Technical Report on

Manufacture of Nb_3Sn Superconducting Wire

by Cold Hydrostatic Extrusion

By

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Introduction

Because of the prominent superconducting properties (T_c, J_c and H_{c2}) of Nb_3Sn wires, their development is being actively pursued in many laboratories in this country and abroad.

One of the most common methods of manufacturing Nb_3Sn multifilamentary superconducting wires is the conventional 'bronze' technique [1], in which the first step involves assembling a number of niobium cores in a bronze sleeve. This assembly is conventionally hot extruded to a smaller cross-section, then drawn and annealed numerous times to form rods. These rods in turn are re-assembled to form a large billet which is again conventionally hot extruded and drawn. Re-assembly and reduction are carried out alternately until a rod with the required number of filaments is obtained. This rod is then drawn to produce a suitable multifilamentary fine wire. Numerous intermediate anneals are required between the passes. Finally a diffusion anneal is done to produce superconducting Nb_3Sn at the niobium-bronze interface.

Since this conventional 'bronze method involves a number of hot formings at approximately 750°C (1380°F) and a number of intermediate anneals at 450-500°C (840-930°F), there is considerable premature diffusion at the bronze-niobium interface leading to the formation of brittle Nb_3Sn. With subsequent deformation micro-fissures may be formed in the brittle Nb_3Sn layer, thus causing a degradation of superconducting properties. Furthermore, in wire drawing the reduction obtainable per pass is quite small.

The process of cold hydrostatic extrusion is an alternative method of manufacturing Nb_3Sn superconducting wire [2]. A group at Battelle [3] has evaluated the feasibility of converting existing conventional extrusion presses to <u>hot</u> hydrostatic extrusion presses capable of extruding 3000 lb. billets. They have also successfully <u>hot</u> hydrostatically extruded multifilamentary Nb_3Sn wires.

On the other hand, Breme and Massat [4] have produced Nb_3Sn wires by a combination of cold hydrostatic extrusion, drawing and forging. Their results indicate that cold hydrostatic extrusion in a suitable process for manufacturing Nb_3Sn wires.

Despite the research being carried out in this field, there has been no direct comparison between cold hydrostatically extruded Nb_3Sn wires and drawn Nb_3Sn wires, in an effort to evaluate the metal forming capabilities of the two processes and to determine the effect of the process itself on the final superconducting properties of the wire.

The objective of this research program is to manufacture monofilament Nb_3Sn superconducting wires by hydrostatic extrusion and thereby study the capabilities and limitations of the process. A direct comparison between hydrostatic extrusion and drawing as a method of producing Nb_3Sn monofilament wires is also intended.

Hydrostatic Extrusion

As mentioned earlier, conventional extrusion involves a number of hot formings at 750°C. As compared to conventional extrusion, hydrostatic extrusion can be performed at room temperature, thus minimizing the danger of premature formation of brittle Nb_3Sn.

Furthermore, in conventional ram extrusion the ram pressure is higher than the extrusion pressure at the die exit. This pressure rise causes frictional resistance along the walls of the chamber. However, in hydrostatic extrusion the billet does not touch the walls of the chamber, and wall friction can be eliminated to permit the extrusion of long billets. In the extreme, excessive lengths of wound wire can be held in relatively small diameter chambers and then be hydrostatically extruded.

Lubrication is of great importance in any metal forming operation. In hydrostatic extrusion good lubrication, which sometimes develops into hydrodynamic lubrication between the die and billet, makes it possible to use dies of small angles that in turn reduce the redundant deformation and the tendency of billets to shear along the die face.

In comparison to wire drawing, hydrostatic extrusion makes larger reductions per pass possible, because the billet is deformed by compressive stress rather than by tensile stresses that are prevalent in wire drawing. With larger reductions the number of required passes and the number of intermediate anneals are minimized, thus again reducing the danger of premature diffusion and its consequent embrittlement. Furthermore, brittle substances tend to be quite ductile under hydrostatic pressure, making it possible to obtain even larger reductions without fracture. This high hydrostatic pressure also causes internal voids and pores to close, thus leading to a sounder product than the one obtained by wire drawing.

The hydrostatic processes do have limitations, however. One of the major difficulties associated with hydrostatic extrusion is inherent in the process and stems from its basic concept, namely the replacement of the ram push by liquid pressure. Liquid is compressible and thus the positive ram push in the process of ram extrusion is replaced during hydrostatic extrusion by a push with a spring. Positive speed control is lost and the likelihood of the stick-slip phenomena has to be faced. Other problems associated with the use of fluid as a pressure medium are the creation of high pressure, sealing problems and changes in volume and viscosity of the fluid under pressure. Despite these limiations the hydrostatic extrusion of Nb_3Sn superconducting wires holds great promise.

Experimental Procedure

a) Material Specifications

Bronze: The Bronze used to form the outer sleeve of the billet was a Cu-13%Sn alloy that had been cast in rod form, homogenized and then swaged down to a 0.375" diameter rod. At this stage it had a hardness of 102 R_B.

The rod was held at 760°C (1400°F) in air for 1 hour and then quenched in water to give a hardness of 45 R_B. This was the optimum heat treatment because a shorter annealing time leads to a high hardness, which allows only a small reduction, and a longer time produces excessively large grains without a compensating decrease in hardness.

It was noted that furnace cooling of samples would lead to excessively large grains and a relatively high hardness. The increase in hardness was due to the precipitation of the ε and δ phases on slow cooling (see the Cu-Sn phase diagram in Fig. 1). Quenching from 760°C (1400°F) would keep the ε and δ phases in solution and thus decrease the hardness.

Niobium: (99.85% purity - supplied by the Kawecki Berylco Industries). Niobium rods to be used for the core were swaged down to the required diameters and then vacuum annealed ($\sim 10^{-6}$ torr) for 4.5 hours at 1200°C (2190°F).

b) Equipment

The Model 6 Monoblock Fluid Extrusion Unit installed at the Institute for Metal Forming, Lehigh University, was used to hydrostatically extrude the monofilaments. Figs. 2 and 3 show the Model 6 Extrusion Unit. This hydrostatic extrusion unit has a chamber that is 14" in length and an inner diameter of 0.5". It can attain a maximum safe pressure of 200 ksi. To contain such high pressures the Bridgman seal and the mitre ring with O-ring seal were used. Figure 4 shows the functioning of these high pressure seals.

The motive fluid used in the hydrostatic chamber was 'Crisco' vegetable oil, which did not degenerate at pressures of 200 ksi and thus was reusable. Either candle wax or beeswax was used as the sealant. The cone or tapered end of the billet was dipped into the molten wax to deposit a thin layer of wax on the cone, which then helped form an initial seal between the billet and the die.

The compressive load on the ram was applied by the 60,000 pound Baldwin Universal Testing Machine. A deflectometer was connected to a strip recorder that plotted the ram displacement versus the applied load. An overall view of the equipment used for hydrostatic extrusion is given in Fig. 5.

c) Assembly of Billets

A 1.5" bit was cut from the heat treated 0.375" bronze rod. One end of the rod was machined to give a cone that matched the die while a hole was drilled through the center of the other end, up to a depth of 1". The hole was thoroughly cleaned with alcohol.

Next the niobium rod to be inserted was polished with fine (#600) emergy paper and cleaned with alcohol. This rod was inserted into the bronze sleeve to give a tight fit, and then the protruding end of the niobium rod was ground off to give a flat end.

Finally, the billet was capped with a flat copper cap using an epoxy resin that could withstand high pressures. The cap prevented the compressed liquid from flowing into the niobium-bronze interface and also prevented the niobium from being squeezed out from the back. (Fig. 6).

Results and Discussion

Data on the hydrostatic extrusion of bronze clad niobium monofilaments are given in Table 1, which gives the dimensions of the original billets, and in Table 2, which gives the extrusion pressures and reductions obtainable per pass. Figure 7 shows the hydrostatically extruded bimetallic product.

To produce the specified monofilament wire, a 99.6% reduction in area was required. As Table 2 shows, use of the Model 6 fluid extrusion unit allowed for a 40-50% reduction in area per pass. In addition, on the average an intermediate annealing was needed after a 75% reduction in area.

Compared to this hydrostatic process, the drawing of bimetallic niobium-bronze wires commonly involves a 30% reduction in area per pass in the early draws and 20% or less as the wire becomes thinner. Intermediate anneals have to be done after every 2 or 3 draws to prevent fracture during drawing. This is equivalent to an intermediate anneal after a reduction in area of about 45%.

The data given above indicate that hydrostatic extrusion of Nb_3Sn superconducting monofilament wires requires fewer passes and fewer intermediate anneals than wire drawing. Fewer intermediate anneals imply that the danger of premature formation of brittle Nb_3Sn in minimized. Further, the use of hydrostatic extrusion to produce Nb_3Sn monofilament wires saves time and effort since the total number of passes are reduced and intermediate annealings are done less frequently.

Frequent Problems Encountered During Hydrostatic Extrusion And Their Solutions

Quite often the fluid pressure would not build up because fluid would leak between the billet and the die. To prevent this the tapered end of the billet had to be shaped so that it fitted into the die to form an effective seal. Shaping the tapered end, especially for fine wires, was a difficult task.

Rigid billets (diameter >0.2") were machined on the lathe to form conic sections. However, the front ends of wires having diameters between 0.1" to 0.2" were swaged to give symmetrically tapered cones. The important point to note is that the cone need not match the semicone angle of the die but should be symmetrical so that it makes a ring of contact with the die. The nose of fine wires having diameters of 0.03" and 0.1" could not be machined or swaged. A suitably designed holder having small jaws was used to grip the wire as it rotated on the lathe. The nose was formed by lightly pressing a dull file against the tip of the wire as it rotated at a high speed. However, a mini-swaging machine (not available in our laboratory) would simplify the nose forming operation for fine wires.

A dangerous situation encountered a few time was the loss of control over the extrusion rate, resulting in a phenomenon known as free extrusion. The billet would pick up speed and shoot out of the chamber like a bullet. One way to control the speed of extrusion was to reduce the ratio of the volume of fluid to the volume of the billet. A larger amount of oil led to free extrusion because there was much more compressed energy stored in the oil. The oil volume was reduced by inserting spacers between the chamber and billet. This filled up the space but did not hinder the movement of the billet (Fig. 8a).

Even if the billet picked up speed, free extrusion was prevented by leaving a conical head on the back of the billet. The large conical head would bring the extrusion to an abrupt stop, since it was too large to be extruded, and would form an effective seal with the die. Sometimes it was not possible to obtain a natural conical head; this required an artificial head to be glued to the rear end of the billet. Figure 8b shows a natural conical head and Fig. 8c illustrates the use of an artificial head.

To obtain good bonding the bronze-niobium interface should remain extremely clean. If the rear end of the billet is left open, pressurized oil can easily infiltrate along the bronze-niobium interface. On large diameter billets this infiltration was prevented by glueing a copper cap on the billet, while fine wires were capped with a drop of solder.

Having overcome these difficulties, the production of hydrostatically extruded wires has become far easier than before.

Properties of Hydrostatically Extruded vs. Cold Drawn Single Core Nb_3Sn Wire

The aim of this investigation was to compare the superconducting properties of Nb_3Sn monofilaments prepared by hydrostatic extrusion with those of the drawn Nb_3Sn monofilaments. The superconducting properties include the critical current J_c and the effect of a bending strain on the degradation of J_c.

One sample 12" in length and 0.0239" in diameter prepared by hydrostatic extrusion was tested. The area ratio of bronze to niobium was 9.77. The sample was heat treated at 725°C (1335°F) for 24 hours to form the Nb_3Sn layer. The preliminary data from superconducting measurements are given below -

Thickness of Nb_3Sn layer = 1.75 μm

Magnetic Field (tesla)	J_c at 4.2 °K under the Magnetic Field (amp/cm^2)
6T	1.15×10^6
8T	6.4×10^5

The above results indicate that the superconducting properties of Nb_3Sn wires could be improved by the use of hydrostatic extrusion. Billets with varying ratio of core to sleeve radius as given in Table 1 are being hydrostatically extruded and will be tested. The measured data will be compared with the results obtained from the conventionally drawn wires with the same composition, niobium-bronze ratio and diffusion treatment. This will provide a critical evaluation of the modified process for manufacturing Nb_3Sn wires using hydrostatic extrusion.

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2 B. Avitzur, "Alternate Manufacturing Technologies for the Production of Multifilamentary Superconducting Wire by the External Bronze Technique," unpublished work.

3 Report submitted by Battelle to the Division of Magnetic Fusion Energy, Energy Research and Development Administration, on "Experimental Evaluation of Hydrostatic Extrusion for the Fabrication of Multifilament Superconducting Wire," (1976).

4 J. Breme and C.H. Massat, "The Effect of Hydrostatic Extrusion on the Superconductive Characteristics of Tin-bronze/Niobium Polymer Conductors," Metallwissenschaft und Technik, Vol. 33, No. 6, June 1979.

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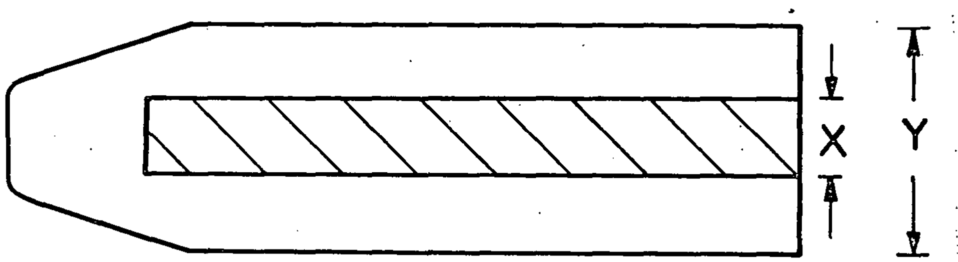
TABLES

FIGURES

TABLE 1

DIMENSIONS OF THE ORIGINAL

BRONZE CLAD NIOBIUM BILLETS



Billet	Initial Diameter 'Y' of Bronze (in.)	Initial Diameter 'X' of Niobium (in.)	Crossectional Area Ratio of Bronze to Niobium
A	0.375	0.248	2.29
B	0.375	0.185	4.11
C	0.375	0.120	9.77

TABLE 2

DATA ON THE HYDROSTATIC EXTRUSION OF
BRONZE CLAD NIOBIUM BILLETS

Extrusion No.	Diameter before extrusion (in)	Diameter after extrusion (in)	Reduction in area (%)	Extrusion pressure (ksi)		
				Billet A	Billet B	Billet C
1	0.375	0.228	63	116	141	146
2	0.228	0.185	34	92	98	127
3	0.185	0.148	36	103	110	140
4	0.148	0.118	36	113	139	141
* Intermediate Anneal: 20 minutes at 450°C (840°F)						
5	0.118	0.0884	43	97	105	106
6	0.0884	0.0682	40	108	113	127
* Intermediate Anneal: 20 minutes at 450°C (840°F)						
7	0.0682	0.0525	40	104	105	112
8	0.0525	0.0406	40	114	132	139
* Intermediate Anneal: 20 minutes at 450°C (840°F)						
9	0.0406	0.0313	40	99	107	112
* Intermediate Anneal: 20 minutes at 450°C (840°F)						
10	0.0313	0.0239	40	120	122	153
Total no. of extrusions	Initial diameter	Final diameter	Total reduction in area	Average pressure		
10	0.375"	0.0239"	99.6%	107	117	130

- Average reduction in area per pass = 41.2%

- Intermediate anneal given after an average reduction in area of 75%

* The intermediate anneal was carried out in an argon gas furnace. After being kept in the furnace for the required time the billet was air cooled. Sometimes a thin oxide layer formed which was sanded off with #600 emery paper.

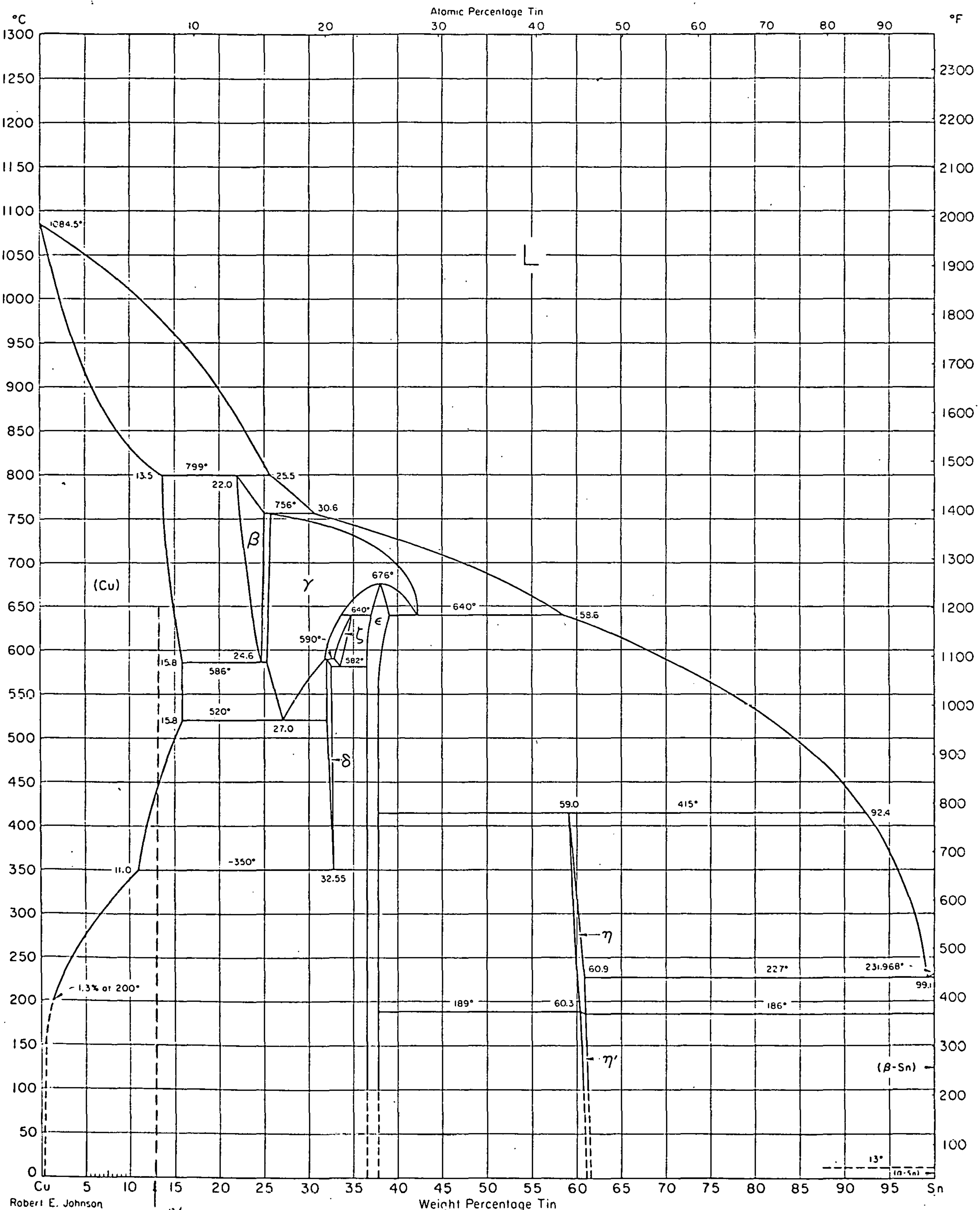


FIG. 1 Copper-tin phase diagram (Courtesy: Metals Handbook Vol. 8)

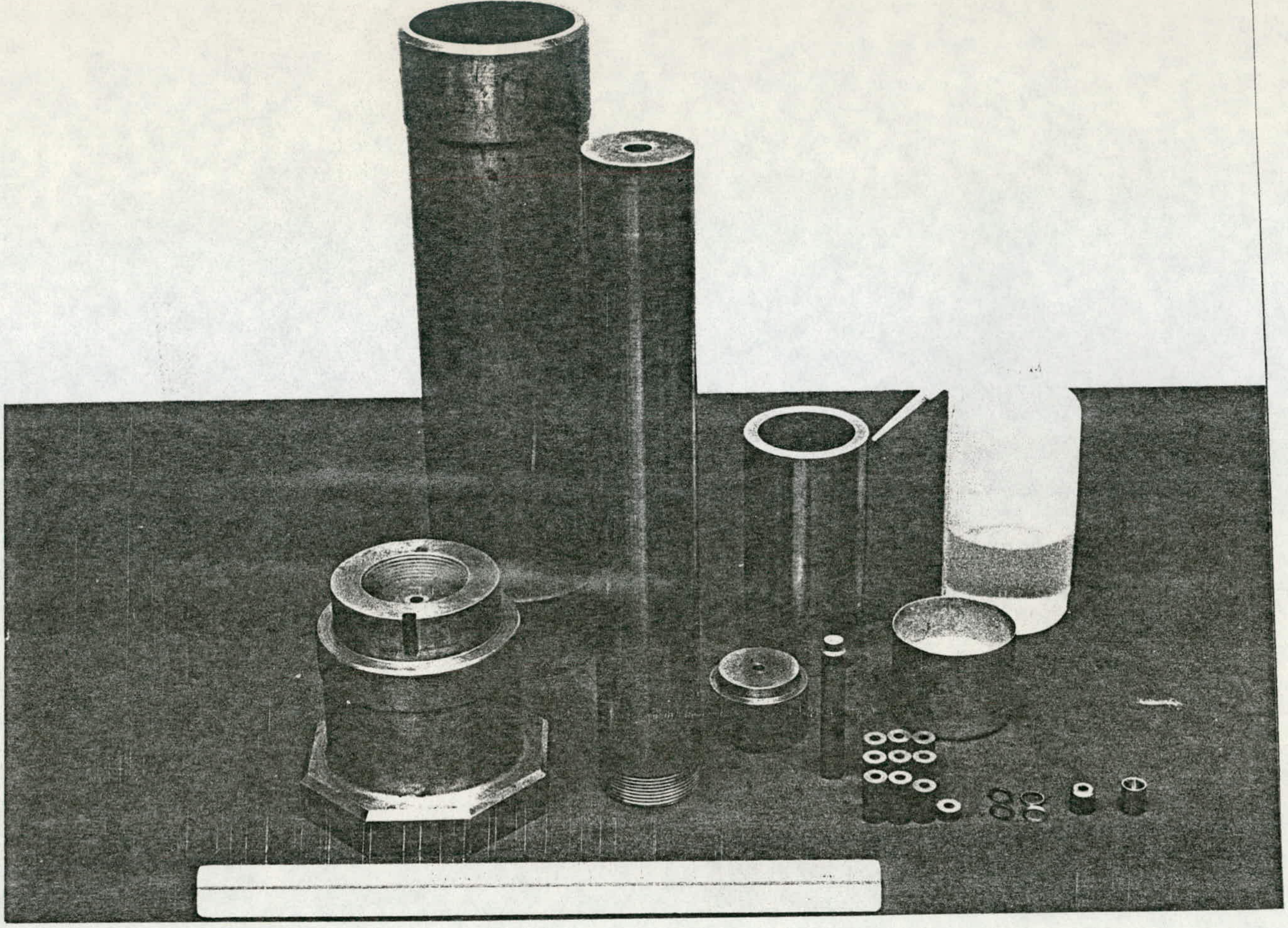


FIG. 2 MODEL 6 FLUID EXTRUSION UNIT

FROM LEFT TO RIGHT: BASE; SHIELD; CHAMBER; RAM ATTACHMENT; RAM; SPACERS; OIL; O-RINGS; MITRE RINGS; DIE HOLDERS; DIE; BEESWAX.

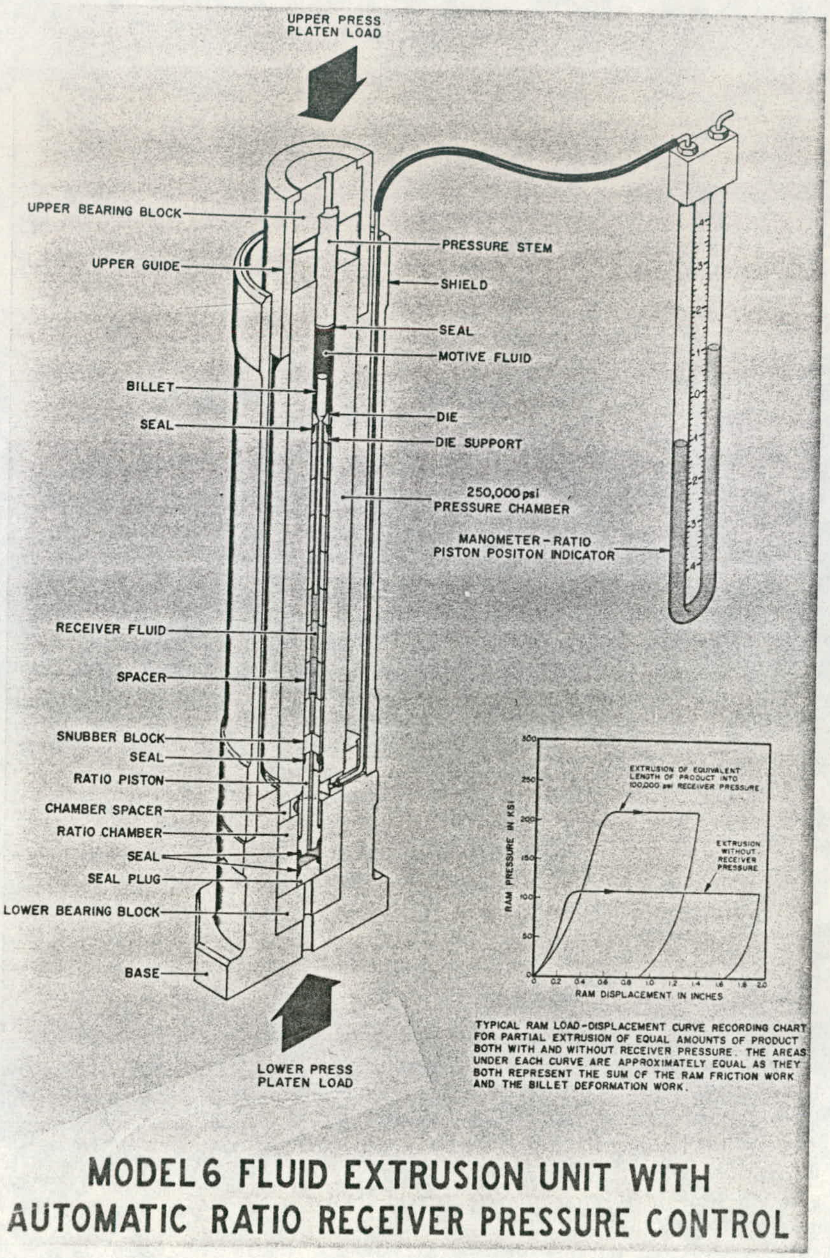
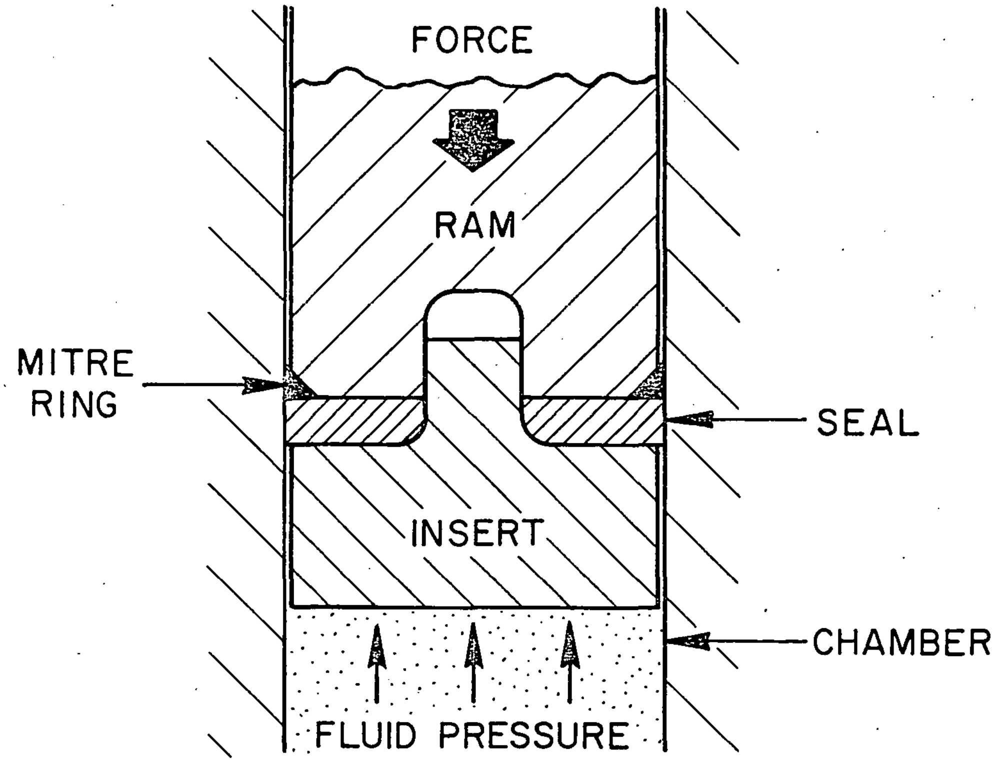
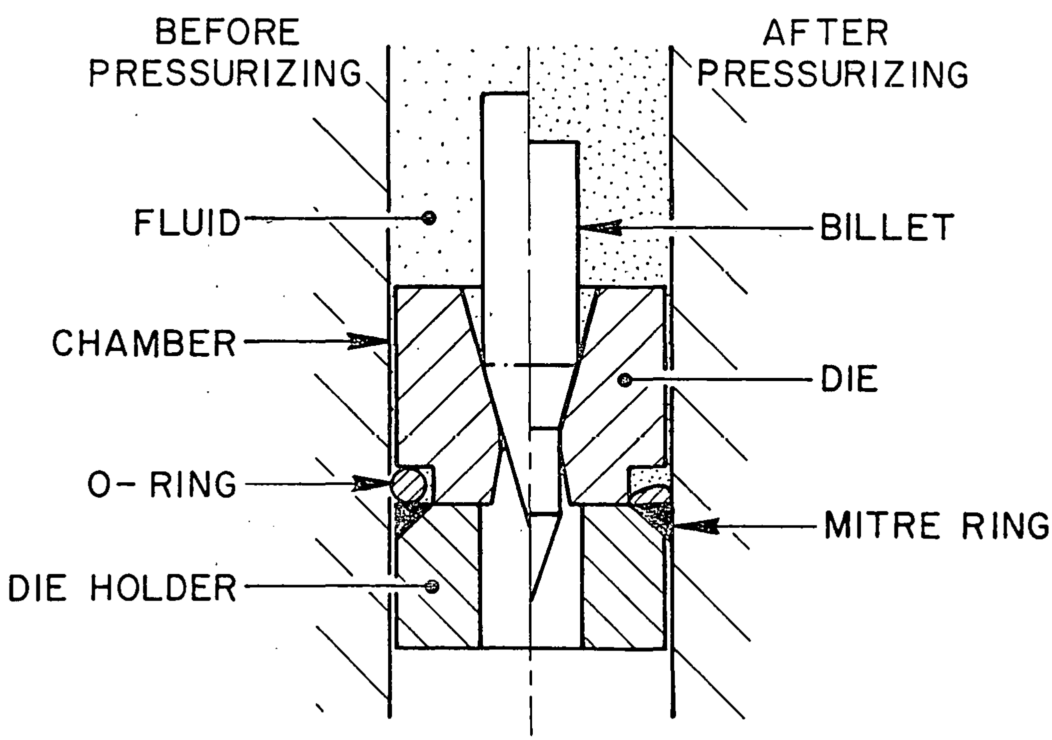


FIG. 3   SECTIONED VIEW OF THE MODEL 6 FLUID EXTRUSION UNIT.

DESIGNED BY DR. A. AUSTEN, BETHLEHEM.



(a)



(b)

FIG.4 SEALS USED ON MODEL 6.
(a) BRIDGMAN SEAL.
(b) MITRE RING WITH O-RING SEAL.

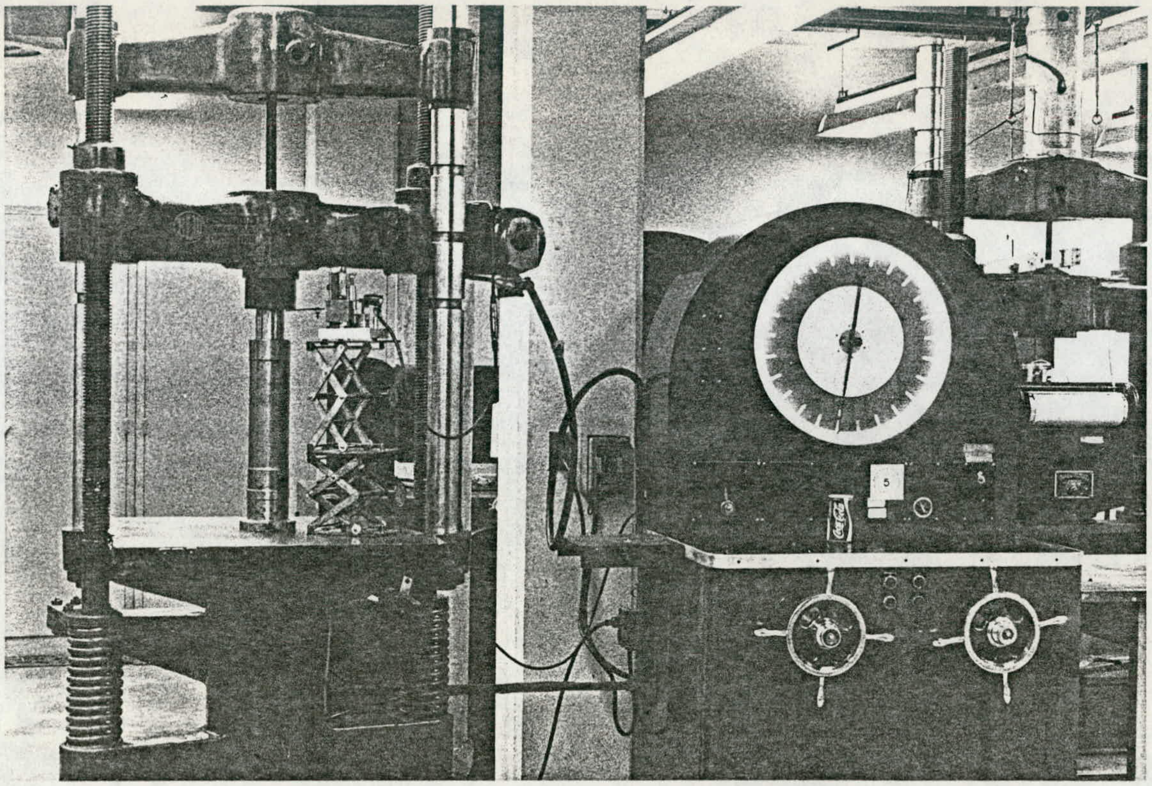


FIG. 5 OVERALL VIEW OF THE EQUIPMENT USED FOR HYDROSTATIC EXTRUSION.

FROM LEFT TO RIGHT - MODEL 6 FLUID EXTRUSION UNIT UNDER COMPRESSION; DEFLECTOMETER ON STAND; BALDWIN CONTROLS; STRIP RECORDER THAT PLOTS APPLIED LOAD VS. RAM DISPLACEMENT.

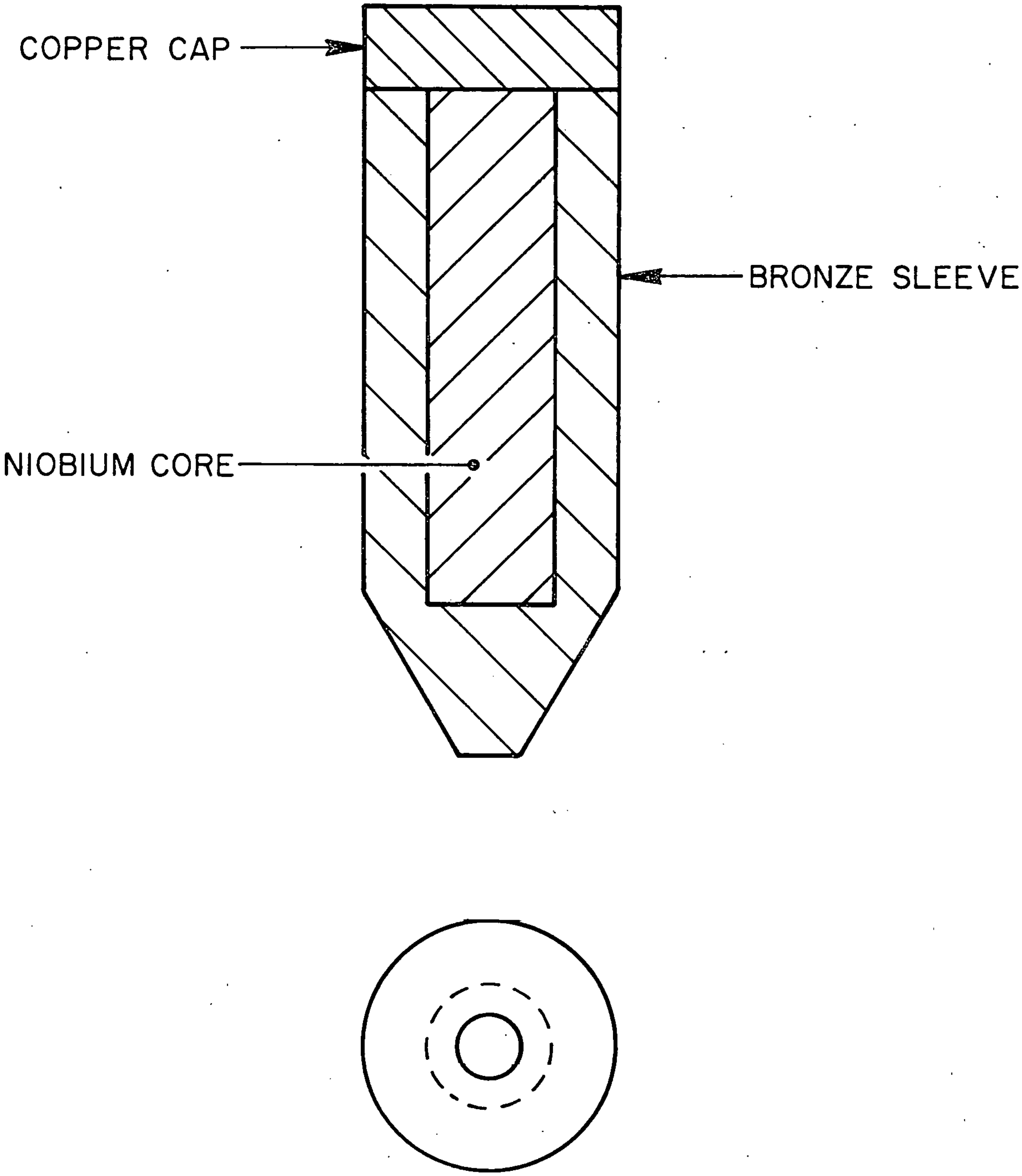


FIG.6 ASSEMBLED BIMETALLIC BILLET.

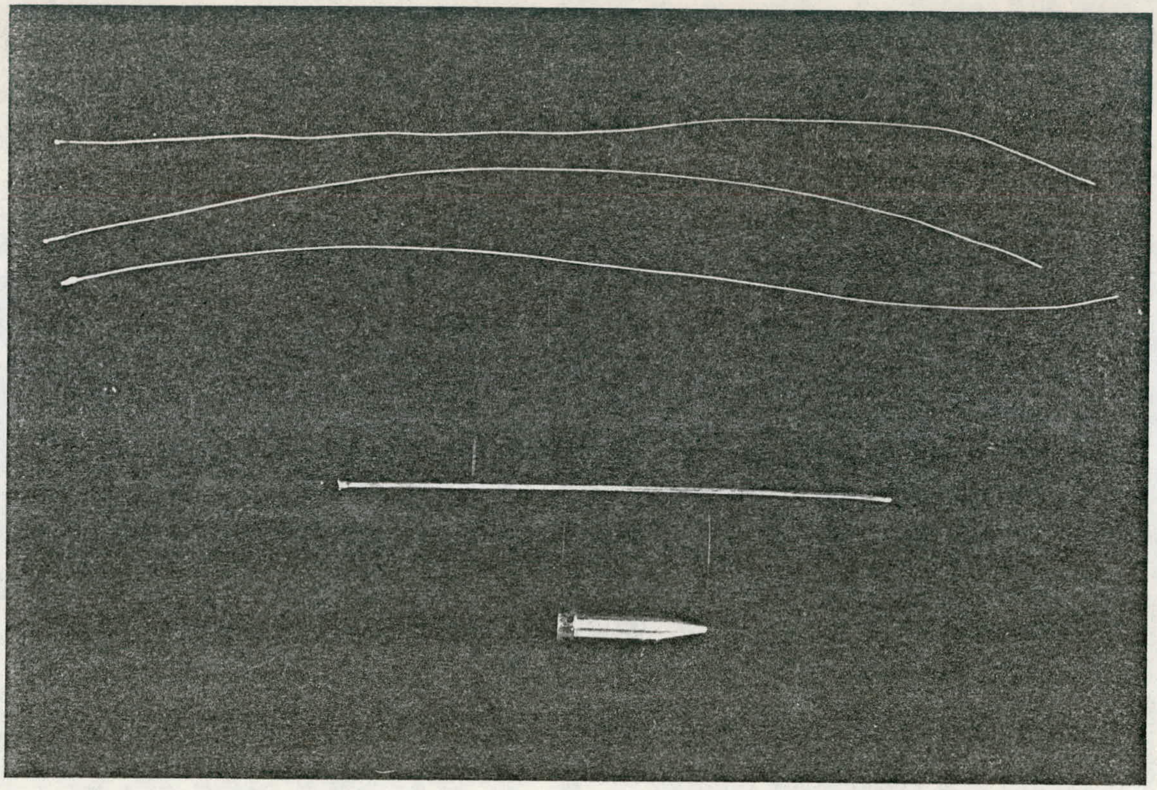


FIG. 7 HYDROSTATICALLY EXTRUDED NIOBIUM/BRONZE WIRE

FROM BOTTOM TO TOP: ASSEMBLED BIMETALLIC BILLET; ONE OF THE INTERMEDIATE PRODUCTS; FINAL PRODUCT OF 0.0239" DIAMETER.

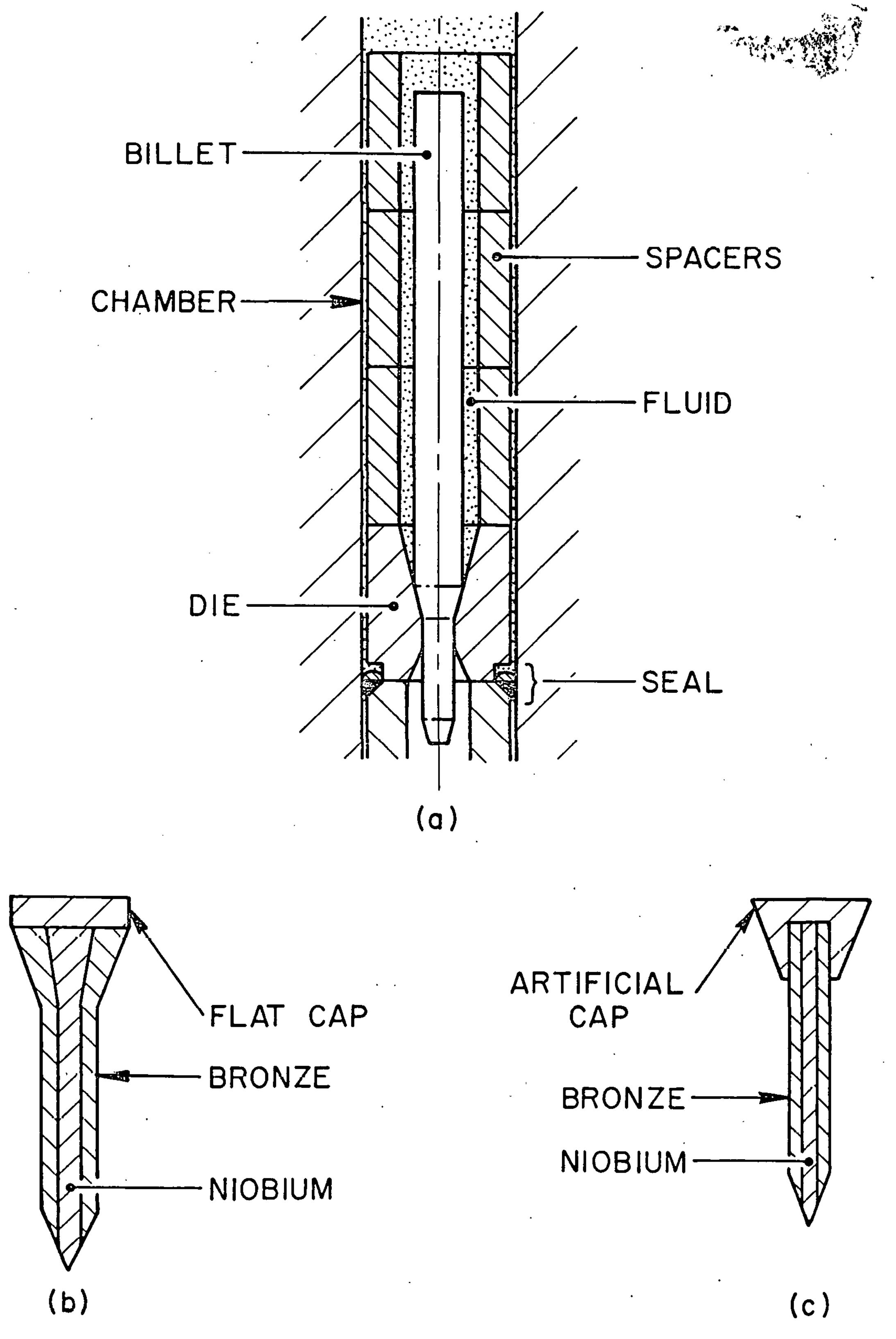


FIG.8 PREVENTION OF FREE EXTRUSION.

(a) INTRODUCING SPACERS TO REDUCE VOLUME OF FLUID.

(b) NATURAL HEAD ON BILLET.

(c) ARTIFICIAL HEAD.