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The Influence of Purity Level on the Mechanical Properties of Hot Isostatically Pressed Beryllium

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energy report

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ABSTRACT

The procurement of a quantity of ultra-pure beryllium powder combined with special handling from powder to billet form resulted in the fabrication of high purity beryllium. The mechanical properties of these billets were contrasted to those of commercial grade billets to determine the influence of impurities and powder processing.

The tensile test results show that the strength values are primarily dependent on the grain size in a behavior predictable by the Hall-Petch relationship. Only a fraction of the strength differential can be attributed to metallic impurities in solution. The grain size is controlled by the powder size distribution. The ductility is dominated by both grain size and oxide content. The fine grained, low oxide billets exhibited the highest ductilities. There is evidence to suggest that oxide distribution has a large influence on the ductility.

The fracture toughness was highest for the high purity beryllium billets.

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[illegible] the help and cooperation of [illegible] sample preparation and testing.

*Dr. London is now with the Naval Air Development Center Warminster, Pa.

THE INFLUENCE OF PURITY LEVEL ON THE MECHANICAL PROPERTIES OF HOT ISOSTATICALLY PRESSED BERYLLIUM

Introduction

Several studies have preceded this work relating the influence of grain size, oxide content and total chemistry to the room temperature mechanical properties of beryllium.[1-4] This study utilized high purity powders of varying compositions and inert gas handling at the Kawecki Berylco facility to fabricate beryllium billets. The result was a fine grain, low impurity material which is difficult to produce. Tests were conducted on this material and contrasted to tests on three commercially available grades of beryllium. This is reflected in the test matrix shown in Figure 1, which enable the study of powder [illegible], chemistry and processing history on the micro-structural and mechanical properties.

	[illegible]		
GRADE	-400	-400+[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
HP-10 [illegible] LEACHED [illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]

Figure 1. Program Matrix - showing the billet identification numbers for each particle size and grade of powder.

Material:

The materials used in this investigation were produced by Kawecki Berylco Industries using a hot isostatic pressing process. The pressing schedule offered the advantage of consolidation at much higher pressures (103 MPa) over conventional vacuum hot pressing and thus lower temperatures to accomplish full densification. The low temperatures minimize grain growth and the isostatic pressure produces a near isotropic material. The high pressures also allow the consolidation of high purity powders to full density. Billet compositions are shown in Table I.

Table I
BILLET COMPOSITION

Billet No.	Grade	Particle Size μm	BeO* %	C† ppm	Fe∇ ppm	Al ppm	Mg▼ ppm	Ni▼ ppm	Mn▼ ppm	Cr▼ ppm	Co▼ ppm	Cu▼ ppm	Si ppm	Ti▼ ppm
301	P-10	-44	1.02	680	1090	410▼	260	160	85	95	<5	75	190▼	190
303	"	-44+10	.64	560	1000	340▼	270	135	90	115	<5	70	175▼	125
305	"	-20	1.89	400	1350	560▼	385	135	95	115	<5	70	180▼	280
307	XP-10	-44	.53	270	950	100▼	50	130	60	60	<5	65	130▼	300
309	"	-44+10	.43	300	950	100▼	50	110	55	50	<5	60	130▼	155
311	"	-20	1.00	300	1000	110▼	45	110	50	90	<5	55	175▼	135
314	P-1	-44	.95	180	170	<20∇	5	50	3	10	50	15	31●	<10
315	"	-44+10	.52	180	140	<20∇	4	110	<3	<5	9	10	46●	<10
317	"	-20	1.02	300	260	25∇	8	105	8	8	25	25	45●	<10
319	P-0	-44	.43	N.D.	70	45∇	5	3	7	<5	<5	15	40●	<10
321	"	-44+10	<.01	250	120	30∇	12	20	12	<5	8	20	50●	<10
322	"	-20	1.16	250	810	35∇	28	25	17	7	8	25	50●	<10

Notes: * Neutron Activation
† Conductometric
∇ Atomic Absorption
▼ Spectrographic
● Wet Chemistry

Powders:

The P-10 powder was derived from magnesium reduced bead by vacuum melting, casting into small ingot form, lathe turning to chips and impact attritioning.

The latter is a process by which chips are fed into a hopper and carried by high velocity, cold, inert gas to a beryllium target. The impact velocity is sufficient to fracture the chips into smaller particles. The result is a powder of varying sizes ranging from sub-micron to >44 μm. Subsequent separation into size ranges (powder classification) produced the fractions used in this study: -44+10 μm, -44 μm, and -20 μm. The XP-10 grade was produced by further processing the P-10 powder using an acid leaching step to reduce impurities. As noted in Table I, the BeO, Al, Mg and to some extent C, Mn and Cr are lowered by this process.

The P-1 powder was processed from electrolytic flake (grade EF-1) and the P-0 powder from twice electrolized flake (EF-0). This step utilizes an electrolytic process to reduce the impurity content of the magnesium reduced bead. The BeO content was minimized by processing the beryllium in an argon environment whenever possible. The final comminution was accomplished in a beryllium-lined ball mill (tungstun carbide balls) filled with argon. Powder classification into the ranges discussed earlier was done in argon.

Consolidation:

The flow diagrams for powder processing to billet form are shown in Figures 2 and 3. The P-10 and XP-10 powders were hot isostatically pressed at a pressure of 103 MPa (15 ksi) at 915°C; the P-1 and P-0 powders at 103 MPa and 1065°C. The higher temperature used to consolidate the latter powders was to ensure full densification. The XP-10 billets were given on additional heat treatment (750°C, 2 hours, air cool) to eliminate the low ductility exhibited in the as-pressed condition.

The schedule used in powder consolidation was to (1): load the powders into a stainless steel cylindrical can, (2) weld the end closed with an attached stem, (3) vacuum bake out the can, (4) pinch off the stem and (5) process the assembly in a HIP chamber at a predetermined pressure, temperature and time schedule.

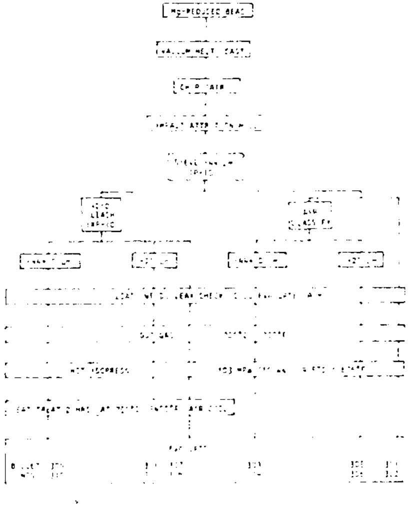


FIGURE 2 FLOW DIAGRAM FOR P-[illegible] AND [illegible] MATERIAL

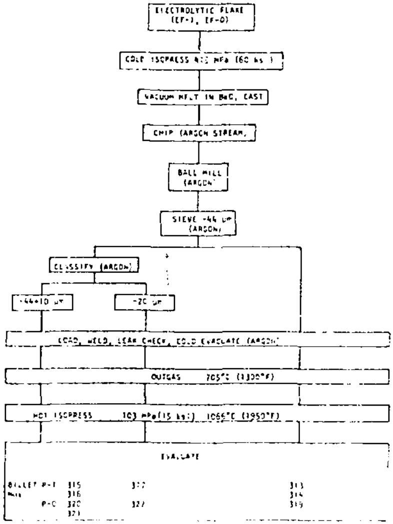


FIGURE 3 FLOW DIAGRAM FOR P-O AND P-I MATERIAL

Experimental Technique:

Tensile tests were conducted on an Instron machine on specimens having a 12.7 mm gage length and 3.56 mm diameter; at a cross head velocity of 8.5×10^{-4} cm/sec. Strain was measured using a 7.62 mm extensometer attached to the specimen. Surface damage from machining was removed by etching 0.08 mm from the surface of all test specimens.

Compressive test data was generated on an Instron machine with cylindrical samples 6.35 mm in length and a 5.00 mm diameter.

Toughness tests were accomplished using a WOL geometry with a thickness of 9.91 mm. The cross head velocity for all tests was 8.5×10^{-4} cm/sec. (Ref. ASTM-E399).

Transmission electron microscopy (TEM) was accomplished on a Phillips 300 EM using a double tilt stage. Foils were electropolished at 273K to final thickness in a solution consisting of 350 ml ethylene glycol, 40 ml HNO_3, 9 ml H_2SO_4 and 9 ml HCL.

Grain size was determined optically using polarized light and a mean linear intercept method[5] at a magnification of 800X. The stage on the optical microscope was rotated 120° after each count to provide three counts for averaging. No attempt was made to determine grain size distributions, nor was a correction factor incorporated for grain morphology.

Test Results:

The tensile properties together with the compressive yield strengths and the grain sizes are shown in Table II.

TABLE II

TENSILE/COMPRESSION DATA

HIP Temperature	Billet No.	Material Particle Size	Tensile Properties σ_{y1}		σ_{y2}		UTS		Elong %	Compression Yield		Grain Size
			MPa	(ksi)	MPa	(ksi)	MPa	(ksi)	%	MPa	(ksi)	(μm)
	[illegible]	P-10/-44μm	474 471	([illegible]) (68.70)	41[illegible] 406	([illegible]) (59.6[illegible])	45[illegible] 444	([illegible]) (64.39)	3.5[illegible] *	413	(59.9[illegible])	6.70
1188 K	313	P-10/44+10μm	430 422	(62.44) (61.30)	375 370	(54.47) (53.66)	431 427	(62.[illegible]) (62.03)	4.[illegible] 4.3[illegible]	36[illegible]	(53.[illegible])	7.86
	316	P-10/-20μm	626	(90.91) **	52[illegible] **	(76.45) **	534 615	(77.52) (89.27)	2.9[illegible] 0.26	548	(79.62)	3.97
1166 K	307	IP-10/-44μm	422 426	(61.22) (61.84)	3[illegible]4 390	(57.[illegible]) (56.56)	47[illegible] 46[illegible]	(68.2[illegible]) (67.2[illegible])	4.54 4.50	39[illegible]	(57.[illegible])	5.71
	309	IP-10/-44+10μm	382 396	(55.45) (57.56)	354 354	(51.3[illegible]) (51.46)	434 436	(62.93) (63.25)	4.63 3.97	291	(42.13)	9.03
(1074 K 2h/AC)**	311	IP-10/-20μm	606	(87.97) **	524 **	(76.02) **	5[illegible] 565	([illegible]) (84.96)	2.9[illegible] 0.27	545	(79.11)	4.76
	314	P-17/-44μm			31[illegible] 343	(46.18*) (50.50*)	402 456	(58.[illegible]) (66.12)	0.66 0.96	435	(63.12)	5.59
1340 K	315	P-17/-44+10μm			2[illegible] 2[illegible]3	(41.52*) (38.86*)	441 432	(64.00) (62.68)	3.[illegible] 2.93	336	(48.74)	7.08
	317	P-17/20μm			464 445	(67.32*) (64.63*)	[illegible] 5[illegible]	([illegible].92) (83.98)	3.92 2.47	5[illegible] 49[illegible]	(73.75) (71.75)	4.44
	319	P-07/-44μm			[illegible] 26[illegible]	(41.30*) (38.60*)	441 435	(63.93) (63.38)	3.26 3.24	364	(52.8[illegible])	9.64
1340 K	321	P-07/-44+10μm			214 220	(31.06*) (31.95*)	3[illegible]9 3[illegible]	(56.50) (56.56)	4.0[illegible] 4.00	291	(42.24)	7.05
	32[illegible]	P-07/-20μm			422 426	(61.20*) (61.75*)	557 543	(85.20) (78.86)	2.93 1.55	520	(75.40)	3.64

*Extensometer slipped
**Post-HIP heat treatment
σ_{y1} = upper yield point
σ_{y2} = (0.02% offset) yield point

The toughness data is shown in Table III. The K_{IC} value is derived by the equation

$$K_{IC} = PS/BW^{3/2} \; f\left(\frac{a}{W}\right) \quad \text{(Ref. ASTM-E399)}$$

where P = load, B = specimen thickness,

S = span, W = specimen depth and

a = crack length.

Table III
TOUGHNESS DATA

Billet No.	Material/ Particle Size µm	K_{IC} MPa-m$^{1/2}$
301	P-10/-44	11.65
303	P-10/-44+10	10.85
305	P-10/-20	9.93
307	XP-10/-44	8.98
309	XP-10/-44+10	14.29
311	XP-10/-20	11.24
314	P-1/-44	15.11
315	P-1/-44+10	14.46
317	P-1/-20	11.47
319	P-0/-44	14.56
321	P-0/-44+10	17.90
322	P-0/-20	16.28

Discussion:

Strength -- The yield point phenomenon, indicated by the value, σ_{Y1} (Table II), for the P-10 and XP-10 grades, has been suggested as due to the metallic impurity, iron.[6] Heat treating at 1023K for 5 hours followed by slow cooling (25°C per hour) to 523K produces a precipitate, removes the iron from solution, and eliminates the yield point. No work was done in this study to dispute or support this claim. However it is noteworthy that only the high metallic impurity grades exhibited this phenomenon.

The tensile yield strengths of the P-10 and XP-10 grades are contrasted to those of the P-1 and P-0 grades as influenced by grain size in Figure 4. The strength dependence on grain size is predicted by the Hall-Petch relationship. The higher yield strengths on the P-10 and XP-10 grades are thought to be due to the solid-solution strenghthening effect of the metallic impurities. This theory is supported by Figure 5, where the grain size dependence has been normalized and the higher impurity billets (P-10, XP-10) are moderately higher in strength. The strength differential is about 100 MPa.

The compressive yield strengths follow the same behavior as in tension but at a consistantly lower value indicating a degree of anisotropy.

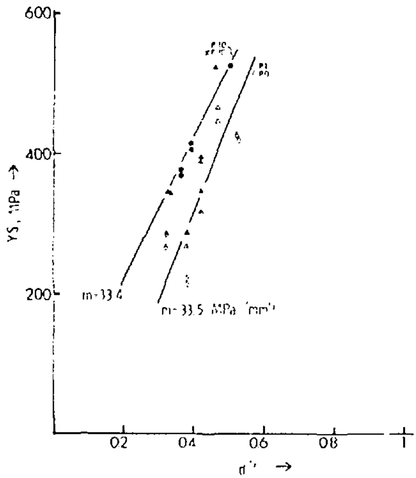


Figure 4 - The influence of grain size on the yield strength.

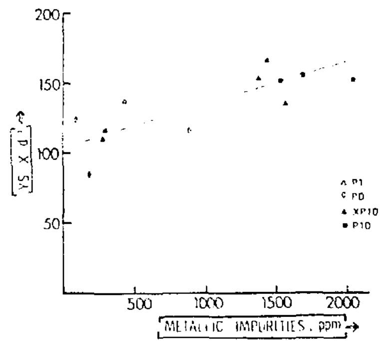


Figure 5 - The influence of metallic impurities on the yield strength (normalized for grain size)

Ductility:

The ductility within a given beryllium grade was influenced strongly by the powder input size; the finest size powder (smallest grain size) consistently yielded the lowest ductility. To infer that grain size is singly affecting the ductility value is an over simplification as the impurity level for a given grade of beryllium goes up as the powder size goes down. The increase in impurities may contribute to the reduction in ductility. Each grade was capable of yielding a high ductility (0.04 strain to failure). In recent studies by Aldinger et al. on similarly processed beryllium grades including P-1, P-10, RR 243* and cast material, it was shown that elongation was dependent on grain size and beryllium oxide content. Figure 6 shows the elongation (normalized for grain size) as influenced by beryllium oxide content. The

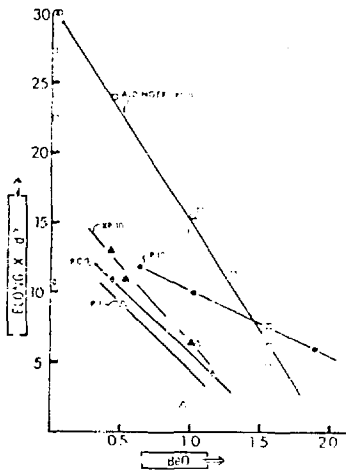


Figure 6 - The influence of beryllium oxide on elongation (normalized for grain size).

*Brush-Wellman Product

since of the fillers [illegible], [illegible] and [illegible] [illegible] data reflecting [illegible]. The [illegible] in the data [illegible] techniques used to [illegible] the [illegible] in the [illegible]. The [illegible], in contrast, is less influenced by [illegible]. The [illegible] from that of the [illegible], [illegible], [illegible] with [illegible] as [illegible] below.

[illegible]

[illegible]

of oxide sizes with isolated regions where the oxides had agglomerated. This is illustrated in Figures 7 and 8.

The P-1, P-10 and HP-10 grades are inherently lower in impurities and oxides because of the methods used in processing the powders. The test results [illegible] that the microstructural differences between these grades and [illegible] are great enough to affect the ductility and toughness behavior.

[illegible]

- [illegible] tested in this study were capable of [illegible] ductility levels regardless of the processing and [illegible].

- [illegible] produced [illegible] and the [illegible].

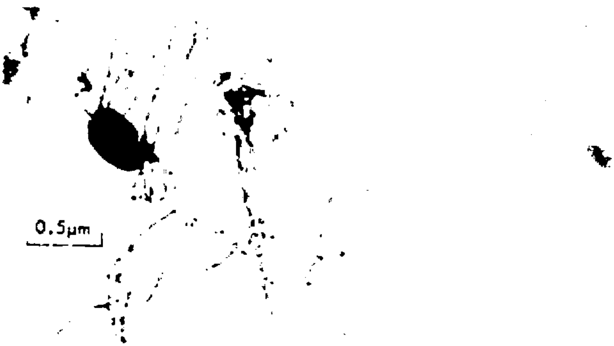
- [illegible] was [illegible] by the grain [illegible] content [illegible] than the [illegible].

- [illegible]

- [illegible] highest for the high purity grade billets. The [illegible] was [illegible] billet #[illegible] which had the lowest beryllium oxide content.


0.5μm
1μm

[illegible]


0.5μm

[illegible]

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