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Investigation of Foamed Metals for Application on Space Capsules

GEORGE C. MARSHALL SPACE FLIGHT CENTER NATIONAL AERONAUTICS AND SPACE ADMINISTRATION HUNTSVILLE, ALABAMA

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Investigation of Foamed Metals for Application on Space Capsules

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ABSTRACT

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Investigations of methods of preparing foamed metals in the foamed, sintered state are summarized. Metals included are aluminum, titanium, nickel, 316 stainless steel, H-11 tool steel, and molybdenum. Thus far, it has not been possible to prepare a foamed, sintered aluminum since no practical method, mechanical or chemical, has been found to eliminate the oxide film around the metal particles. Titanium, although difficult to protect against oxidation, can be reduced and sintered but elimination of oxide contamination between the grain boundaries is hard to prevent and the product cannot be considered ductile.

No unusual difficulties are encountered in preparing foams of nickel, 316 stainless steel, H-11 tool steel, and molybdenum but ductility has not been achieved at densities less than 15 percent of theoretical. Thermal and mechanical properties for the nickel and stainless steel foams are given. Machining of the foamed metals and methods of brazing them to facing sheets of similar and dissimilar metals are described. Preparation of variable density beams in which the core material is of a low density and the encasing metal foam is of a relatively high density can be carried out by one of several methods.

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I. INTRODUCTION

This investigation was initiated to determine the potential usefulness of foamed metals in a number of aerospace applications. Among these are vibration damping systems, lightweight structures where honeycomb may be replaced by foamed metal, and in various components of space capsules. The objectives of the development have been to produce the foamed metals in a ductile state with predictable properties and to develop joining techniques for the materials.

The program reported herein has explored the possibilities of making a number of foamed metals in different percentages of theoretical density and of predicting the densities that would result when experimental foaming conditions were standardized. Among the properties of the products that were to be determined at -320°F, ambient temperature, and at four elevated temperatures, including the maximum useful temperature, were the following:

- 1. Tensile, compressive, and shear strengths
- 2. Vibration damping capacity
- 3. Coefficient of thermal expansion
- 4. Thermal conductivity
- 5. Stability at elevated temperatures

The metals chosen for investigation were aluminum, titanium, 316 stainless steel, H-11 tool steel, nickel, and molybdenum.

All of these except for aluminum were prepared by foaming and sintering. Titanium presents certain difficulties because of oxidation which occurs during presintering and it appears likely that an alternative to the customary method will also be required for this metal.

II. SUMMARY

A foamed metal may be produced by suspending a fine metal powder in a liquid containing setting and foaming agents. The slurry is foamed under conditions such that bubbles of uniform size are distributed uniformly through the suspension. After drying and sintering, a product having the desired porosity and density is obtained. The volatile contaminants are expelled during sintering.

The work described herein has involved a detailed study of the

fabrication and sintering of aluminum, titanium, nickel, molybdenum, H-11 tool steel, and 316 stainless steel.

Foamed aluminum is not yet producible since no practical mechanical, chemical, or thermal means has been found to eliminate the sinter retarding oxide film on the metal particles. Pure aluminum and aluminum alloys of spherulized powders, made by atomization of melts in inert atmospheres, have been tried. Various additives of hydrochloric acid, aluminum chloride, and fluorides were tried in order to break down the oxide film and permit metal-to-metal sintering but these resulted in weak and collapsing cast foams.

The application of powder metallurgy methods in which pressed shapes of aluminum powders and salts or naphthalene was attempted without success.

Titanium foam has been prepared by the standard foaming procedure but the sintered products were weak and brittle because of a thin oxide film on the metal particles. Samples were made in the 30-32% of theoretical density range by presintering for 3 hours at 2300°F and final sintering at 2800°F for 1 hour. Titanium hydride was then added to break down the oxide film at sintering temperatures and permit metal-to-metal sintering. The sintered products again were brittle and weak and an oxide phase appeared to be present.

Nickel foam can be produced by using the standard foaming procedure and proper sintering techniques. Samples of two densities (18 and 27% of theoretical) have been made by: (1) a dewaxing step, (2) a presintering for 3 hours at 2300°F in vacuum, and (3) a final sintering for 20 hours at 2550°F in a vacuum. Tensile, compression, shear, thermal expansion and thermal conductivity tests have been made and the results are presented herein. Samples have been made in the 12-15% density range but all were brittle and weak.

Stainless steel 316 foam has been prepared by the same procedures used for nickel. The only difference between the procedure for nickel and stainless steel is the final sintering for 40 hours at 2550°F in a vacuum. Samples have been made and the same tests as for nickel have been performed and are presented. Samples prepared in the 12-15% density range resulted in brittle and weak foams. Green foams of H-11 tool steel have been fabricated but not all have been sintered. A series of different foam densities has been produced.



Figure 1. Foamed 1100 Series Aluminum in Green State 30X

- 2. Ammonium fluoroborate, NH_4BF_4
- 3. Ammonium bifluoride, NH_4HF_2
- 4. Di-n-butyl ammonium tetrafluoroborate, $(n-C_4H_9)$
- 5. Tetramethylammonium tetrafluoroborate, (CH₃)₄ NBF₄
- 6. Hydrazinium difluoride, N₂H₄.2HF
- 7. Hydrazinium tetrafluoroborate, N₂H₅. BF₆

All of these additives produced too acid a reaction in the mixture and partial collapse of the foamed structure resulted.

Other additives tried included aluminum chloride which was used in conjunction with a non-aqueous system where the suspending medium was absolute alcohol or isopropyl alcohol and the foaming agent was saponin. An alternative also tried was the addition of a small amount of hydrochloric acid to the alcohol suspension. The foams obtained by this method were weak and unstable and the degree of sintering obtained at 1200°F was slight.

Because of the poor results with pure aluminum, attention was turned to the 7178 aluminum alloy (6.8% Zn, 2.7% Mg, 2.0% Cu, 0.3% Cr, Al, balance). This is a wrought alloy used in applications where high strength and good oxidation resistance are required. The 7178 alloy was likewise obtained in -100 mesh and -325 mesh spherulized form. It was considered that this alloy might be more suitable then pure aluminum in preparing a foamed, sintered structure for the following reasons:

- 1. The alumina layer is probably less adherent on the grains because of the presence of the alloying metals.
- The relatively high concentration of zinc in the alloy might be expected to cause mechanical breakage of the oxide film since the vapor pressure of the zinc would be in excess of 400 microns at temperatures over 1100°F.

3. The alloy has a liquidus temperature of 1165°F and a solidus temperature of 890°F. By holding the foamed mass at a temperature not far removed from the liquidus the structure might retain sufficient rigidity to maintain its shape.

Accordingly, a number of formulations of the 7178 alloy powder were foamed, dried, and sintered in vacuum at temperatures up to 1150°F. In several cases, moderately strong sintered products were obtained ranging in density from 17 to 20 percent of theoretical. However, the products were brittle, somewhat powdery, and therefore not suitable. A photomicrograph of a typical sintered alloy is shown in Figures 2 and 3.

Strengthening of the porous aluminum samples was attempted by impregnating them with various corrosive materials which were expected to remove the oxide film at temperatures below the liquidus and convert it to a compound leachable with water, but none of these were successful in obtaining a strong sinter.

Since foaming of the aluminum alloy powders was unsuccessful, an alternative procedure was tried. This involved the pressing of an intimate mixture of graded aluminum or aluminum alloy powder with a nonmetallic material at pressures believed sufficient to cause mechanical breakage of the oxide film on the grains, and thereby to secure metal-to-metal contact of the aluminum grains. The initial mixtures consisted of various percentages by weight of aluminum powder with the balance made up of an inert material which could be volatilized by heating in vacuum. Finely divided sodium chloride was tried first and bars containing various percentages of aluminum powder and salt, pressed at 20,000 psi, were heat treated in vacuum at temperatures in the range of 1150 to 1200°F for 1 hour at a pressure of 0.1 micron. The sublimation of the sodium chloride was incomplete in this temperature interval.

Accordingly, mixtures of aluminum powder and naphthalene, which sublimes at a very low temperature, were prepared and pressed at 20,000 and 40,000 psi. These were heat treated in vacuum at various temperatures up to 1200°F and showed development of a porous, sintered structure. The pores were irregular and bonds between the grains were not well developed probably because of the existence of unbroken oxide layers which prevent sintering.





Figure 6. Sintered Molybdenum, 155X

A suitable method of obtaining a sintered aluminum foam has thus not yet been found.

B. <u>Nickel</u>

The nickel powder was obtained in a form that had been spherulized in an inert atmosphere. In most cases mixtures of -100 mesh and -325 mesh fractions were used. Preparation of the green foam followed the customary procedure and sintering of the material was performed by various methods until the most favorable sequence was found. Two inch foamed cubes were used to determine the initial formulations that would be required to produce a given density during presintering and sintering. Then larger shapes $(2 \times 4-1/2 \times 6 \text{ inches})$ were made.

1. Initial Procedures

The early work was done with both the presintering and sintering carried out in one furnace since it appeared advantageous to avoid moving of the parts after presintering. Cold wall vacuum furnaces were used throughout this phase.

An organic binder is present in the metal foam and this must be removed in a "dewaxing" step before high temperature sintering can be performed. If the binder is volatilized under usual vacuum conditions, part of it will condense on the cold walls of the furnace and the remainder will be drawn into the mechanical and oil diffusion pumps and thus ultimately prevent their operation.

The first expedient tried, therefore, was to seal off the organic vapors as effectively as possible from the furnace itself. This was accomplished as shown diagrammatically in Figure 4.

The dried green foam pieces are placed in a mild steel box, which is loaded into the furnace. The furnace is then sealed and nitrogen admitted to a pressure of about 785 Torr. Heating is started and the exit gas line valve is opened permitting the flow of nitrogen to remove the decomposition products from the furnace. Most of the organics have been eliminated within approximately one hour, when the furnace temperature reaches 500°F.

A minor part of the organics did deposit on the cold walls





of the furnace. The amount of this deposit increases with the number of times this "dewaxing" process is carried out and the furnace shell must be periodically cleaned.

When the foamed piece reached a temperature of 500°F, the nitrogen inlet and exit valves were closed and the furnace pumped down to start the final sintering.

The pressure attained was dependent on whether the furnace had been used previously in the "dewaxing" step. With an uncontaminated furnace the pressures were normally in the range of 0.1 micron.

During the final sintering, the furnace was heated to 2450 - 2550°F under vacuum. Hydrogen was then bled into the furnace for 3-5 minutes to a final partial pressure of 5 Torr. After this period the furnace was pumped down, and the cycle repeated 10 to 20 times at 2450 -2500°F. On completion of the cycling, the hydrogen was pumped out and the furnace allowed to cool overnight under vacuum. When more rapid cooling was desired, the furnace was backfilled with argon or helium.

The following tests were made on the sintered products:

- a. The foamed pieces were examined for color, completeness of sintering, variations in texture, and for cracking, warping, and other defects.
- b. The density was determined by measuring the volume and weight of an accurately cut sample of the material. Values were expressed as the percentage of the theoretical density of the wrought material.
- c. The indentation hardness, in terms of the Brinell Hardness Number (Bhn), was measured by forcing a hardened steel ball of 10 mm diameter into the surface of the foam. The loads used were 10 kg, 25 kg, and 50 kg, depending on the hardness of the material. Measurements of the impressions were made to within 0.25 mm. Brinell Hardness Numbers were then calculated as the ratio of the load in

kilograms to the impressed area in square millimeters. Bhn values ranging from 0.1 to 3.0 were obtained, depending to some extent on the density reached by the sintered foam.

2. Final Procedure

Since carrying out the entire operation of "Dewaxing" and sintering in one furnace had not proven satisfactory, owing to the volatiles condensing on the furnace insulation and cold walls, a twostep procedure was adopted. This involved using a Kanthal-resistor furnace for expelling the volatiles and doing initial sintering.

The Kanthal furnace has a working space of 14 inches in diameter and 17 inches deep and is heated by helical elements recessed in foamed refractory brick. The maximum operating temperature of the furnace is 2300°F and the attainable vacuum is 30 microns since a mechanical pump with no diffusion pump is used. Possibly because of a catalytic effect of the resistance element, which operates with an on-off control, the organic volatiles appeared to decompose in the furnace and form lower molecular weight products. These were partly caught in a large cold trap between the pump and the furnace and partly in the oil of the mechanical pump, with the remainder discharged to the atmosphere. Replacement of the oil in the mechanical pump was required about once a week.

For final sintering of the small specimens, a laboratory-type furnace having a working space of $5 \times 6 \times 12$ inches was employed. For larger specimens which, in the green state, were normally in the form of slabs of $2 \times 4-1/2 \times 6$ inches, a larger furnace having a working space of $8 \times 21 \times 21$ inches was used. Since the volatiles had been expelled during the presintering treatment both of these furnaces could be maintained uncontaminated and gave pressures in the order of 0.1 micron Hg.

Having once determined the formulations required to produce a given density by presintering at 2300°F and final sintering at 2450-2550°F, sufficient quantities of slabs of foamed nickel in two different densities could be prepared for machining test specimens. In each case, the specimens were presintered for 3 hours at 2300°F in vacuum and then subjected to a final sintering at 2550°F in vacuum for 20 hours. The density varied from 18.0 to 19.3% of theoretical for twelve slabs in the lower range of densities and 27.0 to 27.8% of theoretical for ten slabs in the higher range of densities. The Brinell Hardness Numbers of the pieces were 1.6 for the low-density materials and 4.6 for the high-density materials.

It was possible to prepare foamed nickel of 12-15% density but the products could not be considered ductile, whereas those in the range of 18% and higher were uniformly ductile.

C. 316 Stainless Steel

The procedure used in making stainless steel foams was the same as that in preparing nickel. Here again the initial heat treatment involved cycles of vacuum and hydrogen but the final process consisted of only vacuum, since it was found that bright, foamed products could be obtained by a sufficiently prolonged vacuum heat treatment.

Figure 5 shows the increase in density and hardness values as the sintering time is increased from 10 to 50 hours at 2500° F. An approximate plateau in both properties is indicated at a sintering time of 40 hours. The presintering time used in each case was 3 hours at a temperature of 2300° F.

In ten slabs of 316 stainless steel made from identical formulations and sintered for 40 hours at 2500°F, the density ranged from 17.1 to 18.1% of theoretical. A series of tests was made to establish the conditions necessary for producing foamed 316 stainless steel having 27% of theoretical density, using the same presintering and sintering conditions.

Experiments are under way at higher sintering temperatures in order to reduce the excessively long sintering time at 2500°F.

As in the case of nickel, no success has been attained in the production of a ductile 316 stainless steel having a density range of 10-15% of theoretical.

D. H-11 Tool Steel

The tool steel powder was obtained in atomized form in -100 and -325 mesh fractions. Foaming was carried out according to procedures established for nickel and 316 stainless steel and comparable densities were obtained. The tool steel foams, in the green state, must be protected from the atmosphere after leaving the drying cabinet until they are presintered. Moisture absorption results in oxidation and collapse of the structure or





development of cracks during presintering. The presintered material is also stored in a drying cabinet to protect it from the atmosphere until final sintering.

High density as well as low density materials have been fabricated but only the high density materials have been sintered to date.

E. Molybdenum

Various molybdenum powders were investigated to determine those most suitable for preparing specimens free of flaws. Presintering has been carried out in the usual manner at 2300° F. Although the specimens were fragile at this stage, they were strong enough to be transferred to a 4000° F furnace for final sintering. The furnaces used were a horizontal unit with working space of 5 x 6 x 12 inches and a vertical unit with working space of 5-inch diameter and 11 inches deep both capable of 4000° F operation.

It was found to be more difficult with molybdenum than with the other metals tried thus far, to prevent the formation of cracks and other flaws. These are especially apt to occur during the high temperature sintering because of the long interval that exists between the volatilization of the temporary cementing agents and the temperatures at which sintering together of the molybdenum particles begins to occur. Proper grain sizing of the molybdenum powders with increased percentages of fines to promote solid state reaction at as low a temperature as possible, together with improved fixturing techniques have eliminated most of these difficulties. A method of fixturing, that has been especially effective, is to support the presintered molybdenum piece on a foamed zirconia base which has been cut with lateral grooves at close intervals. With this type of support, development of cracks caused by a restraining action of the fixture is less likely to occur.

A photomicrograph of a section of foamed molybdenum is shown in Figure 6.

Sintering at 3800 to 4000°F in vacuum is indicated to be necessary for the molybdenum foams in order to obtain reasonably strong products.

F. Titanium

Titanium powder was procured in grades of -100 and -325 mesh and, as an additional source material, titanium hydride was also used. The purpose of the hydride was to furnish a reducing atmosphere



Figure 6. Sintered Molybdenum, 155X

and thus inhibit oxidation during the furnace treatment.

1. Standard Foaming Procedure

Mixtures of various size gradations of titanium powder and of titanium hydride powder with different proportions of titanium metal were tried in attempts to secure a well-sintered foamed metal, Presintering at 2300°F followed the usual procedure with final sintering at 2600 to 3000°F for periods of 2-3 hours.

Photomicrographs of a foamed titanium section at 32 percent of theoretical density are shown in Figures 7 and 8. It can be noted at the higher magnification that some interstitial material appears at the grain boundaries which will lead to a brittle structure. It was hoped that incorporation of titanium hydride in the mixtures would prevent the formation of contaminants such as lower oxides of titanium but apparently the reducing conditions during presintering are not sufficient to prevent minor oxidation and to make subsequent reduction during vacuum sintering somewhat difficult. However, sufficient indications have been obtained that foamed titanium can be made by the sintering technique although the densities obtained thus far (30-32% of theoretical) have been beyond the limits desired.

IV. MACHINING OF FOAMED METALS

As described in the foregoing section, the green foamed mixtures are initially cast into blanks which undergo large dimensional changes during sintering. In a subsequent portion of this investigation, the possibilities of casting and sintering parts to approximate finished sizes will be investigated.

In the forming operation the surface of the foamed metal often does not exhibit a pore structure that is characteristic of the mass of the material.

Experimentation showed that the foamed metals have a machinability rating of "very easy". Foamed nickel has a rating better than that of C1117, or exceeding 91%. The principal problem in machining, is to prevent smearing the delicate cell walls during the sawing, parting, and other machining operations. Cut-off can be accomplished by an ordinary knife. For example, 1/8" plates can be parted by repeated passes with a knife blade. The resulting cut face appears to be almost completely sealed. Whereas the foamed metal has a mat appearance when it is machined without closing the pores, the parting with a knife blade gives a shiny surface and



Figure 7. Presintered Titanium, 110X



Figure 8. Presintered Titanium, 875X

almost completely closed pores. Equipment for machining foamed metals, which will leave the cell walls in place without distortion, must have tools which are very sharp and the operation must be carried out without creating heat. Sawing can be performed if it is done slowly and with a sharp saw. When a coolant is used, the sawing operation can be at a faster rate.

Samples for physical testing were prepared largely on a band saw. In the case of some of the thermal conductivity specimens, the tolerances were such that circular discs were finished to size using an engine lathe.

Experimentation proved that an 8-pitch band saw blade gave the most satisfactory machining for the preparation of rectangular specimens. The band saw used was a DoAll model 1612-3 Contourmatic Band Saw and Filing Machine. This machine is equipped with a servo feed control and a powered table. The work was clamped on the table in such a way that the clamping did not permit the closing of the cut following the passage of the band saw blade through the material. If the material pinched on the blade, smearing of the cell walls resulted. It was found that very light sawing pressures were required and here the use of a servo feed control was very helpful. The pressure used to move the work into the saw blade is controllable on the DoAll machine and the setting used was 1/2, which represented a pressure on the saw cut of 5 psi. A blade speed of 95 ft per minute was found to give the best cuts. Cuts were made both dry and with coolant but it was felt that in most cases a coolant would be undesirable because of the hazard of contamination. When a coolant was used, one that appeared satisfactory was DoAll's H. D. 660 synthetic cutting fluid.

The general procedure for cutting test specimens was to make cuts on the block to remove the surface which contained sealed pores. Since the smallest dimensions of many of the test specimens was 1/2", certain blocks were sliced into 1/2" plates. From these plates and others of greater thickness, the circular discs, the rods, and the rectangular test specimens were sawed.

The pores at the surface of the foamed metal may be closed by touching the piece lightly against a grinding wheel. This smears the surface of the nickel, stainless steel, and H-11 tool steel very thoroughly, but sealing of the pores is incomplete with molybdenum and still less so with titanium.

V. BRAZING

Sandwich structures were fabricated by brazing similar and dissimilar metals with various brazing alloys. Development of the sandwich materials was based on the following concepts:

Joining Materials

Three sheet materials were selected to correspond with the foamed metals and to provide a range of information regarding bonding similar and dissimilar metals.

The brazing alloys were chosen by the following criteria:

- 1. The material must provide well-diffused, highstrength, heat-resistant, and possibly oxidationresistant joints.
- 2. The braze should be applicable to standard brazing techniques which employ vacuum or an inert gas atmosphere processing of the materials.
- 3. Selections were made from a variety of ready mixed paste materials, powder and vehicle mixtures, and sheet materials cut into preformed shapes.

Table I lists various braze materials which were investigated.

A. Procedures

Various vacuum furnaces were utilized during the development of the sandwich materials. The variables which were investigated for producing well-bonded brazed joints included:

1. Temperature

Recommended brazing temperatures were furnished by the sources of the various brazing materials. The recommended brazing temperatures as well as temperature variations were investigated.

TABLE I. BRAZING ALLOYS

Braze Material	Composition	Recommended Brazing
		Temperature °F.
1		
Nicrobraz 30 ¹	Cr 19.00	2175
	Si 10.00	
	C 0.15 (max)	1
	Ni Balance	
Nicrobraz 125 ¹	Cr 13.45	2150
	B 3.50	
	Si 4.50	
	Fe 4.50	
	C 0.80	
	Ni Balance	
Nicrobraz 130 ¹	B 3.00	2150
	Si 4.50	
	C 0.15 (max)	
	Ni Balance	
Nicrobraz 170 ¹	Cr 11.50	2150
	B 2.50	
	Si 3,25	
	Fe 3.75	
	W 16.00	
	C 0.55	
	Ni Balance	
AMI 100 ²	Cr 19.00	2175
	Si 10.00	
	Ni Balance	
AMI ²	Cr 19.00	2150
	Si 10.00	
	Mn 10.00	
	Ni Balance	
Copper ³	Oxygen Free Copper	2050

Note:

- 1 Powder + vehicle hand mixed
- 2 Paste
- 3 Sheet

2. Time

Various soaking times at the appropriate brazing temperatures were investigated to determine flow characteristics of the braze materials which produced optimum brazed joints.

3. Atmospheres

The vacuum furnaces have capabilities of maintaining maximum vacuum conditions of 0.1 micron throughout a cycle, or of maintaining a controlled atmosphere at various partial pressures of selected inert gases. Partial pressures are normally utilized for two primary reasons:

- a. To reduce vaporization of braze materials,¹ parent materials, or both. The amount of vaporization of an element depends on the atmosphere and temperature conditions. The temperature can be controlled by proper instrumentation and the atmosphere is regulated by the partial pressure system. Control of both variables will minimize or largely eliminate vaporization.
- b. To provide oxidation protection. Oxidation of a material is a time-temperature-atmosphere relationship. By introducing an inert gas into the vacuum furnace during the brazing cycle, the mean-free path of any oxygen which may be present in the vessel is reduced.

4. Fixturing

Fixturing was not stressed during this stage of development because of the relatively simple shapes which were investigated, but proper fixturing techniques will be an important factor in the brazing of the foamed metals. Generally the brazing procedure consisted of taking a piece of refractory sheet metal and placing some braze material on it. The foamed refractory metal was then pressed onto the brazing material and to the sheet metal. This setup was then placed in a vacuum furnace and the furnace evacuated. When the furnace was at a pressure of less than 100 microns Hg, it was rapidly heated to a preheat temperature of approximately 1800°F. Some outgassing accompanies the heat-up. The work pieces were held at the preheat temperature for 10-15 minutes. then rapidly heated to the brazing temperature of usually 2150-2175°F, and held for 5-10 minutes. Outgassing also accompanied this heat-up. After 5-10 minutes at the brazing temperature, the furnace power was cut, and the work quenched with an inert gas. After cooling, the specimens were removed and inspected.

Figure 9 illustrates a typical brazing cycle which includes the variables of time, temperature, and pressure.

B. Results

1. Nicrobraz alloys gave well diffused and high strength joints with the solid metals. However, the brazing of foamed metals presents certain difficulties.

The use of the least possible amount of braze is recommended to produce a strong bond, because undercutting by erosion or reaction of the parent metal may occur at brazing temperatures. However, the quantity of braze necessary for joining foamed materials was found to be far greater than that necessary for joining solid materials, because of the capillary action of the pores in the foamed materials. The braze flowed up into the pores and voids of the foamed material, thus depleting the metal interface of the brazing alloy. Therefore, the problem is to determine the correct amount of brazing alloy to use for a well bonded joint and yet not erode the parent metal.

The erosion which has occurred in various samples has been found to be a function of the quantity of braze applied and the density of the foamed metal. In some cases, where a minimum amount of braze material was utilized, the erosion of the parent metal was not excessive but the bond between the foamed metals and the sheet metals was poor.

It has been found necessary to smear the porous surface of the foamed metal. Smearing the surface minimizes erosion and



Figure 9 Brazing Curve of Temperature and Pressure vs Time

capillary action. However, lower density foamed materials (15-18% of theoretical) retain a large number of open pores in the surface even after smearing. The higher density (27% of theoretical) foamed materials smeared much more readily, thus reducing the capillary action and erosion.

To date, the Nicrobraz materials have eroded 316 stainless steel and nickel foams but readily bonded foamed molybdenum to similar and dissimilar sheet metals. These samples are to be tested for bond strength.

2. Copper Braze

Copper braze was selected for investigation primarily for lower temperature brazing applications. Sheet 0.005 - 0.007 inch thick was used to determine whether or not this thickness would provide an adequate quantity of material for proper brazing, and to determine whether or not this method was applicable for brazing of similar and dissimilar metals.

A relationship between the quantity of brazing material and the porosity of the foamed material was observed. In some cases where low density foamed metals were brazed to solid sheet materials, the porosity of the foamed metal allowed the braze alloy to flow into the voids thus depleting the brazed joint of braze material. Figures 10 and 11 illustrate a well bonded copper brazed joint between a sample of 27% of theoretical density foamed nickel and a section of sheet molybdenum. Figure 10 does not clearly illustrate the diffusion surfaces although the braze material is obvious to the unaided eye. Figure 11 depicts the same area after the mounted brazed sample had been etched with a modified Carapella's Reagent.

Figure 11 clearly illustrates the flow of the braze into the pore structure of the foamed nickel. In this instance, two sheets of .005 - .007 inch thick precut copper sheets were utilized to braze the foamed material.

A destructive test was conducted on a copper brazed sample to determine whether or not the bond between the foamed and sheet metal was adequate. A sample of foamed nickel (27% of theoretical density) was copper brazed to a sheet of nickel using a single precut sheet of 0.005 - 0.007 inch thick copper braze. The specimen was securely clamped so as to restrain the sheet material from



Figure 10. Photomicrograph of Porous Nickel, Copper Brazed to Molybdenum Sheet, 110X



Figure 11. Porous Nickel Foam Copper Brazed to Molybdenum Sheet, 110X. Etched Sample Shown in Figure 10.
moving during the test. A bending moment was then applied to the foamed nickel at a point farthest from the brazed joint. The test specimen fractured in the metal foam just above the brazed joint. The foamed material which remained attached to the sheet appeared uniform and identical to the parent metal which broke away. Thus the fracture occured in the foamed metal and not in the joint between the foam and sheet metal.

VI. VARIABLE DENSITY BEAM

It was originally planned in this investigation that aluminum be used in the development of techniques for making metal foams in which the lowest density was present at the central core of a structure and the highest density was at the exterior surfaces.

Since the preparation of sintered foamed aluminum has so far proven to be impractical by present techniques, nickel was chosen as a substitute.

The original procedure was simply to cast successive layers of lower density foams in a rectangular mold and then to increase the density of the foam in steps after the minimum density had been cast. This procedure was not successful because of interdiffusion of the foam layers of different densities.

A second method involved making the low density foam first, drying and forming it, and then imbedding the section in a high density foam. These sections were presintered and given a final heat treatment at 2500°F. Two problems were encountered in this method of fabrication as follows:

- 1. Maintaining the position of the low-density core when the high-density foam was poured around it proved difficult.
- 2. In presintering and final heat treatment the low density core shrank more than the high-density encasement and the resulting stresses caused cracks to develop at the interfaces of the low and high-density layers.

The methods used to alleviate these conditions were as follows:

- 1. The reversal of the above procedure in that the high-density material was formed into slabs in the dried green state and the lower density foam was then poured into the mold between the slabs.
- 2. Use of the initial procedure, but with specially designed forms which would anchor the low density slab in position, and then troweling of the higher density foam onto both sides of the slab to facilitate integration with the higher density foam which was then poured around the slab.
- 3. Use of the same procedure but with a presintered low-density foam as the core.
- 4. Individual layering of the dried, green foams to give both good integration and accurate positioning of the layers.
- 5. The use of cores having a range in densities in conjunction with a constant high density encasement.

The procedure in Item 3 proved suitable for use in making a core of constant low density with a higher density layer surrounding it. The core material was presintered at 2300°F and positioned in a box mold so that the higher density foam could be applied to it uniformly both by trowelling and by pouring. Drying, presintering, and final sintering of the composite were then performed in the usual manner.

Maintaining parallelism of the porous, low-density core with the outer shell of high-density material presented certain problems. When the variable density blocks were placed with the laminated surfaces horizontal to the hearth, the composite tended to sag. This was largely minimized when the composite was fixtured perpendicular to the hearth. A typical variable density beam is shown in Figure 12 in which the beam has a core of 15.6% theoretical density and an outer shell of 22.2% theoretical density.

The most reproducible results were obtained when the core was presintered prior to encasing it with the higher density foam.



VII. THERMAL AND MECHANICAL TESTS

The thermal and mechanical testing of the test specimens was largely subcontracted to Melpar, Inc., Falls Church, Virginia. Thus far, the property data is confined to foamed nickel and 316 stainless steel. Single test specimens have been tested for mechanical strength properties at various temperatures. Under the extension to this contract, testing of specimens will be performed at least in duplicate.

Post-test specimens as received from Melpar are shown in Figures 13 and 14. Figure 13 shows the mechanical test specimens indentified as follows:

- 1. Broken tensile bar specimen of 27% dense stainless steel.
- 2. Compressive cube specimen of 18% dense stainless steel.
- 3. Shear strength bar specimen of 27% dense stainless steel.
- 4. Thermal stability bar specimen of 27% dense nickel.

Figure 14 shows the vibration damping sample, No. 5 of 18% dense nickel and the thermal conductivity sample, No. 6 of 27% dense stainless steel.

Prior to conducting thermal and mechanical tests on the foamed materials, it was necessary to determine the maximum temperature at which the foamed metals would support themselves under their own weight. A foamed metal bar $(1/2 \times 1/2 \times 5 \text{ inch})$ was placed on two supports with a 4-inch span. Heat was then slowly applied until the sample sagged 0.010" under its own weight. This method was abandoned since some lateral warpage of the specimens occurred. The test specimens were then lightly loaded with a pressure dial deformation indicator and the maximum useful temperature (corresponding to a sag of 0.010 inch) was determined. The results are shown in Table II.

Figure 13. Mechanical Property Test Specimens After Testing



Metal	Density, % of Theoretical	Maximum Useful Temperature °F.
Nickel	15	1675
Nickel Nickel	18 27	2500
316 Stainless St 316 Stainless St	ceel 18 ceel 27	1875 2000

TABLE II. Maximum Useful Temperature of Foamed Nickel and 316 Stainless Steel

A. Tensile Strength

Tensile tests were made using an Instron Tensile Tester. Standard threaded end pieces were machined and brazed to the ends of the foamed metals 1/2 inch in diameter and 4 inches long. In some cases, the specimens failed at the brazed joint and it was necessary to machine sections at the ends of the rods and fasten collets to them. Breakage of the specimen usually occurred near its midpoint.

l. Nickel

The values in Table III were obtained for the two densities of nickel. As shown in the table and in Figure 15, the tensile strengths decrease with increase in temperature in a fairly uniform manner. The tensile strength of pure electrolytic nickel at ambient temperature is 46,000 psi.² It is expected that the tensile strengths of the foamed metals will be low because of their structure, which consists of thin, perforated cell walls and because the tensile strength was determined by dividing the tensile yielding load by the total cross-sectional area of the test bar. This total cross-sectional area included the thin cell walls and porous areas of the bar. The actual tensile strength would be determined by the cell wall cross-sectional area available for load carrying. The comparisons between a solid bar metal and the foamed material would be much closer if the tensile strength could be calculated on this basis.



Figure 15 Tensile Strength vs Temperature for Foamed Nickel

18% Den	sity Nickel	27% Densit	y Nickel
Temperature	Tensile Strength	Temperature	Tensile Strength
°F.	psi	°F	psi
-320	1160	-320	1340
70	990	70	1200
418	620	500	700
836	560	1000	640
1255	580	1500	475
1675	145	2000	225

TABLE III. Tensile Strengths of Foamed Nickel at Various Temperatures

Tensile strength of electrolytic nickel at ambient temperature = 46,000 psi.²

2. Stainless Steel

The density of the foamed metal appears to have a considerable effect on the relationship between tensile strength and temperature in comparing the materials of 18 and 27% density as shown in Table IV. Although the tensile strength of the 27% dense material decreased at a much greater rate than that of the 18% material, the relationship in each case followed a smooth curve as shown in Figure 16. The reason for the difference in tensile characteristics of the 18 and 27% density stainless steel as compared with the parallel behavior of the 18 and 27% nickel is not apparent.

Although 316 stainless steel is a non-hardenable material by the procedures of heating and quenching, annealing and/or sintering with varying cooling rates, may bring about a variation in physical properties. Such variations would be caused by the degree of carbide precipitation with very slow cooling or the induced residual stresses with rapid cooling or quenching.

The tensile strengths of wrought 316 stainless steel





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over a range of temperatures are given in Table IV for comparison.

Wroug Stainl (Ann	ght 316 ess Steel nealed)	F	Foamed 316 Stainless Steel				
100%	Density	18% I	Density	27% I	Density		
Temp.	Tensile	Temp.	Tensile	Temp.	Tensile		
	Strength ²		Strength		Strength		
°F.	psi	°F	psi	°F	psi		
a) a, a, a, a, a,		-320	1100	-320	2850		
70	85,000	70	970	70	2450		
1000	73,000	469	700	500	1880		
1100	70,000	938	465	1000	1100		
1200	67,000	1405	320	1500	380		
1300	65,000	1875	285	2000	265		
1400	51,000						
1500	40,000						

TABLE IV. Tensile Strengths of Wrought and Foamed316 Stainless Steel at Various Temperatures

B. Compressive Yield

Compressive yield values were determined on $1/2 \ge 1/2 \ge 1$ -inch specimens. All of the materials exhibited ductile properties and since no ultimate strength was reached, the yield points (0.2% offset) were taken from the time vs stress curves.

1. Foamed Nickel

The specimens were tested at the temperatures listed in Table V for densities of 15, 18, and 27 percent of theoretical, but the results are erratic. Foamed materials are difficult to evaluate in compression because of the structure of the material. The cushioning effect of the foamed structure results in high stress concentrations at the surface and lower stress concentrations in the core which may partly account for the irregular results.

159	% Density	180	% Density	27 9	% Density
Temp.	Compressive	Temp.	Compressive	Temp.	Compressive
°F	Yield, psi	°F	Yield, psi	°F	Yield, psi
-320	210	-320	410	-320	1170
70	243	70	305	70	1100
418	254	418	296	625	900
836	115	838	304	1250	1350
1255	186	1255	332	1875	1396
1675	223	1675	220	2500	204

TABLE V. Compressive Yield of Foamed Nickel

2. 316 Stainless Steel

The 18 and 27% density 316 stainless steels were evaluated. The highest values were recorded at -320°F in each case. The compressive yield decreased markedly to 500°F and to a lesser extent thereafter as shown in Table VI and presented graphically in Figure 17.

Annealing with varying cooling rates may change the compressive yield of the foamed 316 stainless steel.

C. Shear Strength

Shear strength determinations as performed by Melpar, Inc. were done by a three-point loading technique on specimens having dimensions of $1/2 \ge 1/2 \ge 4$ inches. It should be noted that this type of measurement gives data often referred to as flexural strength.





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	18% Density	279	6 Density
Temp.	Compressive Yield	Temp.	Compressive Yield
°F	psi	°F	psi
-320	1190	-320	3150
70	770	70	1100
469	308	500	855
938	305	1000	730
1405	234	1500	689
1875	59	2000	260

TABLE VI. Compressive Yield of Foamed316 Stainless Steel

1. Foamed Nickel

Values for the 15, 18 and 27 percent nickel are listed in Table VII and presented graphically in Figure 18. The slope of the graph for the 27 percent material is somewhat greater than that of the 18 percent material but the shear strengths of the higher density foam are increased very greatly over those of the lower density foam.

2. Foamed 316 Stainless Steel

Values for the 18 and 27 percent density 316 stainless steel are listed in Table VIII and graphed in Figure 19. The shear strength of the foamed stainless steel of 27 percent density is high (8500 psi) at -320°F but shows a sharp decline at ambient and elevated temperatures. The foamed 18 percent density stainless is low in strength by comparison throughout the entire test temperature interval.

Again any additional heat treatments (annealing with varying cooling rates) would undoubtedly change the shear strength of the foamed stainless steel.

15%	Density	187	% Density	27 %	Density
Temp,	Shear Strength	Temp.	Shear Strength	Temp.	Shear Strength
°F	psi	°F	psi	°F	psi
-320 70 418	127 70 164 	-320 70 418 836 1255 1675	922 719 722 540 276 *	-320 70 625 1250 1875 2500	1985 2542 2180 1278 966 *

TABLE VII. Shear Strength of Foamed Nickel

*Samples at maximum temperature yielded at less than 50 psi.

18%	18% Density		Density
Temp.	Shear Strength	Temp.	Shear Strength
°F	psi	°F	psi
-320	1047	-320	8555
70	1370	70	3821
469	602	500	2256
938	670	1000	2353
1405	449	1500	931
1875	*	2000	*

TABLE VIII. Shear Strength of Foamed 316 Stainless Steel

*Samples at maximum temperature yielded at less than 50 psi.







Figure 19 Shear Strength vs Temperature for Foamed 316 Stainless Steel

D. Thermal Expansion

Thermal expansion measurements were made on $1/2 \ge 1/2 \ge 2$ -inch specimens using a sapphire rod dilatometer. A diagram of the apparatus appears in Figure 20.

The thermal expansion was linear between 70°F and 1900°F for both foamed materials. In the region between -320°F and 70°F the relation between thermal expansion and temperature is non-linear.

1. Foamed Nickel

Table IX summarizes values for the thermal expansion of foamed nickel and the results are illustrated in Figure 21. Values for the thermal expansion of nickel bar stock⁹ are plotted for comparison and fall on the curve for the foam.

2. Foamed 316 Stainless Steel

Table X contains data on the foamed stainless steels at 18 and 27 percent density, and the results are compared to bar stock⁹ in Figure 22. The graph indicates a rather slight deviation in thermal expansion with density.

3. Coefficients of Thermal Expansion

The coefficients of thermal expansion for both foamed nickel and foamed stainless steel were calculated and are shown for comparison with the solid metals in Table XI.⁴ No high temperature values were found in the literature for nickel bar stock. As expected, the coefficients of expansion of the solid and foamed metals do not differ greatly.

E. Thermal Conductivity

Thermal conductivities were determined by means of a radial heat flow technique using stacked discs of the foamed metals. Protection of the specimens from the atmosphere was not quite adequate as indicated by some discoloration of the foamed metals. The discs were 2 inches in diameter and consisted of 14 of 1/2-inch thickness and one of 1-inch thickness.





DILA'FOMETER

Legend

- A Liquid Nitrogen Inlet B Bell Jar
- C Vacuum Port
- D Air Vent
- E Metal Dewar
- F Heater Winding
- G Copper Can H Metal Housing
- K Insulation
- L Sample Aligner (Quartz) M Dial Indicator
- N Quartz Tube

- P Quartz Rod R Specimen S Thermocouple

Diagram of Apparatus for Thermal Expansion Figure 20. Measurements

18% De	ensity Foamed Nickel	27% D	ensity Foamed Nickel
Temp. °F.	Thermal Expansion in/in x 10 ⁻⁴	Temp. °F.	Thermal Expansion in/in x 10 ⁻⁴
- 320 - 168 - 112 - 101 - 4 + 72 91 288 601 903 1328 1674	$\begin{array}{c} -21.80\\ -15.17\\ -12.43\\ -11.31\\ -4.90\\ 0.00\\ 1.37\\ 15.87\\ 42.23\\ 69.12\\ 109.00\\ 142.15\end{array}$	-320 -180 -168 -160 + 3.2 24.8 71.6 324 1000 1500 1900	-22.25 -16.04 -15.42 -14.99 - 4.66 - 3.30 0.00 +18.59 79.64 123.47 160.66

TABLE IX. Thermal Expansion of Foamed Nickel





ty s Steel	31	27% Density 6 Stainless Steel
ermal Expansion in/in 10-4	Temp. °F.	Thermal Expansion in/in 10 ⁻⁴
-27.31 -20.49 -19.64 -15.00 -7.97 -7.82 -7.37 -0.51 0.00 18.57 47.41 73.19 100.90 127.09 167.15	-320 -288 -178 -117 +5 9 72 320 557 900 1175 1526 2003	-29.50 -27.30 -20.41 -15.78 -5.65 -5.60 0.00 22.96 46.25 82.57 115.86 154.28 207.74
	ty Steel ermal Expansion in/in 10-4 -27.31 -20.49 -19.64 -15.00 -7.97 -7.82 -7.37 -0.51 0.00 18.57 47.41 73.19 100.90 127.09 167.15	ty 310 steel 310 ermal Expansion in/in 10-4Temp. °F27.31-320-20.49-288-19.64-178-19.64-178-17.979-7.8272-7.37320-0.515570.0090018.57117547.41152673.19100.90100.902003127.09167.15

TABLE X. Thermal Expansion of Foamed316 Stainless Steel

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Figure 22 Thermal Expansion vs Temperature for Foamed and Bar Stock 316 Stainless Steel

TABLE	XI.	Coeffic	ient	s of	Therr	nal	Expansion
c	of Nic	kel and	316	Stai	inless	Ste	el

Material	Coefficient of Thermal Expansion ⁴
Nickel:	
Bar Stock	7.4 x 10 ⁻⁶ °F (32-212°F)
27% Density Foam	8.75 x 10^{-6} °F (72-1900°F)
18% Density Foam	8.88 x 10 ⁻⁶ °F (72-1675°F)
316 Stainless Steel:	
Bar Stock	11.1 x 10^{-6} °F (32-1500°F)
27% Density Foam	10.45 x 10 ⁻⁶ °F (72-2000 °F)
18% Density Foam	9.1 x 10 ⁻⁶ °F (72-1875°F)

1. Foamed Nickel

Table XII permits a comparison between values for the thermal conductivities of solid nickel⁹ with those of the nickel foams over a range of temperatures and illustrates the insulating value of the foamed metals.

Figure 23 indicates that the thermal conductivity of the foamed nickel decreases to a minimum at about 800-900°F and then increases with temperature. The solid nickel decreases to a minimum conductivity at 700-750°F and then increases linearly with higher temperatures. The minimum conductivity point for the curves is due to the magnetic transformation or Curie temperature of nickel. This is actually a temperature range where the material transforms from ferromagnetic to paramagnetic. Theoretically, the intensity of magnetization at a temperature is a function of the degree of atomic order. At higher temperatures, as the disorder increases because of thermal agitation, a sudden and almost complete collapse of order occurs in the structure and correspondingly in the magnetization and changes the thermal conductivity. Solutes or alloying elements added to magnetic materials linearly affect the Curie temperature of nickel.

For purposes of comparison the values for solid nickel have been multiplied by factors of 0. 18 and 0. 27 to indicate the thermal conductivities that might result at the lower percentages of theoretical. Here again rough comparisons of the insulating qualities of the foamed metal are obtained.

2. Foamed 316 Stainless Steel

Table XIII and Figure 24 allow a comparison between the thermal conductivity values of foamed stainless steel, (18 and 27% of theoretical density) with material of theoretical density⁹.

As in the case of nickel, the values for thermal conductivity of the fully dense stainless steel have been multiplied by 0.18 and 0.27 respectively, to provide rough comparisons with the values for the foamed metal. The insulating gualities of the foamed products are evident in the actual measured values.

F. Vibration Damping

Vibration damping characteristics were evaluated for foamed

TABLE XII. Thermal Conductivities of Solid Nickel and Foamed Nickel

.

kel V
Foamed Nick 18 % Density 19. The
Len I
med Nickel % Density Thermal
Foam 27 % Temp.
ytic Nickel Density Thermal
Electrol 100 % Temp.



Figure 23 Thermal Conductivity vs Temperature for Foamed Nickel

TABLE XIII. Thermal Conductivities of Solid 316 Stainless Steel and Foamed 316 Stainless Steel

ss Steel y	mal tctivity -sec- ^o F/in 02 09 30 63 63 63 96 96
ned Stainle 8 % Densit	Ther Condu BTU/ft ² BTU/ft ² 0.001 0.001 0.001 0.001 0.002 0.002 0.003
Foan	Temp. oF - 62 - 62 -0.4 194 194 365 496 1022 1346 1688
d Stainless Steel % Density	Thermal Conductivity BTU/ft ² sec- ^o F/in 0.00335 0.00347 0.00407 0.00459 0.00565
Foame 27	Temp. ^o F 77 212 581 932 1513
tinless Steel 6 Density	Thermal Conductivity BTU/ft2_sec- ^o F/in 0.0153 0.0245 0.0313 0.0377 0.0455 0.0547 0.0547
316 Sta 100 %	Temp. ^o F 500 1000 1500 2000



Figure 24 Thermal Conductivity vs Temperature for Foamed Stainless Steel

316 stainless steel and foamed nickel and these results were compared to the values for the solid metals.

Vibration damping is an inherent material property caused by the internal friction encountered during motion of a body of material. This property is often referred to in terms of a vibration decay rate or logarithmic decrement which is based on the concept of energy dissipation (determined by amplitude measurement) per cycle of vibration. The amount of damping is usually expressed in terms of percent critical damping (Cc). Critical damping represents the limiting damping value for a particular material above which a body does not vibrate, but gradually creeps back to an equilibrium position or, in other words, is overdamped. It is related to the logarithmic decrement by the equation:

$$\frac{C}{Cc} = \frac{\delta}{2\pi}$$
 (100) References: 5,6,7, &8

C = damping effect of material

Cc = critical damping factor

S = logarithmic decrement as measured by amplitude variations

Table XIV describes the vibration damping characteristics for foamed 316 stainless steel and foamed nickel, and these results were compared to values determined for the wrought materials.

Originally discs of foamed metals 3 inches in diameter and 1/2 inch thick were used for the measurements. These proved unsatisfactory and bars having dimensions of $1/2 \ge 1/2 \ge 4$ inches were used.

Nickel of theoretical density is far superior to 316 stainless steel in vibration damping capacity. As might be expected, the foamed nickel of 18 percent theoretical density has a much higher damping capacity than the material of 27 percent of theoretical density.

Stainless steel in wrought form has a very low damping capacity but this property is greatly increased in the foamed state. It is not clear why the vibration damping capacity of the 27 percent foamed material is higher than that of the 18 percent material but these values have been rechecked and possible sources of error are believed to have been eliminated.

Material	Cc %	Logarithmic Decrement x 10-4
Electrolytic Nickel (theoretical density)	0.201	126.3
27% Density Foamed Nickel	0.211	132.5
18% Density Foamed Nickel	0.340	213.4
Stainless Steel (wrought)	0.014	5.4
27% Density Foamed Stainless Steel	0.192	120.4
18% Density Foamed Stainless Steel	0.101	63.6

TABLE XIV. Vibration Damping Characteristics of Nickel and 316 Stainless Steel

VIII. ANTICIPATED WORK

In the development contemplated for the ensuing year, at least four foamed metals are to be produced and evaluated during the course of the contract extension. The materials are to be mutually agreed on by a representative of Ipsen Industries, Inc. and the contracting officer's technical representative. The foamed metals are to include both low density metals such as molybdenum alloys and the highest strength type of stainless steel. Work will also proceed on H-11 tool steel in order to produce enough material for a mechanical property evaluation of the sintered foamed product.

The following items will be included:

1. Each of the foamed metals will be produced in various densities. Sufficient quantities of the products in each density will be produced to permit evaluation of properties and to demonstrate potential areas of application.

2. Each of the foamed metals will be used in evaluating the relationship between the mechanical properties and density and pore size of the foamed metals. Each of the metals will be produced and evaluated in at least two densities and at least two pore sizes for each density. It has been agreed that the small pore size should be in the range of 0.015 - 0.035 inch and the larger pore size should average 0.060 inch. Also the low density range is to be 10-15% of theoretical and the high density range is to be 20-30% of theoretical. Mechanical properties to be determined at ambient temperature, will include tensile, compressive, and shear strengths, vibration damping capacity, and energy abosrption with tests made at least in duplicate. The resistance of the foamed metals to impact of high velocity projectiles will be evaluated by the test methods provided by the contract officer.

3. Aluminum and titanium or their alloys will be used as core materials in sandwich structures. Foamed molybdenum as a core material clad with molybdenum sheet will also be developed as a sandwich structure. The sandwich structures are to be formed by brazing aluminum and/or titanium facing sheets to each of the foamed metal cores. The structures are to be tested at -320° F, ambient temperature, and two elevated temperatures including the maximum temperature to which the materials making up the composite can be exposed without too severe an effect on properties including tensile, compressive, and flexural strength, and energy absorption. 4. A high-strength stainless steel will be selected to evaluate the effects of the initial particle size and distribution on the tensile and compressive properties of the foamed metal.

5. Two of the foamed metals will be developed and evaluated as variable density beams wherein the lowest density will be at the center and the highest density at the surface. A high strength type of stainless steel is to be one of the selected foamed metals and the other is to be an aluminum alloy. However, titanium or molybdenum may be substituted if found more suitable. Evaluations will be made at ambient temperatures by load deflection bend tests. The foamed metals will be of an intrinsically low-density metal and of a high strength metal.

6. At least two foamed metals will be selected for investigation of foaming and forming methods. Casting of the metal foams into shapes approximating final configurations will be attempted to reduce machining operations. A stainless steel is to be one of the selected metal foams while the most promising of the other metal foams will be selected in the future. Variables including shrinkage and shape limitations will be investigated. Machining studies will include sawing, grinding, drilling, and turning.

7. At least one foamed metal of either titanium or aluminum is to be developed and evaluated at a density of less than 10% of theoretical in a ductile state.

8. Metallographic studies will be made whenever appropriate to illustrate structures of the foamed metals and the nature of bonding in composite structures.

A Program Planning Chart is shown on page 61.

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TASK	1. FOAMED METALS	*A. Aluminum	*B. Titanium	*C. Molybdenum	*D. Stainless Steel	*E. H-11 Tool Steel	2. MATERIAL TESTII	³ . DEVELOPMENT A ³ . FABRICATION STU	A. Sandwich Struc	B. Particle size v Mechanical Pr	C. Variable Densi	D. Fabrication an Forming Tech	E. Low Density M (10% of Theore	4. REPORTS	A. Monthly	B. Quarterly	C. Annual (rough	D. Annual (final d		* Tentative Developme	

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- 1. H. N. Ipsen, Fundamentals of Vacuum Heat Treating, page C-2, 1962.
- 2. S. Hoyt, ASME Handbook Metals Properties, page 371, McGraw-Hill Book Company, Inc., 1954.
- 3. <u>Metals Handbook Volume I Properties and Selection</u> of Metals, 8th Edition, pages 413-415. Published by American Society for Metals.
- 4. S. Hoyt, ASME Handbook Metals Properties, page 63, McGraw-Hill Book Company, Inc., 1954.
- 5. F. Forster, Z. Metalk, 29, 109 (1937).
- 6. F. Forster and Breitfeld, Z. Metalk, 30, 343, (1938).
- 7. F. Forster and W. Koster, Engineer, 166, 626, (1938).
- 8. G. M. Smith and H. D. Berns, <u>Material Research and</u> Standard. 4 (5), May 1964 225-227.
- Goldsmith, Waterman, and Hitschhorn, <u>Handbook of</u> <u>Thermophysical Properties of Solid Materials</u>, Vol. II, <u>I-N-3 & II</u>, D-3, The Macmillan Company, New York, 1961.

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