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A NEW A15 MULTIFILAMENTARY SUPERCONDUCTOR BASED ON THE NIOBIUM-ALUMINUM-SILICON SYSTEM

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ABSTRACT

Based on Powder Metallurgy techniques, a process to fabricate $Nb-Nb_3(Al,Si)$ multifilamentary superconducting wire has been developed. Optimum sintering and infiltration parameters are listed and the methods of mechanical reduction to the wire form are described. Preliminary data indicate that diffusion reaction temperatures as low as 850°C form the A15 superconducting compound $Nb_3(Al,Si)$.

INTRODUCTION

In the past, the increasing demand for power on this planet was met by simply generating more of it. Energy conservation, and the more efficient utilization of existing energy sources had not been practiced until recently. The realization that world reserves of fossil fuels are indeed finite, and the fact that energy prices are sky-rocketing has compelled scientists to investigate new methods to generate and preserve our most precious natural resource-energy.

High field superconductivity research promises numerous technological applications for generating and conserving energy. In the sixty-six years since its discovery by Kamerlingh Onnes[1] superconductivity underwent little progress until the mid 1960's. The technology of electric current without resistance is just now starting to compete in generation, transforming, and transmitting power, propelling ships and levitating trains----with economy and reliability. Some prototype quasi-commercial devices are already in use and approximately 30 million dollars are being spent annually to develop this technology further.[2]

Research and development of superconducting materials is divided into two groups: ductile solid-solution alloys, such as those of niobium and titanium; and A15 (see Fig. 1), intermetallic compounds, such as Nb_3Sn and $Nb_3(Al,Ge)$, which are relatively brittle. The A15 type superconductors boast high magnetic fields with critical temperatures above 20^oK, but demand special fabrication methods owing predominantly to their brittle nature. A critical temperature over 21^oK permits the possibility of using liquid hydrogen rather than liquid helium to cool the superconductor which could make large scale commercial applications much more feasible. For this reason intensive research is being conducted to develop many of the high field superconducting materials to a commercial level. So far this has only been done for V_3Ga and Nb_3Sn while materials thought to have superior properties have gone essentially undeveloped.

At the present time commercial fabrication of A15 structure multifilamentary superconductors utilizes the so called "bronze process".[3] This process is particularly attractive because it imparts to the wire an intrinsic stability and has been effective in the fabrication of Nb_3Sn and V_3Ga composite superconductors. However, the bronze process has been found to be ineffective for aluminum-taining compounds which provide the advantages of much higher critical fields and temperatures. In contrast, the power metallurgy infiltration process developed at the Lawrence Berkeley Laboratory has been used successfully not only for Nb_3Sn,[4] but also for Nb_3Al,[5] $Nb_3(Al,Ge)$,[6] and as now reported for the first time, for $Nb_3(Al,Si)$.

This report concerns the adaptation of the existing Powder Metallurgy Process to fabricate mutlifilamentary Nb + $Nb_3(Al,Si)$ superconducting wire. The success of the Powder Metallurgy Process with other materials[7] does not necessarily guarantee success with seemingly similar materials systems. The design parameters for this system are discussed in the experimental procedure section. The advantages and/or disadvantages are mentioned in the results and discussion section while conclusions and projections for further studies are made in the final section.

EXPERIMENTAL PROCEDURE

I. Processing of the Wire

If industry is to eventually employ high field superconductors, the development of viable commercial methods for its fabrication must be completed. The Powder Metallurgy Process shows great promise of being a workable means for fabricating multifilamentary wire. It requires six steps to go from a powder to the final superconducting wire. These steps are: 1) isostatic compaction of the niobium powder to render the desired porosity and green (i.e. unsintered) strength; 2) sintering of the niobium rod to give it strength and ductility; 3) infiltration of the rod with Al-Si eutectic alloy, thereby filling the pores with an inter-connected network of the eutectic; 4) cladding of the rod to facilitate mechanical deformation; 5) mechanical deformation of the rod to produce a multifilamentary structure of the Al-Si eutectic in the niobium, and 6) diffusion heat treatment to form $Nb_3(Al,Si)$ filaments. A schematic of the process is shown in Fig. 2.

Niobium powder composed of -270 +400 mesh size particles were isostatically compacted in rubber tubes to a pressure of 30 ksi. The porosity of the 3/16" diameter green compact, which is a function of particle size and compaction pressure, was estimated to be approximately 22%. (Fig. 3)

Green compacts were vacuum sintered at 2100°C for 15 minutes at a pressure of 10^{-5} mm. Hg. The sintered core was then infiltrated with the liquid Al-Si eutectic at 580-585°C for 30 seconds (Fig. 4). Both the sintering and infiltration steps were done in a high vacuum Abar furnace, (Fig. 5).

After sintering and infiltration of the core was completed it was cladded with copper or monel to facilitate mechanical deformation. An intermediate sheath of tantalum which served as a diffusion barrier was necessary to prevent any reaction between the outer clad and the infiltrated core during the final heat treatment. A single clad of tantalum would have been inadequate due to its tendency to gall on the wire dies.

The double cladded assemblies were reduced to wire by a combination of deformation processes: swaging, form rolling, and wire-drawing. Swaging of the assemblies was used only to provide a close fit of the cladding materials and to point the wire prior to wire drawing. As a mechanical deformation mode, swaging tends to twist the entire specimen which is an undesirable effect. The degree of deformation taken as the ratio of the original to the final cross section was approximately 1000 to 1.

Although cladding is an essential step in the processing of the wire, it will invariably tend to crack during mechanical deformation. Fracture of the cladding material occurs more often with thicker clads than with thin ones. This is attributed to the larger degree of deformation of a thicker clad which gives rise to strain hardening and residual stress. To relieve these stresses some of the samples were annealed for 5 minutes at 300°C whenever fracture started to occur. The thickly cladded sample #NAS-17 required 5 different heat treatments while the thinly cladded sample #NAS-14 required none at all.

Sections of the finished wire were subjected to several different reaction times and temperatures. Although the heat-treatment parameters to produce the optimum superconducting properties have not yet been determined, it has been found that the Nb-Al-Si system is indeed superconducting and shows promise of good T_c values.

II. Analytical Techniques

Optical microscopy was used to: 1) determine the porosity of the green and sintered compacts; 2) confirm that infiltration of the sintered niobium core with the Al-Si eutectic was complete; 3) identify the various phases present; and 4) verification of multifilamentary structure in the drawn wire. Porosities were determined by a two dimensional point-count method which assumes that cross sectional area ratios are directly proportional to the volume ratios of pores to matrix.[8] Another method to determine porosity entailed sealing the pores of a green compact with epoxy resin, weighing it in water to determine the buoyant force and therefore its volume. Knowing the density of niobium and the weight of the sample, one can easily calculate the porosity. These two methods showed only a 3% difference in porosity values for two different samples fabricated under identical conditions. Once an infiltrated core has been mounted and polished correctly, visual inspection determines if it is completely infiltrated. An anodization method developed by Picklesimer[9] gave a characteristic color to each phase: deep blue for Nb, yellow for Al, brown for Si, light brown for $NbAl_3$ and light blue for Nb3(Al-Si). A photomicrograph of the infiltrated compact is shown in Fig. 4 and the multifilamentary structure of the drawn wire is shown in Fig. 6.

Scanning electron microscopy and EDAX (Energy Dispersive Analysis of X-ray) were used to identify the specific elements for the determination of the compositional relationships in the various phases present.

The critical temperatures T_c were measured by a standard inductive method using a Ge thermistor for the temperature determination with an estimated uncertainty of 0.1K. The reported values correspond to the points showing the highest rate of change of the inductance in the transition (superconducting to normal) region.

RESULTS AND DISCUSSION

I. The Aluminum Silicon Eutectic System

One objective of this project was to develop an A15 multifilamentary superconductor that would exhibit a large degree of ductility during the mechanical deformation stage. At the present time the wire processed by the infiltration process, which shows the best superconducting properties is Nb + $Nb_3(Al,Ge)$.[10] A drawback of this system is its low ductility during mechanical deformation which does not permit large cross-sectional area reductions. This may be due to the brittleness of the Al-Ge eutectic alloy. If another more ductile alloy could be substituted for Al-Ge and still retain the good superconducting properties of the Nb + $Nb_3(Al,Ge)$ system then it would prove to be superior, especially with respect to commercial production.

The infiltrant employed in the fabrication of Nb + $Nb_3(Al,Si)$ wire is the binary aluminum-silicon eutectic (phase diagram Fig. 7). The structure and properties of the eutectic have been reported on by Steen and Hellawell in 1972.[11] Their data show that in the cast condition the Al-Si eutectic exhibits poor ductility, but if it undergoes slow unidirectional solidification, its ductility is greatly improved. Steen and Hellawell have characterized the chill cast microsctructure as fibrous, and the unidirectionally solidifed microsctructure as flaky. Photomicrographs of these two morphologies are shown in Fig. 8 and Fig. 9 respectively. The fibrous specimen represents the chill cast condition of the eutectic. The flaky specimen is a piece of the Al-Si eutectic infiltrant after it had been heated above its melting point of 577°C and slowly cooled in a directional manner. This is achieved by simply raising the resistance heater (item 14, Fig. 5) above the liquid Al-Si eutectic bath (item 13, Fig. 5) and lowering the voltage. This procedure induces a temperture gradient that results in a solid-liquid interface moving upward from the bottom and a flaky form microstructure.

Cooling the infiltrant by the method described above also served another purpose. As the solid-liquid interface travels upward, much of the impurities present migrate along with it. These impurities can then be ground off resulting in an Al-Si infiltrant of a higher purity. The repetition of this step was found to be a necessity in order to successfully infiltrate the sintered niobium compact.

The infiltrant bath was contained in a graphite crucible shown in Fig. 10. The thermocouple to measure the bath temperature was located in this crucible and is assumed to be accurate for steady state conditions.

II. Sintering and Infiltration

Successful infiltration of a porous niobium core with the eutectic Al-Si infiltrant requires certain conditions that must be satisfied. The most critical conditions are: 1) the green compact must be of at least 20%, but no more than 30% porosity; 2) sintering must take place in a vacuum or in an inert gas atmosphere; 3) the eutectic bath must be heated to a temperature only several degrees above its melting point; 4) prior to immersion the sintered compact must be heated to within a certain temperature range. Unless all of the conditions mentioned above are satisfied, failure will result.

Porosity of the green compact is a function of mean particle size and compaction pressure. Niobium powder of -250 +400 mesh particle size purchased from Wah Chang was used. Isostatic compaction at 30 ksi resulted in a porosity of approximately 22%. This shows good agreement with experimental work previously done at MMRD under similar conditions.[12] Complete infiltration would be impossible unless all of the pores were interconnected. A generally accepted rule states that if the porosity is 10% or more the pores are indeed interconnected.

Sintering temperatures of approximately 2150°C for 15 minutes duration appeared to be satisfactory. The compacts were sintered in a high vacuum of 10^{-5} mm Hg to prevent oxidation of the niobium compact.

It was found that if the infiltration time and temperature were too high, then the formation of a secondary brittle phase resulted. Through EDAX analysis this phase was found to be $NbAl_3$ (Fig. 11). To eliminate the undesirable formation of $NbAl_3$ the infiltration time and temperature were optimized. A series of optical micrographs showing the varying amounts of $NbAl_3$ within the core is shown in Fig. 12 a,b,c. The microstructure was found to be extremely sensitive to small differences in infiltration time and temperature. Fig. 12a was infiltrated for 60 seconds at 600°C and it shows a great deal of the brittle intermetallic phase. Fig. 12b shows slightly less formation of $NbAl_3$, because it was infiltrated for only 30 seconds at 600°C. On the other hand the brittle phase is completely absent in Fig. 12c, which was infiltrated for 30 seconds at 580°C, only

$3^{o}C$ above the melting point of the eutectic.

To insure complete wetting of the core it was necessary to heat it prior to immersion. The infiltrant would either fail to soak in or react to form the brittle $NbAl_3$ intermetallic phase if the niobium core was too cool or too hot respectively. Core temperatures from $500^{o}C$ to $580^{o}C$ were found to be satisfactory. A list of sintering and infiltration attempts is described in Table 1.

III. Wire Drawing

One major advantage of the Nb-Al-Si system is the ease with which it reduces to wire. Other systems exhibit a lack of ductility which hinders mechanical reduction. An Nb+(AlSi) sample was drawn from a 3/16" diameter core to a .006" diameter wire at room temperature. This yields a cross-sectional area reduction ratio of 975:1 and an average filament size of only 1 micron.

In past work on niobium alloy systems containing aluminum, reaction temperatures in excess of $1200^{o}C$ were required for the formation of the A15 compound. It has now been discovered that the very fine filaments of the aluminum-silicon eutectic interact with the matrix to form the A15 compound at temperatures as low as $850^{o}C$. This is of tremendous technological importance, in that it makes possible the incorporation of copper to provide the stability necessary for practical superconductors.

Sample #NAS-14 illustrates the importance of good ductility and the resulting filament size. Two pieces of this sample were wire drawn down from the original 3/16" diameter, 1" long infiltrated core. These pieces were reduced to 0.018" O.D. and .012" O.D., which correspond to reduction ratios of 110 and 250 respectively. Optical microscopy observations have confirmed that increasingly finer average filament sizes can be obtained by increasing the reduction ratios. Both sectons of wire were subjected to identical heat treatments: a pre-reaction heat treatment of one hour at 700°C and a reaction heat treatment of six hours at 850°C. The wire that had been more extensively reduced hold a critical temperature of 14.0K, while the larger wire had a T_c of only 11.0K. These results seem to indicate that a smaller average filament size enhances the critical temperatures obtainable for the niobium-aluminum-silicon system.

CONCLUSIONS

The distinguishing characteristics of the Nb-Nb_3(Al,Si) system are: 1) The remarkable ease with which the Al-Si eutectic wets and infiltrates niobium. With other infiltrants (Sn,Ga,Al-Ge) infiltration temperatures as much as several hundred degrees Centigrade above the melting point were required for complete infiltration. It has been demonstrated repeatedly that with the niobium-aluminum-silicon system, complete infiltration takes place at an aluminum-silicon eutectic bath temperature less than 5°C above its melting point, thus eliminating the possibility of any undesirable side reactions. 2) The extraordinary ductility of the infiltrated core permits mechanical deformation reduction ratios on the order of 1000 to 1 at room temperature. This property makes possible the fabrication of long lengths of superconducting wire and/or extremely fine filament sizes. 3) The very fine filamentary micro-structure that can be achieved with the Nb-Al-Si system makes possible the formation of the A15 compound at reaction temperatures as low as 850°C. Smaller filaments also result in higher critical temperatures than larger ones.

The Powder Metallurgy infiltration process has been successfully adapted to the niobium-aluminum-silicon system. Fabrication of flexible Nb+Nb_3(Al,Si) multifilamentary superconducting wire with the unique characteristics of high reduction ratio and low reaction temperature is of tremendous technological importance. Preliminary data indicate that with heat treatment optimization this sytem shows promise of high critical temperature values.

RECOMMENDATIONS FOR FURTHER STUDY

The primary objective of this study was to determine if a new A15 multifilamentary superconductor based on the niobium-aluminum-silicon system is feasible. By adapting this system to the powder metallurgy process it has been shown that it is not only possible to fabricate multifilamentary superconducting wire, but there are several distinct advantages characteristic of this system.

The superconducting properties have yet to be optimized for the $Nb-Nb_3(Al,Si)$ wire. A critical temperature of 17^oK has been measured in one instance, but this was at a relatively high reaction temperature and time, (1200^oC for 5 minutes). Lower critical temperatures have been obtained for reactions at 850^oC for 3 to 6 hours. It is in this area of low temperature reactions that more research should be directed.

Another aspect of the process that should be noted, but not necessarily studied more extensively concerns the cooling rates inside the infiltrated core once infiltration has taken place. As has been discussed previously it is desirable to slow cool the Al-Si eutectic in order to obtain a ductile flaky microsctructure rather than a brittle fibrous microstructure. Once the infiltrated core is raised out of the Al-Si eutectic bath there is no way to control the resulting cooling rates inside the Abar furnace. This has the deleterious effect of producing different cooling rates throughout different sections of the core itself. It is logical to assume the core's outer layers are cooling at a faster rate than the inner layers and one would expect to observe the respective fibrous and flakey microstructures associated with these different cooling rates. This is indeed the case and is shown in Fig. 13, which is slow cooled and by Fig. 14, which is fast cooled. In both figures, "a" is a photomicrograph of just the eutectic and "b" is a photomicrograph of the infiltrated niobium matrix showing the similar eutectic morphology within the pore. These two figures are from the same sample and the fact that the fibrous structured pores were only predominant near the outer layers of the core substantiates the assumption that a cooling rate gradient exists in a radial direction from the core center.

One last recommendation for future study concerns the possibility of using the Al-Si eutectic alloy as a potential fibre reinforced composite. Composite reinforcement, which is defined as the load transfer from the aluminum matrix to the silicon fibres when subjected to uniaxial tension or compession, does exist for the Al-Si eutectic if the Si fibres are grown by unidirectional solidification. It has also been shown that under specific reaction conditions the A15 compound can be formed concentrically around the eutectic filaments in the multifilamentary superconductors.[14] Depending on the relative brittleness of the silicon fibres and the $nb_3(Al,Si)$ filaments there is a possibility of load transfer to the silicon fibres rather than to the $Nb_3(Al,Si)$. A one micron diameter silicon fibre has a theoretical ultimate tensile strength of 555,000 psi. This could have the effect of increasing the tensile strength of the multifilamentary wires tremendously.

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TABLE I

Sample No.	Vacuum (mmHg)	Sinter Temp. (0°C)	Sinter Time (min)	Infiltrate Temp. (0°C)	Infiltrate Time (sec)	Backfill atmosphere	Core Temp (°C)	Comments
NAS-05	10^{-5}	2100	15	650	60	Helium	750	Large amount $NbAl_3$ formed around pores
NAS-08	10^{-5}	2250	15	600	65	Helium	700	Medium amount $NbAl_3$ formed around pores.
NAS-09	10^{-5}	2200	15	600	60	Helium	500	Small amount $NbAl_3$ formed around pores
NAS-10	10^{-5}	2200	15	600	30	Helium	500	Very small amount $NbAl_3$ formed around pores.
NAS-11	10^{-5}	2200	15	600	35	Helium	600	Small amount $NbAl_3$ formed around pores
NAS-12	10^{-5}	2200	15	580	30	Helium	400	Lack of pores on the perimeter of cross section. Sample thrown out.
NAS-13	10^{-5}	2200	15	580	40	Argon	500	Very small amount $NbAl_3$ formed around pores.
NAS-14	10^{-5}	2200	15	580	30	Helium	500	Total absence $NbAl_3$ around pores.
NAS-15	10^{-4}	2150	15	580	30	Helium	500	Sample not infiltrated.
NS-16	10^{-4}	2200	15	585	30	Helium	500	Sample not infiltrated.
NS-17	10^{-5}	2200	15	585	30	Helium	580	Total absence of $NbAl_3$ around pores.

FIGURE CAPTIONS

1) Unit cell of the A15 structure.

2) The infiltration process for producing multifilamentary superconducting wire.

3) Green compact of -270+400 mesh size niobium powder isostatically compressed at 30,000 psi.

4) Photomicrograph of an Aluminum-Silicon eutectic filled pore within a niobium matrix. Sample #NAS-14

5) Schematic diagram of Abar furnace; 1) extension tube, 2) tantalum rod, 3) back filling port, 4) electrical leads, 5) heating element, 6) niobium specimen, 7) radiation shields, 8) water cooled wall, 9) W-5% Re vs. W-26% Re thermocouple junction, 10) vacuum connection, 11) quartz tube, 12) graphite crucible, 13) liquid Al-Si eutectic alloy, and 14) resistance heater.

6) Longitudinal view of multifilimentary niobium-aluminum-silicon wire, a) sample #NAS-14 wire drawn down to 0.018 inches O.D. and heat treated for one hour at 700^{o}C, b) same as "a", but twice the magnification.

7) Phase diagram for binary aluminum-silicon alloy.

8) Quenched aluminum-silicon eutectic alloy as received from M.M.R.D. machine shop. Note the fibrous micro-structure.

9) Slow cooled aluminum-silicon eutectic alloy. Note the flaky microstructure.

10) Graphite crucible used to contain the liquid aluminum-silicon eutectic bath during heat treatment and infiltration.

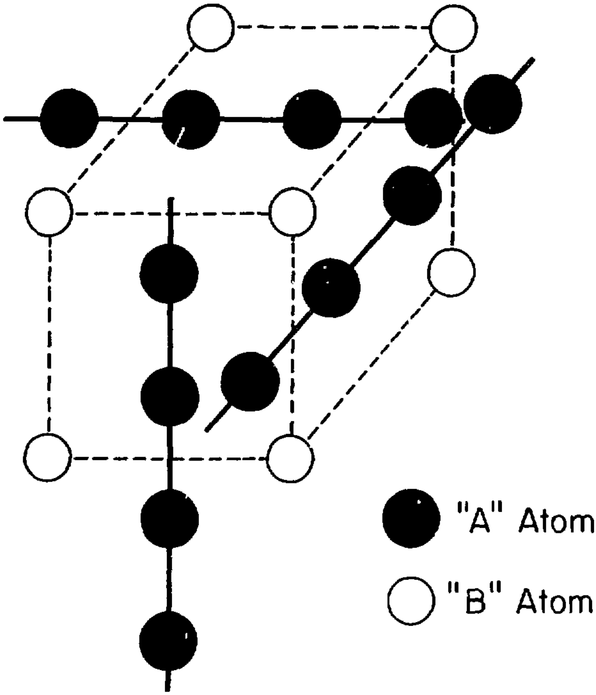
FIGURE CAPTIONS (Continued)

11) An infiltrated compact showing an EDAX analysis of the secondary $NbAl_3$ phase which forms under non-ideal infiltration conditions. Sample #NAS-05. 1: $NbAl_3$; 2: Nb

12) Series of optical photomicrographs illustrating how infiltration parameters affect the formation of the brittle $NbAl_3$ phase; a) sample #NAS-8, sintered at 2250°C for 15 minutes, infiltrated at 600°C for 65 seconds, sintered compact heated to approximately 750°C prior to immersion; b) sample #NAS-10, sintered at 2200°C for 15 minutes, infiltrated 600°C for 30 seconds, sintered compact heated to approximately 500°C prior to immersion; c) sample #NAS-14 sintered at 2200°C for 15 minutes, infiltrated at 580°C for 30 seconds, sintered compact heated to approximately 500°C prior to immersion.

13) Aluminum-Silicon eutectic alloy. a) The flaky form micro-structure is characteristic of slow unidirectional cooling.
b) The flaky form Aluminum-Silicon eutectic alloy within a well infiltrated niobium matrix.

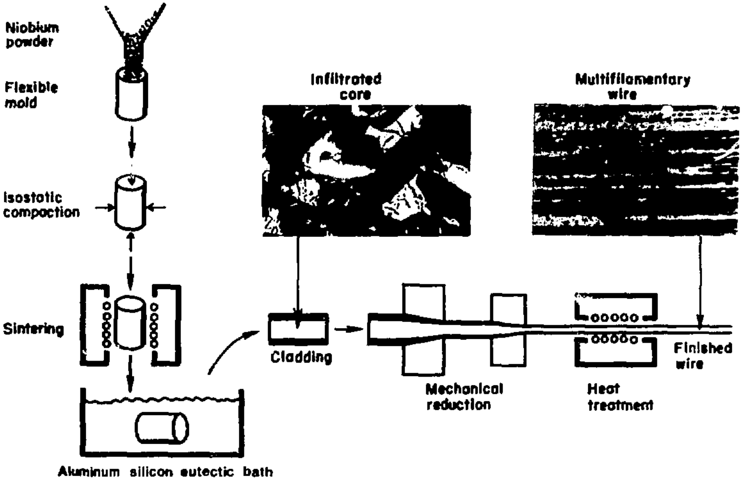
14) Aluminum-Silicon eutectic alloy. a) The fibrous form micro-structure is chracteristic of quenched or fast cooled specimens.
b) The fibrous form Aluminum-Silicon eutectic alloy within a well infiltrated niobium matrix.



A15 structure
formula type A_3B

Figure 1.

XBL 763-2568



THE INFILTRATION PROCESS FOR PRODUCING MULTIFILAMENTARY SUPERCONDUCTING WIRE

Figure 2. XBB 770-10876

Figure 3

XBB 770-12180

Figure 4.

XBB 760-10504

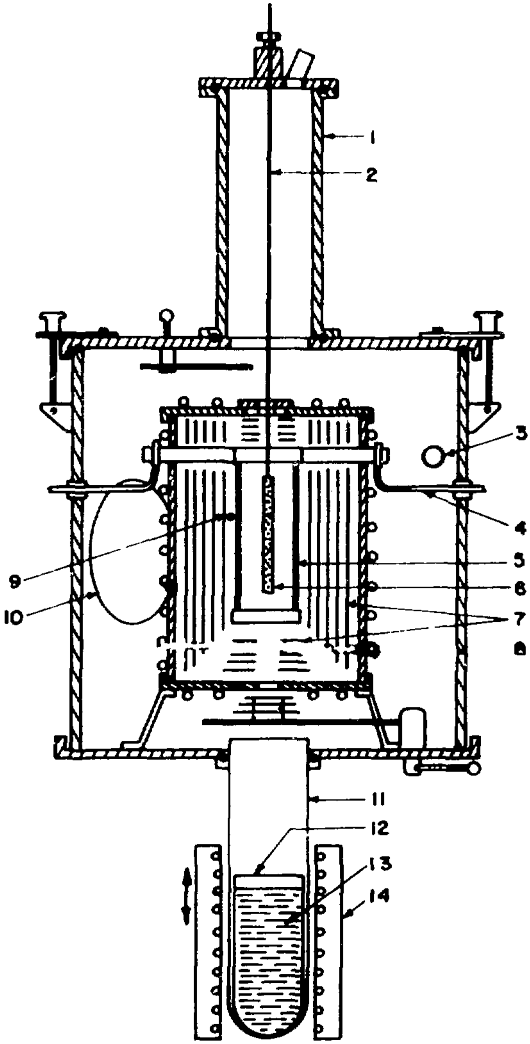
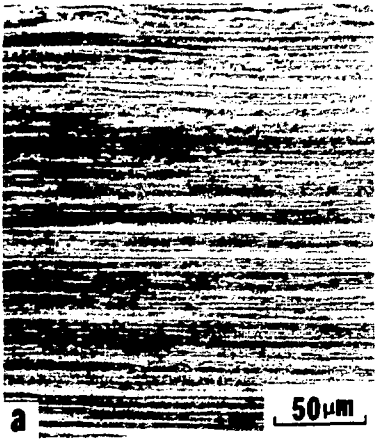


Figure 5.

XBL 7210-7043



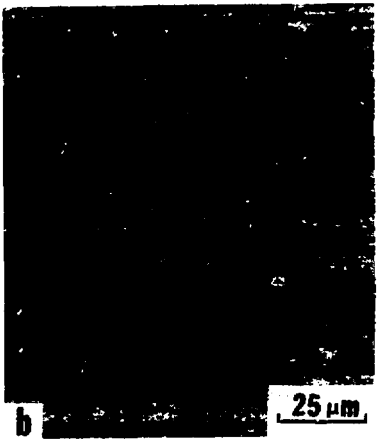


Figure 6.

XBB 760-10500

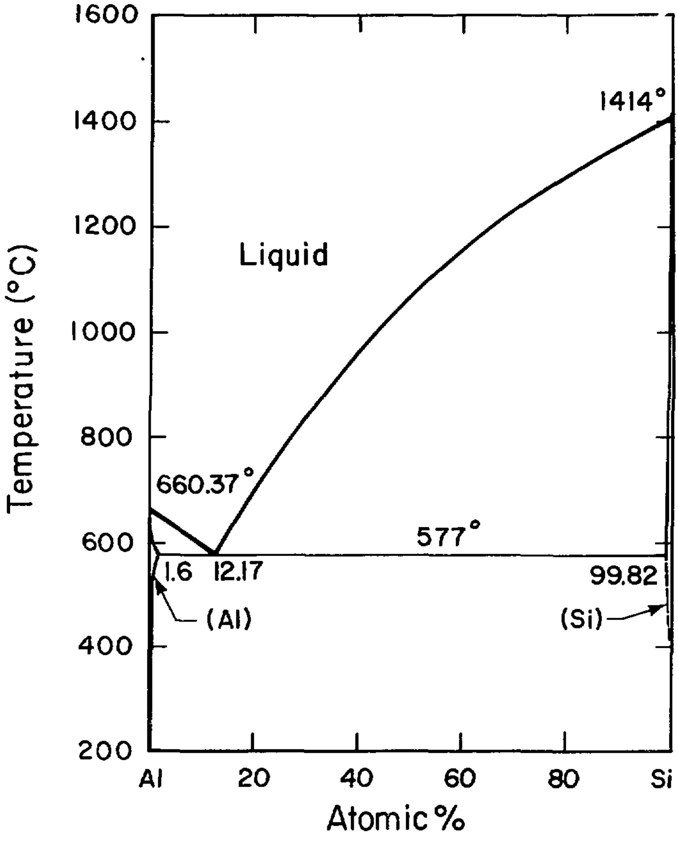


Figure 7. XBL 777-1278

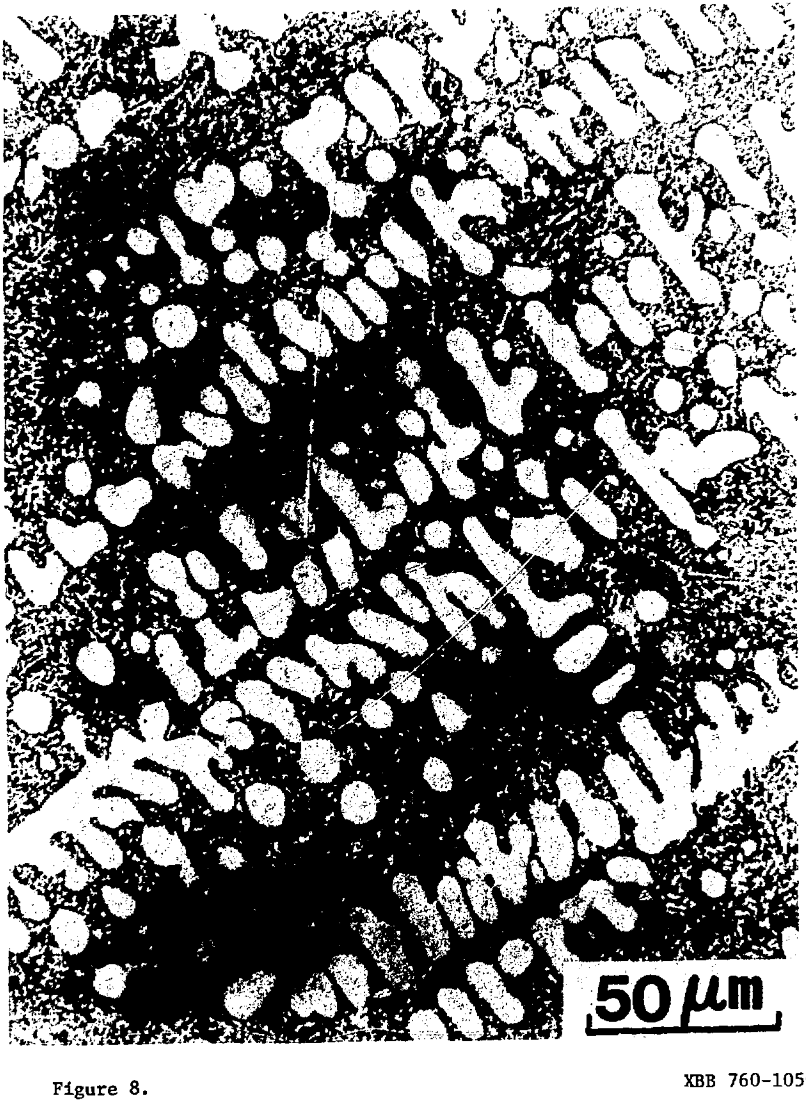


Figure 8.

XBB 760-10505

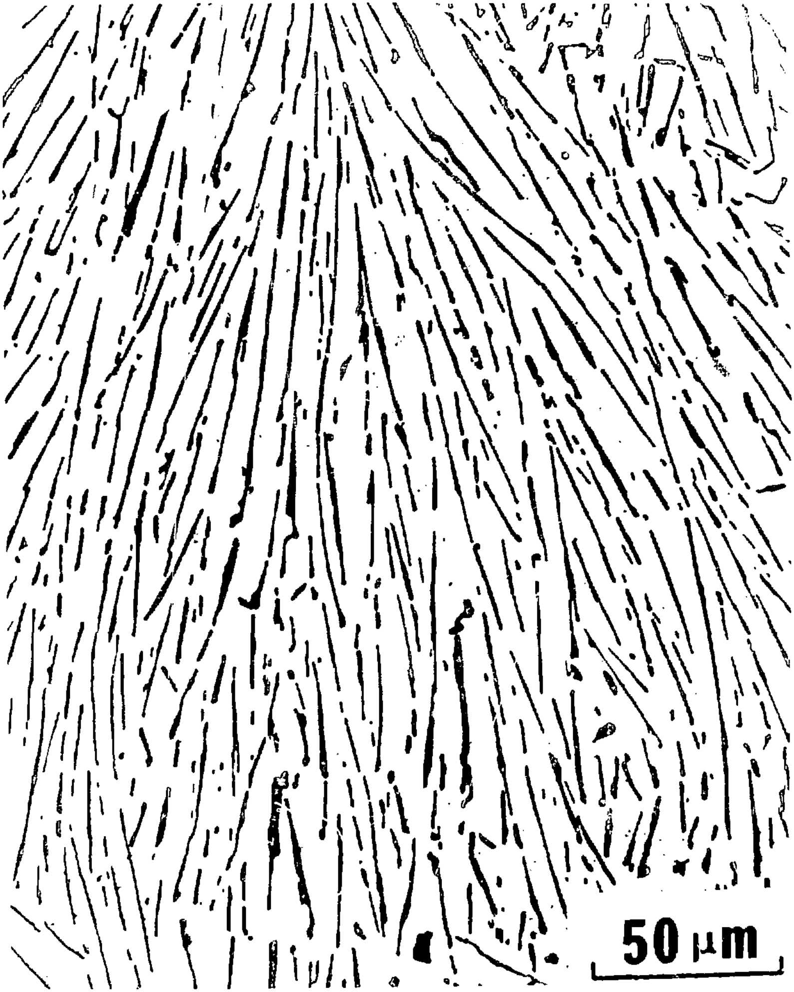


Figure 9.

XBB 799-9415

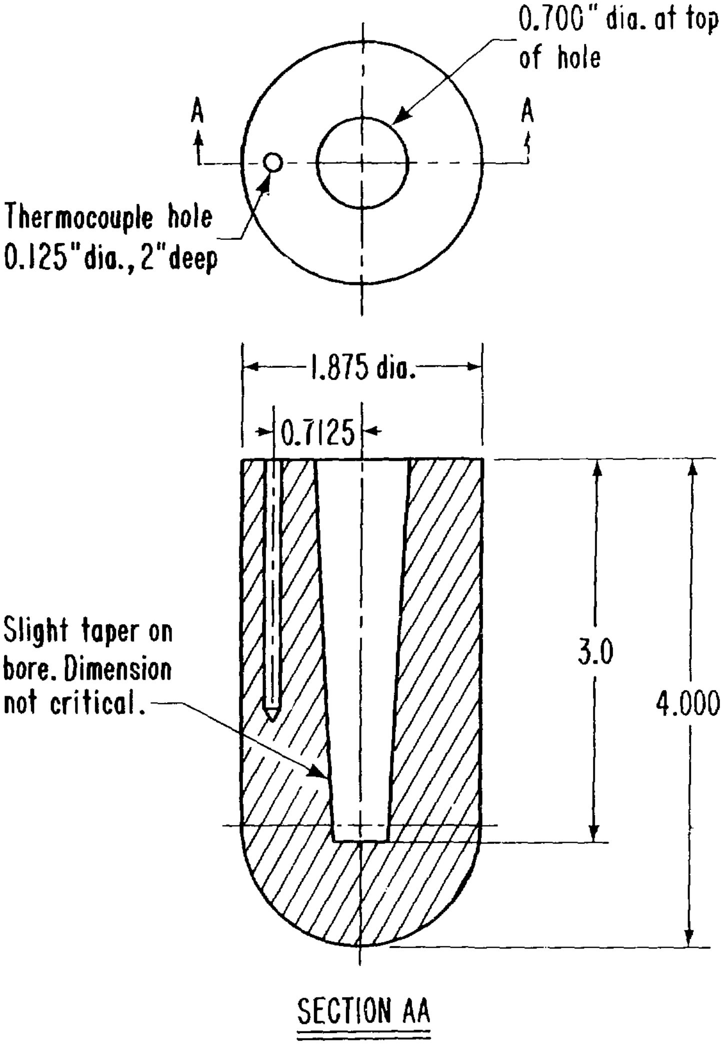


Figure 10.

XBL 7712-11077

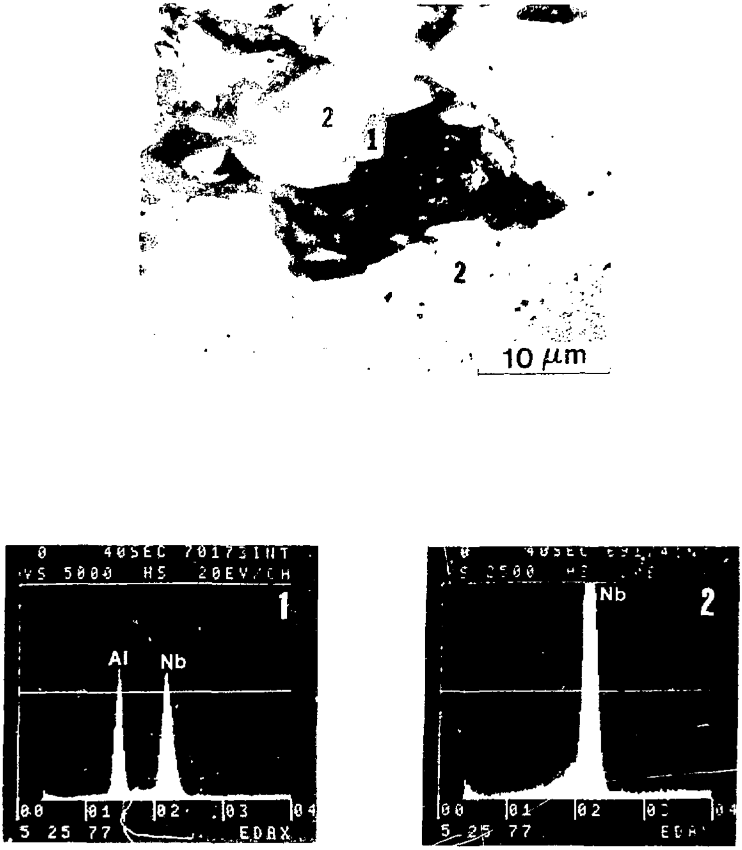


Figure 11.

XBB 770-11724

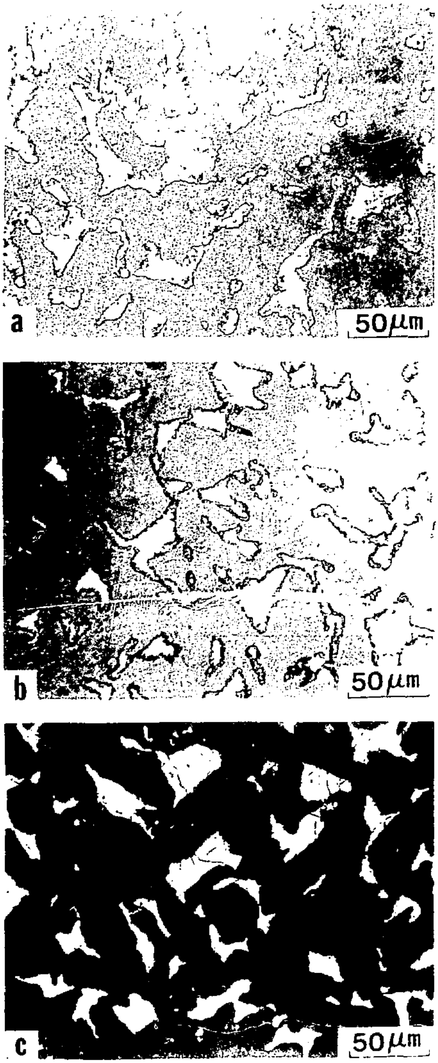
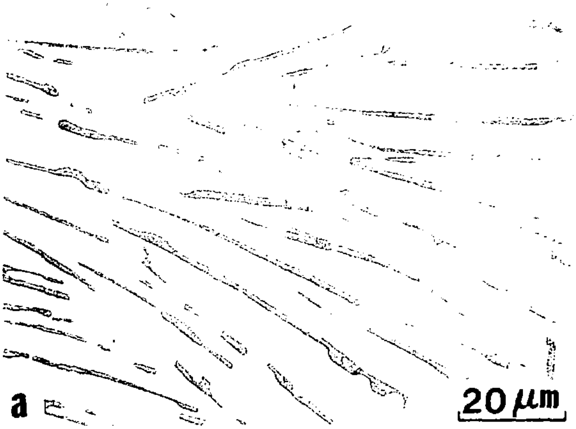


Figure 12.

XBB 760-10591



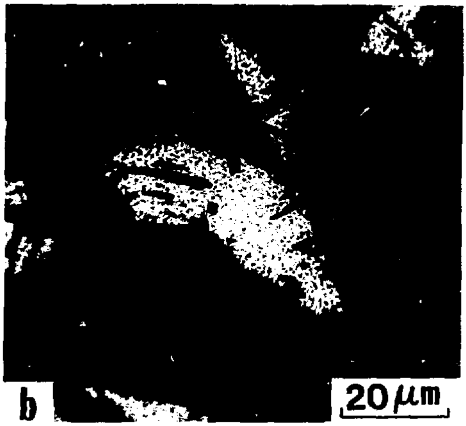


Figure 13. XBB 760-10502

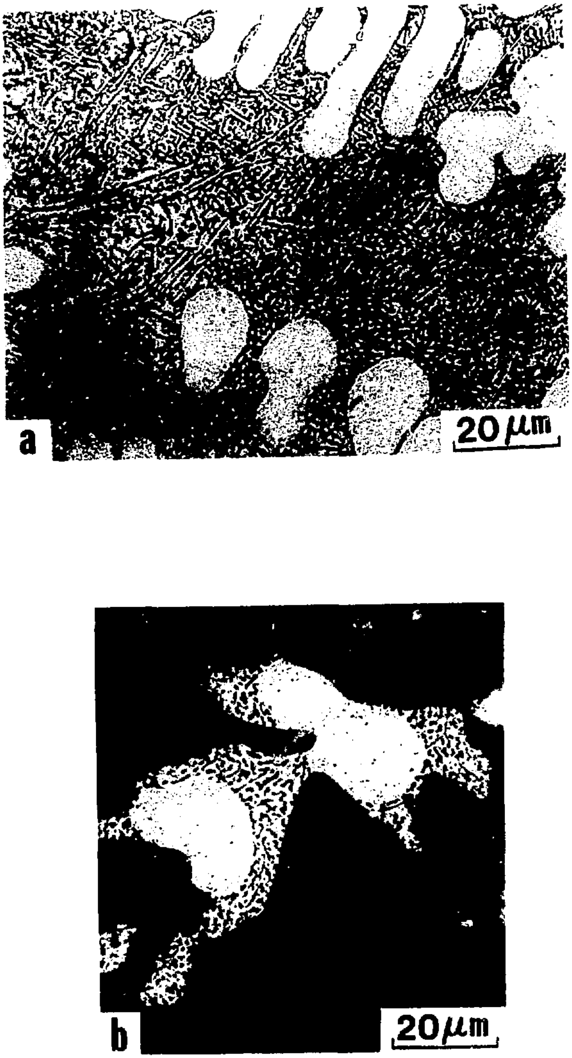


Figure 14. XBB 760-10503