AN INVESTIGATION OF A THERMOPLASTIC-POWDER METALLURGY PROCESS FOR THE FABRICATION OF POROUS NIOBIUM RODS

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AN INVESTIGATION OF A THERMOPLASTIC-POWDER METALLURGY PROCESS FOR THE FABRICATION OF POROUS NIOBIUM RODS

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ABSTRACT

The feasibility of using a thermoplastic-powder metallurgy technique for the fabrication of porous niobium rods was investigated. Some early problems were overcome to successfully extrude the polymer coated niobium powder into long lengths. The effects of certain process variables were investigated. Residual porosity and extrusion pressure were found to be regulated by the polymer fraction. The procedures for taking the extruded polystyrene-niobium rods through the heat treatments to the final, tin infilatrated, stage are explained.

INTRODUCTION

The numerous applications of superconductors are being expanded each year. Socially beneficial areas in which these materials can be used advantageously include energy conversion, mass transit systems, and power transmission lines. The superconducting compounds known should be used to realize their full potential. Currently the conductors having the best properties include Nb_3Sn, Nb_3Al, and Nb_3Ge. These A-15 compounds are brittle and require special fabrication techniques. Since Nb_3Sn was the first high field superconductor discovered, the greatest bulk of work has been done on its development.

A great deal of interest has been taken in a unique powder metallurgy approach[1] to the fabrication of A_3B type superconducting tape and wire. This process involves infiltration of a porous niobium core by submerging it in a liquid bath of the alloying metal (in this case Sn). The advantageous multifilamentary structure is obtained through mechanical deformation. Superior superconductors have been fabricated using this approach.

Long lengths of superconducting wire or tape could be used in many systems. The inherent brittleness of these compounds has raised many obstacles to the production of long lengths. A continuous process that would make possible the production of unlimited lengths would be especially desirable.

The objective of this work was to demonstrate the feasibility of another powder metallurgy approach to a continuous production process. Pickus[2,3] developed a technique to allow forming of powdered materials, metallic or not, as though they were a thermoplastic. He formed many brittle materials by coating the powders with polystyrene, forming the mixture while heated, and finally subjecting the specimen to a timed heating cycle. The heating cycle had basically two stages. The first, lower temperature stage, volatilized the plastic leaving the sample in its as-formed configuration. The second stage sintered the powder to its final density. Some materials, requiring higher sintering temperatures, had to have the second stage divided into two parts. The first part, a partial sintering, would usually be done immediately following and in the same oven as the plastic removal. Partial sintering was required to give the sample enough strength to be handled for mounting in the high temperature, final sintering, furnace. The high temperature sintering usually had to be done in a vacuum while the first part could be done in an inert atmosphere. Equipment considerations dictated that the polymer removal be done in a furnace that was easily cleaned. Materials originally processed by this method include niobium, tungsten carbide/cobalt mixtures and aluminum oxide. This procedure can be controlled to yield specimens having close to theoretical density or a specified amount of residual porosity. Small (0.8 mm, 1/32 in.) spherical fountain pen tips have been formed from ruthenium and platinum alloys using this approach. A sketch of how this concept could be adapted for the continuous production of infiltrated cores is presented as figure 1. Mechanical deformation and further heat treatment would be necessary to make these cores into multifilamentary superconducting wire. The extrusion apparatus used in this work was not of the type shown in figure 1, but was accessible to the author and served the purpose of establishing the feasibility of the process.

HISTORICAL BACKGROUND

Kamerlingh Onnes liquified helium for the firt time in 1908, opening up a new area of low temperature research. It had been thought for many years that metals exhibit less electrical resistance at lower temperatures. In 1911 Onnes took this assumption one step further with an interesting discovery. While working at the low temperatures possible with liquid helium he found that mercury lost all electrical resistance at 4.1°K. This phenomenon became known as superconductivity. Two years later Onnes discovered that lead became superconducting at 7.2°K. He realized the possibility of generating large magnetic fields by constructing solenoids using superconducting materials. He found, however, that small magnetic fields destroyed the superconductivity.

In 1933 Meissner and Ochsenfeld[4] made another surprising discovery. They found that while in the superconducting state a conductor does not allow magnetic lines of flux to penetrate it. This is called the Meissner effect and implies that a current is developed on the surface of the sample which shields the interior from the magnetic field. This condition seemed to correspond to perfect diamagnetism. Meissner added a new parameter for the evaluation of superconductors.

Two discoveries approximately 50 years after Onnes' breakthrough generated new enthusiasm for the study of superconductors. In 1954 Matthias[5] discovered the high critical temperature of the intermetallic compound Nb_3Sn to be 18°K. Later, in 1961, Kunzler et al.[6] found Nb_3Sn to be the first compound to remain superconductive in high magnetic fields. A new era had begun in the study of superconductivity. The disappointment of Onnes had been displaced.

Currently, study of superconductors is done in laboratories all over the world and includes investigation of applications, fabrication processes, and new materials. Applications being considered involve electrical power generation and transmission, internal circuitry of electronic equipment (including computers), and economical production of large magnetic fields for use in levitated transit systems and new energy processes.

High field superconductors having the A-15 structure, like Nb_3Sn, are inherently brittle. Extensive research has been done in the field of conductor fabrication to allow use of these compounds. The most widely used superconductor is an alloy of niobium and titanium (T_c=10°K). This alloy does not have the excellent high field properties of other known superconductors but is ductile and can be formed using conventional techniques. Although Nb_3Sn does not have the highest critical temperature of all known compounds (Nb_3Ge has a T_c of 23°K),[7,8] it is the only conductor of its type for which practical wire and tape fabrication processes exist.

The first usable A-15 superconductor was made using a powder core process[9] at Bell Telephone laboratories. This process involved filling a niobium tube with a mixture of niobium and tin powders and then reducing the tube to the desired wire size. The wire had to be formed into its final shape before the superconducting core was formed by a heat treatment. The necessity of winding the wire before carrying out the diffusion reaction was a serious drawback which, when considered along with the occurrence of magnetic instabilities, led to the abandonment of this technique.

Chemical vapor deposition was used by Hanak[10] to deposit Nb_3Sn on a hot substrate of Hastalloy wire or ribbon. Apparently the economics of this RCA process overshadowed its basic technical soundness and encouraged its developers to seek a new approach.

The General Electric process, described in 1966 by Benz,[11] provided a way to fabricate a superconductor that could be wound after the reaction forming Nb_3Sn was complete. A niobium tape was passed through a tin bath and then heated to the reaction temperature (950°C - 1000°C) to form a thin layer of Nb_3Sn. Layers of copper and stainless steel were sandwiched with those of the superconducting compound to provide stability and mechanical strength. The G.E. diffusion process is the basis of fabrication procedures currently in use for commercial production of flexible superconducting tape.

Tachikawa[12] developed multifilamentary wire in Japan in 1967. Neither the Bell Telephone process or the G.E. process produced multifilamentary superconductors. The value of having many small filaments has been investigated by the Rutherford Laboratory and others. It was suggested that filaments on the order of 10 microns are necessary for intrinsically stable superconductors. Tachikawa's procedure is now known as the "bronze process". The multifilamentary structure was achieved by repeated drawing operations. Originally Tachikawa drilled a number of axial holes in a bronze cylinder and filled them with niobium wire. The bronze cylinder was then mechanically reduced to the desired size and cut into sections. These sections were inserted in holes drilled in another bronze cylinder which was also mechanically reduced. Intermediate annealing was required to prevent cracking of the bronze. The procedure was repeated until the desired size and number of niobium filaments was obtained. A diffusion reaction at 600-700°C then formed Nb_3Sn at the interface of the niobium and bronze matrix. The thickness of the Nb_3Sn layers was normally less than 5 microns. Modifications of this procedure currently provide the basis for commercial fabrication processes.

A new powder metallurgy approach, previously mentioned in this report, has been used at the Lawrence Berkeley Laboratory to fabricate multifilamentary Nb_3Sn superconducting tape and wire. This procedure, which has already undergone extensive investigation and optimization[13,14,15] involved compaction of niobium powder, sintering of the green compact, and infiltration of the porous niobium rod with molten tin. The interconnected tin-filled pores were formed into fine filaments by mechanical reduction prior to a final heat treatment which formed the superconducting phase. Using this process Hemachalam[16] was able to fabricate Nb_3Sn superconducting wire with higher current density at high magnetic fields than commercially available wire.

The economic production of long lengths (hundreds of feet) of A-15 superconducting wire has been a problem. Hemachalam was only able to make 6 meter lengths by reducing 0.48 cm rods. Research was done at the Lawrence Berkeley Laboratory by MacLeod[17] to determine the feasibility of scaling up the existing process used by Hemachalam to produce long lengths of Nb_3Sn multifilamentary wire. It was hoped that by increasing the diameter of the infiltrated cores from 0.48 cm (3/16 in.) to 1.27 cm and 1.90 cm (1/2 in. and 3/4 in.) it might be possible to produce longer lengths through mechanical reduction. Hydrostatic extrusion and wire drawing were investigated as possible means of reduction. Many problems were encountered and further evaluation was required.

EXPERIMENTAL PROCEDURE

Niobium-tin superconducting wire has been successfully fabricated by the infiltration process previously described. It was envisioned that if continuous lengths of porous niobium cores of the same diameter (0.48 cm) could be made by other means, then the established procedures could be followed to process these cores into multifilamentary superconducting wire. With this in mind it was decided to use a 0.48 cm extrusion die in the powder-thermoplastic process previously mentioned. The specific purpose of this project was therefore to investigate the problems associated with extruding a niobium rod 0.48 cm (3/16 in.) in diameter with a suitable porosity fraction.

This polymer-powder metallurgy process involves four steps to go from a powder to an infiltrated niobium rod. These steps are: 1) coating the powder with a suitable thermoplastic (in this case polystyrene); 2) extrusion of the coated powder into a rod of specified diameter; 3) heat treatment to remove the plastic and sinter the niobium; 4) infiltration of the rod with tin to fill the network of inter-connected pores. The heat treatment was broken up into two stages. A low temperature (up to 1300°C) treatment done in an argon atmosphere was intended to volatilize the polystyrene and sinter the niobium enough to allow its handling. The second stage of the heat treatment was done in a vacuum (10^{-5} mm Hg) furnace. While suspended in this evacuated chamber, the porous niobium rods were heated to 2200-2250°C for several minutes. This high temperature sintering gave the rods strength and ductility. Fabrication of A-15 phase superconducting wire from these cores would involve tin infiltration, mechanical reduction, and another heat treatment.

I. Coating the Powder With Thermoplastic

The thermoplastic was chosen on the basis of its overall properties. Oxides and contaminating residue formed when vaporizing the polymer had to be kept at minimal levels. In view of previous work, polystyrene, a pure hydrocarbon, was chosen as the thermoplastic best suited. Extrusion was found to require less pressure if the polystyrene was modified by the addition of a plasticizer. The plasticizer used was an eutectic mixture of diphenyl and diphenyl ether (27% diphenyl and 73% diphenyl ether). To facilitate its addition the polystyrene was prepared as a 3% solution in toluene. To allow the proper weight of plasticizer to be measured as a volume its specific gravity was determined (0.936 cc/g). Fifty grams (quantity limited by the dimensions of the extrusion apparatus) of -400 mesh niobium powder was weighed out on a dial balance. (All weight measurements were made using the same dial balance which was accurate to ± 0.0005 grams.) The niobium powder and the desired volume of polystyrene-toluene solution were mixed in a pyrex container. The polystyrene fraction was calculated as follows:

$$\frac{\text{W/o polystyrene}}{100} = \frac{\text{Wt. polystyrene}}{\text{Wt. poly.} + \text{Wt. niobium}}$$

The effects of altering the thermoplastic fraction were investigated. Earlier work suggested a starting point of 7 1/2 W/o polystyrene. Originally many different metals were formed by this method. One of the first materials tried was an alloy of ruthenium and platinum which required a polystyrene fraction equal to 51/4% of the total weight of the mixture. It was observed that when changing substances (assuming the powder mesh to be in the same range) good results were obtained if the percentage of polystyrene was increased inversely with the specific gravity of the material.

A graduated cylinder was used to measure out the desired volume of polystyrene-toluene solution. A pyrex dish was used to hold the mixture of solution and niobium powder over a hot plate while it was continuously hand-stirred. The entire evaporation process was carried out under a hood to provide protection from the toxic toluene vapors. When there was just a little solution left the desired amount of plasticizer was added to the mixture using a 1 cc glass syringe. The weight of the plasticizer used was 1/5 the weight of the polystyrene. Plasticizer was added at this late stage to prevent excessive loss during evaporation. A short while later the mixture became rubbery making further stirring ineffective. The material was then placed in an oven at 115°C for one hour to remove all residual solvent. At this stage the substance was hard and had to be cut into small chunks to effectively fit into the extrusion apparatus cylinder. Figure 3 (opposite the long, as extruded, rod) shows the mixture just before it was placed in the cylinder.

II. Extrusion

Extrusion of the powder-plastic mixture was done at low temperature (165°C) and pressures below 27 ksi. The apparatus used was a die set and heater of the type often used for mounting specimens for metallographic work. A photograph of this equipment is shown in figure 2. An extrusion die has been substituted for the base plug and a support has been added to position the die toward the center of the die cylinder where temperatures were more accurately monitored. A voltage regulator was used between the heater and the power supply to keep the temperature between 160°C and 170°C. The temperature was measured by a thermocouple placed in a well which had been drilled in the die plunger (fig. 2).

The extrusion apparatus was positioned in a hand-operated hydraulic press with a maximum fluid pressure of 3000 pounds and a cylinder diameter of 3 inches. Since the extrusion cylinder had a diameter of 1 inch, the maximum available pressure was 27 ksi. When the thermocouple indicated 165°C had been reached, the powder-plastic mixture was placed in the extrusion cylinder. The coated powder was allowed to heat for at least five minutes to insure its uniform temperature. Pressure was exerted until extrusion was complete. Once the material began passing through the die the pressure remained constant until all of it had been extruded. Mixtures with 7 1/2% polystyrene required the maximum pressure of 27 ksi. An example of a specimen after extrusion is shown in figure 3. The length of the rod was limited by the capacity of the die cylinder. After forming, the rod was allowed to cool while hanging from the die.

III. Sintering

As mentioned previously, the sintering procedure was done in two stages. The first stage was intended to remove the polymer and leave a pure niobium rod with enough strength to be handled and mounted in the high temperature, vacuum, furnace. A horizontal mullite tube furnace (figure 4) with an argon atmosphere was used to accomplish the polystyrene vaporization and partial sintering of the niobium. The inert gas was circulated through the tube to carry away the contaminating vapors. To prevent pollution of the laboratory, the fumes leaving the gas tight tube were passed through a series of oil baths. Since the samples went through a period in the heating cycle when they lacked enough strength to support their own weight they were laid in a tantalum support. The dimensions of the hot zone limited the length of these supported sections to about 13 cm (5 in.). The heating cycle was as follows: room temperature to 400°C in approximately one hour; held at 400°C for one hour to vaporize the polystyrene; raise the temperature to 1300°C over a period of 30-45 minutes; kept at this temperature of 1300°C for one hour to partially sinter the niobium. After being subjected to this heat treatment the rods appeared uniform, straight, and had enough strength to be mounted for final sintering at over 2000°C. Figure 5 shows a sample after this first stage of heat treatment.

The second stage of the sintering procedure was done in an Abar furnace (fig. 6). Tantalum wire was used to hang the niobium rods vertically in the hot zone which was long enough to sinter samples of about 5 cm (2 in.). Sintering was done at a temperature of 2200°C to 2300°C for 15 minutes at a pressure of 10^{-5} mm Hg.

IV. Infiltration

Infiltration of the porous niobium rods was done in the Abar furnace immediately after the high temperature sintering operation. After sintering, the samples were allowed to cool to the temperature of the molten tin bath (650°C to 700°C) before being submerged. The pressure inside the furnace was raised from 10^{-5}mm Hg to just below atmospheric after the specimens were completely immersed. Infiltration time was one minute.

V. Metallography

The niobium-tin samples were mounted in bakelite and polished for optical microscope viewing. An anodic oxidation process was used to identify the niobium and the tin and some of their intermetallic compounds. Niobium turned light blue, tin became yellow, Nb_3Sn showed as dark blue or violet, and Nb_6Sn_5 was distinguished by its reddish brown color.

RESULTS AND DISCUSSION

Early attempts at extrusion of niobium-polystyrene rods failed. Either short lengths would extrude or no material at all would pass through the die. It appeared as though the mixture would be compressed into a solid piece when the material began to flow. Raising the extrusion temperature and/or the thermoplastic fraction did not help. These first experiments were done using a powder of -270 mesh. It was found that when finer powder of -400 mesh was used, the extrusion proceeded well. A comparison of the pore configuration was made between two samples, both containing 7 1/2 W/o polystyrene, made with these different mesh powders. Figure 7A shows a section of the specimen prepared using -270 mesh niobium powder (as received). Figure 7B is a photograph of 7 1/2 W/o polymer specimen made with the finer, -400 mesh powder. The sample made with the coarser powder seemed to have a wider range of pore sizes and the average volume of a single pore appeared larger. All of the data reported in this paper was taken from samples prepared with -400 mesh niobium powder.

After successfully fabricating the porous niobium rods through this extrusion process, the objective of this work became to see if the porosity (and therefore the relative amount of tin) could be controlled by altering the polymer fraction. Also of interest was the effect raising the plastic content had on the extrusion pressure. The benefits of adding plasticizer were evaluated by preparing all mixtures with and without.

Several samples were prepared and extruded. The data has been plotted graphically in figures 8 and 9. The polymer fraction was varied by 1/2 W/o and each mixture was prepared with and without plasticizer. It can be seen that the addition of plasticizer caused a significant reduction in the pressure required for extrusion while having little effect on the porosity. Figure 9 suggests that the pore volume fraction was controlled by the polymer content as was the extrusion pressure (figure 8). A linear relation seemed to exist in both cases. Each sample extruded to its full length (limited by the capacity of the extrusion apparatus cylinder) with enough strength to support its own weight while hanging from the extrusion die as it cooled. The capacity of the extrusion press limited the minimum polystyrene fraction that could be investigated. Samples having 7 1/2 W/o polymer required the maximum pressure of 27 ksi even when they were mixed using plasticizer. When specimens were prepared without plasticizer, at least 8 1/2 W/o polymer was required for extrusion using less than the maximum pressure.

Figures 10 and 11 were included to show the structure of a typical specimen as it was taken through the three basic stages of processing. The first stage might be called the 'as extruded' condition. Figures 10A and 11A show the arrangement of the niobium powders within the polystyrene after extrusion. Both of B figures show the structure of the partially sintered pure niobium rods after the first stage of the heat treatment. These rods have been infiltrated with resin to allow polishing. Stage three was considered as the final condition of fully sintered niobium rods infiltrated with tin (figs. 10C and 11C). These photographs show that some Nb-Sn intermetallics formed during the infiltration process.

A portion of the cross sectional area measuring approximately 0.25 cm by 0.18 cm (in the center of 0.48 cm diameter rod) is presented in figures 12 through 15. These photographs were prepared to allow an estimation of the overall pore density and size distribution. The porosities listed to the right of the photographs were measured by comparing the weight in water of resin-infiltrated niobium cores with their weight in air before infiltration. With the exception of some large pores the cross sections generally exhibited even pore distribution.

CONCLUSIONS

Extrusion of thermoplastic-coated powders is a feasible approach to the production of porous rods. The extrusion pressure requirements can be low enough to be within the limits of continuous extrusion equipment. Porosity fractions seem to be controlled by the amount of polystyrene and can be predicted from a linear relationship. The range of porosities obtained included values that would allow efficient conversion of niobium and tin into Nb_3Sn.

It appears that the maximum powder size is an important parameter in this process. Possibly the range of powder sizes should also be considered. Further investigation might well include a series of experiments where only the powder size was varied.

This process may be a good way to produce other porous pieces. Sample batches that did not extrude but were compressed at 27 ksi exhibited uniform pore distribution. Many parts such as filters could be effectively manufactured by this coated-powder procedure.

It is also envisioned that porous tubes could be fabricated through this extrusion technique. If an effective way of supporting the tubes during the polymer volatilization could be devised this may be an advantageous method of producing hollow shapes. It has been suggested that when fabricating superconductors using the infiltration process a core of copper may be beneficial during wire drawing as well as enhancing the performance of the conductor.

These experiments have provided a starting point and a reason for further investigation of the application of this polymer-powder process to the fabrication of porous rods. Parameters such as materials used on extrusion friction surfaces, extrusion die geometry, polymers used to coat the powders, and coating techniques are a few areas where refinement could be rewarding.

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REFERENCES

1. M. R. Pickus and J. L-F. Wang, Paper presented at the International P/M conf. 1976, LBL 5121, May 1976.
2. M. R. Pickus, Tips for Fountain Pens-A Case History on the Adaptability of the Powder Metallurgy Process, Int. Journal of Powder Metallurgy 5 (3), 1969.
3. M. R. Pickus, M. Wells, A Technique for Extrusion Forming of Brittle and Refactory Compositions, Powder Metallurgy, Vol. 8, No. 16, 1965.
4. W. Meissner and R. Ochsenfeld: Naturwiss 21, 787 (1933).
5. B. T. Matthias, T. Geballe, S. Geller, E. Corenzwit, Phys. Rev. 95, 1435 (1954).
6. J. E. Kunzler, E. Buchler, F. S. L. Hsu and J. H. Wernick, Phy. Rev. Lett. 6, 89 (1961).
7. J. R. Gavaler, Appl. Phys. Letters 23, 480 (1973).
8. L. R. Testardi, J. H. Wernick, W. A. Royer, Solid State Comm. 15, 1 (1974).
9. J. E Kunzler, E. Buchler, F. S. L. Hsu and J. H. Wernick, Phys. Rev. Lett. 6, 89 (1961).
10. J. J. Hanak, K. Strater and G. W. Cullen, RCA Reviews 25, 342 (1964).
11. M. G. Benz, IEEE Trans. on Magnetics, Vol. Mag. -2 No. 4, 760 (1966).
12. K. Tachikawa and S. Fukuda, Trans. Nat. Research Inst. Metals 9, 39 (1967).

13. B. N. P. Babu, M.S. Thesis, LBL-437, December 1971.

14. T. N. Garrett, M.S. Thesis, LBL-448, December 1971.

15. K. Hemachalam, M.S. Thesis, LBL-1108, June 1972.

16. K. Hemachalam, D. Eng. Thesis, LBL-4181, February 1976.

17. G. E. Macleod, M.S. Thesis, LBL-6622, June 1977.

FIGURE CAPTIONS

1. Schematic diagram of the infiltrated rod fabricating process.

2. Photograph of the extrusion apparatus. From left to right: heater; plunger, with thermocouple well drilled in the center; cylinder; extrusion die; die positioning piece.

3. Photograph of the as-mixed (above scale) and as-extruded (below scale) polystyrene-niobium material.

4. Schematic diagram of the tube furnace used for polymer removal and partial sintering.

5. Photograph of a length of pure niobium rod after the polystyrene removal and partial sintering.

6. 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 tin, and (14) resistance heater.

7. (a) an as-infiltrated extrusion: prepared with 7 1/2 W/o polystyrene, -270 mesh niobium powder; final sintering at 2200-2300°C for 15 minutes; tin infiltration done at 650-700°C for one minute with a preheated niobium rod. (b) same as sample described in 7a except prepared using -400 mesh powder.

8. Extrusion pressure vs W/o polystyrene for samples prepared with/without plasticizer.

9. Percent volume fraction porosity vs W/o polystyrene for samples prepared with/without plasticizer.

10. Photographs of a sample prepared using 7 1/2 W/o polystyrene. (a) after extrusion (dark areas are polystyrene, lighter areas are niobium). (b) after the first stage of the heat treatment procedure (polymer removal and partial sintering). The dark areas are infiltrating resin used to facilitate polishing. The lighter areas are niobium. (c) a niobium-tin as-infiltrated extrusion.

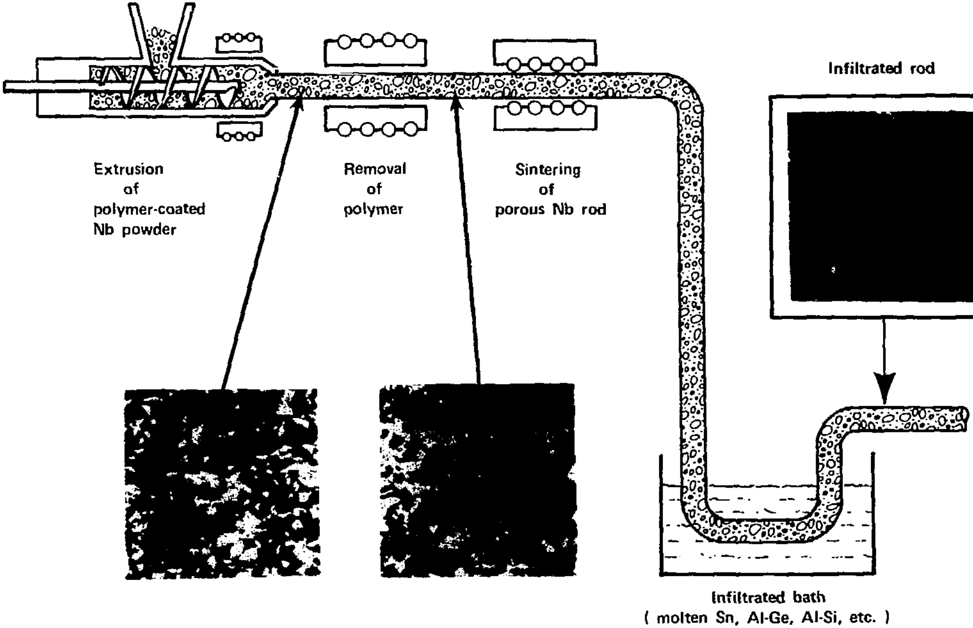
11. Photographs of a sample prepared using 8.0 W/o polystyrene. (a), (b), and (c) same as described in 10a, 10b, and 10c.

12. Niobium-tin as-infiltrated samples prepared using plasticizer.

13. Niobium-tin as-infiltrated samples prepared using plasticizer.

14. Niobium-tin as-infiltrated samples prepared without plasticizer.

15. Niobium-tin as-infiltrated samples prepared without plasticizer.



XBB779-8861-A

A CONTINUOUS PROCESS FOR
PRODUCING INFILTRATED Nb ROD FOR FABRICATING MULTIFILAMENTARY SUPERCONDUCTING WIRE

Fig. 1

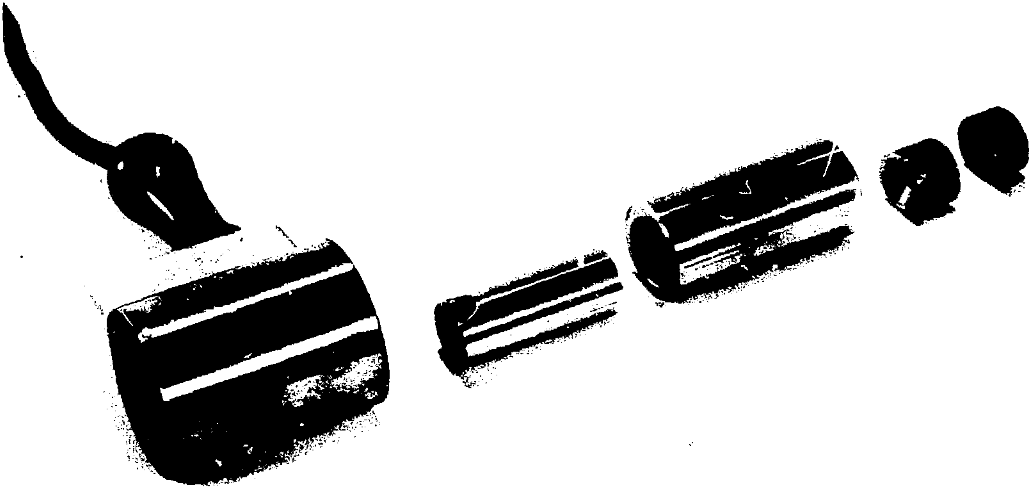


Fig. 2

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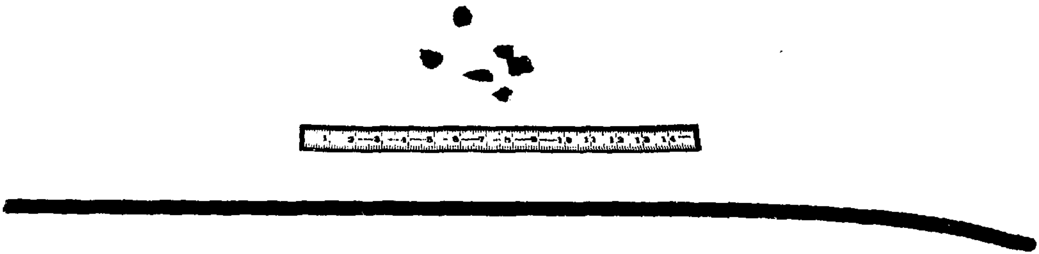

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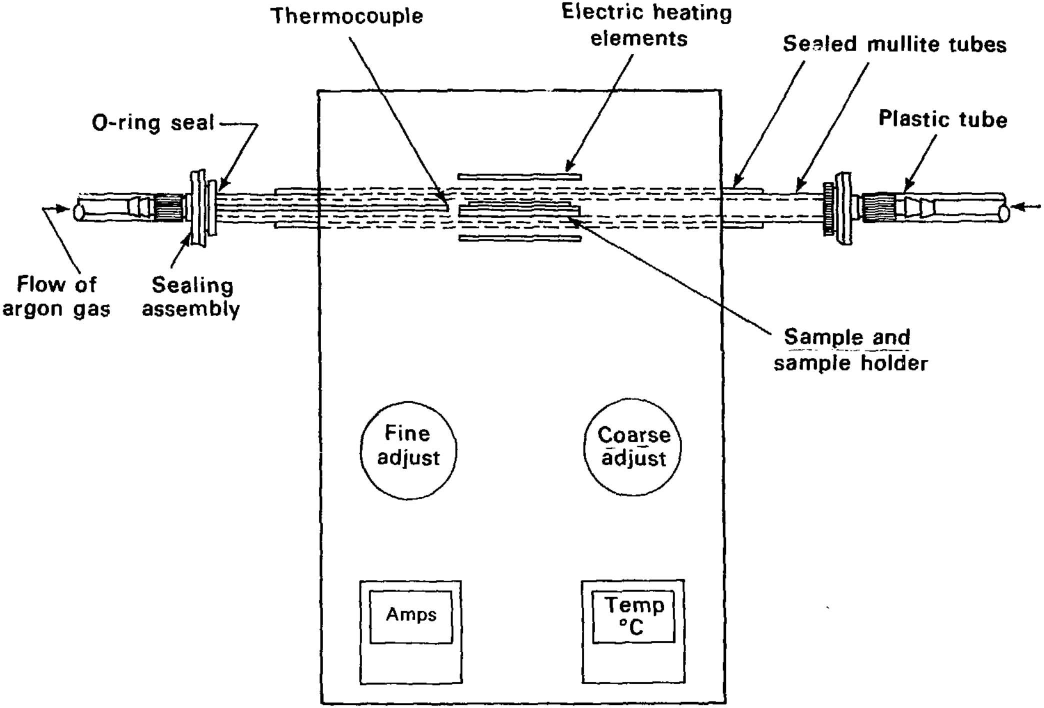
Fig. 3



XBL 781-4

Fig. 4

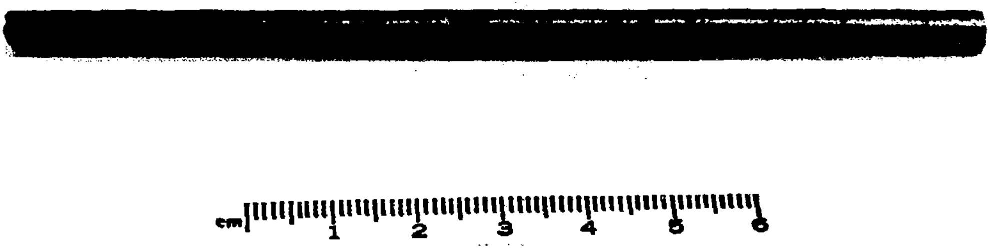


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Fig. 5

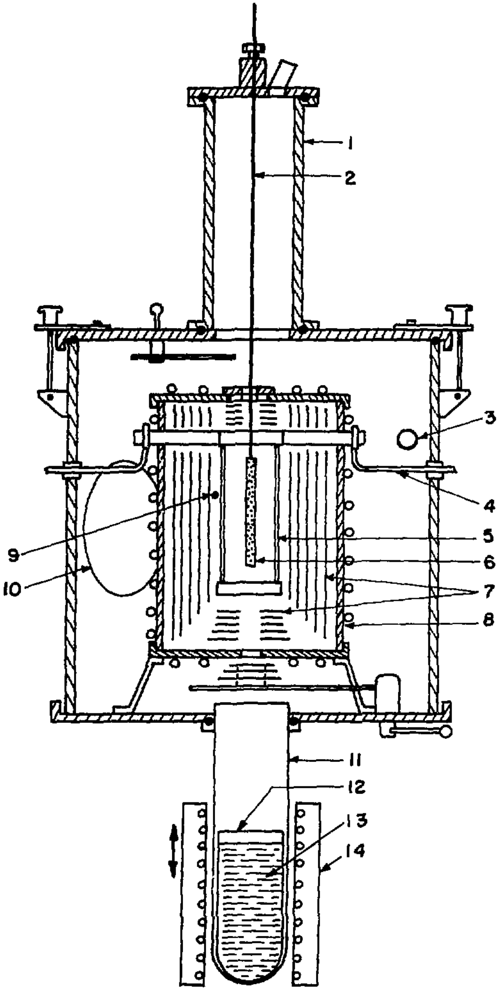

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Fig. 6

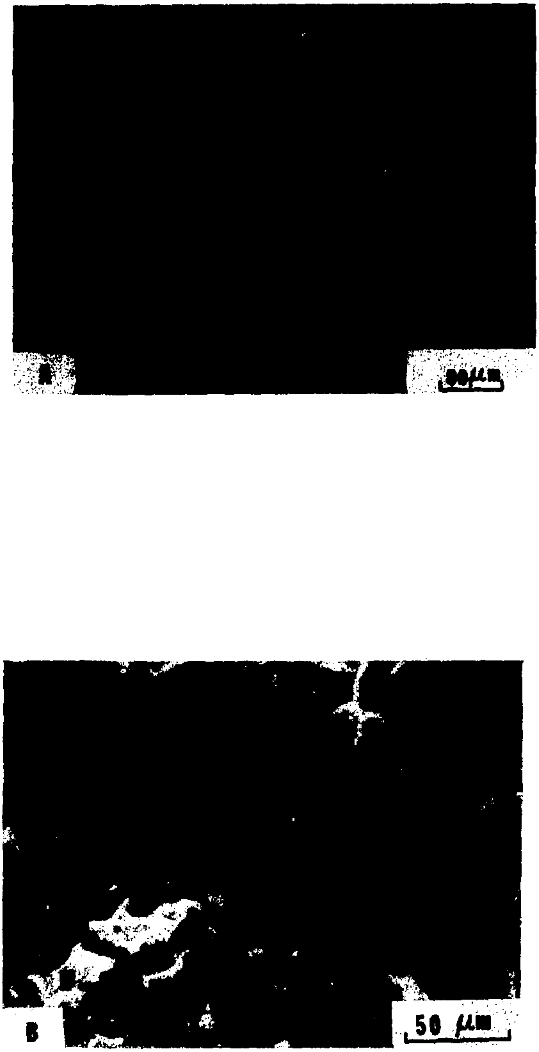

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Fig. 7

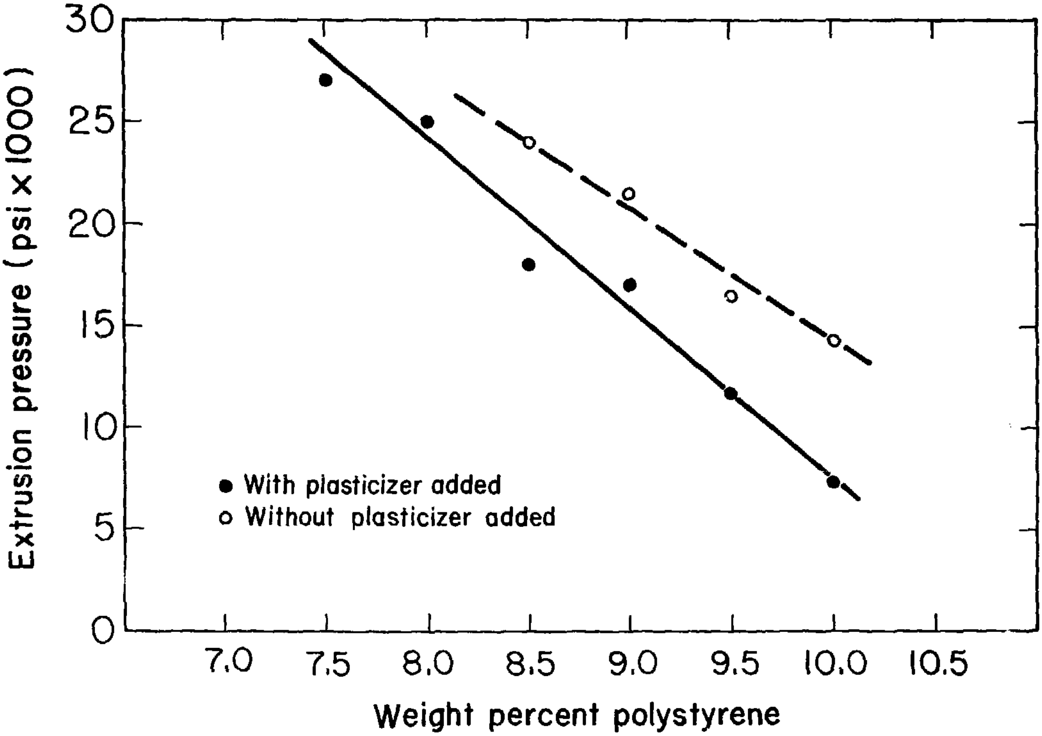

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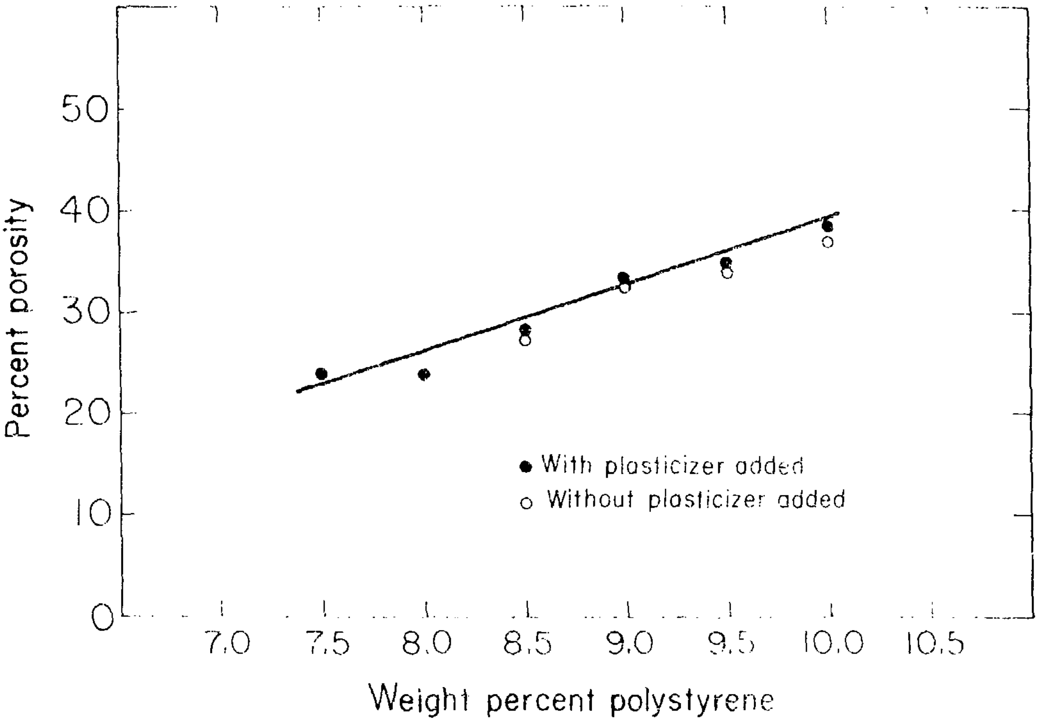
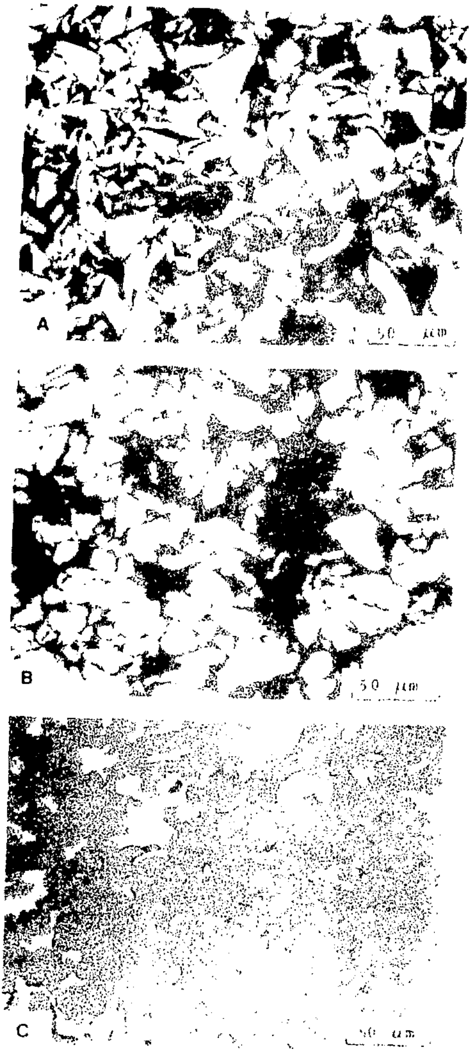
Fig. 8



XBL 781-6

Fig. 9

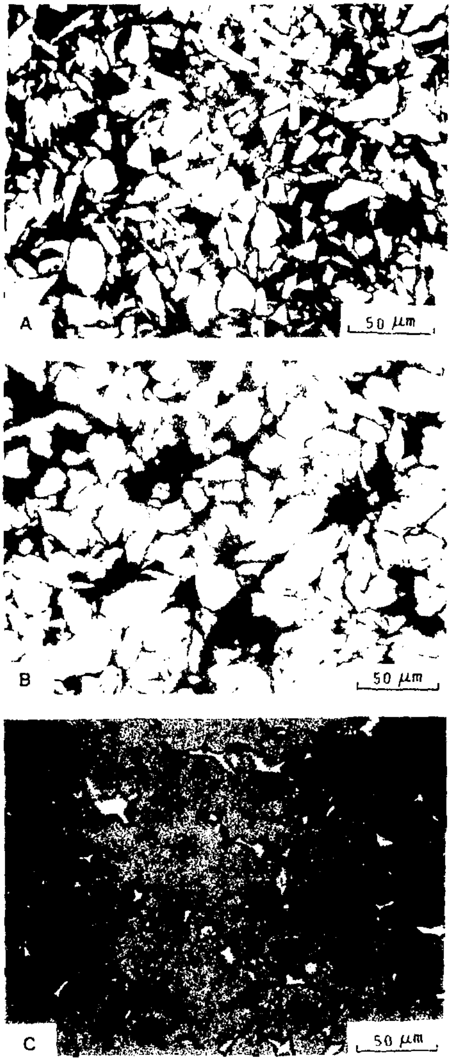
7.5 %



YBF [illegible]

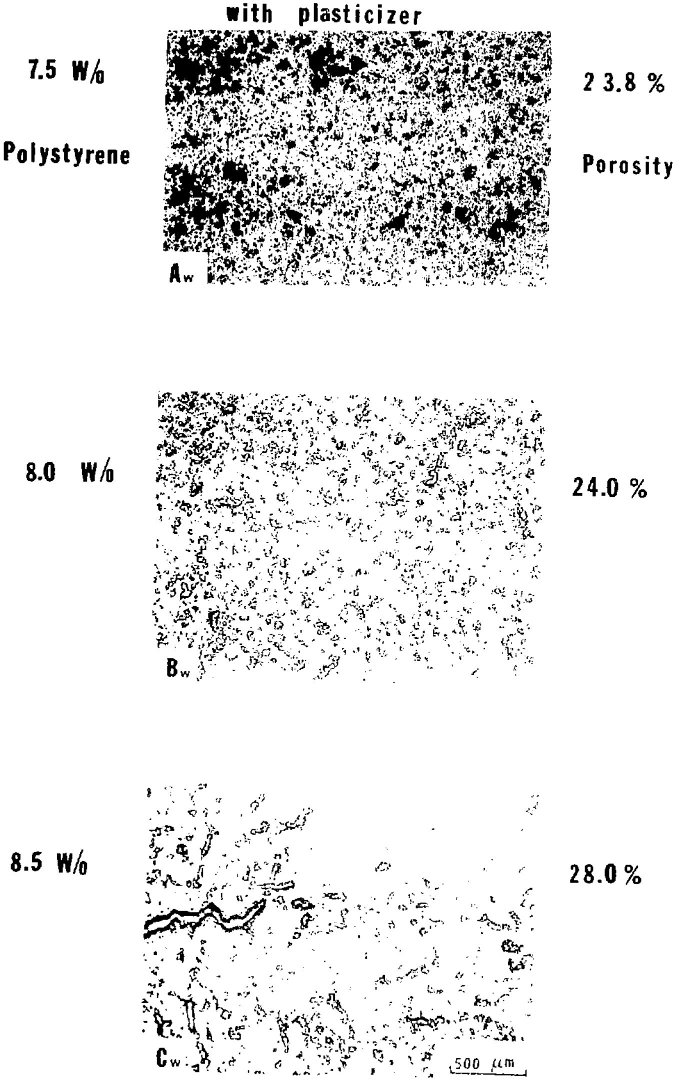
[illegible]

8.0 %



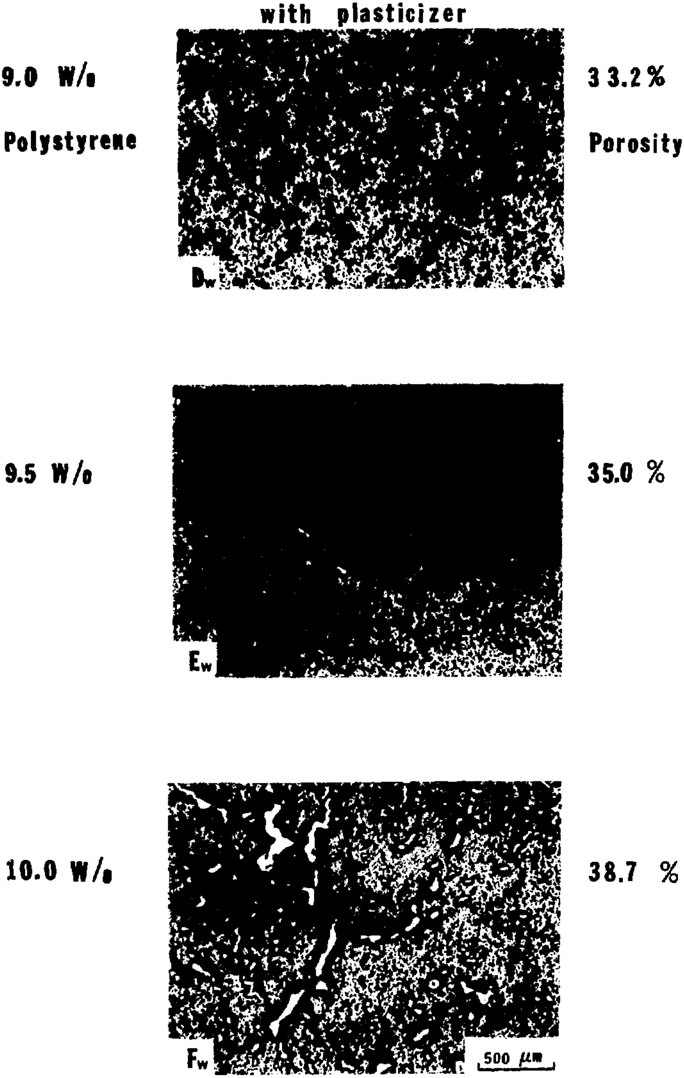
XBB770-11517

Fig. 11



XBB770-11930

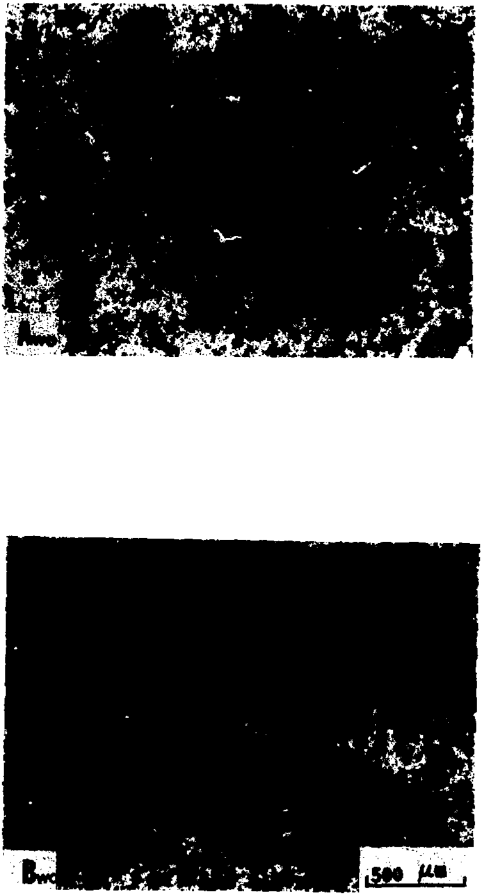
Fig. 12



XBB770-11927

Fig. 13

without plasticizer



XBB770-11929

Fig.14

without plasticizer

9.5 % **34.5 %**

Polystyrene **Porosity**

10.0 % **37.0 %**

XBB770-11928

Fig. 15