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# DEVELOPMENT OF TECHNIQUES FOR THE EXTRUSION OF BERYLLIUM TUBING

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concord, massachusetts

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Nuclear Metals, Inc.
Concord, Massachusetts

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Technical Director

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## I. Introduction

Under Sponsor Agreement S-68, Nuclear Metals, Inc. has worked on the development of extrusion techniques for the production of beryllium tubing in the size range of 0.40" OD to 0.75" OD with 0.03" to 0.10" wall. Three specific sizes were extruded during the development work: 0.4" OD x 0.03" wall, 0.75" OD x 0.03" wall, and 0.75" OD x 0.10" wall. Two methods of extrusion were used. In one, the filled billet method, the hollow beryllium core in the extrusion billet was filled with a steel filler that was removed after extrusion. In the other method, a hardened steel mandrel was used to form and control the ID of the tube. In a third method a stationary stepped mandrel would have been used, having a large cross section except for a reduced tip positioned in the die; this was not attempted because of the success with the other methods and the inherent mechanical difficulties associated with a stepped mandrel.

Extrusion billets were prepared from the starting material, Brush -200 mesh QMV powder, in three ways: powder was packed cold into the extrusion billet can; powder was packed cold into a can, pre-extruded, and machined into extrusion billet cores; powder was packed cold into a can, hot compacted, and machined into extrusion billet cores. The latter method was used for most of the extrusions. Figure (1) presents diagramatically the production processes studied. The production of the small-size tubing, 0.4" OD x 0.03" wall, was studied the most extensively.

## II. Variables Examined

### A. Starting Material

The starting material for tube production was Brush -200 mesh QMV beryllium powder. Analysis of the powder used, presented in Table I, showed it to have a lower iron content than customarily found.

### B. Preparation of Cores for Extrusion Billets

All cores were produced in one of three ways:

#### 1. Cold compacted directly into extrusion billet can

The extrusion billet can was prepared in the same way as that for a machined solid beryllium core (Figure 2). One end was left open, the powder poured in, then compacted at 74 tsi. The powder was compacted in 1/4" - 1/2" thick layers, and the steel end plug welded on after the can was filled. A pin-hole was left in the weld for gas to escape during heating.

This process was the simplest of the three tried, but it produced extruded tubes of doubtful quality. Compared with the smooth, even surfaces of tubes produced from hot compacted or pre-extruded cores, the surfaces of these tubes were much poorer, having a rough, speckled appearance. The surfaces could be improved by etching. It has not been determined whether the pitting discovered during the pickling of some of these tubes was caused by a defect in the tubing, by iron picked up during packing, or some defect in the pickling operation.

2. Cold compacted into steel can and pre-extruded

The powder was cold compacted at 21 tsi into a heavy-walled steel can. An end plug was welded on, and the can was heated to 1950$^{o}$F prior to extrusion. For the 0.4" OD tubes, pre-extrusion was at a reduction of 8.4X; for the 0.75" OD tubes, 3.75X. Each extruded rod was pickled and machined into several extrusion billet cores, with some loss of beryllium caused by the extrusion defects at both ends of the rod. This method was tried both because of its production potentialities and to determine its effect on the preferred orientation in the tubing.

3. Cold compacted into steel can and hot compacted

The powder was cold compacted at 70 - 85 tsi into heavy-walled steel cylinders. After an end plug was welded on, the cans were heated to 1950$^{o}$F and hot compacted. A sketch of the arrangement for hot compacting is shown in Figure (3). The compacts were cooled slowly to prevent cracking, then pickled and machined. One core was obtained from each compact. The larger compacts were made with stainless steel cores to reduce the amount of beryllium necessary. Stainless steel was used in place of mild steel because it has a larger coefficient of expansion than beryllium, and cracking of the beryllium was thereby minimized. The majority of cores were made by hot compacting.

C. Type of Extrusion

For all extrusions the beryllium was canned in steel, and the can components oxidized to provide a barrier between the steel and beryllium. A sketch of the extrusion billet for a mandrel extrusion is shown in Figure (2).

1. <u>Filled Billet Technique</u>

This technique is used when it is advantageous to produce tubing without the use of a mandrel. The extrusion billet contains a steel filler which is removed after extrusion, leaving the desired beryllium tube behind. If mild steel is used for the filler, it must be pickled out - a long slow process. Moreover, depending on the relative dimensions of the beryllium tube and the mild steel filler, cracking will occur in the beryllium following extrusion because of the restraint imposed by the mild steel with its lower coefficient of expansion. Core removal has been expedited, and the cracking caused by thermal contraction reduced, by the use of Hadfield's manganese steel for the filler material. This steel has a higher coefficient of thermal expansion than beryllium and, in addition, work-hardens so readily that when a bar is pulled in tension it will neck down uniformly along its length before failure, rather than necking locally until failure occurs. The Hadfield's manganese steel filler in the extruded beryllium tube is easily removed after it is stretched in a drawbench. A difficulty of this process is that, unless the extrusion is absolutely straight, there is a high probability that the beryllium will crack while the filler is being stretched, because of the accompanying straightening action.

In a filled billet, the amount of shear that occurs in the beryllium during extrusion can be controlled by the positioning of the beryllium in relation to the outside diameter of the billet. The most shear occurs at the outer surface; the least at the center. Because of the ease with which variables could be changed and the relative certainty of success, the first extrusions were made by the filled billet technique.

2. <u>Mandrel Technique</u>

Later, extrusions were made with a small, hardened steel mandrel to control the ID of the tube. This method is simple because there is no need to remove a filler material. Cracking during cooling and the stretching of the filler are eliminated. Mandrel failure, however, is a serious problem, particularly with the smaller ID tubing. The high temperature of the beryllium will cause heating of the mandrel and failure, because of the steel's reduced high-temperature strength. Since the danger of mandrel failure increases with increasing time of contact between the beryllium and the mandrel, and with increasing stress in the mandrel, high extrusion speeds and decreased reductions will help increase mandrel life. All extrusions were made at ram speeds of 140 - 150 in/min, the fastest available on our extrusion presses. The steel from which the mandrels are made affects their ability to withstand the severe service conditions. An AISI T-1 type steel with especially low carbon content* hardened to Rockwell-C 60 proved to be the most satisfactory. Mandrels made from AISI-H-21 and AISI T-2 steels were also tried.

D. Variation of Temperature

The extrusion temperature was varied from 1650°F to 1950°F to determine its effect on extrusion conditions and the preferred orientation of the tubes. Two effects on extrusion conditions were noted. At lower temperatures the extrusion reduction had to be reduced when mandrels were used. When the steel can had an appreciable area compared to the beryllium, cracking was also found to be affected by the extrusion temperature. During cooling, the steel held the beryllium in tension. At the higher temperatures the difference in thermal contraction was sufficient to crack the beryllium.

E. Variation of Extrusion Reduction

The extrusion reduction was varied from 6X to 23X, in order to determine the effect on room temperature properties and preferred orientation of the tubing. Generally, higher reductions were favored because of the greater amount of tubing produced per extrusion. When extrusions were made with a mandrel, the reduction had to be reduced in some cases to minimize mandrel failure. The results of examination for preferred orientation are described below.

F. Annealing Treatment

Other work at NMI indicated that some improvement in properties might result from a long-time heat treatment at 600°C. Samples of tubing were heated for twenty-four hours or one week at 600°C, then burst-tested. In those

* This corresponds to an AISI-H-26 steel but is sold as a low carbon version of the AISI T-1 type steel.

from the one-week test, there was no change in strength compared to samples from the same tube that were not heat treated. The twenty-four-hour test indicated a decrease but there was some indication that this was influenced by the position of the samples in the tube from which they were cut.

## III. Tests Performed

The tubes extruded under these differing conditions of temperature, reduction, core preparation, and extrusion technique were examined in several ways: burst and crush tests were used to determine the room temperature mechanical strength, dimensional measurements were made to determine the variation in diameter and wall thickness of the tubing, and x-ray examination was used to determine the basal plane distribution relative to a tangent to the tube.

### A. Crush Tests

Crush tests of tubing samples gave erratic results. The tubing was etched before testing to remove 0.003 to 0.005 inch from each surface, but some microcracks may have remained to cause the erratic results. The maximum values may be a better indication of true strength than the average value, since some of the low values may be due to cracks that were not seen.

In this series of tests a one-inch long sample is crushed between parallel plates and the force required to cause failure is noted.

1. Effect of Extrusion Temperature on Crush Strength of Tubing Extruded by the Filled Billet Technique at 23X Reduction. (0.40 inch OD x 0.03 inch wall tubing)

| Temperature ($^{o}F$) | Crush Force (lbs) | | |
|---|---|---|---|
| | Max. | Min. | Av. |
| 1600 | 72 | 35 | 54 |
| 1725 | 90 | 70 | 80 |
| 1825 | 126 | 50 | 82 |
| 1950 | 88 | 35 | 61 |

2. Effect of Shear and Reduction on Crush Strength of Tubing Extruded by the Filled Billet Technique; Extrusions at 1825°F. (0.40 inch OD x .03 inch wall tubing

| Reduction Ratio | Crushing Force (lbs) | | | | | |
|---|---|---|---|---|---|---|
| | Maximum Shear, Thin Steel Can | | | Decreased Shear, Heavy Steel Can | | |
| | Max. | Min. | Av. | Max. | Min. | Av. |
| 23 | 126 | 50 | 82 | | | |
| 15 | 71 | 42 | 56 | | | |
| 10 | 79 | 42 | 64 | 111 | 80 | 94 |
| 6 | 70 | 45 | 57 | 73 | 68 | 71 |

3. Crush Strengths Obtained from Tubing Extruded Over a Mandrel at 21X Reduction, 1900°F. (0.40 inch OD x .03 inch wall tubing): Maximum, 125 lbs; Minimum, 86 lbs; average, 110 lbs.

B. Burst Tests

Burst tests were made on 0.40" OD x 0.03" wall tubing. Six-inch lengths were used to minimize any end effects. Hydrostatic pressure was built up in oil which filled the inside of the tube until failure occurred.

| Type of Extrusion | Type of Core | Ext. Temp. (°F) | Reduction Ratio | Hydrostatic Pressure on ID (psi) | Calc Hoop Stress (psi) | Remarks |
|---|---|---|---|---|---|---|
| Filled Billet | Hot Compacted | 1725 | 23 | 4910 | 34,200 | |
| | Hot Compacted | 1825 | 23 | 4700 | 31,800 | |
| | Hot Compacted | 1825 | 10 | 3715 | 23,800 | Billet prepared for decreased shear |
| Mandrel | Hot Compacted | 1900 | 21 | 5680 | 43,500 | Average of 6 tests |
| | Cold Compacted | 1900 | 19 | 4010 | 30,000 | |
| | Pre-extruded | 1900 | 19 | 1590 | 12,800 | Average of 5 tests |

| Material | Treatment | Hydrostatic Pressure on ID (psi) | Calc Hoop Stress (psi) |
|---|---|---|---|
| Mandrel extrusion; Hot compacted core; extruded at 1900°F; 21X Red | Annealed 1 day 600°C | 4505 | 33,500 |
| | Not annealed (same tube) | 5320 | 39,900 |
| | Annealed 1 week 600°C | 6240 | 45,100 |
| | Not annealed (same tube) | 5915 | 43,900 |

All tubes except the tubes made from pre-extruded cores broke into several pieces at failure. The tube made from pre-extruded cores split longitudinally at failure and failed at very low levels.

C. Dimensional Measurements

| Type | Spread Between Max + Min Value Measured (in) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | OD | | | | Wall Thickness | | | |
| | Per Measured Cross-section | | Per Tube | | Per Measured Cross-section | | Per Tube | |
| | Av. | Max. | Av. | Max. | Av. | Max. | Av. | Max. |
| 1. 0.4" OD x 0.03" wall<br>Filled Billet<br>Hot Compacted Core 6-23X Red | 0.001 | 0.005 | 0.003 | 0.009 | 0.002 | 0.004 | 0.003 | 0.005 |
| Filled Billet<br>Decreased Shear<br>Hot Compacted Core 6-15X Red | 0.003 | 0.006 | 0.009 | 0.012 | 0.001 | 0.003 | 0.003 | 0.004 |
| Mandrel Extrusion<br>Hot Compacted Core 20X Red | 0.001 | 0.002 | 0.003 | 0.003 | 0.005 | 0.006 | 0.006 | 0.006 |
| Mandrel Extrusion<br>Pre-extrusion Core 19X Red | 0.002 | 0.004 | 0.004 | 0.004 | 0.005 | 0.009 | 0.007 | 0.009 |

| Type | Spread Between Max + Min Value Measured (in) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | OD | | | | Wall Thickness | | | |
| | Per Measured Cross-section | | Per Tube | | Per Measured Cross-section | | Per Tube | |
| | Av. | Max. | Av. | Max. | Av. | Max. | Av. | Max. |
| Mandrel Extrusion<br>Cold Compacted Core<br>19X Red | 0.002 | 0.007 | 0.005 | 0.008 | 0.005 | 0.008 | 0.007 | 0.009 |
| 2. 0.75" OD x 0.03" wall*<br>Mandrel Extrusion<br>Hot Compacted Core<br>20X Red | 0.003 | 0.005 | 0.005 | 0.007 | 0.003 | 0.007 | 0.006 | 0.008 |
| 3. 0.75" OD x 0.10" wall*<br>Filled Billet<br>Hot Compacted Core<br>14X Red | 0.004 | 0.007 | 0.009 | 0.012 | 0.004 | 0.006 | 0.008 | 0.008 |
| Mandrel Extrusion<br>Hot Compacted Core<br>10X Red | 0.006 | 0.010 | 0.012 | 0.013 | 0.005 | 0.008 | 0.008 | 0.008 |

* The 0.75" OD tubes were extruded with heavy steel cans on the OD, whether on a mandrel or from a filled billet, because of tooling limitations.

The values obtained seem to indicate that a steel can of heavy OD increases slightly the spread between OD measurements, but does not affect the wall measurements.

D. Preferred Orientation in Extruded Beryllium Tubes

Because of the extreme dependence of mechanical properties on preferred orientation in beryllium, texture studies were performed on the extruded tubes. The relatively small wall thickness of most of the beryllium tubes extruded imposed geometrical problems on the choice of technique for determining a pole figure. Accordingly, thin beryllium wire specimens (see Figure 4) were machined from the tubes and used in conjunction with the Norton Rod method of texture analysis to determine the (0001) basal plane orientation with respect to the extrusion direction and tube tangent. Figure (5) illustrates the mechanics of the Norton Rod technique as applied to samples taken from the tubes. To determine the random intensity level, a wire of similar size was machined from a hot pressed block of beryllium and used as a random specimen. All the wire samples were machined, by means of a high-speed grinder mounted on a lathe, to a diameter of 0.0280 $\pm$ 0.0020 inch, then etched in an aqueous solution of sulfuric acid to 0.0235 $\pm$ 0.0005 inch. The random sample was rotated at approximately 30 rpm to obtain the random intensity level using the Bragg angle associated with the (0002) basal plane reflection ($50.92^{\circ} = 2\theta$). The wire samples taken from the extruded tubes were rotated at approximately 3 rph to determine the degree of preferred orientation relative to the hot-pressed standard. This experimental technique resulted in an (0002) diffraction intensity profile as a function of the angle between the direction in question and the tube tangent. As shown in Figure (6), this basal plane intensity trace is equivalent to an equatorial profile on the normal (0001) basal plane pole figure and, because of the complete radial symmetry characteristic of the extrusion process, an entire (0001) basal pole figure may be constructed from this data. It should be noted that a Wulff net must be used to plot the pole figure in conjunction with the basal plane intensity profile. To simplify the data presentation, only intensity profiles were used. The corresponding pole figures were not constructed from these basal plane profiles.

Since only part of all the tubes have had their textures analyzed at the time of report preparation, the data obtained to date will be summarized and a complete analysis presented in the form of a supplementary letter to this report, to be mailed immediately following the end of the contract period.

1. <u>The Effects of Extrusion Temperatures</u>

At 23:1 reduction, and by means of the filled billet technique, four extrusions were made at 1600, 1725, 1825, and 1900$^{\circ}$F respectively. The extrusions made at the three highest temperatures exhibited crystallographic textures which were essentially of the same type, although the intensity varied slightly from specimen to specimen. The extrusion made at 1600$^{\circ}$F exhibited a duplex basal plane texture. In all cases, however, the plane parallel to the tube tangent contained the lowest basal plane population.

2. The Effects of Extrusion Reduction

An extrusion temperature of 1825°F and a filled billet technique were employed in this series of experiments. Two distinct types of extrusions were made. In the first type, the amount of shear during the extrusion process was kept constant and reductions of 6:1, 10:1, and 15:1 were employed. This constant amount of shear during extrusion was achieved by utilizing one die size and varying the liner size to effect changes in reduction. The clad thickness was kept constant and the core diameter changed. Effectively, the maximum basal plane intensity increased with the reduction ratio in almost a linear fashion. Within the limits of experimental error, the texture remained relatively constant and minimum basal plane populations were observed in a plane parallel to the tube tangent.

In the second type of experiment to investigate the effects of reduction ratio on preferred orientation, a constant billet size and a varying die size were used to effect reduction ratios of 6:1, 10:1, and 15:1. Essentially, the amount of shear to which each billet was subjected during extrusion decreased with decreasing reduction ratio. The textures obtained in these experiments differed extensively, especially in the case of the high reduction extrusion, from those obtained by other techniques. That is, the maximum basal plane population occurred in a plane parallel to the tube tangent. The maximum basal plane population increased in proportion to the reduction ratio. Essentially, these results imply that the extrusion geometry greatly affects the degree and type of preferred orientation developed during the extrusion process.

In addition to these experiments, the texture of a tube extruded over a mandrel at 1900°F and a 21:1 reduction ratio was investigated. This specimen exhibited the highest degree of preferred orientation observed. The basal plane population parallel to the tube tangent was as low as 1/2 random, and plane populations as high as 15 random were observed parallel to the radial direction.

## IV. Results

Three sizes of tubing were produced: 0.40" OD x 0.03" wall, 0.75" OD x 0.03" wall, and 0.75" OD x 0.10" wall. All the methods shown in Figure (1) were used to produce 0.4" OD tubing. The effects of temperature, reduction, and shear were examined with filled billet extrusions. The lowest degree of preferred orientation was found in tubes extruded by the filled billet method from hot compacted cores within a heavy OD can that decreased the amount of shear in the beryllium during extrusion. Tubes extruded at the lower temperatures and lower reductions also had less preferred orientation. Tubes produced by extrusion with a mandrel from hot compacted cores had a high degree of preferred orientation, but also had the highest burst strengths at room temperature. Although mandrel failure caused some extrusions to fail, those extrusions which were successful were generally free from cracks. Sections of tubing produced in this way were sent to Oak Ridge for evaluation, since they appeared to represent the product of the best production procedure.

The 0.75" OD x 0.03" wall tubing was produced from hot compacted powder by extrusion over a mandrel. No filled billet extrusions were made because mandrel extrusions were simpler to process and because the ID of this tubing was large enough to permit the use of mandrels with little danger of mandrel failure. Because of equipment limitations it was necessary to extrude at reductions in the range of 15 to 25X. Tubes extruded at the lower reduction cracked more severely than those at higher reductions, presumably because the heavy steel sheath held the beryllium in tension during cooling. Tubes were extruded at 1950$^{\circ}$F at first, but this was lowered later to reduce the danger of cracking and to improve the surface appearance.

The 0.75" OD x 0.10" wall tubing was extruded by both the filled billet and the mandrel techniques. The ID of this size tubing is on the border line for extrusion with a mandrel; even at 10X, the low reduction tried, the mandrels were red-hot after extrusion. Filled billet extrusions at 14X were more successful. Perhaps because of the larger size they were straighter and it was easier to remove the Hadfield's manganese steel cores.

TABLE I

Analysis of Beryllium Powder
by Brush Beryllium Company

| | | Lot. No. | |
|---|---|---|---|
| | | V-5298 | V-5681* |
| % | Be | 98.9 | 99.1 |
| | BeO | 0.90 | 0.96 |
| | C | 0.09 | 0.07 |
| ppm | B | 1.6 | |
| | Al | 250 | 400 |
| | Cd | <.7 | |
| | Cr | 90 | |
| | Fe | 840 | 1000 |
| | Li | 1 | |
| | Mg | 70 | 200 |
| | Mn | 70 | |
| | Ni | 170 | |
| | Ag | 3 | |
| | Ca | <100 | |
| | Co | 1 | |
| | Cu | 120 | |
| | Mo | <20 | |
| | Pb | <10 | |
| | Si | 300 | 200 |
| | Zn | <100 | |

* Incomplete analysis furnished

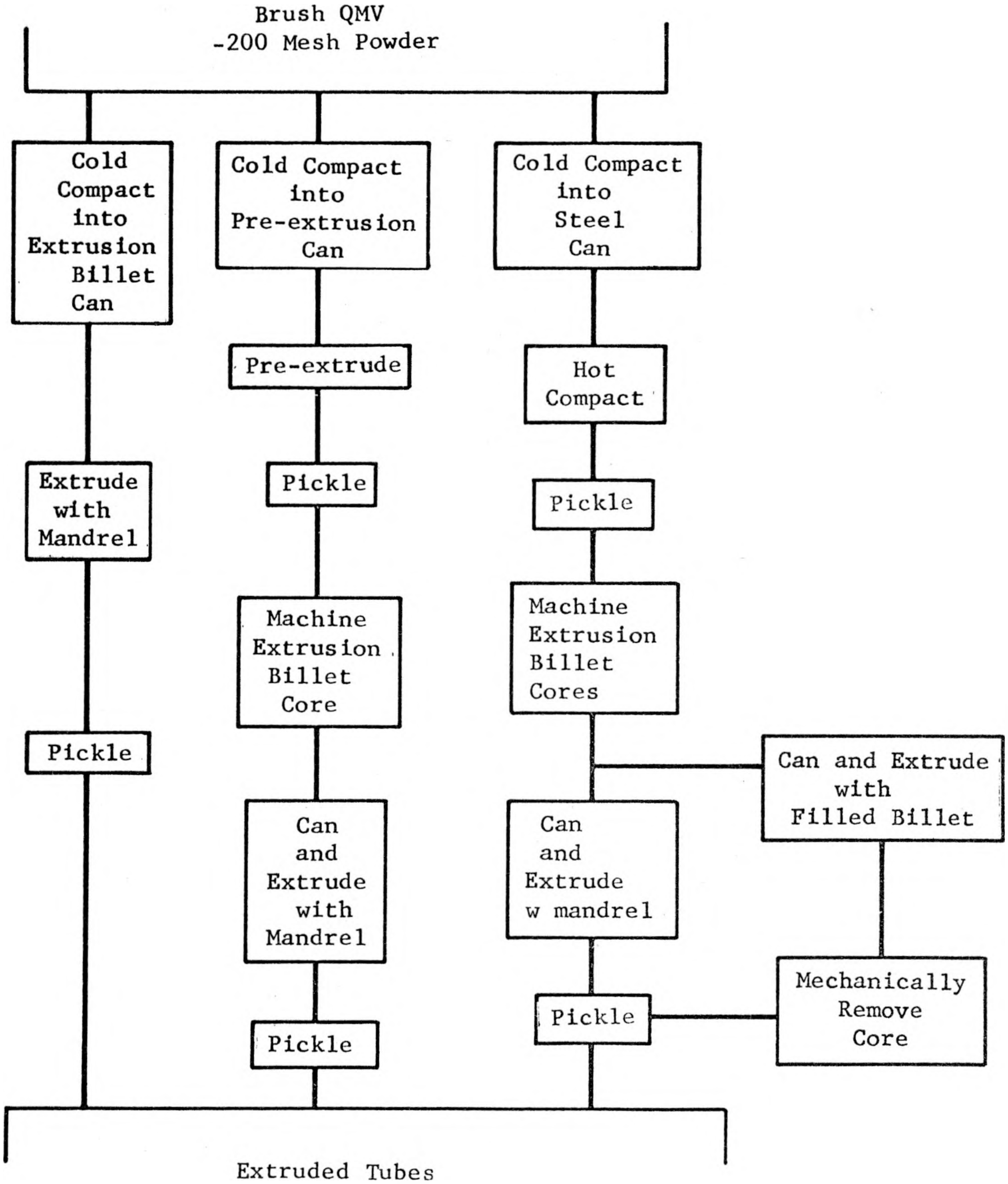


Fig. 1 - Production Methods Studied
RA-1622

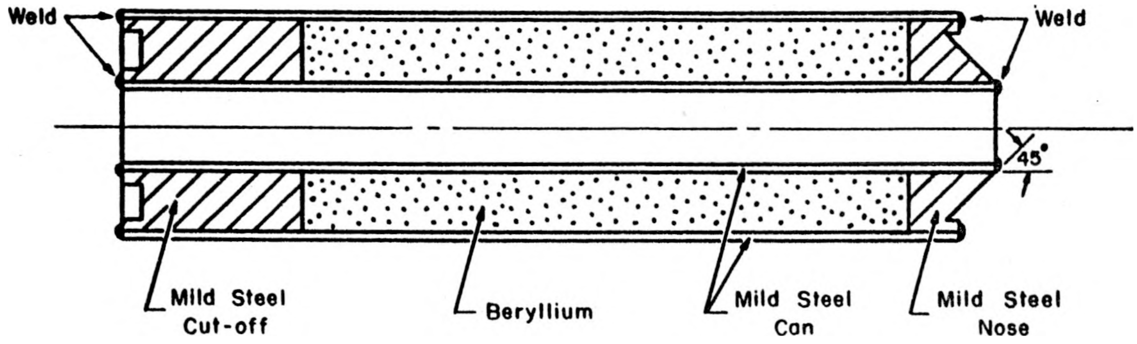


Fig. 2 - Sketch of Extrusion Billet

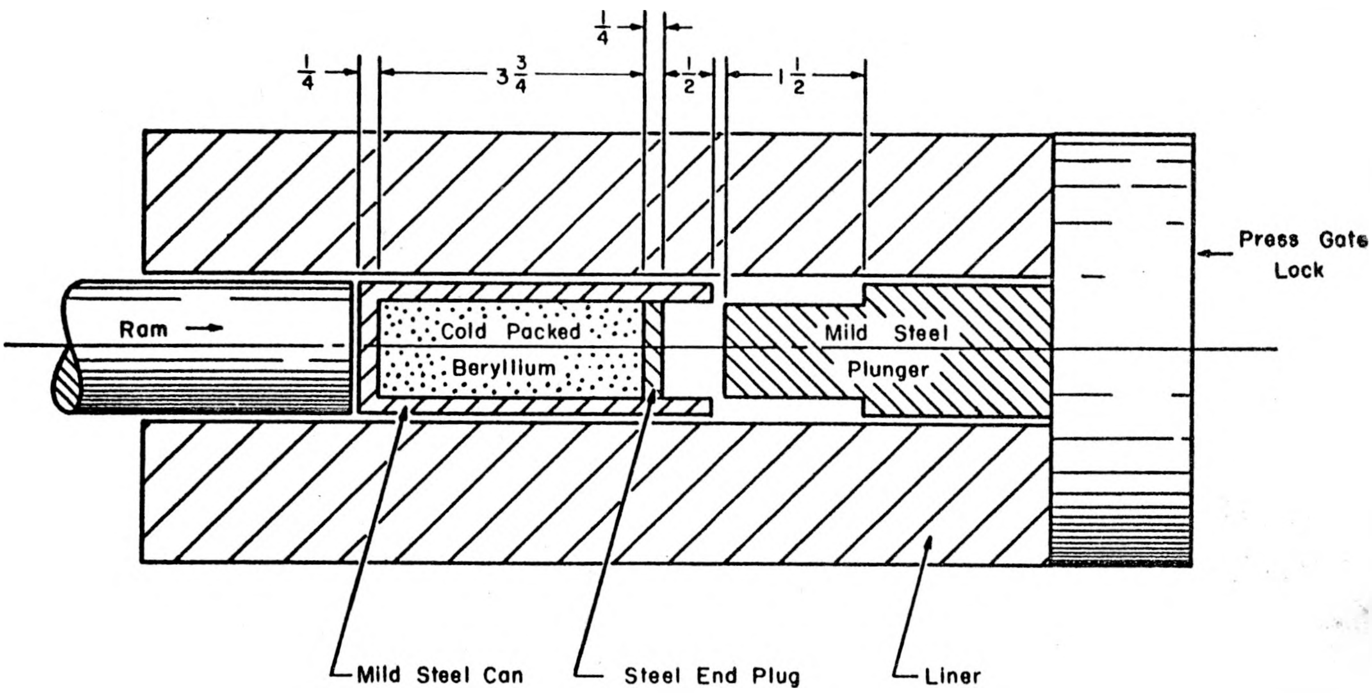


Fig. 3 - Schematic Drawing of Hot
Compacting Procedure

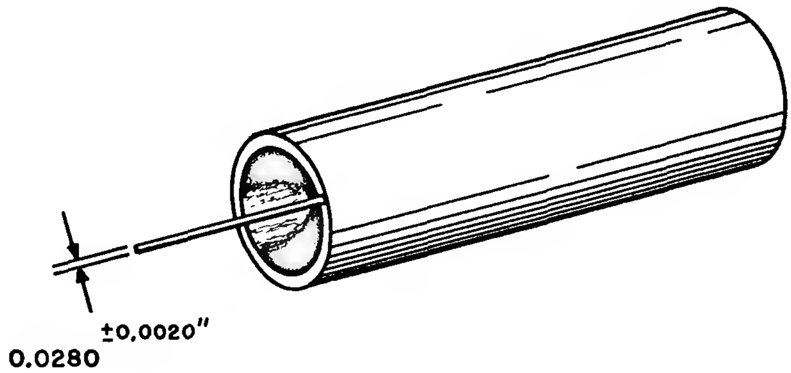


Fig. 4 - An illustration of the geometric relationship between the machined wire specimen used for texture analysis and the extruded tubing.
RA-1625

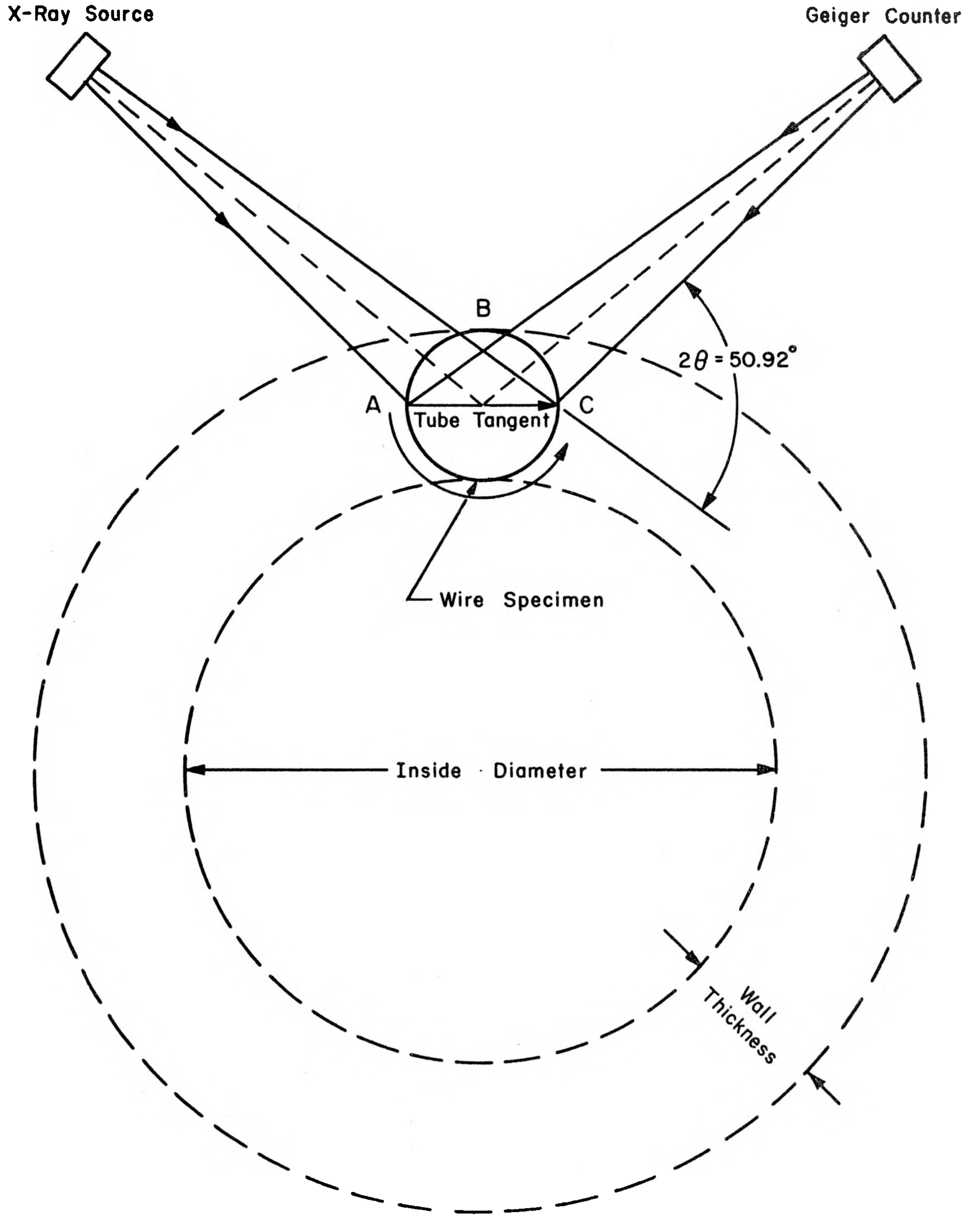


Fig. 5 - A schematic illustration of the Norton Rod method of texture analysis as applied to the wire samples taken from the extruded tubes. RA-1626

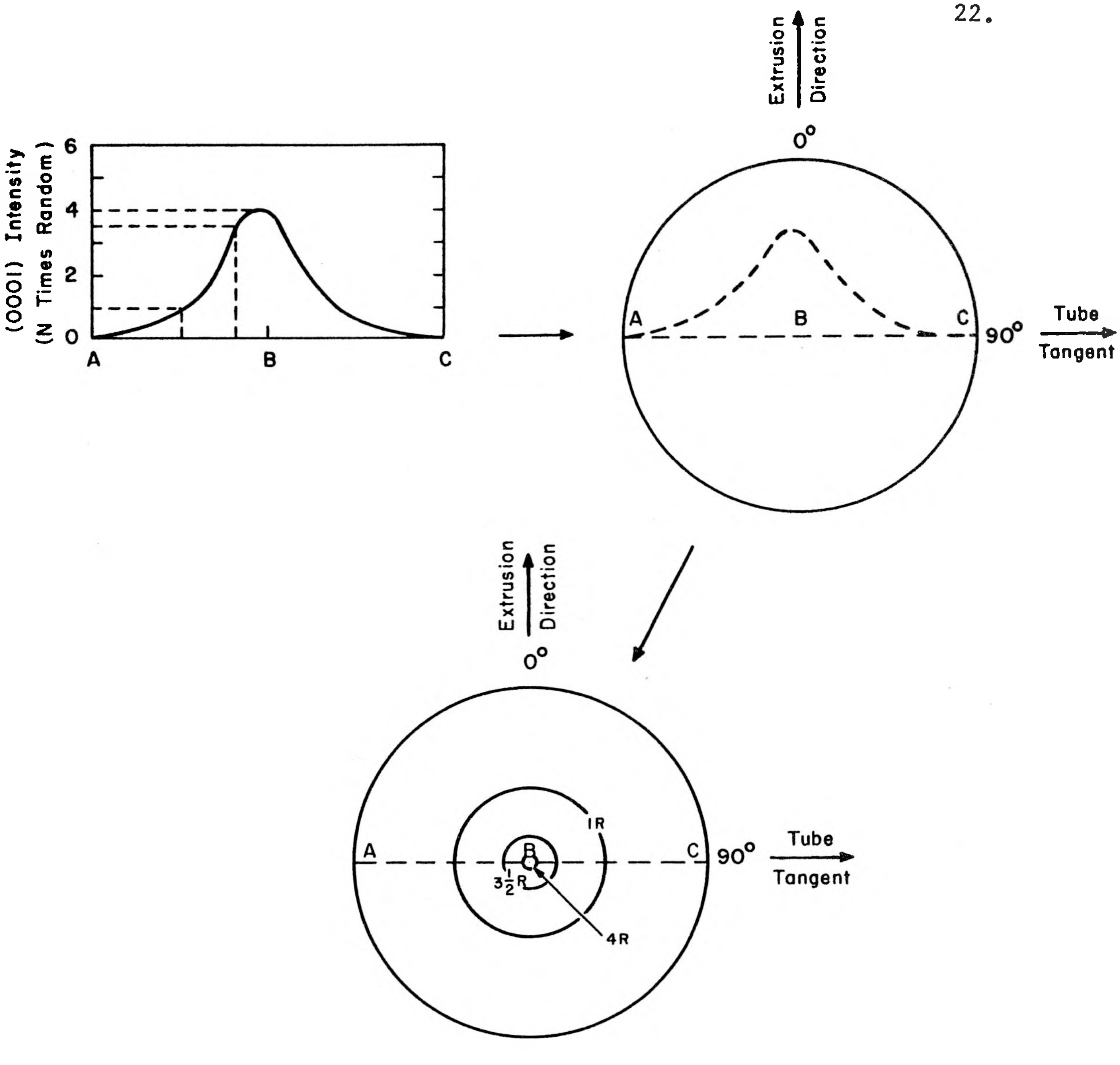


Fig. 6 - A schematic illustration of the relationship between the (0001) intensity profile (upper left) and the resulting (0001) pole figure (lower center). RA-1627