastly higher numbers of vacancies, their behavior should differ from that which is seen in conventionally prepared materials. It has been observed that pure aluminum splats do contain significantly larger numbers of dislocations in a tangled configuration than do conventionally quenched solid materials. Al - 5% copper splats also appear to have an altered dislocation configuration.

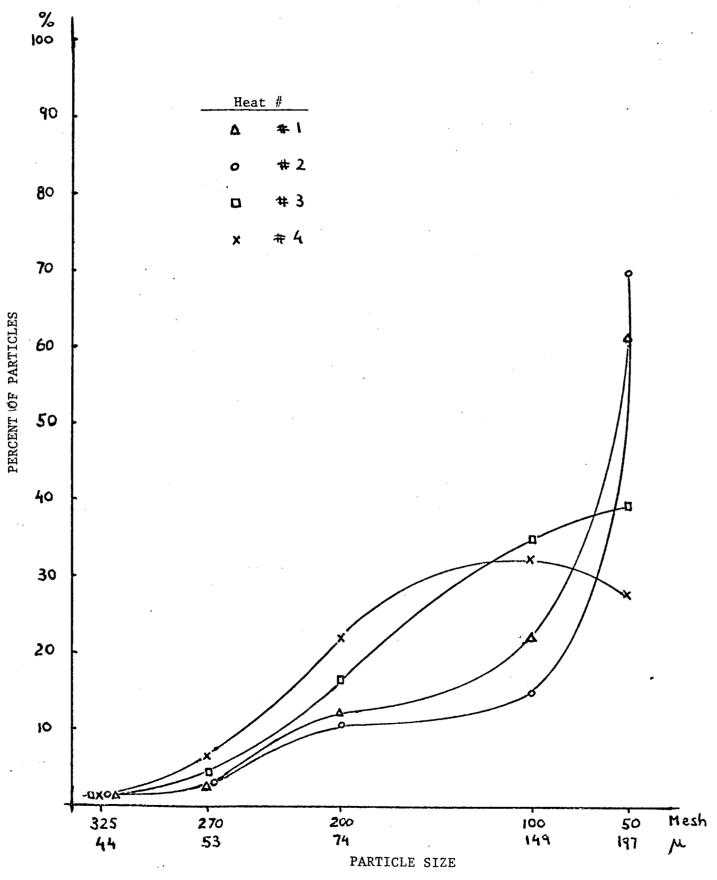
5. <u>Copper-base alloy systems</u> - The work in the Cu-Be and Cu-Si systems previously described has been evaluated and a publication is being prepared.

6. Study of Atmospheric corrosion of Al and Al alloy splat-cooled materials - In the course of the present work on aluminum alloy splats, it was found that unusually rapid corrosion occurred at room temperature, far in excess of that normally observed with conventionally prepared coarser aluminum powders. It was found that atmospheric  $SO_2$ concentrations greater than 0.15 ppm severely corroded splat foils of pure Al and Al alloyed with Ni, Mn, Fe, Co, and Pd. In the Boston area, concentrations of this magnitude occur frequently between the months of November and May. X-ray diffraction patterns of splat foils made during this period of time showed numerous unidentified phases. In many cases, however, alpha  ${\rm Al}_2{\rm S}_3$  and beta  ${\rm Al}_2{\rm S}_3$  phases could be identified from X-ray diffraction patterns. A mechanism for the formation of these compounds has been proposed. Other likely corrosion products are sulfates, sulfites, and thiosulfates. Comparative corrosion rate studies of splat-cooled alloys were made by exposing samples both in the Boston area and in a suburban area of lower New Hampshire, where the atmosphere is relatively free of SO2. The changes in these two areas have been recorded. Attempts have also been made to simulate the corrosion reactions in the laboratory using commercially prepared SO, gas. The specimens were unaffected by the presence of dry SO, alone.

There is concern over these reaction rates because other splat cooled alloys, based on Cu, Ni, Co, or Fe, and other materials, might also be strongly affected by the atmosphere conditions which prevail.

#### Future Work

The atomization units are essentially ready to start producing significant quantities of material. On this basis, deliberations are currently under way on the selection of the most suitable alloy system for the production of bar stock and measurement of properties. Of most immediate interest are alloys in which grain size can be controlled in the near micron or submicron size range, and where solubilities can be changed drastically, as with aluminum-silicon and aluminum-copper, thereby allowing the production of dispersion strengthened or highly modified aged alloys.



Distribution of splat-cooled powders in Figure 1. preliminary trials with Pb-Zn alloy.