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X-ray determination of lattice parameters and thermal expansion of potassium bromate

The lattice parameters of potassium bromate ($KBrO_3$) have been determined by different investigators (Swanson *et al.* [1], Mery [2] and Wyckoff [3]) but only at room temperature. No data are available on the temperature variation of the lattice parameters either at low or high temperatures. Also thermal expansion of $KBrO_3$ has not previously been measured. Hence it was decided to investigate the properties of $KBrO_3$ with the object of determining the lattice parameters and thermal expansion as a function of temperature between 302 and 512 K.

Powder samples of $KBrO_3$ with a specified purity of 99.99% were obtained from Riedel Dettan Ag Seelze, Hannover, Germany. To obtain uniform particle size these powder samples were filtered through a 44 μm sieve.

A symmetrically focusing back-reflection camera of 15 cm diameter was used for obtaining powder photographs at elevated temperatures with filtered CuKα radiation. The specimen holder and heater were of such a design that the specimen could be heated to and maintained at any desired temperature. The temperature of the specimen could be measured to an accuracy of ± 0.8 K with the help of Pt–13% Rh thermocouple and a commercial temperature controller. The powder photographs were obtained at thirteen intervals between 302 and 512 K.

The X-ray powder photograph showed ten intense lines in the angular range from 72° to 82° i.e. $(6\,1\,2)_{\alpha_1\alpha_2}$, $(6\,1\,1)_{\alpha_1\alpha_2}$, $(4\,2\,6)_{\alpha_1\alpha_2}$, $(4\,0\,8)_{\alpha_1\alpha_2}$ and $(3\,3\,6)_{\alpha_1\alpha_2}$. The $KBrO_3$ has a rhombohedral structure. In evaluating the lattice parameters of $KBrO_3$ using Bragg reflections, it was assumed that rhombohedral structure is a special case of hexagonal structure. The a and c lattice parameters were evaluated at different temperatures using Cohen's analytical method [4]. Independent measurements and calculations were made on each film and the average values obtained from these are given in Table I.

Wykoff [3] and Mery [2] quoted the room temperature values of the lattice parameters but without any reference points or experimental

TABLE I Lattice parameters of potassium bromate at various temperatures

Temperature (K)	a_T (Å) (± 0.0003)	c_T (Å) (± 0.0009)
302	6.0170	8.1935
324	6.0232	8.1978
341	6.0279	8.2015
358	6.0341	8.2056
379	6.0390	8.2089
394	6.0436	8.2148
400	6.0456	8.2167
410	6.0503	8.2255
425	6.0554	8.2301
446	6.0611	8.2365
462	6.0661	8.2448
486	6.0760	8.2601
512	6.0841	8.2747

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TABLE II Lattice parameters of potassium bromate at room temperature

Serial number	a (Å)	c (Å)	Reference
1	6.0170 ± 0.0003	8.1935 ± 0.0009	Present work
2	6.0057	8.1408	[3]
3	6.014	8.156	[1]
4	6.008	8.1842	[2]

details. Similarly Swanson *et al.* [1] have found lattice parameters at room temperature using X-ray diffraction data taken up to the Bragg angle, $\theta = 55°$. All three measurements of the lattice parameters lack precision and there is considerable disagreement between the three values. In the light of these facts, it is not possible to compare the precisely determined lattice parameters of this work with that of all the three earlier workers [1–3]. For the sake of completeness, the present results along with earlier measurements are given in Table II.

The variation of lattice parameters with temperature are shown in Figs 1 and 2. They are non-linear and the least square fitting to these data points gives the following equations

$$a_T = 5.9473 + (177.2525 \times 10^{-6})T + (17.5871 \times 10^{-8})T^2 \quad (1)$$

$$c_T = 8.2023 - (120.5561 \times 10^{-6})T + (10.0381 \times 10^{-10})T^3 \quad (2)$$

TABLE III Thermal expansion coefficients of $KBrO_3$ at various temperatures

T (K)	α_a ($\times 10^6$ K^{-1})	α_c ($\times 10^6$ K^{-1})
302	47.66	18.79
324	48.97	23.84
341	49.97	27.99
358	50.98	32.36
379	52.22	38.04
394	53.11	42.30
400	53.46	44.04
410	54.05	47.01
425	54.94	51.62
446	56.18	58.33
462	57.13	63.67
486	58.55	72.02
512	60.08	81.55

where a and c are expressed in Å and T in K. It is observed that c_T increases with T more than a_T. The thermal expansion coefficients α_a and α_c

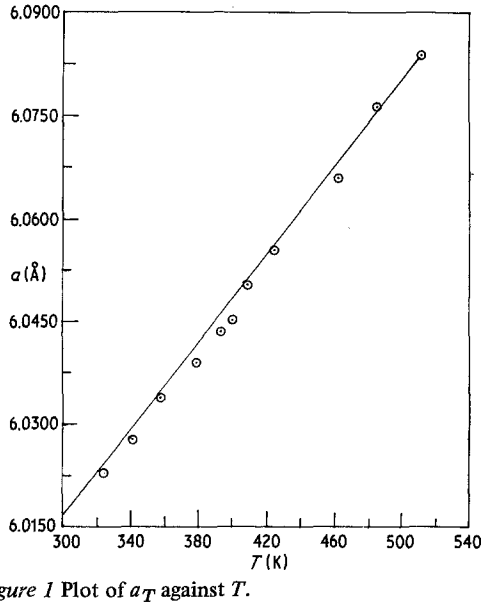

Figure 1 Plot of a_T against T.

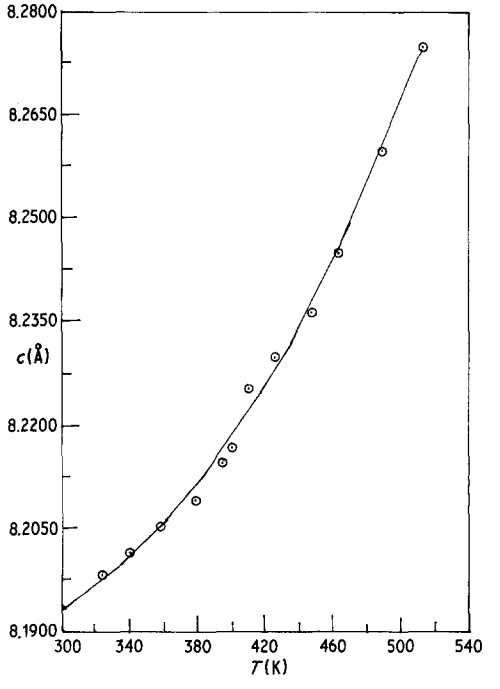

Figure 2 Plot of c_T against T.

obtained by differentiation of a_T and c_T with respect to temperature are

$$\alpha_a = (29.80 \times 10^{-6}) + (5.91 \times 10^{-8})T \quad (3)$$

$$\alpha_c = -(14.70 \times 10^{-6}) + (3.67 \times 10^{-10})T^2 \quad (4)$$

where α and T are expressed in K^{-1} and K respectively. The error in α is estimated to be less than 2.5% per 10 K. The present results (Equations 3 and 4) suggest that α_a varies linearly with temperature and α_c varies non-linearly with temperature. The rate of variation of α_c with temperature is much faster than that of α_a with temperature. This is because the slow variation of α_a with temperature maintains the crystal structure. The variation of α_a and α_c with temperature are presented in Table I. The thermal expansion data of α_c obtained in this work by X-ray diffraction shows anomalous behaviour at higher temperatures (above 390 K). The anomalous behaviour of α_c may be attributed to point defects in crystals.

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Consolidation of Si_3N_4 by shock compression

Silicon nitride (Si_3N_4) has been considered a prime candidate for high temperature engineering materials. So far, the materials have been fabricated with densification aids. The strength of sintered materials at high temperature was lessened due to the softening of intergranular phases containing densification aids. The consolidation of Si_3N_4 without a densification aid has been tried by hot isostatic pressing (HIP) [1], high pressure hot pressing (HPHP) [2, 3] and heating under high nitrogen pressures [4].

The present note reports the results of attempts to consolidate Si_3N_4 by shock compression without a densification aid.

Two kinds of Si_3N_4 powders were used as starting materials, made by AME* and GTE†. Both powders did not densify by hot pressing at 1750° C for 1 h under a pressure of 15 MN m^{-2} without a densification aid. The powder was compacted in a stainless steel capsule at 800 MN m^{-2} to form a disc 4 mm thick and 12 mm in diameter giving a density of about 2.04 g cm^{-3}. The disc was shock-compressed by an explosive plane-wave generator, sometimes called a "mousetrap". The details of the shock experiment are reported elsewhere [5]. The impact pressure induced in

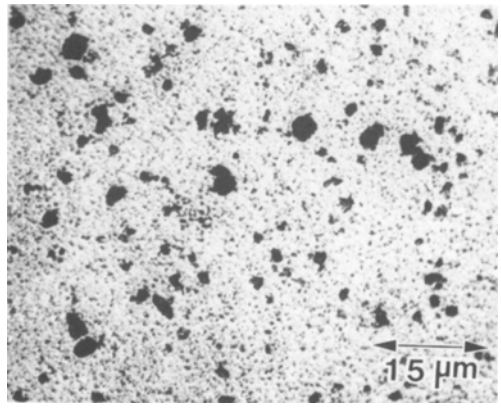

Figure 1 Optical micrograph of polished surface of shock compressed Si_3N_4.

*Advanced Materials Eng., England, high purity grade, α content = 71%.
†GTE Sylvania, USA, SN-502, α content = 91%.

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