16

⑤ 649400

64-10

AD No. 432452
DDC FILE COPY

NP-13425
Report Number

432452

DDC
MAR 13 1964
RECEIVED
TISIA B

United States Atomic Energy Commission
Division of Technical Information

OTI-131(May 1962)
16p $1.60

USAEC Division of Technical Information Extension, Oak Ridge, Tennessee

UNCLASSIFIED

NP

13425

1 OF 2

DTIE
MICROCARD
ISSUANCE
DATE

1/24
1964

(5) 649700

N-13425

(6)

BERYLLIUM RESEARCH AND DEVELOPMENT PROGRAM

(7)

QUARTERLY PROGRESS REPORT TO
AERONAUTICAL SYSTEMS DIVISION
FOR THE PERIOD
JANUARY 1, 1963 THROUGH MARCH 31, 1963

(11) AUG~~UST~~ 1963

NUCLEAR METALS, INC.
WEST CONCORD, MASSACHUSETTS

(15) Contract No. AF 33(616)-7065
(17) Task Nos. 73518 and 735104

A1

ABSTRACT

The strength and ductility above room temperature of the hot-pressed Brush and Pechiney powders being studied at Lockheed show the usual behavior for beryllium. This material exhibits little response to aging heat treatments. Extruded Brush and Pechiney beryllium possess greater strength and ductility in the transverse direction than in the longitudinal direction at temperatures below about 20°C. This appears to be due to twinning in the longitudinal specimens.

Tensile specimens are being prepared at Nuclear Metals from single crystals of SR Pechiney and of single distilled beryllium. These crystals were obtained from metal melted and slowly cooled in BeO crucibles.

TABLE OF CONTENTS

LIST OF FIGURES

I. INTRODUCTION

This report describes the progress made on the Beryllium Research and Development Program for the period January 1, 1963 through March 31, 1963. Work at all Contractor sites has been terminated except for work at Lockheed and Pechiney. Final reports from these two Contractors are expected on August 1, 1963. The final report from New England Materials Laboratory is expected on June 21. The first draft of the final reports has been received from Franklin Institute and from National Research Corporation. The first draft of the final reports for the work carried out at Nuclear Metals is expected July 1, 1963.

II. PROGRESS AT SUBCONTRACTOR SITE

A. Subcontract No. 5 - Lockheed Missiles and Space Company - M. I. Jacobson and E. E. Underwood - Metallurgical Factors Affecting the Ductile-Brittle Transition in Beryllium.

1. Introduction

During the past quarter, the research effort was concentrated on completing tests on hot-pressed Brush and Pechiney beryllium, and obtaining tensile data on extruded samples. Tensile tests were made over the range -195°C to 100°C for the extrusions. The results obtained were unexpected in that at low temperatures, the transverse samples exhibited better strength and ductility than the longitudinal samples. This behavior can be attributed to twinning.

Professor John Dorn of the University of California, Berkeley, has been engaged as a consultant on this project to aid in the interpretation of the data.

2. Hot-Pressed, Randomly Oriented Material

a. Tensile Tests

Tensile tests were completed on samples machined from hot-pressed blocks of Pechiney and Brush powder. The results are summarized in Figs. 1 - 3 and are typical of beryllium with a random orientation. In the figures, the grain sizes corresponding to the sample designations used are as follows:

Designation	Mean intercept grain diameter, microns
PEC-1	13.2
PEC-2	11.0
PEC-3	10.3
QMV-4	26.5
QMV-5	18.9
QMV-6	9.7

The mean intercept diameter represents the mean distance across a grain, averaged over all angles and positions and over all three dimensions. The mean diameter is the physically significant distance transversed by the dislocations in a polycrystalline aggregate of grains randomly oriented in space.

In general, there was considerably more scatter in the data for the Pechiney material, particularly for the two larger grain sizes. As many as four samples were tested at one temperature for a single grain size in order to establish a suitable curve. The problem is particularly apparent for the PEC-1 samples at 400°C, where the elongation values ranged from 3 to 12 per cent. As a result, the curve of elongation vs temperature (Fig. 3) for PEC-1 is shown as a band at 400°C. This temperature probably represents the ductile-brittle transition temperature for PEC-1, in which case some specimens would be expected to fail in a brittle fashion and others in a ductile fashion.

Comparison between the properties of the Brush and Pechiney material is complicated because the grain sizes are not exactly the same. However, for the PEC-3 and QMV-6 samples, the grain sizes are 10.3 and 9.7 microns, respectively, which is close enough for a comparison to be made. If the properties of these are examined in Figs. 1 and 2, it will be noted that the ultimate and yield strengths are slightly higher for the Brush material.

Comparison of the elongation vs temperature curves, Fig. 3, shows that the elongation of the Brush beryllium rises gradually to 400°C, so there does not appear to be a particular temperature that could be identified as a ductile-to-brittle transition temperature. On the other hand, the elongations for the Pechiney samples increase rapidly after reaching a certain temperature, and in two cases, decrease after obtaining a maximum. The decrease is believed to be due to the onset of intergranular failure; this will be checked metallographically.

Overall, the higher purity Pechiney material does not appear to offer any advantages over the Brush QMV material in the temperature regions investigated.

An attempt to analyze the data for the Brush QMV material in terms of Cottrell's ductile-brittle transition theory for body centered cubic metals was not successful (January 1963 Letter Report). This is not too surprising if one considers that the assumptions made for b.c.c. metals are not particularly applicable to h.c.p. metals. Moreover, no sharp transition from ductile-to-brittle behavior was observed for the Brush samples, the assumption being made that the ductile-brittle transition temperature was that for which the elongation was 3 per cent. The Cottrell theory predicted a variation of transition temperature, Tc, to square root of grain size, $d^{1/2}$, of $\delta T_c/\delta d^{1/2} = 1060°C/mm^{1/2}$, whereas the observed value was $2500°C/mm^{1/2}$. Complete details of the calculation will be presented as an appendix to the final report.

The Cottrell treatment has not been applied to the Pechiney material as yet, but is not expected to be particularly helpful, one reason being there is not a sufficient spread in grain sizes to permit a valid analysis.

b. Effects of Aging and Pre-Straining Treatments on Mechanical Properties

As described in the December Quarterly Report, heat treatments of 6 hours at 800°C, followed by furnace cooling, did not significantly alter the room temperature tensile properties of the Brush or the Pechiney material. Brush samples annealed at 800°C for 1 hour, air cooled, and aged for 40 minutes at 600°C also showed no improvement.

A heat treatment in which samples were annealed for 1 hour at 1000°C, air cooled, and aged for 96 hours at 700°C gave the following room temperature properties:

Designation	0.1% Offset Yield Strength psi	Ultimate Tensile Strength psi	Elongation in 1.5", %
PEC-1	27,900	30,500	0.7
PEC-2	29,400	33,600	0.8
PEC-3	34,400	42,400	1.1
QMV-4	25,300	27,400	0.7
QMV-5	29,400	33,200	0.8
QMV-6	38,500	44,800	0.9

Again, the properties are substantially the same as those for untreated samples. If any redistribution of metallic impurities has resulted from the heat treatments used to date, it has not been sufficient to affect the room temperature properties.

If impurities are responsible for hindering dislocation movement in beryllium at room temperature, then straining at an elevated temperature should free the dislocations from impurity atmospheres, and provide a high number of freely moving dislocations to improve room temperature ductility. A series of samples was therefore strained 2 per cent at 400°C and then tested at room temperature, with the following results:

Designation	0.1% Offset Yield Strength psi	Ultimate Tensile Strength psi	Elongation in 1.5", %
PEC-1	32,500	33,700	0.5
PEC-2	31,000	31,000	0.4
PEC-3	40,400	42,300	0.5
QMV-4	30,000	30,400	0.5
QMV-5	33,800	34,400	0.5
QMV-6	44,300	49,600	1.1

This treatment results in a slight increase in yield strength and a slight decrease in ductility. Unfortunately, serrated stress-strain curves were observed at 400°C, particularly for the coarse-grained QMV samples, which is indicative of repeated yielding. Thus, at 400°C, impurities diffuse so rapidly that any dislocations that were freed were immediately relocked, and room temperature ductility was not improved.

A few tests are planned in which pre-straining would be done at a lower temperature, say 200°C, to see whether or not the idea has any merit.

3. Extruded Material

a. Tensile Tests

The results of tensile tests made to date on Brush and Pechiney extrusions are shown in Figs. 4 - 9. The curves drawn through the points are not necessarily final, as some additional testing remains to be done. Since most of the samples exhibited fair ductility at room temperature and 100°C, it was necessary to test at subzero temperatures in an effort to determine whether or not a ductile-brittle transition temperature exists.

The measurements necessary for determination of grain size in the extruded samples have not been completed, and the samples are designated according to the mesh size of the original powders as follows:

Designation	Origin	Mesh Size
QMV-1	Brush	- 60 + 80
QMV-2	Brush	-150 + 200
QMV-3	Brush	-325
PEC-4	Pechiney	- 50 + 120
PEC-5	Pechiney	-120 + 200
PEC-6	Pechiney	-200 + 350

An 'L' after the sample designation indicates that the specimen axis is parallel to the extrusion direction, in which case slip should be primarily on prism planes. A 'T' indicates a sample with its axis at 45 degrees to the extrusion direction, in which basal slip should be predominant. That this is so is indicated on the curves of yield strength vs temperature, Fig. 5, where it is obvious that the yield strengths for the longitudinal specimens are considerably higher than for the transverse.

An interesting phenomenon may be noted in connection with the curves of elongation vs temperature, Figs. 6 and 9. When longitudinal and transverse samples of the same extrusion are compared, such as QMV-3-L and QMV-3-T, there exists a temperature below which the elongations for the transverse samples are greater than for the longitudinal samples. For example, this temperature is 10°C for samples taken from extrusion QMV-3. The reason for this did not become apparent until tests were made at liquid nitrogen temperature, -195°C. At this point, the longitudinal QMV samples

exhibited jagged stress-strain curves characteristic of twinning, while the transverse samples did not. Subsequent metallographic examination proved that this was the case; the longitudinal samples had a heavy band of twins along the central portion of the sample. Only a very few twins could be seen in the transverse samples.

Further metallographic examination will be made of samples tested at higher temperatures to determine the extent of twinning.

The lower ductility of the longitudinal samples, when compared to the ductility of the transverse samples below a certain temperature, is therefore believed to be due to premature failure caused by the generation of large numbers of twins and by the initiation of cracks at twin-matrix interfaces, or at the intersections of twins and grain boundaries.

Figure 6 also indicates an anomalously low ductility for the longitudinal samples tested at -75°C. This temperature was obtained with a dry ice-acetone bath. Temperatures in the vicinity of -150°C were obtained by immersion in isopentane cooled with liquid nitrogen. It is possible that the acetone adversely affected ductility while the isopentane did not. This will be checked by testing at -75°C in isopentane.

The behavior of the yield stress vs temperature curves, Fig. 5, for the Brush samples is also interesting. The rate of decrease of yield strength with temperature for the longitudinal samples, which are supposed to deform primarily by prismatic slip, is not nearly as great as we would expect on the basis of single crystal studies. As a matter of fact, the rate of decrease is not much greater than for the transverse samples. Again, this is believed to be due to the generation of twins, which would act first as a stress relief mechanism and then as a crack nucleation mechanism. Strictly speaking, the "yield stress" curves are actually curves of flow stress vs temperature at 0.1 per cent strain, and it may not be valid to draw comparisons between these curves and the critical resolved shear stress curves for single crystals.

The curves of tensile strength vs temperature for the Brush samples, Fig. 4, are also indicative of twinning. In general, the tensile strength of the longitudinal samples decreases below room temperature and the tensile strength of the transverse samples increases. An exception is at -195°C, where the tensile strengths for QMV-1-T and QMV-2-T show a decrease.

The curves for the Pechiney samples do not present as clear a picture and the data are merely presented without comment at the present time. More testing and metallographic work will have to be done to try and determine the reasons for the anomalies on the curves.

As yet, no attempt has been made to analyze the data for the extruded samples in terms of theoretical treatments of the ductile-brittle transition theory. Also, no tests have been made on heat-treated or prestrained samples.

b. <u>Pole Figure Studies</u>

A basal pole figure for QMV-2 was presented in the Fourth Quarterly Progress Report and a prism pole figure in the February Letter Report. The textures indicated in these figures are believed to be representative of all of the extruded material. An additional pole figure will, however, be made for one of the Pechiney extrusions to check this point.

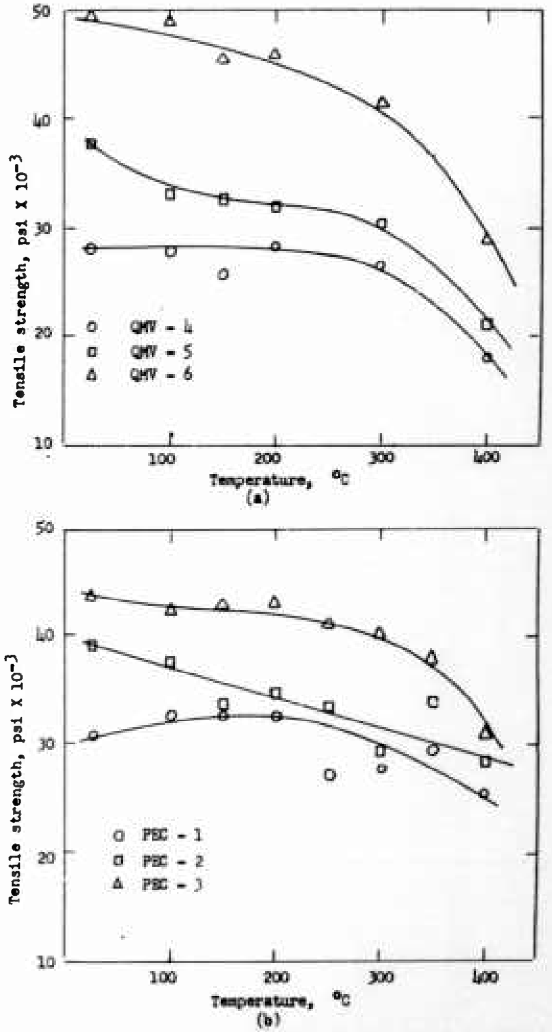

FIGURE 1. Tensile strength vs. temperature for hot-pressed beryllium. (a) Brush, (b) Pechiney.

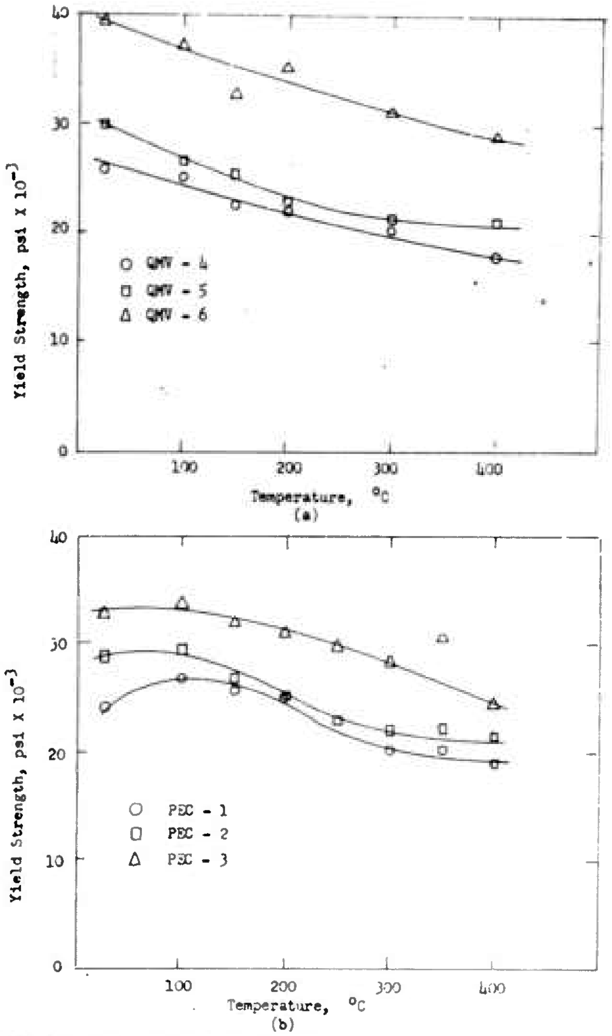

FIGURE 2. Yield strength vs. temperature for hot-pressed beryllium.
(a) Brush, (b) Pechiney.

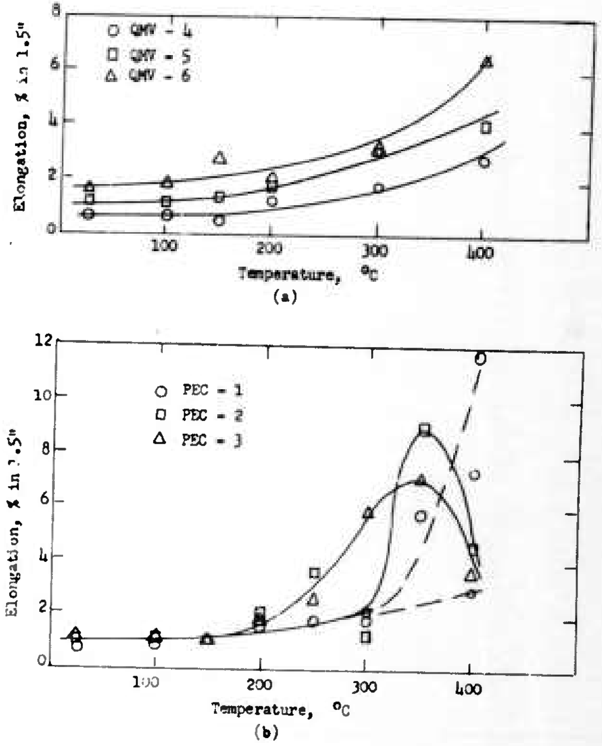

FIGURE 3. Elongation vs. temperature for hot-pressed beryllium.
(a) Brush, (b) Pechiney.

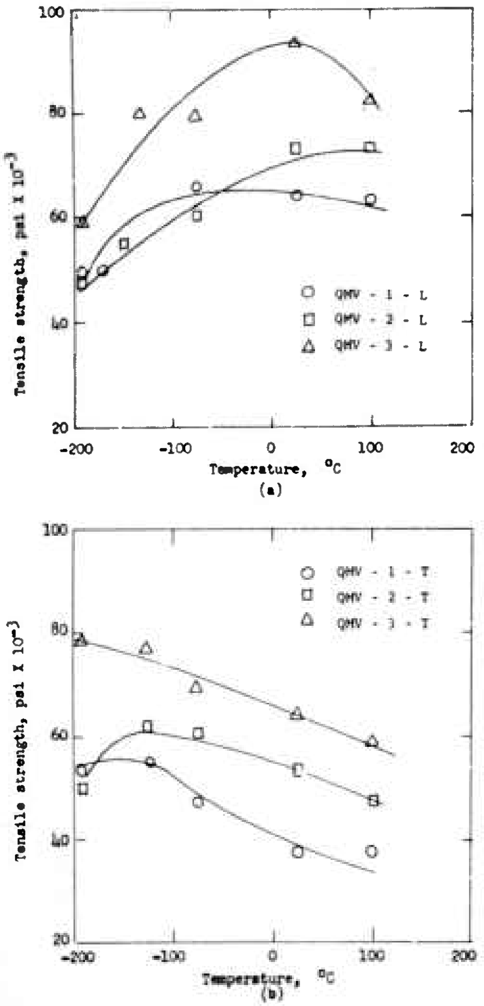

FIGURE 4. Tensile strength vs. temperature for extruded Brush beryllium. (a) Longitudinal, (b) Transverse.

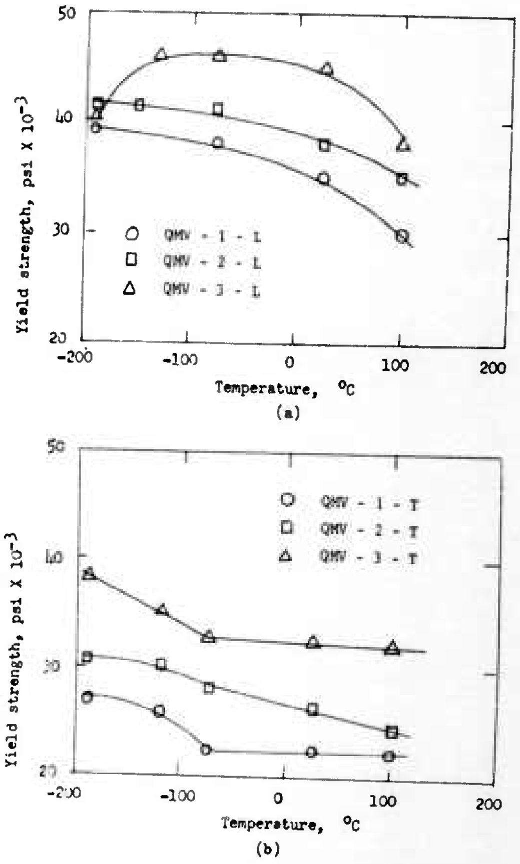

FIGURE 5. Yield strength vs. temperature for extruded Brush beryllium. (a) Longitudinal, (b) Transverse.

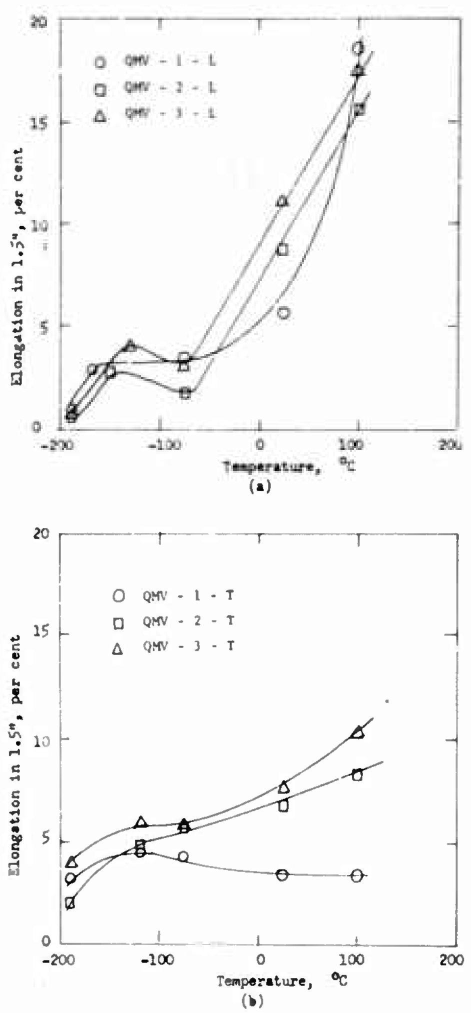

FIGURE 5. Elongation vs. temperature for extruded Brush beryllium. (a) longitudinal, (b) Transverse.

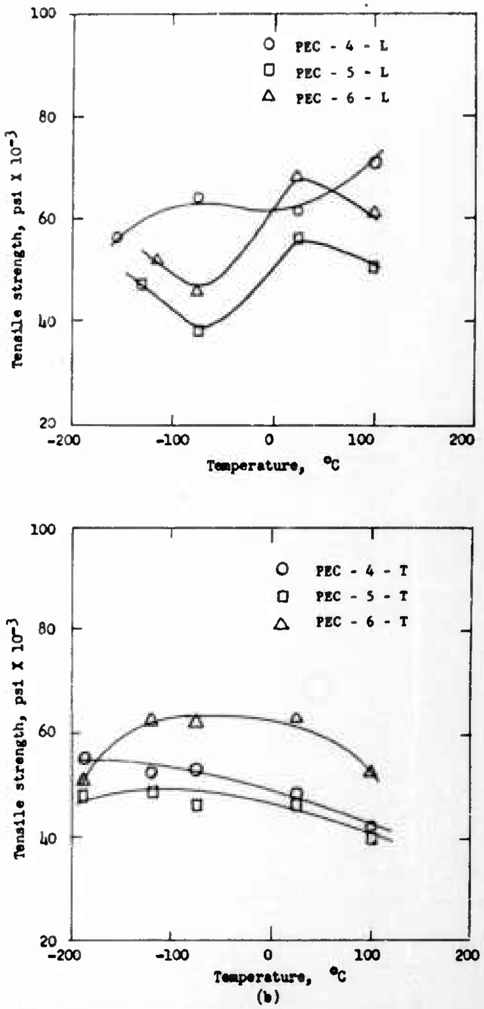

FIGURE 7. Tensile strength vs. temperature for extruded Pechiney beryllium. (a) Longitudinal, (b) Transverse.

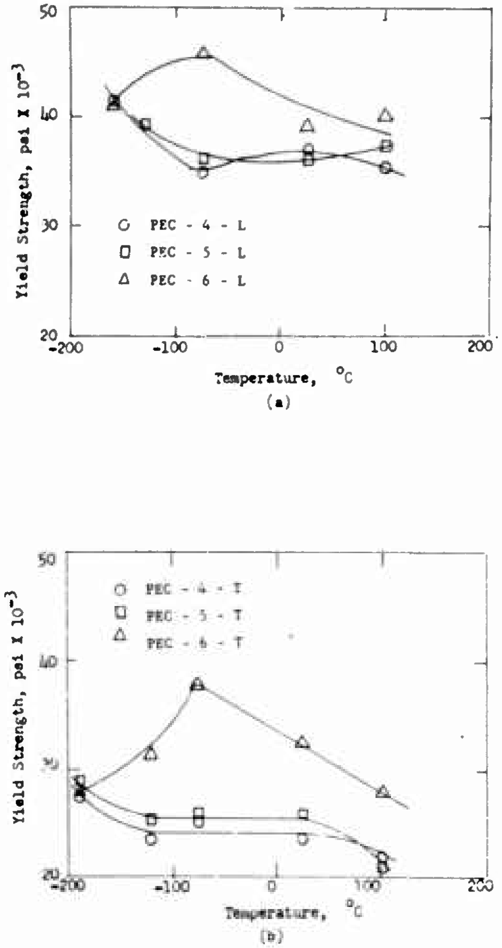

FIGURE 8. Yield strength vs. temperature for extruded Pechiney beryllium. (a) Longitudinal, (b) Transverse.

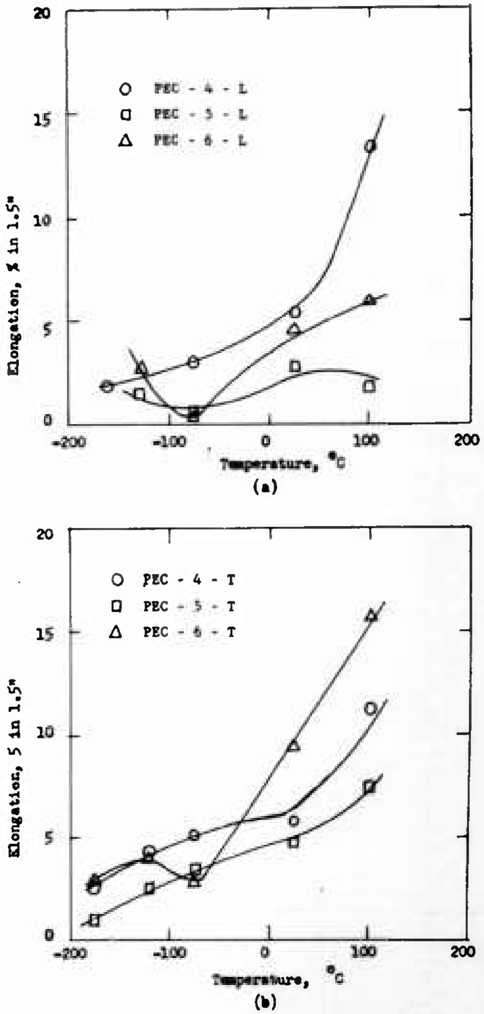

FIGURE 9. Elongation vs. temperature for extruded Pechiney beryllium. (a) Longitudinal, (b) Transverse.

III. WORK AT NUCLEAR METALS, INC.

A. Evaluation of High Purity Single Crystals Grown From the Melt - S. H. Gelles

1. Introduction

This program is aimed at determining the basal slip characteristics at room temperature of high purity single crystals of beryllium grown from the melt in a beryllium oxide crucible. Two purity levels are under investigation, SR grade Pechiney beryllium and single distilled metal produced at Nuclear Metals, Inc.

2. Experimental Work

a. Production of Large Single Crystals Grown From the Melt

As previously reported(1) in calibration experiments aimed at ensuring a proper temperature gradient from the top to bottom of the tantalum-wound furnace used for growing large crystals, it was established that, with the electrical circuit used, insufficient power was available to the cover plate. The tantalum-wound furnace and cover were therefore rewired to produce a furnace containing three separate zones: the cover; top portion of the cylindrical furnace; and bottom portion. A calibration experiment was conducted with the new circuit. The titanium block previously described(1) was heated to approximately 1390°C and then slowly cooled in such a way as to maintain a temperature gradient from the top to the bottom of the block, through the beryllium solidification temperature range. The temperature conditions sought were approximately realized in this experiment and it was felt that the operating conditions were well enough known to attempt the production of large beryllium crystals by solidification in a temperature gradient.

Consequently, 625 grams of vacuum melted SR grade Pechiney beryllium were placed in a crucible approximately 2-3/4 inches diameter by 4-1/8 inches high. The crucible and metal were set in the distillation apparatus as in the calibration run described above. A schematic diagram of the equipment is shown in Fig. 10. Temperature was monitored by two thermocouples placed at the bottom center of the crucible. The beryllium was heated above its melting point in a vacuum of $\approx 10^{-6}$ mm of Hg and allowed to solidify from the bottom up over a period of approximately 12-1/2 hours. At this point, the power to the furnace was switched off. The casting produced in this experiment (Fig. 11) had a number of large crystals with the bottom portion containing essentially two large crystals over a height of approximately 1 inch. As may be seen from Fig. 11, the casting has a line of demarcation approximately 1/4 of the way up its height, and a blow hole at its side just above this line. This is an indication that the metal did not completely solidify as intended. It appears that the billet solidified from the bottom up to this line and from downward to this line.

In an attempt to grow large crystals from distilled metal, an effort was made to counteract this situation by increasing the power to the top cover plate. Using a procedure similar to that used for the SR metal, 266 grams of distilled metal were set into the vacuum chamber. As a result of the increased power to the cover plate of the furnace, the temperature of the metal increased to approximately 1500°C (estimated on the basis of the calibration runs) at which point the melt was allowed to cool by decreasing the power to the bottom of the cylindrical section of the furnace. The casting was allowed to cool over a period of approximately 16-1/4 hours. The power was then turned off.

As a result of the smaller amount of material in this experiment, because of the bulkiness of the distilled beryllium strips, a much smaller casting was obtained than with the SR metal. The size of the crystals in this casting were quite a bit smaller than in the SR casting, and it was therefore questionable whether a crystal had been made which would be large enough for a tensile sample when cut out at the correct orientation for basal slip.

b. Tensile Sample Preparation

Large crystals of both the SR and distilled beryllium castings were isolated by cutting with a "Cimcool" - cooled silicon carbide cut-off wheel followed by etching to remove the surface damage. Crystals which had some promise of providing tensile blanks of the proper orientation (basal plane 45° to the tensile axis) were oriented using a Unicam X-ray Goniometer and a back-reflection Laue technique. Two crystals were trepanned from the SR casting and one from the distilled metal casting. Their designations are given in Table 1. The samples listed in Table 1 were entirely monocrystalline with the exception of DSC-4-1, which had a small secondary crystal at the center of its length.

Tensile samples are now being prepared by spark machining and electrolytic etching techniques from the above samples. These will be "dumbbell type" samples with a 0.5-inch gauge length and 0.100-inch gauge diameter.

c. Resistance Ratio Measurements

The ratios of resistance at room temperature to that of liquid helium temperature ($R_{R.T.}/R_{4.2^\circ K}$) have been measured on the three trepanned tensile blanks mentioned. The results are shown in Table 1. The values shown are considered to be within the range of values usually obtained for materials of these purity levels. The difference between the values for the two SR crystals may be due to an anisotropic distribution of impurities or imperfections.

d. Future Work

During the next report period, tensile samples will be fabricated from the tensile blanks and mechanical testing at room temperature will be carried out. These results will be correlated with the purity levels of the materials and compared to materials prepared by zone-refining techniques. A final report will then be prepared.

TABLE 1

Single Crystal Orientations and Resistance Ratios

Sample	ϕ_o *	λ_o **	$R_{R.T.}/R_{4.2^\circ K}$
1SR-1-1***	90°	19°	98
SR-2-1	43°	53°	68
DSC-4-1	55°	35°	298

* ϕ_o Angle between basal pole and tensile axis.

** λ_o Angle between tensile axis and closest $<1120>$.

*** This sample was inadvertently oriented for prism slip rather than basal slip.

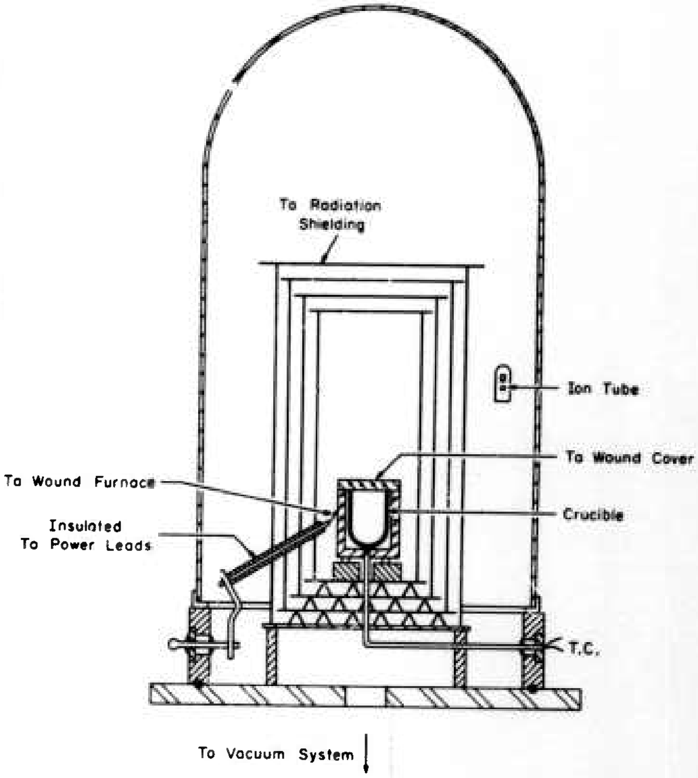

Figure 10. Schematic diagram of apparatus used for growing large beryllium single crystals.

Figure 11. View of casting of Pechiney SR grade metal showing large crystals

REFERENCES

(1) Quarterly Progress Report to the Aeronautical Systems Division for the Period October, November and December 1962 on the Beryllium Research and Development Program, NMI-9528.

END