SUMMARY REPORT

on

CHARACTERISTICS OF COMMERCIAL VACUUM-
HOT-PRESSED BERYLLIUM III--EFFECT OF
HEAT TREATMENT AND PRESSURIZATION ON
THE PROPERTIES OF HOT-PRESSED BLOCK HY

to

UNIVERSITY OF CALIFORNIA
LAWRENCE LIVERMORE LABORATORY

August, 1974

by

S. H. Gelles

AEC Contract W-7405-eng-92
SACL 151/004

BATTELLE
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

MASTER

TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT.	vi
INTRODUCTION.	1
EXPERIMENTAL.	2
Sample Preparation	2
Sample Characteristics	5
Radiographic Analysis	5
Optical Metallography	5
Density Measurements.	8
X-ray Diffraction Analysis.	8
Transmission Electron Microscopy and Selected Area Diffraction	13
Tensile Tests.	18
Fractography	20
Pressurization Experiments	32
Objective	32
Procedure	35
Results	36
Discussion.	36
CONCLUSIONS AND RECOMMENDATIONS	40
Effect of Heat Treatment on Hot-Pressed Block HY	41
Structural Changes.	41
Mechanical Property Changes	41
Fractography	41

TABLE OF CONTENTS
(Continued)

	<u>Page</u>
Pressurization Studies.	42
ACKNOWLEDGEMENT.	43
REFERENCES	44

LIST OF TABLES

Table 1. Densities of Heat-Treated Samples from Hot-Pressed Block HY	9
Table 2. X-ray Powder Pattern of As-Received HY Samples	10
Table 3. X-ray Powder Pattern of Heat-Treated DY Samples.	11
Table 4. Tensile Properties of Heat-Treated, Hot-Pressed Block HY	21
Table 5. Tensile Properties of Heat-Treated, Hot-Pressed Block HY at a Strain Rate of 38.5/Min	22
Table 6. Effect of Pressurization to 21,000 Atm (309,000 psi) on the Tensile Properties of Nonheat-Treated, Hot-Pressed Block HY and Hot Isostatically Pressed P21	37

LIST OF FIGURES

Figure 1. Position of Heat-Treatment Slabs in Billet HY (Diametrical Section).	3
Figure 2. Sample Layout in Heat-Treated HY Slabs	4
Figure 3. Enlargements of Radiographs from Identical Areas of Hot-Pressed Block HY	6
Figure 4. Photomicrographs of Longitudinal Sections of As-Received and Heat-Treated Specimens from Hot-Pressed Block HY	7
Figure 5. Transmission Electron Micrographs of Hot-Pressed Block HY	14
Figure 6. Transmission Electron Micrographs of As-Received Hot-Pressed Beryllium Block HY	15

LIST OF FIGURES
(Continued)

	<u>Page</u>
Figure 7. Transmission Electron Micrographs of Hot-Pressed Beryllium Block HY, Heat-Treated Condition.	16
Figure 8. Transmission Electron Micrographs	19
Figure 9. Transmission Electron Micrographs	19
Figure 10. Comparison of the Transverse Strengths of As-Received and Heat-Treated Hot-Pressed Block HY ($\dot{\epsilon} = 0.0385/\text{Min}$).	23
Figure 11. Comparison of the Longitudinal Strengths of As-Received and Heat-Treated Hot-Pressed Block HY ($\dot{\epsilon} = 0.0385/\text{Min}$).	24
Figure 12. Effect of Heat Treatment on the Total Elongation of Hot-Pressed Block HY ($\dot{\epsilon} = 0.0385/\text{Min}$)	25
Figure 13. Effect of Heat Treatment on the Reduction in Area of Hot-Pressed Block HY ($\dot{\epsilon} = 0.0385/\text{Min}$)	26
Figure 14. Effect of Strain Rate on the Transverse Strength of Heat-Treated Hot-Pressed Block HY	27
Figure 15. Effect of Strain Rate on the Longitudinal Strength of Heat-Treated Hot-Pressed Block HY	28
Figure 16. Effect of Strain Rate on the Total Elongation of Heat-Treated Hot-Pressed Block HY.	29
Figure 17. Effect of Strain Rate on the Reduction in Area of Heat-Treated Hot-Pressed Block HY.	30
Figure 18. Photomicrographs of Sections Parallel to the Tensile Axis of Specimens from As-Received Hot-Pressed Block HY Tested at Room Temperature and at 500 F (250X).	31
Figure 19. Replica Electron Micrographs of Fractured Surfaces of Tensile Samples Taken from As-Received Hot-Pressed Block HY Tested at Room Temperature (5100X)	33
Figure 20. Replica Electron Micrographs of Fractured Surfaces of Tensile Samples Taken from As-Received Hot-Pressed Block HY Tested at 500 F (5100X).	34

LIST OF FIGURES
(Continued)

	<u>Page</u>
Figure 21. Comparison of the Stress-Strain Behavior of Hot-Pressed Block HY in the As-Received and Pressurized Conditions.	38
Figure 22. Transmission Electron Micrographs of As-Received Hot-Pressed Beryllium Block HY Pressurized at 21,000 Atmospheres.	39

ABSTRACT

Continuing work to characterize hot-pressed beryllium block HY which had shown brittle behavior in the pressing direction at 500 F (260 C) has been concerned with the effect of heat treatment and pressure treatment on the structure and tensile properties of the metal. The heat treatment consisted of 8 hours at 1090 C (1994 F) followed by a cool at a linear rate to 750 C (1382 F) in 8 hours and then cooling to room temperature in 24 hours at a linear rate. Tensile properties were evaluated at room temperature, 300 and 500 F (149 and 260 C, respectively), at strain rates of 0.0385 and 38.5 per minute.

The heat treatment reduced the yield and ultimate tensile strengths, improved the tensile ductility, and somewhat reduced the tendency for embrittlement at the slower strain rate. However, at a strain rate of 38.5 per minute, pronounced embrittlement was still observed in the pressing direction at both 300 F (149 C) and 500 F (260 C).

Structural changes accompanying the heat treatment consisted of a small increase in grain size (8.8 to 11.2 μ), a small decrease in density (1.853 to 1.851 g/cm³), and changes in the size and type of minor phases present in the structure.

The pressurization treatment to 21,000 atmospheres (308,600 psi) was found to restore the ductility of the as-received samples of hot-pressed block HY. This observation has pinpointed the source of embrittlement in this material and in a hot isostatically pressed P-21 sample similarly treated as a deficiency in mobile dislocations probably due to solute, precipitate, or inclusion pinning.

SUMMARY REPORT

on

CHARACTERISTICS OF COMMERCIAL VACUUM- HOT-PRESSED BERYLLIUM III--EFFECT OF HEAT TREATMENT AND PRESSURIZATION ON THE PROPERTIES OF HCL-PRESSED BLOCK HY

to

UNIVERSITY OF CALIFORNIA
LAWRENCE LIVERMORE LABORATORY

from

BATTELLE
Columbus Laboratories

by

S. H. Gelles

August, 1974

INTRODUCTION

This is a summary report on SANL 151/004 for the period August through December, 1972. The work was conducted in the Materials Processing Division, H. D. Hanes, Chief. Liaison with Lawrence Livermore Laboratory was through Messers. M. Weiss, P. Landon, and J. Hauber. Previous related work has been summarized in two BCL reports to Lawrence Livermore Laboratory.^(1,2) The objective of the work was to determine the effect of a heat treatment on the mechanical properties and structural characteristics of a special hot-pressed block designated HY. It had been found in the last phase of this study⁽²⁾ that this material was embrittled at 500 F at a strain rate of 0.0385 per minute and at both 300 and 500 F at a strain rate of 38.5 per minute. The heat treatment consisted of 8 hours at 1090 C followed by a cool at a linear rate to 750 C in 8 hours followed subsequently by a cool at a linear rate to room temperature in 24 hours. Mechanical properties were evaluated at strain rates of 0.0385 per minute and 38.5 per minute, at room temperature, 300 and 500 F. At a strain rate $\dot{\epsilon} = 0.0385/\text{min}$, the heat treatment reduced both the yield and ultimate tensile strengths while the ductility somewhat improved in the embrittlement region. At $\dot{\epsilon} = 38.5/\text{min}$, the

yield and ultimate tensile strengths increased and showed pronounced embrittlement effects at both 300 and 500 F.

Structural changes on heat treatment consisted of a small increase in grain size (8.8 to 11.2 μ), a small decrease in density (1.853 to 1.851 g/cm³), and a reduction in the size and type of second-phase particles.

Lastly, it was established through a series of pressurization experiments that the ductility of the embrittled hot-pressed block HY could be restored by a room-temperature pressurization to 21,000 atmospheres. This observation coupled with fractographic studies pinpoints the basic reason for the embrittlement as a lack of mobile dislocations probably caused by solute or precipitate pinning.

EXPERIMENTAL

Sample Preparation

Four slabs nominally 5/16 inch thick by 1-1/2 inches wide and either 6-1/2 or 8-1/2 inches long were machined from hot-pressed block HY. The position of the two longitudinal and two transverse slabs in relationship to the hot-pressed block is shown in Figure 1. The slabs were machined to uniform dimensions and radiographed as described below in order to obtain a measure of the inclusion distribution. After radiography the slabs were cleaned in acetone, distilled water, and finally in alcohol. They were then individually wrapped in tantalum foil and sealed in fused silica ampoules at a partial pressure of argon amounting to approximately 170 Torr. This corresponds to approximately 1 atmosphere at the highest temperature (1090 C). The slabs were given the following heat treatment: 3 hours at 1090 C followed by a cool at a linear rate to 750 C in 8 hours and a final cool at a linear rate to room temperature in 24 hours.

After heat treatment the slabs were once again radiographed and machined into sample blanks in accordance with the diagram shown in Figure 2. Some of the samples were used for tensile tests while others

FIGURE 1. POSITION OF HEAT-TREATMENT SLABS IN BILLET HY (DIAMETRICAL SECTION)

FIGURE 2. SAMPLE LAYOUT IN HEAT-TREATED HY SLABS

See Figure 1

were used for metallography, density measurements, electron microscopy, and X-ray diffraction. The structural changes resulting from the heat treatment are summarized in the next section.

Sample Characteristics

Radiographic Analysis

As indicated above, the beryllium slabs were radiographed before and after heat treatment. Relatively low-energy, 14-kv X-rays were used in a vacuum path setup. The radiographs so obtained were enlarged five times and examined to determine the change in inclusion content and distribution. The technique used is similar to one developed at the Lawrence Livermore Laboratory.

The radiographic analysis showed a marked decrease in the visible inclusion density, especially with the very finest inclusions. This change in inclusion content is illustrated in Figure 3 where identical regions of one of the hot-pressed slabs is displayed before and after heat treatment. It may be noted from this figure that a large number of the finer inclusions have disappeared with heat treatment, whereas some of the larger inclusions appear to remain unaltered.

Optical Metallography

Metallographic examination was carried out on three samples as shown in Figure 2. Longitudinal and transverse sections of each were viewed. There was little difference noted among samples and sections. Grain-size measurements were made by the linear intercept technique and showed that a small amount of grain growth had occurred with the heat treatment ($8.8 \pm 0.3 \mu$ for the as-received compared to $11.2 \pm 1.5 \mu$ for the heat-treated material). Comparative microstructures of the as-received and heat-treated samples are shown in Figure 4.

5X

As Received

5X

Heat Treated

FIGURE 3. ENLARGEMENTS OF RADIOGRAPHS FROM IDENTICAL AREAS OF HOT-PRESSED BLOCK HY

Black specks are inclusions. The density of fine inclusions has markedly decreased with heat treatment.

As-Received
Specimen HYB11L

0G601

Heat-Treated
Specimen HYCC9T

4G256

FIGURE 4. PHOTOMICROGRAPHS OF LONGITUDINAL SECTIONS OF
AS-RECEIVED AND HEAT-TREATED SPECIMENS FROM
HOT-PRESSED BLOCK HY

Polarized light.

Density Measurements

Archimedian density determinations of six heat-treated samples were made in the same manner as previously described⁽¹⁾. The location of the samples used in the density determinations is also shown in Figure 2. The results of the density determinations are presented in Table 1 where comparison is also made with the average density previously measured in the as-received samples. A small decrease in density with heat treatment is to be noted. This is presumably due to either the transformation of foreign phases into more equilibrium structures or due to a small amount of swelling which could be produced by gas-generating reactions.

X-ray Diffraction Analysis

A sensitive Debye-Scherrer powder technique was applied to the identification of the phases present in the as-received and heat-treated samples. Powder samples were prepared from the as-received and heat-treated hot-pressed block by first cutting a small section from the block and fragmenting it in a closed-end beryllium cylinder by hammering on it with a beryllium rod. Both the cylinder and rod were made from hot-pressed block HY. Chromium K α radiation was used at power settings of 25 kv and 40 ma using a vacuum path and approximately a 10-hour exposure. This technique produces films with low-background intensity and great sensitivity to minor amounts of impurity phases.

The d spacings and observed intensities as well as the probable identification of the diffraction lines are listed in Tables 2 and 3 for the as-received and heat-treated samples respectively. Tables 2 and

TABLE 1. DENSITIES OF HEAT-TREATED SAMPLES
FROM HOT-PRESSED BLOCK HY

Sample No.	Density, g/cm ³
HYBL1-5	1.851
HYBL1-15	1.852
HYAA-11T	1.852
HYAA-17T	1.851
HYDL2-7	1.851
HYCC-11T	1.852
Heat-Treated Av $\pm \sigma$	1.8514 $\pm$ 0.0005
As-Received Av $\pm \sigma$	1.8530 $\pm$ 0.0003

TABLE 2. X-RAY POWDER PATTERN OF AS-RECEIVED HY SAMPLES

d, Å	Observed Intensity	Phase Identification	Reference Diffraction Pattern					
			Be	BeO	FeAlBe ₄	MBe ₁₂	MBe ₁₃	Be ₂ C
5.1	5	MBe ₁₃ MBe ₁₂				5.12(60)	5.16(35)	
3.65	10	MBe ₁₃ MBe ₁₂				3.64(100)	3.65(100)	
3.49	20	AlFeBe ₄			3.49(100)			
3.20	5	--						
3.09	10	--			3.01(10)		2.98(50)	
2.94	3	MBe ₁₃						
2.77	5	--						
2.69	20	--						
2.58	3	MBe ₁₃ MBe ₁₂				2.57(100)	2.58(30)	
2.51	10	Be ₂ C						2.50(95)
2.34	35	BeO		2.337(91)			2.31(95)	
2.27	3	MBe ₁₂				2.29(50)		
2.19	25	BeO		2.189(61)				
2.14	20	AlFeBe ₄			2.14(100)			2.17(2)
2.06	35	BeO		2.061(100)		2.12(15) 2.10(50)	2.10(45)	
1.98	90	Be	1.97(20)			1.96(25)		
1.905	10	--						
1.83	15	AlFeBe ₄			1.83(85)	1.83(50) 1.81(100)	1.82(10)	
1.795	90	Be	1.79(14)					
1.735	100+	Be	1.73(100)		1.74(5)	1.71(20)	1.74(10)	
1.595	10	BeO		1.598(22)		1.63(10) 1.62(40) 1.56(50)	1.72(10) 1.63(30)	
1.535	10	Be ₂ C			1.51(5)			1.535(100)
1.495	3	--						
1.485	3	--						
1.463	3	--						

TABLE 3. X-RAY POWDER PATTERN OF HEAT-TREATED BY SAMPLES

d, Å	Observed Intensity	Phase Identification	Reference Diffraction Pattern				
			Be	BeO	FeBe ₁₁	MBa ₁₂	MBa ₁₃
10.8	5	FeBe ₁₁			10.7(55)		
5.45	5	FeBe ₁₁			5.36(65)		
5.10	10	MBa ₁₂ , MBa ₁₃				5.12(60)	5.16(35)
3.61	20	MBa ₁₃			3.57(100)	3.64(100)	3.65(100)
		FeBe ₁₁ , MBa ₁₂					
3.31	3	FeBe ₁₁			3.39(25)		
3.20	12	--					
3.10	15	--					
2.93	3	MBa ₁₃			2.976(65)		2.98(50)
		FeBe ₁₁					
2.78	5	--					
2.68	18	--					
2.57	5	MBa ₁₃				2.57(100)	2.58(30)
		MBa ₁₂					
2.50	10	FeBe ₁₁			2.528(50)		2.50(95)
		Be ₂ C					
2.34	35	BeO		2.337(91)			2.31(95)
2.28	5	MBa ₁₂				2.29(50)	
2.19	25	BeO		2.189(61)	2.165(5)		2.17(2)
						2.12(15)	
2.06	30	BeO		2.061(100)	2.067(45)	2.10(50)	2.10(95)
					2.029(5)		
1.98	90	Be	1.97(20)			1.96(25)	
1.905	18	--			1.928(20)		
1.861	3	--					
1.830	5	MBa ₁₂			1.839(60)	1.83(50)	1.82(10)
		FeBe ₁₁				1.81(100)	
1.790	85	Be	1.79(14)		1.787(85)		
1.735	100	Be	1.73(100)				1.74(10)
					1.697(30)	1.71(20)	
					1.636(5)	1.63(10)	1.72(10)
1.595	7	BeO		1.598(22)	1.599(5)	1.62(40)	1.63(30)
1.533	10	MBa ₁₃			1.531(1)	1.56(50)	1.535(100)
		MBa ₁₂					
1.495	4	--					
1.480	4	--			1.488(15)		
1.458	5	--					

3 reveal the presence of BeO , Be_2C , MBe_{12}^* , and MBe_{13}^* common to both the as-received and heat-treated samples.

The major difference noted in the diffraction patterns of the two types of samples was the presence of the ternary compound FeAlBe_4 in the as-received sample and its complete disappearance and replacement in the heat-treated sample by FeBe_{11} . Although there are many diffraction lines that are common to both patterns, the presence or absence of the diffraction lines at 10.8 Å (FeBe_{11}) and 3.49 Å (FeAlBe_4) are among the most distinctive.

In addition to the compounds identified above, a number of extra diffraction lines appear in the patterns of both the as-received and heat-treated samples. These are at d spacings of 3.2, 3.09-3.10, 2.77-2.78, 2.68-2.69, 1.905, 1.495, 1.480-1.485, 1.458-1.460 Å, etc. These extra diffraction lines could not be attributed to any of the known inclusions in beryllium or to compounds containing beryllium. Further effort is required for the solution of this pattern.

The X-ray diffraction results confirm those made earlier by other investigators^(3,4) that if a high-solutionizing temperature ($> 900^\circ\text{C}$) is used, the precipitate that forms during cooling tends to be FeBe_{11} rather than the ternary, FeAlBe_4 , which forms after solutionizing at relatively low temperatures.

This phenomenon may be explained in terms of the diffusion of iron to regions well away from the grain boundaries during the high-temperature diffusion anneal. This process reduces the iron content in the vicinity of the aluminum which tends to be segregated at the grain boundaries even after the high-temperature anneal. Also since the iron concentration is now

* The MBe_{12} group of compounds is tetragonal with $a = \sim 7.2$ and $c = \sim 4.2$. This compound is formed in a number of binary beryllium systems (M can be chromium, manganese, titanium, etc.). The MBe_{13} is cubic with $a \sim 10.3$ Å. In this case, M can be magnesium, calcium, uranium, cerium, etc. Presumably in both cases, these elements can substitute for one another and form a ternary compound having the same structure. There is no easy way to distinguish which element or elements make up the compound, save, isolating individual inclusions and analyzing them either chemically or by means of the electron beam probe.

relatively high in the matrix, there is more of a tendency to form the binary compound FeBe_{11} after cooling from the highest annealing temperature. Low annealing temperatures do not allow the iron to diffuse far enough away from the aluminum to prevent the ternary compound from forming during cooling. Thus, the ternary compound present in the as-received HY hot-pressed block appears to be a nonequilibrium precipitate resulting from the fact that the solute elements have not been well dispersed throughout the structure but tend to segregate at the grain boundaries where the local composition is quite different from that of the alloy as a whole.

Transmission Electron Microscopy and Selected Area Diffraction

Transmission electron microscopy of the as-received and heat-treated specimens was carried out on thin sections. Chemical milling and electrolytic polishing procedures were used to produce the thin sections as previously described.⁽²⁾

The major change in the structure of hot-pressed block HY on heat treatment is clearly shown in Figure 5 where it may be seen that most of the coarser particles present in the as-received material are either completely or partially dissolved during the heat treatment. It is probable that during the cooling portion of the heat treatment some precipitation has occurred but in a form finer than the original inclusions.* These observations are consistent with the radiographic observations discussed above and illustrated in Figure 2.

Higher magnification photomicrographs of the as-received and heat-treated specimens are presented in Figures 6 and 7, respectively. Both specimens show relatively low dislocation densities (Figures 6a and 7a).

* It should be pointed out that the transmission electron microscopy observations are most sensitive in detecting finer inclusions ($< 2 \mu$). The coarser inclusions or precipitates probably are removed from the specimen during the thinning operation.

10 μ

2500X

As Received

2513

2500X

b. Heat Treated

2660

FIGURE 5. TRANSMISSION ELECTRON MICROGRAPHS OF HOT-PRESSED BLOCK HY

FIGURE 6. TRANSMISSION ELECTRON MICROGRAPHS OF AS-RECEIVED HOT-PRESSED BERYLLIUM

100

15,000X

2688

FIGURE 7. TRANSMISSION ELECTRON MICROGRAPHS OF HOT-PRESSED
BERYLLIUM BLOCK HY, HEAT-TREATED CONDITION

b.

c.

FIGURE 7. (CONTINUED)

However, some isolated regions showed heavily tangled dislocations and evidence of pinning by fine precipitates (Figure 6b), whereas another region showed relatively straight dislocations and dislocation loop formation (Figure 6c). One region of the heat-treated specimen contained a large concentration of fine precipitates (Figure 7b). A modulated structure similar to that caused by stacking faults is also observed in this region. Evidence of stacking faults in beryllium containing 1.2 weight percent iron has been reported.⁽⁵⁾ Also a low stacking fault energy has been reported for beryllium containing 0.15 weight percent iron after solutionizing at 920 C and quenching.⁽⁶⁾

A limited number of selected area electron diffraction analyses were performed in the program. Although several patterns have been taken of particles in the as-received and heat-treated specimens, only a few have been completely analyzed. In some cases we have been aided by the fact that particle networks extended into the hole produced by the electro-polishing procedure so that diffraction patterns were more readily obtained without interference from the matrix. Such a network is illustrated in Figure 7c. Aside from the BeO ring patterns detected in regions containing a large quantity of second-phase particles (Figures 8a and b), only one other pattern was completely analyzed. This is illustrated in Figure 9a and corresponds to the relatively massive particle of Figure 9b. The "d" spacings and angles between the diffracting planes correspond to the tetragonal MBe_{12} lattice (See footnote, p. 12). Additional effort will be required for solution of the other diffraction patterns taken during the course of this study.

Tensile Tests

Tensile tests were conducted on samples of the heat-treated portion of the vacuum-hot-pressed block. The location of the samples in the block is shown in Figure 2. Specimen preparation, design, and testing procedures were the same as previously described.^(1,2) The tests were conducted in duplicate at room temperature, 300, 500, and 700 F, in both the longitudinal (pressing) and transverse directions at a strain rate of 0.0385/min. In addition, tests were conducted at a strain rate of

52,800X

2750

a. Particle Cluster in
Specimen HYC12T,
As Received

2751

b. Corresponding Selected Area
Diffraction Pattern

FIGURE 8. TRANSMISSION ELECTRON MICROGRAPHS

50,000X

2529

a. Particles in Specimen HYC12T,
As Received

2528

b. Selected Area Diffraction
Pattern of Particle

FIGURE 9. TRANSMISSION ELECTRON MICROGRAPHS

38.5/min at room temperature, 300 and 500 F, again in duplicate in both the longitudinal and transverse directions. The results of these tests are summarized in Tables 4 and 5 and in Figures 10 through 17.

As shown in Figures 10 through 13, there is a general reduction in strength parameters (σ_{UY} , σ_{LY} , and σ_{UT}) produced by the heat treatment. In spite of the reduction in strength, however, only a small difference between the upper yield strength and ultimate tensile strength is shown by the longitudinal samples tested at 500 F. However, even at this test condition the ductility was somewhat improved over the as-received sample. Improvement in ductility with heat treatment was generally found under all test conditions (Figures 12 and 13).

The effect of strain rate on the tensile properties of heat-treated hot-pressed Block HY is shown in Table 5 and Figures 14 through 17. Increasing the strain rate from 0.0385/min to 38.5/min consistently increased both the yield and ultimate tensile strengths and lowered the ductility. The presence of a nil-ductility region is evident at the higher strain rates (See Figures 13 and 14). This results from the more rapid increase in yield strength with strain rate than the corresponding increase of the ultimate tensile strength.

Fractography

Fractured tensile samples of as-received hot-pressed beryllium Block HY were examined in order to determine whether any differences could be discerned between samples exhibiting "normal" ductility and those exhibiting nil ductility (longitudinal sample tested at 500 F). Four samples were examined. Two of these had been tested at room temperature (HYA1T and HYB20L), and two had been tested at 500 F (HYC1T and HYB2L). The fracture surfaces were examined both by scanning and replica electron microscopy. In addition, sections were cut parallel to the tensile axis and metallographically polished in order to examine the fracture surfaces on edge.

The bright light and polarized light optical micrographs presented in Figure 18 show that the fracture in all cases occurs both by

TABLE 4. TENSILE PROPERTIES OF HEAT-TREATED, HOT-PRESSED BLOCK HY
 $\dot{\epsilon} = 0.0385/\text{MIN}$

	σ_{LY} , ksi	σ_{UY} , ksi	$\Delta\sigma$, ksi	$\sigma_{0.2YS}$, ksi	σ_{UTS} , ksi	Total Elong., pct	Reduction in Area, pct	Stress-Strain Curve Type
<u>Room Temperature</u>								
HYAA8T	44.2	47.5	3.3	45.8	57.4	2.9	3.9
HYCC6T	45.0	48.4	3.4	45.0	57.8	3.1	3.4	
HYBL1-12	41.8	42.7	0.9	41.8	50.9	1.6	2.4	
HYDL2-6	42.8	44.3	1.5	43.2	50.8	1.4	1.7	
<u>300 F</u>								
HYAA4T	45.5	46.8	1.3	44.3	54.2	(c)	7.8
HYAA6T	44.8	46.5	1.7	43.4	53.4	7.0	7.6	
HYAA20T	45.5	47.4	1.9	45.7	53.8	6.8	6.7	
HYBL1-18	44.4	46.6	2.2	45.3	49.2	2.1	2.6	
HYBL1-16	42.6	44.3	1.7	34.3	47.8	2.3	0.1(d)	
<u>500 F</u>								
HYAA14T	40.0	41.5	1.5	37.8	44.9	9.5	13.4
HYAA16T	41.2	42.4	1.2	34.8	45.3	12.8	17.8	
HYBL1-10	--	43.5	--	25.4(a)	43.5(b)	2.6	5.0	
HYDL2-8	--	43.4	--	37.0	43.4(b)	2.0	4.1	
<u>700 F</u>								
HYAA-2T	38.0	41.0	3.0	15.6(a)	41.9	16.8	44.8
HYCC-12T	35.6	39.3	3.7	32.5(a)	40.4	15.6	40.6	
HYBL1-14	36.8	39.2	2.4	35.2(a)	40.5	12.2	28.2	
HYDL2-12	39.1	41.6	2.5	34.3	41.1	8.1	19.0	

(a) Microstrain.

(b) Sample failed at a stress below UY. Failed in Luders strain region.

(c) Extensometer slippage.

(d) Sample failed in shoulder region.

TABLE 5. TENSILE PROPERTIES OF HEAT-TREATED HOT-PRESSED BLOCK NY
AT A STRAIN RATE OF 38.5/MIN

	σ_{LY} , ksi	σ_{UY} , ksi	$\Delta\sigma$, ksi	σ_{UTS} , ksi	Total Elong., pct	Reduction in Area, pct	Stress-Strain Curve Type
Room Temperature							
HYAA12T	46.8	49.6	2.8	63.4	3.1	4.3
HYCC10T	46.5	51.2	4.7	65.3	3.2	4.1	
HYBL1-8	46.7	49.2	2.5	55.6	1.7	2.7	
HYBL1-20	48.5	52.1	3.6	58.6	1.9	2.1	
300 F							
HYAA18T	48.4	50.4	2.0	57.6	3.7	4.8
HYAA22T	49.1	51.5	2.4	57.1	3.5	4.2	
HYBL1-2	(a)	(a)	(a)	45.6(a)	0.4	-0.8(b)	
HYDL2-2	(a)	(a)	(a)	46.0(a)	0.5	-1.6(b)	
500 F							
HYCC-2T	46.0	48.9	2.9	50.8	5.1	5.6
HYCC-14T	46.7	48.0	1.3	51.6	5.1	6.7	
HYBL1-6	(a)	(a)	(a)	37.5(a)	0.3	-3.2(b)	
HYDL2-10	(a)	(a)	(a)	47.1(a)	0.5	0.8(b)	

(a) Failed before yielding.

(b) Failed in shoulder area.

FIGURE 10. COMPARISON OF THE TRANSVERSE STRENGTHS OF AS-RECEIVED AND HEAT-TREATED HOT-PRESSED BLOCK HY ($\dot{\epsilon} = 0.0385/\text{MIN}$)

FIGURE 11. COMPARISON OF THE LONGITUDINAL STRENGTHS OF AS-RECEIVED AND HEAT-TREATED HOT-PRESSED BLOCK NY ($\dot{\epsilon} \approx 0.0385/\text{MIN}$)

FIGURE 12. EFFECT OF HEAT TREATMENT ON THE TOTAL ELONGATION OF HOT-PRESSED BLOCK HY ($\dot{\epsilon} = 0.0385/\text{MIN}$)

FIGURE 13. EFFECT OF HEAT TREATMENT ON THE REDUCTION IN AREA OF HOT-PRESSED BLOCK HY ($\dot{\epsilon} = 0.0385/\text{MIN}$)

FIGURE 14. EFFECT OF STRAIN RATE ON THE TRANSVERSE STRENGTH OF HEAT-TREATED Hot-Pressed Block HY

FIGURE 15. EFFECT OF STRAIN RATE ON THE LONGITUDINAL STRENGTH OF HEAT-TREATED HOT-PRESSED BLOCK HY

FIGURE 16. EFFECT OF STRAIN RATE ON THE TOTAL ELONGATION OF HEAT-TREATED HOT-PRESSED BLOCK HY

FIGURE 17. EFFECT OF STRAIN RATE ON THE REDUCTION IN AREA OF HEAT-TREATED HOT-PRESSED BLOCK HY

46264

a. Room Temperature Transverse, B.L.

46263

b. Room Temperature Transverse, P.L.

46262

c. Room Temperature Longitudinal, B.L.

46261

d. Room Temperature Longitudinal, P.L.

46258

e. 500 F Transverse, B.L.

46257

f. 500 F Transverse, P.L.

46260

g. 500 F Longitudinal, B.L.

46259

h. 500 F Longitudinal, P.L.

FIGURE 18. PHOTOMICROGRAPHS OF SECTIONS PARRALLEL TO THE TENSILE AXIS OF SPECIMENS FROM AS-RECEIVED HOT-PRESSED BLOCK BY TESTED AT ROOM TEMPERATURE AND AT 500 F (250X)

transgranular cleavage and intergranular failure. There appears to be very little difference in the fracture appearance among the four samples with the possible exception of the sample showing nil ductility (Figure 18g and h) in which there is evidence of crack formation in regions somewhat displaced from the main fracture surface. In spite of this, cracking occurred relatively close to the main fracture surfaces compared to what has been observed in other hot-pressed beryllium blocks (See Figure 24 of Reference 1). The reason for this concentration of cracking near the main fracture surface is probably related to the relatively low-crack stability expected in this material. Thus, the first microcrack that forms in this material probably leads to catastrophic failure. In other tougher materials, stable cracks that ultimately link up to cause sample failure can form throughout the gauge length.

Scanning and replica electron microscopic observations on the fracture surfaces confirm (1) the similarity of the fracture surfaces of all of the samples observed and (2) the mixed mode of failure. Replica electron micrographs of the fractured surfaces are shown in Figures 19 and 20. In addition to the above, many regions on all the failure surfaces contained a large concentration of particulate matter. These particles are, to a large extent, the second phase shown in the micrographs of Figures 5, 6, and 7 and which would generally be situated on the original beryllium powder particle surfaces.

In conclusion, little difference has been found in the appearance of the fracture surfaces present on tensile samples of the as-received hot-pressed Block HY tested at room temperature and 500 F. The longitudinal sample which exhibited nil ductility when tested at 500 F shows no distinct fracture surface features. Thus, a change in fracture mode cannot explain the occurrence of nil ductility in hot-pressed beryllium Block HY.

Pressurization Experiments

Objective

The objective of this portion of the work was to determine in a critical experiment whether solute or precipitate pinning of dislocations

2579

2582

a. Transverse Sample HYA1T

A2554

A2567

b. Longitudinal Sample HYB20L

FIGURE 19. REPLICA ELECTRON MICROGRAPHS OF FRACTURED SURFACES OF TENSILE SAMPLES TAKEN FROM AS-RECEIVED HOT-PRESSED BLOCK HY TESTED AT ROOM TEMPERATURE (5100X)

2610

2616

a. Transverse Sample HYC1T

2570

2574

b. Longitudinal Sample HYB2L

FIGURE 20. REPLICA ELECTRON MICROGRAPHS OF FRACTURED SURFACES OF TENSILE SAMPLES TAKEN FROM AS-RECEIVED HOT-PRESSED BLOCK HY TESTED AT 500 F (5100X)

was an important contribution to brittleness in hot-pressed beryllium block. The approach taken was to create mobile dislocations in samples of hot-pressed block which exhibited brittle behavior. The dislocations were to be produced by subjecting the samples to a high hydrostatic pressure at room temperature, releasing the pressure, and then tensile testing at ambient pressure under conditions at which the brittle behavior was otherwise observed. Mobile dislocations are produced as a result of the shear stresses generated by application of a high hydrostatic pressure to a polycrystalline material having anisotropic linear compressibilities. The experiment outlined is unambiguous. The only change in structure expected as a result of the pressurization is a change in dislocation density or configuration. There are no complications such as changes in the kind and amount of precipitates or inclusions or changes in grain size as might be expected with experiments involving thermal treatments.

Procedure

Two types of materials were investigated in this part of the study: hot pressed Block HY in the as-received condition and a hot isostatically pressed P21 material. The former exhibits nil ductility in the longitudinal direction at 500 F, whereas the latter was found to exhibit brittle behavior at room temperature in the as-hot isostatically pressed condition.

Sample blanks approximately 5/16 inch square by 1-1/4-inch length were pressurized in a Bridgman-Birch 30 kilobar apparatus as previously described.^(1,2) Several samples of hot-pressed Block HY, some in the longitudinal and some in the transverse directions, and two samples of P21 were pressurized at 21,000 atm (309,000 psi). After pressure release, the blanks were machined into tensile samples and etched. The specimens from the pressurized hot-pressed Block HY were tested at 500 F, while those from the P21 hot isostatically pressed material were tested at room temperature. One of the pressurized HY sample blanks was used for the preparation of specimens for observation

in the electron microscope. Procedures for the tensile testing and electron microscope sample preparation and observation were similar to those described previously. (1,2)

Results

The results of tensile testing of the pressurized HY and P21 samples are presented in Table 6 where they are compared with unpressurized samples tested under the same conditions. Three significant effects are to be noted:

- (1) The brittleness present in both the HY material and the P21 material is removed by the pressurization.
- (2) The shape of the stress-strain curve is generally changed from one exhibiting a yield point to a more rounded type.
- (3) The effective yield strength of the material is lowered by the treatment.

All these effects are demonstrated in Figure 21 where the stress-strain curve for the pressurized Sample HYD2L is compared to two stress-strain curves for the as-received material similarly tested.

Transmission electron micrographs of the pressurized sample from hot-pressed Block HY are shown in Figure 22. These micrographs should be compared to those from the as-received hot-pressed Block HY shown in Figures 5 and 6. It may be seen from these micrographs that the dislocation density which was relatively low in the as-received material has dramatically increased in the pressurized sample and that the dislocations are in general much longer and straighter than in the as-received material.

Discussion

The pressurization experiments show that the embrittlement which occurs in hot-pressed Block HY at 500 F is caused by a lack of

TABLE 6. EFFECT OF PRESSURIZATION TO 21,000 ATM (309,000 PSI) ON THE TENSILE PROPERTIES OF NONHEAT-TREATED, HOT-PRESSED BLOCK HY AND HOT ISO-STATICALLY PRESSED P21. $\dot{\epsilon} \approx 0.385/\text{MIN}$

Sample No.	Temperature, F	σ_{LY} , ksi	σ_{UY} , ksi	$\Delta\sigma$, ksi	$\sigma_{0.2YS}$, ksi	σ_{UTS} , ksi	Total Elong. pct	Reduction in Area pct	Stress-Strain Curve Form
<u>Unpressurized Hot-Pressed Block HY</u>									
HYC1T	500	46.7	49.1	2.4	43.0	50.8	6.2	8.5	
HYC15T	500	46.0	48.1	2.1	47.4	49.7	10.3	17.6	
HYB2L	500	(a)	(a)	(a)	(a)	44.3 (a)	<0.1	(c)	
HYB8L	500	(a)	(a)	(a)	(a)	45.1	0.2	(c)	
HYB18L	500	(b)	47.1	(b)	45.7	47.1	1.3	2.0	
<u>Pressurized Hot-Pressed Block HY</u>									
HYA9T	500	--	--	--	26.4	48.7	10.4	15.2	
HYA19T	500	--	--	--	25.4	49.2	10.4	14.4	
HYB5L	500	--	--	--	22.0	47.2	3.6	5.3	
HYD2L	500	--	--	--	27.0	46.7	3.9	5.2	
<u>Unpressurized Hot Isostatically Pressed P21</u>									
P-21C	Room Temp	(a)	(a)	(a)	(a)	61.9	0.15	(d)	
<u>Pressurized Hot Isostatically Pressed P21</u>									
P-21A	Room Temp	--	--	--	51.8	65.8	2.4	3.3	
P-21B	Room Temp	--	--	--	51.4	65.3	2.7	3.1	

- (a) Failed before yielding.
 (b) Lower yield strength not reached.
 (c) Failed away from minimum cross section.
 (d) Not measured.

FIGURE 21. COMPARISON OF THE STRESS-STRAIN BEHAVIOR OF HOT-PRESSED BLOCK HY IN THE AS-RECEIVED AND PRESSURIZED CONDITIONS

FIGURE 22. TRANSMISSION ELECTRON MICROGRAPHS OF AS-RECEIVED
HOT-PRESSED BERYLLIUM BLOCK BY PRESSURIZED AT
21,000 ATMOSPHERES

mobile dislocations which, in turn, causes an excessively high yield strength. This behavior can be interpreted in terms of the effect of mobile dislocation density on the stress-strain behavior of crystalline solids. (7)

Most of the small number of dislocations present in the as-received material are in the form of tangles from which they are difficult to move or are pinned by precipitates or inclusions or perhaps by components in solid solution. The pressurization and depressurization have been found to create mobile dislocations mainly as a result of the shear stresses induced in this anisotropic material due to grain boundary constraints. It is evident from the mechanical behavior that little or no repinning of the dislocations occurs during heating to and testing at 500 F.

The same general phenomena appear to explain the brittleness at room temperature in the hot isostatically pressed P21 metal and its elimination by the pressurization treatment. However, the two cases may differ in the details.

Although we now understand the general cause for the embrittlement in both the HY block and P21 hot isostatically pressed beryllium, we still do not understand the specifics regarding the low mobile dislocation density in these pressings. Further research effort should be expended to determine the pinning mechanism(s) and specific impurities involved.

CONCLUSIONS AND RECOMMENDATIONS

The following conclusions can be made on the basis of this study:

Effect of Heat Treatment on Hot-Pressed Block HY

Structural Changes

The solutionizing heat treatment followed by a slow cool dissolves the larger inclusions present in the as-received material and leads to the precipitation on cooling of FeBe_{11} rather than the FeAlBe_4 originally present. MBe_{12} , MBe_{13} , BeO , Be_2C , and other unidentified phase(s) are present in both the as-received and heat-treated condition. An increase in grain size and a decrease in density result from the heat treatment.

Mechanical Property Changes

At a strain rate of 0.0385/min, yield and ultimate tensile strengths are reduced by the heat treatment, whereas the ductility generally increases. However, some vestiges of the 500-F embrittlement in samples tested in the pressing direction remain. Most of the improvement in ductility can be explained in terms of the decreased yield strength due to the larger grain size. An increase in strain rate to 38.5/min increases yield and ultimate tensile strengths in the heat-treated samples and reduces the ductility. Embrittlement is evident at both 300 and 500 F in samples tested in the pressing direction.

Fractography

The characteristics of the fracture surfaces of tensile samples of as-received HY material tested at room temperature and 500 F in the pressing and transverse directions show that there is no change in the basic fracture mechanism under the test conditions investigated.

Pressurization Studies

The pressurization studies coupled with the fractography show that the cause of embrittlement in the HY hot-pressed block is due to the lack of mobile dislocations. Embrittlement at room temperature in hot isostatically pressed F21 beryllium powder can be similarly explained. The details of the pinning mechanisms and impurities involved are not known.

The research described above and in the previous reports^(1,2) have uncovered a number of unusual phenomena. It is recommended that research be conducted to supply information to answer the following questions concerning commercially pure vacuum hot-pressed beryllium block:

- (1) What is the mechanism causing the lack of mobile dislocations in embrittled metal?
- (2) Is the lack of mobile dislocations caused by solute or precipitate pinning? If so, identify the impurity causing the phenomenon.
- (3) How does the grain size affect the pinning mechanism?
- (4) Is the increase in yield strength with temperature observed in some of the vacuum hot-pressed beryllium associated with the pinning reaction or is it a different phenomenon?
- (5) What is the mechanism for this increase in yield strength with temperature?
- (6) Can pressurization be applied in a practical way to produce and retain a ductile material?
- (7) What is the reason for the unusual strain rate dependency of the flow stress observed in one of the commercial hot-pressed materials?

ACKNOWLEDGEMENT

The author wishes to express his appreciation to A. Webster and J. Schofield of BCL for their support in the area of sample preparation and tensile testing, respectively, and to J. Mueller of BCL for supplying samples of hot isostatically pressed P21 beryllium. In addition, he wishes to express his thanks to H. Weiss, J. Hauber, and P. Landon of LLL and N. Borch of LASL for their helpful discussions during the course of this work and for their continued support.

REFERENCES

- (1) Gelles, S. H., and Peterson, J. H., "Characteristics of Commercial Vacuum Hot-Pressed Block", Report to University of California, Lawrence Livermore Laboratory, AEC Contract W-7405-eng-92 (December, 1971), BMI-X-629.
- (2) Gelles, S. H., "Characteristics of Commercial Vacuum Hot-Pressed Beryllium, Part II", to University of California, Lawrence Livermore Laboratory, AEC Contract W-7405-eng-92 (December, 1972), BMI-X-639.
- (3) Rooksby, H. P., "Intermetallic Phases in Commercial Beryllium", Journal of Nuclear Materials, 7 (1962), 205.
- (4) Croutzeilles, M., et al., "Beryllium Research and Development Program--Identification of Inclusions and Precipitates of Different Origins", Report ASD-TDR-62-509, Vol. VII (February, 1965).
- (5) Scott, V. D., and Lindsay, H. M., "Some Electron Microscope Observations on Precipitation in Beryllium", Proceedings of the Second International Conference, Philadelphia, Pennsylvania, October 15-17, 1964, Beryllium Technology, 1, Gordon and Breach, New York (1966).
- (6) Ranzetta, G. V. T., and Scott, V. D., "The Effect of Thermal Treatment on the Metallographic Structure of Beryllium and Dilute Beryllium Alloys", Journal of Nuclear Materials, 10 (1963), 113.
- (7) Hahn, G. T., "A Model for Yielding with Special Reference to the Yield Point Phenomena of Iron and Related BCC Metals," Acta Met., 10 (1962), 727.