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β'-sialons are solid solutions with the structure of β-Si_3N_4. The general formula is $Si_{6-z}Al_zO_zN_{8-z}$. Silicon and aluminium occupy 6h sites (x, y, $\frac{1}{4}$) in the hexagonal cell with spacegroup $P6_3/m$ [1, 2]. Nitrogen and oxygen are present on 6h and 2c sites (1/3, 2/3, 1/4). Gillot *et al.* [3] have performed neutron diffraction studies of sialons with $z = 2.0$, 2.9 and 4.0. Because of the different neutron diffraction scattering amplitudes of nitrogen and oxygen the atomic distribution of these atoms over the 6h and 2c sites could be determined. The authors find there is a definite trend towards a preferential occupation by oxygen of the 2c sites. Gillot *et al.* also report that the profile refinement gives the best fit with the experimental data when it is assumed that vacancies are present on the silicon and aluminium positions. The vacancy concentrations of 3.5% and 5% observed for the metal-6h sites for $z = 2.0$, 2.9 and 4.0 suggest that the actual composition of β'-sialon should be written as $Si_{6-3/4z}Al_{2/3z}O_zN_{8-z}V_{z/12}$, where V represents a vacancy. The calculated vacancy concentrations corresponding to the latter formula would be 2.8%, 4.2% and 5.5%, respectively. This somewhat unexpected result led us to a reinvestigation of the β'-sialon structure.

In fact the problem is related to the question whether β'-sialons can be prepared from $Si_3N_4 + Al_2O_3$ or from $Si_3N_4 + Al_2O_3 + AlN$. Gillot *et al.* have used the first method to prepare their samples. From later studies of the phase diagram it follows that three components are necessary to produce β'-sialon. Thus, the sample of Gillot *et al.* probably contained small amounts of second phases. Because such phases are often present in an amorphous state they are difficult to observe. A small number of weak impurity peaks is present in the diffraction patterns of Gillot *et al.*

We have chosen a different approach, using carbothermal conversion of kaolinite to produce sialon powders. A sample with $z = 3$ was prepared, according to the reaction

$$3Al_2(OH)_4Si_2O_5 + 5N_2 + 15C \rightarrow 2Si_3Al_3O_3N_5 + 15CO + 6H_2O$$

Details of the preparation method are published elsewhere [4]. Because of the presence of small amounts of impurities in the kaolin, used as starting product, minor amounts of second phases $FeSi_2$ and TiN are present.

A neutron diffractogram has been obtained at 293 K on the powder diffractometer at the HFR reactor at Petten. Neutrons of wavelength $\lambda = 0.25790$ (3) nm were obtained by using the beam reflected from the (1 1 1) planes of a single crystal of copper and the λ/n contamination was reduced to less than 0.1% by means of a pyrolytic graphite filter. Soller slits with a divergence of 30′ were placed between the reactor and in front of the four ^3He counters. The pattern was analysed by means of the Rietveld profile refinement technique [5]. The scattering amplitudes used were taken from Koester and Yelon [6].

The quality of the fit is given by the expression $\chi^2 = \Sigma\, w_i[y_i\,(\text{obs}) - y_i(\text{calc})]^2/v$, where y_i(obs) and y_i(calc) are the observed and calculated values of the ith measuring point, w_i being its statistical weight and v representing the degrees of freedom. The value obtained is $\chi^2 = 19$. This rather high value is ascribed to the presence of extra peaks in the diffractogram because of the impurities already mentioned. The value for the often used reliability index R defined as $R = \Sigma[I(\text{obs}) - I(\text{calc})]/\Sigma I(\text{obs})$ is 2.9%.

The lattice parameters calculated from the neutron diffraction data are: $a = 0.76805$ (4) nm, $c = 0.29750$ (2) nm. The coordinates of the 6h sites for silicon and aluminium are $x = 0.1675$ (8) and $y = -0.2395$ (12). The coordinates of the 6h sites for oxygen and nitrogen are $x = 0.3330$ (7) and $y = 0.0317$ (3).

The calculated diffraction profile gives the best fit with the experiments when it is assumed that all sites are occupied. The site occupancy is shown in Table I. The table also gives the calculated occupancy for a random distribution of nitrogen and oxygen over the 6h and 2c sites. From these data it is evident that oxygen atoms show a preference for 2c sites and nitrogen for 6h sites. It is interesting to make a comparison with β-Si_3N_4 and Si_2N_2O. In Si_3N_4 there are SiN_4 tetrahedra consisting of three nitrogen on h-sites and one nitrogen on a c-site. The Si_2N_2O structure can be derived from the β–Si_3N_4 structure by replacing the nitrogen on the c-site by an oxygen atom. We then

TABLE I Values of the site occupancy for oxygen and nitrogen atoms on the 6h and 2c sites, determined from neutron diffraction amplitudes at room temperature for $Si_3Al_3O_3N_5$

Site	Atom	Observed occupation	Random occupation
6h	O	1.73 (3)	2.25
6h	N	4.27 (3)	3.75
2c	O	1.27 (3)	0.75
2c	N	0.73 (3)	1.25

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have pseudo-planar SiN_3 groups connected with each other by Si–O–Si bonds. In β'-sialon we find an intermediate case.

The bond lengths obtained are 0.1743, 0.1746 and 0.1816 nm, while those obtained by Gillot *et al.* are 0.1730, 0.1739 and 0.1745 nm. We do not have any explanation for the value of 0.1816 nm.

Summarizing we can say that our measurements confirm the earlier conclusion of Gillot *et al.* concerning the site preference of oxygen and nitrogen atoms. We have not found any indication for the presence of structural vacancies in the β'-sialon structure, however. This is in accordance with the generally accepted formula $Si_{6-z}Al_zO_zN_{8-z}$ for β'-sialon.

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