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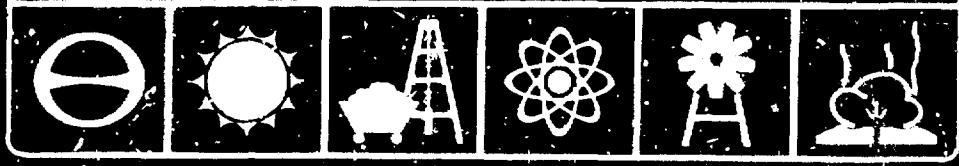
REGISTER

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

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**Sandia Laboratories
energy report**



THE PRACTICAL USE OF

MASTER

¹⁰ See, for example, the discussion of the 1992 Constitutional Conference in the *Journal of African Law* 36 (2002).

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P. 2. 1. 2. 3. 4.

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.

THE BIRDS OF THE BAHAMAS

The first question whether the *U.S. Constitution* is a "Contract" is
settled by the fact that it is a "Contract" in the sense that it is a
compact between the several States, and that the *U.S. Constitution* is
the result of the compact, and that it is a "Contract" in the sense that
the compact creates a "Contract" between the several States, and
the compact creates a "Contract" between the several States and the
people of the United States.

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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.^[1-3] This study utilized high purity powders of varying compositions and inert gas handling at the Elweck Beryllco 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 grade, size, chemistry and processing history on the microstructural and mechanical properties.

Program Matrix		Billet Identification	
Grade	Size	1-44410	2-44411
1-44410	100	1-44410-100	2-44411-100
1-44410	150	1-44410-150	2-44411-150
1-44410	200	1-44410-200	2-44411-200
1-44410	300	1-44410-300	2-44411-300
1-44410	400	1-44410-400	2-44411-400
1-44410	500	1-44410-500	2-44411-500
1-44410	600	1-44410-600	2-44411-600
1-44410	700	1-44410-700	2-44411-700
1-44410	800	1-44410-800	2-44411-800
1-44410	900	1-44410-900	2-44411-900
1-44410	1000	1-44410-1000	2-44411-1000
1-44410	1200	1-44410-1200	2-44411-1200
1-44410	1400	1-44410-1400	2-44411-1400
1-44410	1600	1-44410-1600	2-44411-1600
1-44410	1800	1-44410-1800	2-44411-1800
1-44410	2000	1-44410-2000	2-44411-2000
1-44410	2200	1-44410-2200	2-44411-2200
1-44410	2400	1-44410-2400	2-44411-2400
1-44410	2600	1-44410-2600	2-44411-2600
1-44410	2800	1-44410-2800	2-44411-2800
1-44410	3000	1-44410-3000	2-44411-3000
1-44410	3200	1-44410-3200	2-44411-3200
1-44410	3400	1-44410-3400	2-44411-3400
1-44410	3600	1-44410-3600	2-44411-3600
1-44410	3800	1-44410-3800	2-44411-3800
1-44410	4000	1-44410-4000	2-44411-4000
1-44410	4200	1-44410-4200	2-44411-4200
1-44410	4400	1-44410-4400	2-44411-4400
1-44410	4600	1-44410-4600	2-44411-4600
1-44410	4800	1-44410-4800	2-44411-4800
1-44410	5000	1-44410-5000	2-44411-5000
1-44410	5200	1-44410-5200	2-44411-5200
1-44410	5400	1-44410-5400	2-44411-5400
1-44410	5600	1-44410-5600	2-44411-5600
1-44410	5800	1-44410-5800	2-44411-5800
1-44410	6000	1-44410-6000	2-44411-6000
1-44410	6200	1-44410-6200	2-44411-6200
1-44410	6400	1-44410-6400	2-44411-6400
1-44410	6600	1-44410-6600	2-44411-6600
1-44410	6800	1-44410-6800	2-44411-6800
1-44410	7000	1-44410-7000	2-44411-7000
1-44410	7200	1-44410-7200	2-44411-7200
1-44410	7400	1-44410-7400	2-44411-7400
1-44410	7600	1-44410-7600	2-44411-7600
1-44410	7800	1-44410-7800	2-44411-7800
1-44410	8000	1-44410-8000	2-44411-8000
1-44410	8200	1-44410-8200	2-44411-8200
1-44410	8400	1-44410-8400	2-44411-8400
1-44410	8600	1-44410-8600	2-44411-8600
1-44410	8800	1-44410-8800	2-44411-8800
1-44410	9000	1-44410-9000	2-44411-9000
1-44410	9200	1-44410-9200	2-44411-9200
1-44410	9400	1-44410-9400	2-44411-9400
1-44410	9600	1-44410-9600	2-44411-9600
1-44410	9800	1-44410-9800	2-44411-9800
1-44410	10000	1-44410-10000	2-44411-10000
1-44410	10200	1-44410-10200	2-44411-10200
1-44410	10400	1-44410-10400	2-44411-10400
1-44410	10600	1-44410-10600	2-44411-10600
1-44410	10800	1-44410-10800	2-44411-10800
1-44410	11000	1-44410-11000	2-44411-11000
1-44410	11200	1-44410-11200	2-44411-11200
1-44410	11400	1-44410-11400	2-44411-11400
1-44410	11600	1-44410-11600	2-44411-11600
1-44410	11800	1-44410-11800	2-44411-11800
1-44410	12000	1-44410-12000	2-44411-12000
1-44410	12200	1-44410-12200	2-44411-12200
1-44410	12400	1-44410-12400	2-44411-12400
1-44410	12600	1-44410-12600	2-44411-12600
1-44410	12800	1-44410-12800	2-44411-12800
1-44410	13000	1-44410-13000	2-44411-13000
1-44410	13200	1-44410-13200	2-44411-13200
1-44410	13400	1-44410-13400	2-44411-13400
1-44410	13600	1-44410-13600	2-44411-13600
1-44410	13800	1-44410-13800	2-44411-13800
1-44410	14000	1-44410-14000	2-44411-14000
1-44410	14200	1-44410-14200	2-44411-14200
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1-44410	14600	1-44410-14600	2-44411-14600
1-44410	14800	1-44410-14800	2-44411-14800
1-44410	15000	1-44410-15000	2-44411-15000
1-44410	15200	1-44410-15200	2-44411-15200
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1-44410	16400	1-44410-16400	2-44411-16400
1-44410	16600	1-44410-16600	2-44411-16600
1-44410	16800	1-44410-16800	2-44411-16800
1-44410	17000	1-44410-17000	2-44411-17000
1-44410	17200	1-44410-17200	2-44411-17200
1-44410	17400	1-44410-17400	2-44411-17400
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1-44410	20400	1-44410-20400	2-44411-20400
1-44410	20600	1-44410-20600	2-44411-20600
1-44410	20800	1-44410-20800	2-44411-20800
1-44410	21000	1-44410-21000	2-44411-21000
1-44410	21200	1-44410-21200	2-44411-21200
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1-44410	23000	1-44410-23000	2-44411-23000
1-44410	23200	1-44410-23200	2-44411-23200
1-44410	23400	1-44410-23400	2-44411-23400
1-44410	23600	1-44410-23600	2-44411-23600
1-44410	23800	1-44410-23800	2-44411-23800
1-44410	24000	1-44410-24000	2-44411-24000
1-44410	24200	1-44410-24200	2-44411-24200
1-44410	24400	1-44410-24400	2-44411-24400
1-44410	24600	1-44410-24600	2-44411-24600
1-44410	24800	1-44410-24800	2-44411-24800
1-44410	25000	1-44410-25000	2-44411-25000
1-44410	25200	1-44410-25200	2-44411-25200
1-44410	25400	1-44410-25400	2-44411-25400
1-44410	25600	1-44410-25600	2-44411-25600
1-44410	25800	1-44410-25800	2-44411-25800
1-44410	26000	1-44410-26000	2-44411-26000
1-44410	26200	1-44410-26200	2-44411-26200
1-44410	26400	1-44410-26400	2-44411-26400
1-44410	26600	1-44410-26600	2-44411-26600
1-44410	26800	1-44410-26800	2-44411-26800
1-44410	27000	1-44410-27000	2-44411-27000
1-44410	27200	1-44410-27200	2-44411-27200
1-44410	27400	1-44410-27400	2-44411-27400
1-44410	27600	1-44410-27600	2-44411-27600
1-44410	27800	1-44410-27800	2-44411-27800
1-44410	28000	1-44410-28000	2-44411-28000
1-44410	28200	1-44410-28200	2-44411-28200
1-44410	28400	1-44410-28400	2-44411-28400
1-44410	28600	1-44410-28600	2-44411-28600
1-44410	28800	1-44410-28800	2-44411-28800
1-44410	29000	1-44410-29000	2-44411-29000
1-44410	29200	1-44410-29200	2-44411-29200
1-44410	29400	1-44410-29400	2-44411-29400
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1-44410	32600	1-44410-32600	2-44411-32600
1-44410	32800	1-44410-32800	2-44411-32800
1-44410	33000	1-44410-33000	2-44411-33000
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1-44410	33600	1-44410-33600	2-44411-33600
1-44410	33800	1-44410-33800	2-44411-33800
1-44410	34000	1-44410-34000	2-44411-34000
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1-44410	35000	1-44410-35000	2-44411-35000
1-44410	35200	1-44410-35200	2-44411-35200
1-44410	35400	1-44410-35400	2-44411-35400
1-44410	35600	1-44410-35600	2-44411-35600
1-44410	35800	1-44410-35800	2-44411-35800
1-44410	36000	1-44410-36000	2-44411-36000
1-44410	36200	1-44410-36200	2-44411-36200
1-44410	36400	1-44410-36400	2-44411-36400
1-44410	36600	1-44410-36600	2-44411-36600
1-44410	36800	1-44410-36800	2-44411-36800
1-44410	37000	1-44410-37000	2-44411-37000
1-44410	37200	1-44410-37200	2-44411-37200
1-44410			

Material:

The materials used in this investigation were produced by Kawecki Beryllco 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 ^a %	C ^b ppm	Fe ^c ppm	Al ppm	Mg ^d ppm	Mn ^e ppm	Cr ^f ppm	Co ^g ppm	Cu ^h ppm	Si ppm	Ti ⁱ ppm	
301	P-10	<4	1.02	680	1090	410 ^j	260	160	85	95	<5	75	190 ^j	190
310	*	<4-10	.64	560	1000	310 ^j	270	135	90	115	<5	70	175 ^j	125
305	*	<20	1.09	400	1350	560 ^j	385	135	95	115	<5	70	180 ^j	280
307	P-10	<4	.53	270	950	100 ^j	50	130	60	60	<5	65	130 ^j	300
312	*	<4-10	.43	300	950	105 ^j	50	110	55	50	<5	60	130 ^j	155
311	*	<20	1.00	300	1040	110 ^j	45	110	50	90	<5	55	175 ^j	135
314	P-1	<4	.95	180	170	<20 ^j	5	50	3	10	50	15	31 ^j	<10
315	*	<4-10	.52	180	140	<20 ^j	4	110	<3	<5	9	10	46 ^j	<10
317	*	<20	1.02	300	260	25 ^j	8	105	8	8	25	25	45 ^j	<10
319	P-0	<4	.43	N.D.	70	45 ^j	5	3	2	<5	<5	15	40 ^j	<10
321	*	<4-10	<.01	250	120	30 ^j	12	20	12	<5	8	20	50 ^j	<10
322	*	<20	1.16	250	810	35 ^j	28	25	17	7	8	25	50 ^j	<10

Notes: ^a Neutron Activation

^b Conductometric

^c Atomic Absorption

^d Spectrographic

^e 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 electrolyzed 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 commination was accomplished in a beryllium-lined ball mill (tungsten 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 an 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.

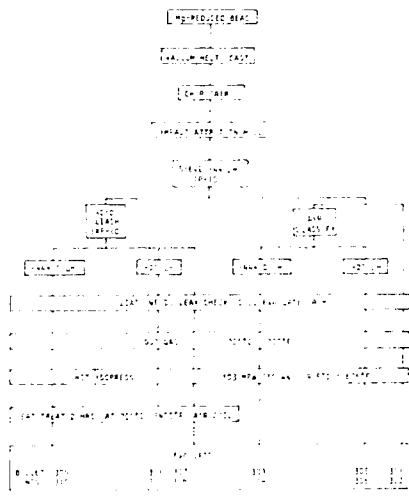


FIGURE 2 FLOW DIAGRAM FOR E-10 AND E-11 MATERIALS.

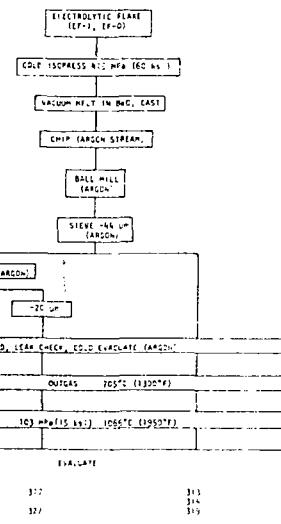


FIGURE 3 FLOW DIAGRAM FOR E-10 AND P-11 MATERIAL

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

WPS Temperature	Gage No.	Material Particle Size	Tensile Properties			Yield Strength psi	Compression Strength psi	Grain Size μ
			E_{UTS} psi	σ_{UTS} psi	ϵ_{UTS} %			
1100 K	12	$P_2O_5/44\text{ wt}\%$	474 (61.44)	46.8 (6.91)	0.4 (0.01)	474 (61.44)	413 (59.96)	6.10
	13	$P_2O_5/44\text{ wt}\%$	430 (61.44)	37.5 (5.47)	0.4 (0.01)	430 (61.44)	315 (52.10)	7.06
	19	$P_2O_5/20\text{ wt}\%$	626 (92.01)	52.1 (7.65)	0.4 (0.01)	626 (92.01)	540 (79.12)	3.97
1150 K	12	$P_2O_5/44\text{ wt}\%$	422 (61.44)	34.6 (5.11)	0.4 (0.01)	422 (61.44)	316 (57.80)	5.71
	19	$P_2O_5/44/20\text{ wt}\%$	392 (55.45)	35.4 (5.13)	0.4 (0.01)	392 (55.45)	291 (47.11)	5.03
(1104 K) 1100 K/AC	31	$P_2O_5/10/20\text{ wt}\%$	608 (87.97)	52.4 (7.62)	0.4 (0.01)	608 (87.97)	545 (79.11)	4.76
	24	$P_2O_5/44\text{ wt}\%$	317 (55.45)	40.2 (5.81)	0.4 (0.01)	317 (55.45)	238 (41.12)	5.59
1180 K	12	$P_2O_5/44/10\text{ wt}\%$	299 (55.45)	48.1 (7.05)	0.4 (0.01)	299 (55.45)	315 (46.74)	5.08
	19	$P_2O_5/44\text{ wt}\%$	454 (61.44)	49.6 (7.97)	0.4 (0.01)	454 (61.44)	347 (51.75)	4.44
1420 K	12	$P_2O_5/44\text{ wt}\%$	251 (55.45)	43.5 (6.89)	0.4 (0.01)	251 (55.45)	264 (51.81)	9.84
	19	$P_2O_5/44\text{ wt}\%$	254 (55.45)	37.9 (5.82)	0.4 (0.01)	254 (55.45)	260 (51.74)	7.05

*Interspace alloyed

**1100 K heat treated

E_{UTS} = 2000 psi yield stress

σ_{UTS} = 100000 stress at 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 strengthening 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 consistently lower value indicating a degree of anisotropy.

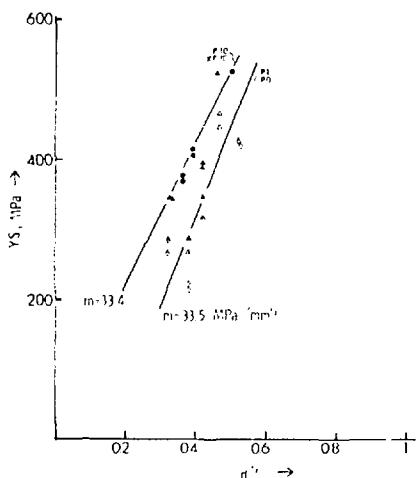


Figure 4 - The influence of grain size on the yield strength.

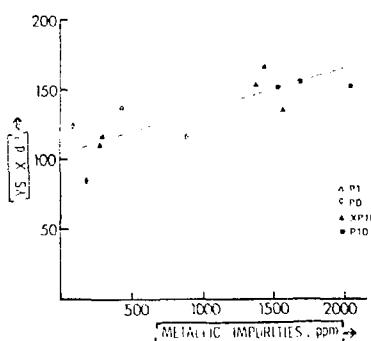


Figure 5 - The influence of metallic impurities on the yield strength (normalized for grain size).

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 F-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

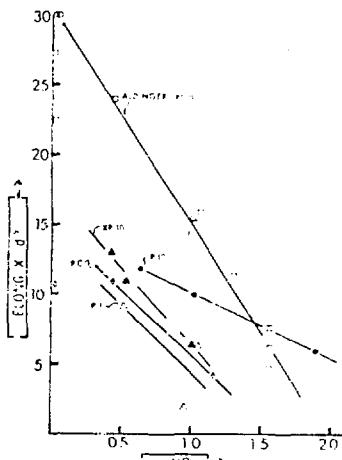


Figure 6 - The influence of beryllium oxide on elongation (normalized for grain size).

*Brush-Wellman Product

Some of the trailers (4-3, 14-1, 14-2) and one for the first flight were made reflecting a constant temperature. The rest of the data is to be fitted by techniques used to analyze the data in the case of constant. The first flight, in contrast, is less influenced by steady-state. The difference in density is that of the air (1.22), the weight of which with their original source is well beyond prediction.

RESULTS

The first flight was conducted at a constant temperature of 20°C. The second flight was conducted at a constant temperature of 15°C. The third flight was conducted at a constant temperature of 10°C. The fourth flight was conducted at a constant temperature of 5°C. The fifth flight was conducted at a constant temperature of 0°C. The sixth flight was conducted at a constant temperature of -5°C. The seventh flight was conducted at a constant temperature of -10°C. The eighth flight was conducted at a constant temperature of -15°C. The ninth flight was conducted at a constant temperature of -20°C.

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

The P-1, P-2 and P-10 grades are inherently lower in impurities and oxides because of the methods used in processing the powders. The test results indicate that the microstructural differences between these grades and the P-10 grade are great enough to affect the ductility and toughness behavior.

Conclusions

* All the Ti-6Al-6V-2Cr-2Mo tested in this study were casting of orthopedic implants. The ductility levels regardless of the impurities and purity levels.

* The Ti-6Al-6V-2Cr-2Mo casting with 10% V was the most ductile. This casting which is consistently produced from cleaned additive and the casting process.

* The aluminum oxide and titania content was estimated by the alumina and titania dissolution method. The purity content was determined by the acid dissolution method using the Ti-137 tracer. The purity content was 99.9% for all the samples tested.

* The Ti-6Al-6V-2Cr-2Mo casting with 10% V had the highest ductility and toughness.

* The Ti-6Al-6V-2Cr-2Mo casting with 10% V had the highest ductility and toughness. The ductility value was estimated by casting P-10 which has the lowest purity, impurity content.



Figure 1. Effect of Fe₃O₄ density, particle size, and distribution on the orientation of the fiber axis in the magnetorheological fluid. (a) 0.5 μm; (b) 1 μm.



Figure 2. Effect of Fe₃O₄ particle size on the orientation of the fiber axis in the magnetorheological fluid.

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