The X-ray pattern of low-temperature cristobalite.

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CRISTOBALITE is found as the first product in the conversion of quartz following a high-temperature heating, and it constitutes a large percentage of the crystalline material in most manufactured silica bricks. It is present also in fireclay products, the amount being dependent upon the nature of the raw material and the conditions of firing. In the majority of these products the cristobalite is only poorly developed, i.e. the crystals are only perfect over distances of the order of about 1×10^{-6} cm. This value is derived from the diffuseness of the high-order reflections on the X-ray powder photograph. On the other hand, the crystals of cristobalite are well developed in the hot face of an open-hearth silica roof-brick. This crystal growth is due undoubtedly to a high flux or mineralizing content of the slagged working face in conjunction with a temperature of 1500–1700° C. It has been our experience that the X-ray powder pattern of this cristobalite is not changed, as regards both relative line intensities and lattice dimension, by the nature or amount of the constituents present.

X-ray studies of single crystals by Nieuwenkamp[1] have already shown that low-temperature or α-cristobalite is tetragonal. The present work confirms tetragonal symmetry, gives indexed powder data, and deduces accurate lattice dimensions.

A sample of the cristobalite zone from a silica roof-brick was ground in an agate mortar. The powder was repeatedly digested in hydrochloric acid and washed until only traces of impurities could be detected in the solution. Powder photographs were taken of two samples in a cylindrical camera, diameter 19 cm., using cobalt Kα radiation; one sample only 0·3 mm. in diameter packed into a capillary tube to obtain sharp lines for the longer spacings, and the second sample diameter 0·8 mm. prepared with Canada balsam solution to obtain an intense line pattern of the shorter spacings in a reasonable time.

Table I. Powder data for low-temperature cristobalite. Diameter of specimen 0·3 mm. Camera diameter 19 cm. Taken with filtered cobalt radiation $K\alpha_1$ 1·78529, $K\alpha_2$ 1·78919 kX.

Intensity.	Sin θ. Observed.	Sin θ. Calculated.	Indices.	Interplanar spacing kX units.	Intensity.	Sin θ Observed.	Sin θ Calculated.	Indices.	Interplanar spacing kX units.
vs	0·2212	0·2217	101	4·03	m	0·4642	0·4642	113	1·924
m	0·2849	0·2856	111	3·13	m	0·4784	0·4786	212	$1·866_5$
ms	0·3145	0·3152	102	$2·83_5$	vw	0·5090	0·5092	220	$1·754_5$
s	0·3595	0·3601	200	$2·48_1$	w	0·5176	0·5175	004	$1·726_5$
w	0·3628	0·3630	112	$2·46_1$	wm	0·5295	0·5294	203	$1·687_5$
wm	0·4228	0·4229	211	2·112	vw	0·5481	0·5479	104	$1·630_5$
wm	0·4432	0·4434	202	2·015	m	0·5554	0·5554	301	$1·608_5$

[1] W. Nieuwenkamp, Zeits. Krist., 1935, vol. 92, p. 82. [M.A. 6–329.]

Intensity.	Sin θ Observed.	Sin θ Calculated.	Indices.	Interplanar spacing kX units.
w	0·5593	0·5592	213	1·597$_5$
w broad	0·5701	0·5694	310	1·569
		0·5712	222	1·564
m	0·5841	0·5839	311	1·530
m	0·5990	0·5989	302	1·491$_5$
wm	0·6253	0·6254	312	1·428$_5$
w	0·6307	0·6305	204	1·4168
w	0·6404	0·6403	223	1·3951
wm	0·6554	0·6552	α_1	1·3627
	0·6568	0·6566	α_2 214	
vw	0·6617	0·6615	α_1	1·3494
	—	—	α_2 321	
vw	0·6648	0·6646	α_1	1·3431
	—	—	α_2 303	
wm	0·6712	0·6709	α_1	1·3305
	0·6726	0·6724	α_2 105	
wm	0·6888	0·6886	α_1	1·2963
	0·6903	0·6901	α_2 313	
wm	0·6985	0·6983	α_1	1·2784
	0·7000	0·6998	α_2 322	
vw	0·7256	0·7255	α_1	1·2304
	—	—	α_2 224	
w	0·7314	0·7312	α_1	1·2209
	0·7329	0·7326	α_2 401	
wm	0·7420	0·7418	α_1	1·2034
	0·7437	0·7434	α_2 410	
w	0·7559	0·7558	α_1	1·1812
	—	—	α_2 323	
w	0·7615	0·7613	α_1	1·1725
	0·7632	0·7630	α_2 215	
vw	0·7689	0·7688	α_1	1·1610
	—	—	α_2 314	
vw	0·7743	0·7742	α_1	1·1529
	—	—	α_2 331	
w	0·8060	0·8059	α_1	1·1076
	0·8078	0·8077	α_2 332	
m	0·8150	0·8149	α_1	1·0954
	0·8166	0·8167	α_2 421	
		0·8163	α_1	1·0935
	0·8182	0·8181	α_2 116	
vvw	0·8229	0·8226	α_1	1·0852
	—	—	α_2 225	
vw	0·8298	0·8296	α_1	1·0760
	—	—	α_2 324	
vvw	0·8371	0·8371	α_1	1·0664
	—	—	α_2 413	
wm	0·8452	0·8451	α_1	1·0563
	0·8470	0·8470	α_2 422	
vw	0·8564	0·8562	α_1	1·0427
	0·8582	0·8581	α_2 333	
w	0·8611	0·8611	α_1	1·0367
	0·8630	0·8630	α_2 315	
wm	0·8933	0·8932	α_1	0·9993
	0·8952	0·8952	α_2 423	
wm	0·8997	0·8996	α_1	0·9922
	0·9016	0·9016	α_2 500	
m	0·9043	0·9042	α_1	0·9872
	0·9063	0·9062	α_2 414	

vs very strong, s strong, ms moderately strong, m moderate, wm weak moderate, w weak, vw very weak, vvw just visible.

Indices of other lines are 501, 325, 334 and 107, 511, 502, 306, 424, 316, 405, 207, 521, 503, 415, and 217 (θ = 82·00° (α_1) 83·00° (α_2)).

The extrapolated values[1] of the cell dimensions of α-cristobalite at 22° C., corrected for refraction, are a 4·9615, c 6·9054 kX; $a:c$ = 1:1·3918.

Summary. Cristobalite taken from the hot face of a used silica brick from an open-hearth furnace roof is found to be well crystalline with tetragonal symmetry. The line pattern with cobalt Kα radiation has been indexed and the lattice dimensions of the unit cell are a 4·9615, c 6·9054 kX units.

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[1] Nieuwenkamp's values, a 7·02, c 6·92 kX (7·02 = $\sqrt{2} \times 4{\cdot}965$).