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SOME PROPERTIES OF BERYLLIUM OXIDE

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ABSTRACT

A literature search was made to determine what information is available on the properties of beryllia. The primary objective was to determine the best values for the properties involved in the thermal stress parameter. Comparison of data from various investigators indicates that thermal expansion of beryllia is independent of density. The wide variation in the thermal conductivity values contained in the literature indicates that substantial investigation of the thermal conductivity of beryllia is required. Graphs and tables are presented which summarize and compare data from various sources. A bibliography is given for some of the major items of the stress parameter (thermal expansion, thermal conductivity, and modulus of elasticity) as well as for general properties of beryllia.

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INTRODUCTION

A literature search to determine the information available on the properties of beryllia was made by the Technical Information Division. The primary purpose of this search was to determine from the literature the best values for the thermal stress parameter, τ . From Timoshenko,⁵

$$\tau = \sigma K(1 - \nu)/E\alpha = CQ,$$

where σ is the strength, K the thermal conductivity, E the modulus of elasticity, α the coefficient of expansion, ν Poisson's ratio, C the geometrical factor, and Q the power through the specimen.

The strength term in the parameter is the most difficult to evaluate because it is a function of stress distribution and loading rate as well as of many variables of the material itself. Based on the published properties of beryllia (K , E , α , and ν), and the thermal stress results for which $\sigma = CQ$, an effort is being made to determine empirically the best method of strength measurement for thermal stress calculations.

A table and curve for quick conversion between Fahrenheit and Centigrade temperatures, and a curve relating percent of theoretical density to measured densities in g/cc are given in Fig. 1. In the density curve of Fig. 1, 3.008 g/cc is used as the theoretical density. This value has been confirmed by x-ray results from General Electric, ANPD; Atomics International (AI); and LRL.*

THERMAL EXPANSION

Comparison of data from various investigators indicates that the thermal expansion of beryllia is independent of density. According to the x-ray data

* Letter on x-ray density of beryllium oxide from S. Carneglia, AI, to T. Merkle, LRL, dated 4-13-59, with accompanying comments from D. Sands and A. Zalkin of LRL.

Fig. 1. Temperature and density conversion scales.

from Klein,¹⁵ the expansion is isotropic from 20°C to 1000°C since the lattice parameter ratio, c/a , remains constant in this range. Klein states that his c/a data above 1000°C may show a slight downward trend, but there is too much scatter to be conclusive. In extruded beryllia, there is a strong tendency for the c -lattice parameter to parallel the extrusion axis. Up to 1000°C, isotropic behavior is substantiated by Etherington's¹² values on extruded beryllia, which fall very close to the average values in Table I. Above 1000°C, the data of Engberg and Zehms¹¹ on extruded material at 1000°C indicate a difference of about 500×10^{-6} in. If this value is subtracted from Engberg and Zehm's results for higher temperatures, then only a slight increase of expansion remains for extruded beryllia over hot-pressed beryllia. Generally, above 1000°C both the x-ray data and the data on extruded beryllia are subject to some suspicion and require verification. The unpublished x-ray results of a subsequent investigation at Atomics International by Taylor indicate that the c/a ratio remains constant at 1.620 from room temperature to 1708°C. It is likely that the expansion of beryllia is isotropic up to about 2050°C, at which point the beryllia is severely cracked by a process which is not fully understood.

The following discussion of high-temperature expansion results is quoted from Engberg and Zehms:

"The thermal expansion of BeO was found to be approximately linear over the temperature range from 1000 to 2000°C except for the net contraction, presumably caused by sintering, at about 1900°C for the ...hot-pressed ...samples. Above 2050°C, BeO exhibited a very large expansion... This large expansion was observed in all BeO samples heated above 2050°C. The rate of expansion for most of the samples was so large that simultaneous displacement measurements were impossible. Visual examination of the cooled samples revealed that the samples had bent and cracked, the physical dimensions had enlarged, and the color had changed to a bright milky white."

The average values listed in Table I and used for the curve in Fig. 2 are unweighted averages of the values in each temperature column, excluding the data of Crawford et al.,⁹ and of Mason and Seibel.¹⁶ Crawford et al., state: "...no great effort was made to obtain precise thermal expansion coefficients on the samples which were measured." Since their values tend to be relatively low, it seems reasonable to exclude them from the averages. The data of Mason and Seibel are above the averages by a nearly constant amount,

Table I. Thermal expansion data (microinches/inch)

Table 1. Thermal expansion data (microns/inch)																				
Ref. No.	Test Method	Bulk Density (g/cc)	Specimen Formation	Temperature (°C).																
				-130	-80	-20	100	200	300	400	500	600	700	800	900	1000	1200	1400	1600	1800
7	X-Ray		Powder				640	1410	2230	3090	3910	4820	5680	6640	7540	8550	10700			
8	Dilatometer	(a)3.004	Hot Pressed				400	1100	1900	2800	3800	4800	5300	6900	8000	9100				
9	Dilatometer	2.8	Hot Pressed				300	800	1600	2400	3300	4200	5200	6100	7000					
11	Dilatometer	(d) --	Hot Pressed													8600	11100	13600	16400	19000 Sintered
	Dilatometer		Hot Pressed													8800	11400	14000	16600	18400 17900
	Dilatometer	2.94	Hot Pressed													8600	11200	13600	16100	18600 20700
	Dilatometer	2.76	Extruded													9300	11900	14500	17200	19800 22500
12		2.10	Extruded					1000		2600		4500		6600		8800				
		2.16	Extruded					1000		2600		4600		6700		8900				
13	Dilatometer	--	Hot Pressed				570	1370	2180	2950	3810	4710	5710	6730	7810	8950	11300			
14	Interferometer	--						1000		2600		4500		6500						
	Dilatometer	--						1200		2800		4800		6900		9200	11600	14000	16600	
15	X-Ray	--	Hot Pressed				580	1200	1850	2500	3300	4170	5180	6180	7460	8570	11200	14000	16000	
16	Dilatometer	1.74 to 2.16									4300	5300	6300	7300	8300	9500	12000	14600		
		2.02	Dry Pressed				410	1070	1830	2700	3520	4500	5490	6530	7570	8670				
		2.14	Dry Pressed				410	1050	1320	2660	3510	4450	5420	6500	7570	8660				
		(b)1.75	Dry Pressed				400	1050	1800	2640	3500	4410	5450	6370	7500	8610				
(c)	Interferometer	(b) --					400	1050		2720										
18	Dilatometer	2.89	Hot Pressed				340	1040	1940	2710	3580	4500	5460	6500	7540	8630				
	Dilatometer	1.50	Hot Pressed				500	1120	1960	2630	3670	4670	5630	6710	7670	8540				
10	Dilatometer																			
				-380	-300	-160	460		1960											
Averages (Excluding Refs. 3 & 10)				-380	-300	-160	460	1120	1950	2710	3620	4570	5540	6600	7630	8780	11300	13950	16500	18950

(a) Specific gravity, (b) contained 12.3% Be₂C as an impurity, (c) reported values of J. B. Austin, (d) dash indicates that value was not given.

Fig. 2. Thermal expansion versus temperature.

600×10^{-6} in. to 760×10^{-6} in. Their report indicates no clear reason for this on the basis of either instrumentation or technique. However, they do not give the temperature reference point for zero expansion. Their data on material other than BeO indicates that this point may not have been definitely established, but varied with each test. The low-temperature data from Day¹⁰ have zero expansion at -140°C . Since the other data show zero expansion between 18°C and 25°C , I have referred Day's data to zero expansion at 20°C by using his diagram and expansion coefficients.

The coefficient of expansion has been established by dividing the expansion curve into a series of straight lines:

Temperature range ($^{\circ}\text{C}$)	200 to 500	600 to 900	1000 to 1600
Coefficient of expansion ($^{\circ}\text{C}^{-1}$)	8.35×10^{-6}	10.20×10^{-6}	12.85×10^{-6}

THERMAL CONDUCTIVITY

A composite curve for thermal conductivity versus temperature is shown in Fig. 3. Above 600°C the curve is from Taylor.³⁹ Taylor's curve was extrapolated to 100°C by using the curves of Franci and Kingery,²⁵ and Powell.³⁶ The dashed curve is an extrapolation to 100% theoretical density based on the data from Taylor,³⁹ and from McGill and Smith.³³ Using these curves, linear interpolations can be made to determine thermal conductivity for material between 90% and 100% of theoretical density. This suggestion is made only with the reservations noted later in this section.

The following passage from Taylor³⁹ introduces the theory of thermal conductivity of insulators:

"The theory of thermal conductivity of insulators, developed by Debye²³ and extended by Peierls,³⁵ states that the conductivity should be expressed by the equation

$$K = (1/3) CV\ell\rho,$$

where C is the specific heat, V is the phonon velocity, ℓ is the mean free path of the phonons, and ρ is the density. The mean free path may be divided into two terms given by the equation

$$1/\ell = (1/\ell_P) + (1/\ell_B),$$

where ℓ_P is the mean free path associated with phonon-phonon scattering, and ℓ_B is the mean free path associated with phonon-boundary and phonon-lattice

Fig. 3. Thermal conductivity versus temperature.

imperfection scattering. At extremely low temperatures, the phonon-phonon scattering is small and may be neglected, in which case ℓ equals ℓ_B and is constant. At these temperatures then, K is proportional to C which, in turn, is proportional to T^3 . At temperatures up to those where the value of specific heat approaches the Dulong-Petit value (0.476 cal/(gram °K) for beryllia), the relationships of specific heat and mean free paths are more complicated, but the net result is for the conductivity to reach a maximum value and then begin to decrease with a continued increase in temperature. For BeO, the maximum value of conductivity occurs somewhere between 100 and 300°K.³¹ ... According to Debye and Peierls, the mean free path is proportional to $1/T$ at high temperature, so that the thermal resistivity at high temperatures should be directly proportional to T .³²

A plot of thermal resistivity versus temperature is shown in Fig. 4. For beryllia, the proportionality between thermal resistivity and temperature seems to hold from about 600 to 1400° C. The reported results leave some question concerning the slope of the linear portion of the curve. This could be due to some unrecognized variable between the samples which affects the slope, but I think it is caused mainly by differences in testing method and technique. Taylor's values have been used in the composite curve since his method of radial heat flow is sound and his technique seems to be carefully developed. Taylor claims that comparison of his results for molybdenum and molten copper with other investigators indicates his accuracy to be 5%; however, one should be cautious about placing a high level of confidence in his values until they are verified by an independent investigation.

There have been numerous attempts to explain the observed deviations from the $1/T$ relationship above 1600° C.^{22, 29, 31, 32} Those who are interested should refer to the brief discussion by Taylor³⁹ of these theories in relation to his data. Taylor concludes that theories claiming the increase of thermal conductivity above 1600° C results from a radiation contribution to heat transfer are insufficient to explain the observed deviations from the $1/T$ relationship. He thinks it is likely that an activation process is occurring.

Kingery et al.,³⁰ have attempted to extrapolate conductivity values to 100% theoretical density with the linear relation.

$$K_{100\%} = \frac{K_{\text{measured}}}{1 - \text{porosity}}$$

Powell³⁶ has produced the only available data over a wide range of densities.

Fig. 4. Thermal resistivity versus temperature.

He has plotted conductivity as a function of density and attempts to draw straight lines through his values for constant temperatures up to 500°C; however, his lines indicate a much steeper slope than the relation used by Kingery. Similar plots of the conductivity results from Table II substantiate the steeper slope, and also indicate that the relation between conductivity and density very likely is non-linear. Therefore, the linear interpolation suggested in the first paragraph of this section should not be used over a wide range of densities.

The wide variation in thermal conductivity values between investigators indicates that substantial investigation of the thermal conductivity of beryllia is still required. There is special need for verification of high-temperature thermal conductivity values, and for additional values over a wide range of densities. Also, the effects of other variables, such as grain size and preferred orientation of grains and porosity, should be evaluated.

MODULUS OF ELASTICITY

Emphasis is placed on dynamic modulus measurements, since this is the modulus applicable to elastic behavior. Fig. 5 contains the available curves of E_t/E_0 versus temperature. The use of the curve from Atomic International⁴⁷ is recommended. The modulus at a particular temperature, E_t , can be determined from the ratio in Fig. 5 and the room temperature modulus, E_0 , given by Fig. 6 for the bulk density of interest.

The dynamic modulus values in Fig. 5 may be regarded as the upper limit of the modulus of elasticity because plastic behavior appreciably lowers the apparent modulus, especially above 1000°C. As a result, low-frequency dynamic-modulus and static-modulus data will be substantially lower at high temperatures than the curves shown in Fig. 5. Thermal stress measurements are usually of such a duration as to allow some plastic deformation to occur; therefore, some adjustment may be required for the modulus of elasticity values used in the thermal stress parameter. It may be necessary to make a static measurement of the modulus using loading rates similar to those seen by the thermal stress specimen.

The magnitude of the effect of plastic behavior is shown in Fig. 5 by the static measurements of Ryskewitch,⁵² and the two highest values from a series of high-temperature "short-time" bend tests made by Battelle Memorial Institute.⁴² The two high-temperature points on Fig. 5 from Atomic

Table II. Thermal Conductivity (watts/cm-°C)

Temperature (°C)														Bulk density g/cc	Spec. for- mation	Test method	Ref. No.
100	200	300	400	500	600	700	800	1000	1200	1400	1600	1800					
			0.671		0.356		0.237	0.180	0.158					2.7	Slip cast	Prolate spheroidal envelope	(a) 19
2.09	1.66	1.23	0.893	0.636	0.460									2.86	Slip cast	Longitudinal comparative heat flow	(a) 25
								0.190	0.165	0.145	0.137	0.145		2.7 to 2.86	Slip cast	Prolate spheroidal envelope	(a) 34
		1.17	0.867	0.652	0.480	0.376	0.299	0.212	0.163					(b) 2.99	Hot pressed	Longitudinal comparative heat flow	21
1.58	1.13	0.845	0.666	0.541	0.462	0.406								2.62	Hot pressed	Longitudinal heat flow	24
									0.336					2.96	--	--	26
	0.810	0.650	0.540	0.470	0.415									2.87	Hot pressed	Steady state	27
0.196														2.88	--	Transient heat flow	28
1.70	1.19	0.902	0.686	0.546										2.60	Hot pressed	Longitudinal comparative heat flow	33
0.54	0.41	0.34	0.285	0.25	0.235									1.85	Slip cast		36
1.02	0.755	0.575	0.475	0.40	0.35									2.3		Longitudinal comparative heat flow	36
1.95	1.37	1.075	0.86	0.69										2.82	Hot pressed		36
1.85	1.41	1.11												3.01			36
1.59														2.97	Hot pressed	Longitudinal comparative heat flow	37
					0.630		0.390	0.287	0.226	0.186	0.174	0.178		2.99	Hot pressed		39
					0.520		0.348	0.262	0.211	0.174	0.149	0.131		2.86	Hot pressed	Radial heat flow	
(c) 0.001 (-270)		0.025 (-263)		0.144 (-253)		1.18 (-223)		3.6 (-183)						2.94	Slip cast	Longitudinal heat flow	(c) 20
									(c) 1.82 (70)					3.0	Hot pressed	Longitudinal comparative heat flow	38

Note: watts/(cm-°C) = 0.2388 cal/(sec-cm-°C) = 57.78 BTU/(hr-ft-°F)

(a) These MIT data have been compiled by Kingery et al.³⁰

(b) Specific gravity

(c) Temperature (°C) listed in parenthesis

Fig. 5. Elastic modulus (E_t/E_0) versus temperature.

Fig. 6. Room temperature dynamic elastic modulus (E) versus bulk density.

International⁴⁰ were from material which was very close to theoretical density. These relatively low results from AI probably indicate either that they were low-frequency measurements, or that beryllia with very high density may be more sensitive than lower density beryllia to the temperature effect on its modulus.

POISSON'S RATIO

As with the modulus of elasticity, dynamic determinations of Poisson's ratio, ν , are of primary interest. The modulus of elasticity, modulus of rigidity, and Poisson's ratio are determined by the ultrasonic pulse technique.⁵¹ This method uses the velocities of a direct elastic wave and an elastic wave reflected from the specimen's surface to calculate the various quantities. Three runs utilizing this technique have been made at Atomics International on a specimen with a density of 3.008 g/cc.^{**} All three runs yielded the same value for Poisson's ratio; $\nu = 0.204$. These are the only dynamic results available.

The values of Poisson's ratio calculated from static measurements are considerably higher than the Atomics International results. Ryshkewitch⁵² reports $\nu = 0.34$ (20°C) and $\nu = 0.35$ (1000°C) for sintered beryllia having a specific gravity of 2.7 to 2.8 g/cc. The major value to us of these results is that they indicate Poisson's ratio remains constant with temperature. Above 1000° C, creep will become significant and static tests should give results increasing to a limiting value of $\nu = 0.5$.

^{**}Telephone conversation with S. Carneglia on April 13, 1961.

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