REPORT ON MEETING ON A PROGRAM RELATING TO FABRICATING BERYLLIUM ROD

Cleveland, Ohio

March 6 and 7, 1952

The meeting was held in the offices of the Brush Beryllium Company on the dates above, both meetings beginning at 9:30 AM and extending to 5:00 PM. E. R. Jette acted as chairman.

Those attending the meetings were:

E. C. Sargent* and E. W. Wallaeger - AEC.
N. W. Bass, W. W. Beaver, Bengt Kjellgren, C. W. Schwenzfeier, and Keith Wikle - Brush.
E. R. Jette, B. L. Moore and J. M. Taub - LASL.
A. R. Kaufmann, T. T. Magel and D. H. Woodard - MIT.

*Present at March 6 meeting only.

The chairman presented an agenda for discussion. The agenda items agreed upon are given in the following paragraphs. The limitations to the extent of agreement are indicated where appropriate. A few items, particularly from Part E, which were dropped as unnecessary or undesirable, are left out of the main body but are summarized at the end.

A. The Purpose of the Meeting

1. To reach an agreement between the four parties concerned on the organization and administration of a project involving research and development leading to the manufacture of shock-resistant Be rod. The parties concerned are: the Cleveland Office of the Atomic Energy Commission, laboratories at MIT under the direction of Dr. A. R. Kaufmann, the Brush Beryllium Company, and the Los Alamos Scientific Laboratory.

2. a. To determine the general objective and scope of the program and

   b. To determine a suitable sub-division among the three laboratories concerned for the initial stages of the program.

3. To decide upon the tests and testing methods to be employed by the several laboratories.

B. Organization and Administration

1. It is agreed that this work be done under contracts already existing between the Atomic Energy Commission and Massachusetts Institute of Technology (Contract No. AT(30-1)-981) and with the Brush Beryllium Co. (Contract No. AT(30-1)-541).

2. It is agreed that any financial arrangements or rearrangements necessary be made by the Los Alamos Scientific Laboratory through the Santa Fe Operations Office.

3. It is agreed that the Los Alamos Scientific Laboratory assume the responsibility for such overall technical direction as may be necessary.

4. It is agreed that the Los Alamos Scientific Laboratory assume the responsibility for approving or, if need be, inaugurating changes in the program, together with making the appropriate financial arrangements.

5. It is agreed that direct communication and consultation between the Laboratory personnel at Los Alamos, MIT and Brush Beryllium Company be agreed to by the AEC Cleveland Office on all program and technical matters with the exception of such items as may increase the cost of the project which is in turn covered by the preceding items 3 and 4.

6. It is proposed that Brush Beryllium Company and A. R. Kaufmann estimate the total quantity of beryllium metal and salts required to carry out this program for the first year of operation, and that the Cleveland Office of the AEC arrange for the allocation of this total quantity at the beginning of the program. All metal or salts returned as scrap should be credited against this allocation.

Note:

a. This item occasioned much discussion. It was finally agreed that the total allotment for work at both Brush and MIT should be 1000 pounds of Be calculated as metal.

b. MIT wants 500 pounds of metal for its experimental extrusion work but out of this will be taken about 250 pounds to meet the current purchase order from Los Alamos (Purchase Order No. BF-230 which will be issued shortly).

c. It was agreed by Brush that each "lot" or batch (~40 lbs.) of powder shipped to MIT would be made from a single casting and that different batches would not be mixed to get a better average composition.

d. Brush wants 500 pounds of Be metal equivalent but in various forms such as metal, hydroxide, sulfate, etc., since its program involves investigations of several methods to prepare high purity Be. They will withdraw these materials from current stocks as needed and return for credit any scrap material resulting.

e. No agreements were reached as to the financial arrangements involved in the transfer of metal or salts to this program. The opinion seemed to be that LASL should pay for the metal delivered to MIT for this program and should receive credit for scrap metal, e.g. butt ends, turnings, etc., when returned to Brush. The situation regarding the handling of materials for use by Brush was more complicated since several forms of Be were to be used. Much of the discussion centered on how values were assigned to the scrap or reused materials. Since these values may vary from $10 to $60 or more per pound, this is a detail which has to be worked out.

f. How the Brush contract with the NYOO through its Cleveland branch was to be modified to fit the present program, and the part LASL was to play in the financial arrangements, were discussed in connection with this item. No decisions were made except that representatives from the LASL Purchasing Department and the Cleveland branch would have to work out the details.

C. Objective and Scope of the Work

1. The object of this work is to develop a method of manufacture of shock-resistant Be rod 7/8" in diameter. (Since much of the Brush equipment is based on 1" diameter bars, they would be permitted to do their experimental extrusions with this size.) It is believed that shock-resistant in this case means that the metal must have an elongation of 20% or more and a breaking strength of 80,000 p.s.i. or more, both of these values being considered as guides rather than goals. The testing conditions will be stated in another place. (Part E below)

2. The scope of the work in general may be described as:

a. Research and development on methods of making Be metal of suitable purity and conversion to metal powder of suitable particle size distribution.

b. Research and development of methods for making 7/8" diameter rod from powders produced in (a) by extrusion techniques.

D. The General Division of the Work in the Initial Program

1. Brush Beryllium Company will investigate methods of obtaining suitably pure metal by chemical and/or electrochemical means, following in general the outline as given in Part III-b of their proposal to R. J. Van Gemert dated December 19, 1951. This outline follows:

High Purity Beryllium

The technique described above of warm pressing, sintering and warm extruding can be used as a final method of fabrication for evaluating Be powders produced by a number of methods. With this method there is no loss through machining and, consequently, small amounts of powder can be made up into 1/2" or larger bars without difficulty. It is proposed that three methods for experimental production be used, for which the bulk of the equipment is now available. These are:

a. Electrolysis of Fused Salt

Beryllium chloride will be electrolyzed at 400-500°C in a lithium chloride-sodium chloride flux in an attempt to produce high-purity flake for fabrication studies.

b. Small Scale Reduction Studies

Production of small amounts on a bench scale of pure beryllium pebbles through the reduction of beryllium fluoride of high purity with magnesium. For this method, beryllium basic acetate will be fluorinated to beryllium fluoride. The basic beryllium acetate can be prepared as the purest of any beryllium compound now known and methods for its preparation have been established. The beryllium fluoride will be reduced by magnesium electrodes which will spark through a gap filled with beryllium fluoride, reducing the beryllium fluoride to magnesium fluoride and beryllium metal, which can then be separated and attritioned under a controlled atmosphere. There should be no wall contamination by this method in that the wall would be lined with pure beryllium fluoride which will not be melted in the process.

c. Pilot Plant Operation

During the earlier work on reduction of beryllium chloride with sodium, a pilot plant for the production of beryllium powder with controlled grain size was developed (by Brush Beryllium Co.) but not put into operation. Should the combination of grain size and high purity be shown advantageous, it is proposed that this pilot plant be activated for the production of pure powder with controlled grain size. This method involves the transportation of metered amounts of beryllium chloride and sodium by means of a stream of helium passing through the respective containers of these materials into a reaction chamber. By making the reduction process isothermal, control of the size of the metal particles formed can be exercised. The sodium-chloride-beryllium product can be immediately taken from the reaction chamber and the sodium chloride removed by extraction in sodium glycolate without further heating.

2. Brush Beryllium Co. will investigate methods of preparing powders of Be metal suitable for compacting and extrusion.

3. A. R. Kaufmann will conduct investigations on the extrusion process as outlined in paragraphs 1 and 3 in the MIT proposal to the New York Office of Operations and approved by them January 21, 1952 (Ref. TA:MCK: AC approved additional work on Kaufmann's Be program as outlined in letter to MacMillan from Kaufmann, 9 January, 1952). These paragraphs state:

a. A more intensive study of the factors responsible for high ductility in extruded beryllium powders. This will consist of a laboratory investigation using metallographic and X-ray diffraction methods and a careful study of the particle size distribution and impurity content of various batches of powder. Extrusion will be carried out under various conditions in an effort to obtain more knowledge of the subject.

b. Extruded high ductility beryllium will be forged into the shape you require in the hope that this operation will promote greater ductility at the critical point. Dies for this work are being made.

4. Brush Beryllium Co. will investigate further the methods of "warm" and "hot" pressing and extrusion and combinations thereof, as well as conducting extrusions of metal thus treated as outlined in Part III-a of the proposal mentioned above in D-1. This states:

a. Powder Metallurgy

The fastest way of producing variant experimental materials appears to be that of warm pressing extrusion billets, sintering and warm extruding. It is proposed that finer powders than have heretofore been made (less than 10 microns) be fabricated by this method. These powders can be made experimentally by grinding in a dry box under an atmosphere of helium. In this way the purity of the powder, especially from oxygen, and the fineness of the particles can be controlled. The orientation of crystallographic faces can be accomplished in the manner which has produced satisfactory or, at least, promising results to date (warm and hot extrusion). On a smaller scale, it is proposed to investigate the effect of particle size on hot extrusion of warm pressed powders, but it is believed that the work could progress much faster, and more variables could be studied, using the warm extrusion method as a prime instrument of procedure.

5. Brush Beryllium Co. and A. R. Kaufmann are to coordinate their extrusion programs.

6. Brush Beryllium Co. will supply A. R. Kaufmann with beryllium powders of regular commercial grades (cf. Note c to Item B-6 above), as well as special powders resulting from D-1 and D-2 in amounts sufficient for the work under D-3.

7. Both Brush Beryllium Co. and A. R. Kaufmann will supply process information, physical and chemical test data as requested and specified by LASL, as outlined in another place. (Cf. Appendix I)

8. LASL will evaluate the results of the physical and chemical tests and perform such additional tests as may be necessary to establish the suitability of the materials for their ultimate usage. Typical samples of each of the rods made will be held for LASL and shipped to LASL on request.

9. LASL will, if desired, assist in the instrumentation for some of the data requested, and may be able to lend equipment for this purpose to Kaufmann and Brush Beryllium Co.

E. Tests and Testing Methods on the Bars

1. The predominant tests so far have been the ultimate tensile or breaking strength and percent elongation. Occasionally, the density and hardness have been measured. Microscopic studies of grain size, inclusions etc., have been made in a number of cases.

2. The following tests were agreed upon to be made routinely on metal from at least three widely separated sections of the bar:

   a. Ultimate tensile strength.

   b. Yield strength (0.2% offset).

   c. Percent elongation.

   d. Density.

   e. Microscopic examination including measurement of grain size distribution on both transverse and longitudinal sections.

   f. Preferred orientation.

3. Since tests 2a, -b and -c involve test specimens different from usual sizes, and since Be is particularly notch sensitive, agreement must be reached on the exact shop methods to be used in making the test pieces, including the etching method. Both Brush and MIT have standard methods but they are far from identical. The standard methods used by Brush and MIT are given in Appendices II and III. What method is to be used will depend upon the outcome of comparative tests.

4. For tests 2a, -b and-c, the following points require agreement:

   a. Type of grip.

   b. Reference points for elongation measurement.

   c. Strain rate.

   No agreements were reached on these details.

5. The density (test 2d) should be measured by loss of weight in a liquid of known density and results corrected to 25°C.

6. On the microscopic examination (test 2e) it was agreed that the routine tests should include two photomicrographs of longitudinal sections of bar.

   a. Polarized light with the crossed Nichols at 45° to the extrusion direction at a magnification of 250.

   b. Bright light at a magnification of 500.

Items Omitted from Original Agenda

1. Hardness Tests.

   Experience has shown that these give no important information related to quality.

2. Bend tests as used at LASL.

   No one except LASL seemed interested in these. LASL will probably continue its work with these.

3. Simple impact test.

   No agreement could be reached as to what the best form might be. LASL will try to find a test more comparable to conditions of ultimate use.

Report prepared by:

E. R. Jette
CMR-Division Leader
Los Alamos Scientific Laboratory

ERJ:vwl

SECURITY INFORMATION

APPENDIX I

Information Needed for Process of Manufacturing Beryllium Rod

I. Introduction

1. The shape desired is 7/8" diameter rod. MIT will make this size but Brush may make 1" since their dies, cans, etc., are set up on this basis.

2. The minimum length of rod extruded should not be less than 10' and preferably not less than 12'-14'.

3. The word "manufacturing" as used here implies that the method is sufficiently well standardized and described so that rod of a given set of characteristics can be made as often as desired without producing large proportions of off-standard metal.

4. The whole chain of operations leading to the production of the Be powder is equally as important as the method of forming it into a billet and the method of extrusion (or other fabrication method).

5. While Part IV refers only to extrusion, and to some extent Part III also, the equivalent information is to be supplied if other fabrication methods are used.

II. Manufacture of Be Powder

1. Method of producing the metal to be used in making the powder, including source and analysis of raw material. It is understood that the normal starting point in making powder is Be "pebbles" which are melted and cast into billets.

2. Melting and casting practice, including time of melting, holding time at pouring temperature, pouring temperature, melting refractory, mold material, gas atmosphere or vacuum conditions, type of heating (gas-fired, resistance, high frequency, etc.), and type of slag. If other methods of producing powder are used, such as vapor phase reduction, the corresponding information is to be given.

3. Method of producing the powder, e.g., grinding conditions, atmosphere, etc.

4. Chemical analysis of the powder, including nitrogen, oxygen, "insoluble matter", fluorine or other elements characteristic of the process for making the original raw metal.

5. The "insoluble matter" should be identified by X-ray diffraction methods (cf. III-4).

6. Particle size distribution including distribution below 325 mesh - by electron microscope if necessary.

III. Method of Making Billet

Note: If the billet is made by pressing the powder into a can which is then sealed and the assembly put through the heating and extrusion processes, certain of the following items of information cannot be supplied, e.g., 3, ~~4 and 6.~~

1. Method of making billet.

   a. Temperature including heating and holding time.

   b. Pressure including holding time at pressure.

   c. Compression ratio.

   d. Atmosphere around powder. Was powder degassed or otherwise treated prior to canning or pressing?

   e. Canning material, including weight, thickness and method of making and sealing can.

2. Other operations, e.g., rolling, forging, scalping, etc., if used.

3. Density of final billet; may be obtained by calculation from weight of powder and dimensions of can.

IV. Method of Extrusion

1. Heating time and temperature of billet. Time must be sufficient to get uniform temperature throughout billet.

2. Time from withdrawal from furnace to start of extrusion. Order of assembly of the extrusion components, e.g., lubrication, insertion of die, insertion of cone, insertion of can, graphite block, etc. Temperatures of heated components of assembly should be noted, if important.

3. Extrusion temperature, if different from 1.

4. Reduction in area.

5. Ram speed as function of time during extrusion.

6. Extrusion pressure, preferably as a function of time.

7. Temperature of rod as it emerges from die.

8. Cooling time down to, e.g., 250°C. This should be recorded continuously. A possible alternate to this would be to run the rod into a trough filled with a quenching oil.

9. Cooling conditions; e.g., resting on runout table, enclosed in pipe, in special atmosphere?

10. Straightening method used, if any. (Technique should avoid necessity for straightening.)

11. Type and shape of die, material of die, initial temperature of die, use of graphite or other materials between billet and ram, lubrication of die, if any.

12. Quenching temperature and other conditions, if quenching is done.

13. Condition of can after extrusion (wrinkling, etc.).

14. Condition of iron cone at front of extrusion can.

15. Abnormal conditions such as failure to complete the extrusion, jamming of press, slow assembly, etc.

V. Analytical Methods

1. Descriptions of the analytical methods used should be distributed to each of the laboratories concerned.

2. Unusual features of the analyses should be noted on the data sheets if important.

ERJ:vwl

Appendix II

~~Brush~~

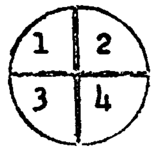
MACHINING OPERATIONS FOR CASE TENSILE SPECIMEN

as used by Brush Beryllium Co,

Material: Y-5480, Warm Pressed and Hot Extruded to 1.6" round.

1. Radiac

From 1.6" dia. by 1.625 long, quarter as shown:



2. Lathe

A. Operational Sequence

Holding each sector in four jaw chuck, the excess stock was turned off half length to .510" dia. on all three pieces.

By holding the pieces in a collet, the other halves were turned to .510". The pieces were faced to length of 1.500" and center drilled on both ends. Then the pieces were held between centers, using a special dog for driving and a live center in the tail stock. The roughing operations were as follows:

1. Rough .300"± .001" dia. on all pcs. to .310"
2. Rough .150"± .001" dia. on all pcs. to .160"
3. Rough in .156" step on all pcs. to .250"
4. Rough gage length to .500"

The finish operations were as follows:

1. Finish turn .510" dia. to .501"
2. Finish turn .300" dia. to .301" and face 5/32 shoulder
3. Finish turn .150" dia. to .151" and gage length to .600"

B. Speeds, Feeds and Depth of Cut

1. Roughing .500", .300", .150" dia.
   450 RPM
   58.5 S.F.M.
   .003" Feed

   The excess stock of quarter section to .510 dia. is removed in two cuts

   Roughing .300" dia.
   1st Cut .100"
   2nd " .060"
   3rd " .030"

Roughing .150" dia.

1st	Cut	.070"
2nd	"	.040"
3rd	"	.025"
4th	"	.015"

2. Finishing .500", .300", .150" dia.

450 RPM
58.5 S.F.M.
.003" Feed

The remaining .010" stock on each dia. is turned off in four cuts

1st	Cut	.005"
2nd	"	.003"
3rd	"	.001"
4th	"	.001"

3. Heat Treatment and Polishing

A. Polish (Lathe)

The following dias. are polished in lathe, .500" and .300", using No. 1 paper for roughing and 600 for finish.

Drill Press

The .150" dia. is longitudinally polished, using No. 1 polishing paper

B. Heat Treatment

750° C. for 20 min. hanging specimens vertically in carbon jig. No. furnace atmosphere used.

C. Polish (Drill Press)

The .150" dia. is finish polished with No. 600 paper by longitudinal method removing oxide caused by heat treating.

D. Chemical Polish

Each sample is chemically polished in the following bath for 60 seconds at 110-120° C.

Chemical Polish for Beryllium

Conc. ortho-phosphoric acid	450 ml.
" sulfuric acid	26-1/2 ml.
Chromic Anhydride	53-1/4 g.

Add CrO_3 to ortho-phosphoric acid. Heat to 150° C. to dissolve CrO_3. Cool to 110° C. and add H_2SO_4.

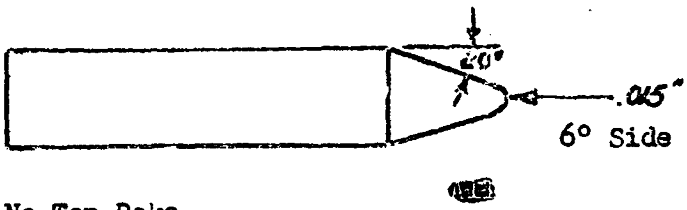
4. Tools

Turning Tools

3/8 Sq. Kennametal Carbide Tip Tool K-6 Grade for all Operations

Rough all dia.

20° on both sides



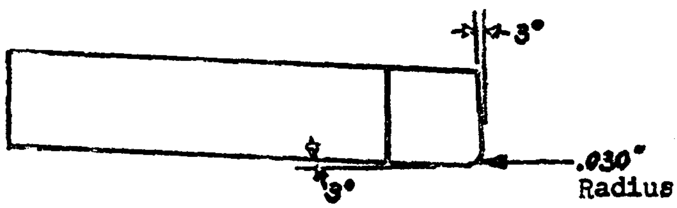
Radius approx.

6° Side Clearance All Around Tool

No Top Rake

This enables tool to cut in both directions.

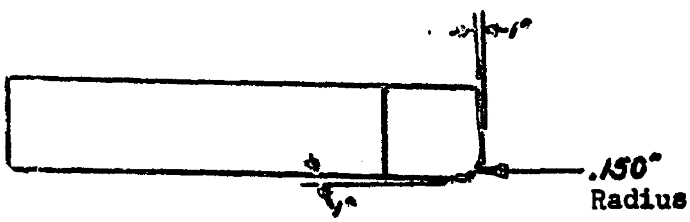
Finish .500, .300 dias. and face 5/32 step



6° Side clearance

6° End clearance

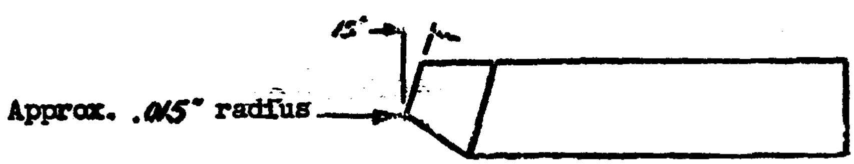
Rough and finish turn .150 dia. and gauge length



6° side and end clearance

No top rake.

Facing Tool



6° Side clearance

2° Back rake

Appendix III

MIT

INSTRUCTIONS FOR MACHINING TENSILE SPECIMENS

as used by A.R. Kaufmann's laboratory at MIT

1. Cut pieces off extruded rod roughly 4 15/16" long

2. Chuck in collet and face ends to 4 13/16" long

3. Layout centers in each end and center both ends same depth. Chuck combined drill and countersink in headstock, hold specimen in left hand with one end on tailstock dead center.

4. Place between centers and turn down to .743" (major diam.) for 3/4" -16NF-2 thread and chamfer the corners.

5. Grind 3/4"-16NF-2 thread, 1 1/2" long on both ends, using the following schedule:

   Norton Wheel Specification 38A120-J7VBE 18 x 3/8 x 9 E face.

   Run wheel spindle at 2290 R.P.M.

   Work speed 20 R.P.M.

   Important: Dress the wheel face after each cut; two dressings of .001" each.

   Depth of successive cuts:

first pass	.010" deep
second pass	.010" deep
third pass	.010" deep
fourth pass	.005" deep
fifth pass	.003" deep
sixth pass	.002" deep

We use an Ex-Cell-o Style No. 33 thread grinder.

6. Return to lathe and turn down gage length using a lathe speed of approximately 300 rpm. Hold work in .750 collet using .003 brass shim stock as a bushing to make up difference in diameters. Support free end on tailstock center.

   (a) Make two grooves about 1/16" wide with parting tool to .400" diam. Linear dimension over outside of grooves .990".

   (b) Rough out solid metal between grooves to .400" diam. using .005" deep cuts only.

   (c) Again make two grooves as before 1/16" wide to .260" diam. maintaining the .990" linear dimension.

   (d) Turn out solid metal between grooves to .260" diam. using .005" deep cuts only.

   NOTE: Bear in mind that depth of damage (twinning and micro-cracks) roughly equals depth of cut. All lathe tools are Grade 883 Carboloy.

   (e) Turn all radii using 3/8" radius tool, using plunge cuts. Leave .010" on the diameters and lengths for grinding allowance.

   NOTE: Turn radii nearest the collet only in first set-up. Then reverse work piece and turn remaining radii. In this way the wide cuts are supported close to the collet.

7. Finish specimen on cylindrical grinder. Brown & Sharpe No 1 10-12,000

   (a) Norton Wheel Specification 39C100-M7V

   Work speed set at 633 R.P.M.

   Dress 3/8" radius on each side of wheel and grind gage length (using .0005" depth of cut) to within .001" of finished diameter.

   NOTE: Do not traverse wheel the full inch or it will break down on the radii. Stay about 1/16" short.

(b) Grind the two radii adjacent to gage length by backing wheel away and then using plunge cut.

(c) Then bring gage length to finished .250" diam. by traversing cut.

(d) Then finish the two outer radii by plunge cuts.

NOTE: The above mentioned speeds and feeds are only suitable for 3/4" -16NF size specimens. Speeds and feeds must be modified to suit other diameters.