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Sialons are solid solutions derived from Si_3N_4 by partial replacement of Si by Al and of N by O. The so-called α'-sialons have the structure of α-Si_3N_4 (space group P31c) and the general formula $Me_xSi_{12-(m+n)}Al_{m+n}O_nN_{16-n}$, with $x \leq 2$ [1]. If the valency of the metal Me is v, electroneutrality requires $x = m/v$. Earlier X-ray diffraction (XRD) studies [2] have shown that the Me cations occupy the large interstitial 2(b)-sites at positions (1/3, 2/3, z) and (2/3, 1/3, $z + 1/2$). The Si and Al occupy 6(c) sites, while O and N are distributed over 2(a), 2(b) and 6(c) sites. In the XRD studies a statistical distribution over the available sites was assumed. It is known, however, from neutron diffraction studies on the closely related β'-sialons that both Si/Al and the O/N ions show a site preference [3, 4]. We therefore performed a neutron diffraction study on an yttrium α'-sialon to investigate a possible site preference in this system.

Samples with starting composition $Y_{0.5}Si_{9.75}Al_{2.25}O_{0.75}N_{15.25}$ ($m = 1.5$, $n = 0.75$) were prepared by reaction sintering of a mixture of Si_3N_4 (LC12, Starck), AlN (grade C, Starck) and Y_2O_3 (99.99% purity, Ventron). The powders were ball-milled with silicon nitride balls in ethanol, dried, pressed first uniaxially (1 MPa) and then isostatically (250 MPa). The pellets were fired in a gas pressure furnace, for 60 min at 1800 °C under 0.5 MPa nitrogen, followed by 30 min at 1900 °C under 10 MPa nitrogen. The total weight loss was <2 wt%. XRD analysis showed that the sintered samples consisted of α-sialon as the only crystalline phase. Scanning (SEM) and transmission (TEM) electron microscopy analysis revealed that a few % of an Y-rich amorphous grain boundary phase was present in the sintered samples. However, energy-dispersive X-ray (EDX) analysis did not indicate any measurable deviation of the bulk grains from the starting composition. Samples were powdered for the neutron diffraction experiments.

A neutron diffractogram (Fig. 1) was obtained at 295 K on the powder diffractometer HB5 at the High Flux Reactor at ECN Petten (The Netherlands). Neutrons of wavelength $\lambda = 0.257\,18$ nm were obtained by reflection from a Cu(1 1 1) single-crystal monochromator. Higher-order wavelength contributions were removed using three pyrolytic graphite filters. The incident and reflected beams were collimated using 30′ Soller slits. The structural parameters were obtained from the Rietveld refinement method (program DBW3.2S, version 8804). The scattering lengths used were, in units of 10^{-12} cm, 0.775 for Y, 0.4149 for Si, 0.3449 for Al, 0.5805 for O and 0.930 for N. The background was refined using a fifth-order polynomial. Due to high correlations between the site occupancy numbers

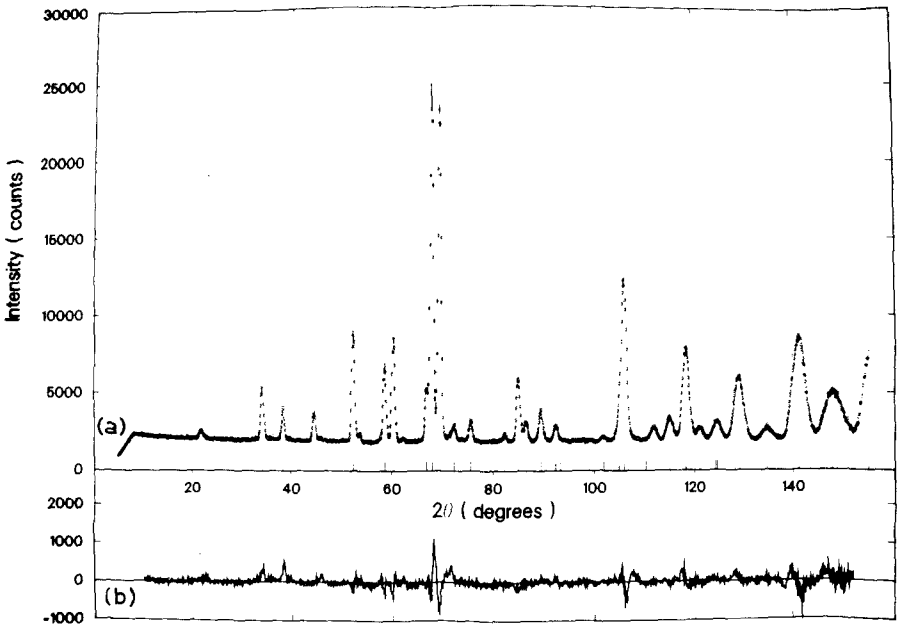

Figure 1 (a) Rietfeld refinement pattern for Y α'-sialon and (b) the difference between the observed and calculated intensities.

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TABLE I Results of the Rietveld refinement for Y α'-sialon with $m = 1.5$ and $n = 0.75$.

Atom	Position	x	y	z	Site occupancy, g
Y	2(b)	1/3	2/3	0.243(6)	0.5
Si(1)	6(c)	0.506(1)	0.078(1)	0.220(2)	5.8(3)
Al(1)	6(c)	0.506(1)	0.708(1)	0.220(2)	0.2(3)
Si(2)	6(c)	0.163(1)	0.2486(9)	0.010(3)	3.9(3)
Al(2)	6(c)	0.163(1)	0.2486(9)	0.010(3)	2.1(3)
N,O(1)	2(a)	0	0	0	2
N,O(2)	2(b)	1/3	2/3	0.651(2)	2
N,O(3)	6(c)	0.3424(5)	−0.04080(4)	−0.014(2)	6
N,O(4)	6(c)	0.3156(5)	0.3175(5)	0.253(2)	6

$R_p = 2.87\%$, $R_{wp} = 3.75\%$, $\chi^2 = 4.20$, $R_B = 3.20\%$, overall temperature factor $B = 0.0036(5)$ nm^2, $a = 0.781\,55(2)$ nm, $c = 0.569\,63(1)$ nm.

and the individual isotropic temperature factors, an overall temperature factor was applied. For the Rietveld analysis, space group P31c was assumed. Two weak peaks in the neutron diffractogram corresponding to $d = 0.332$ and 0.247 nm could not be assigned to the α'-sialon phase and were therefore omitted from the refinement. They originate from small traces of β-Si_3N_4, and can be assigned to the (200) (100%) and (210) (93%) reflections, respectively. Table I gives both the atomic positions and the site occupancy as well as the cell parameters, the overall temperature factor and the fit quality parameters. Taking into account the difference in composition between our sample and the sample used by Izumi *et al.* [2] for the X-ray refinement, there is a good agreement in the atomic positions. The statistical occupancy numbers for Si and Al would be 4.875 and 1.125, respectively. However, from Table I it is evident that aluminium ions show a preference for the sites with $x = 0.163$, whereas silicon prefers the position with $x = 0.506$. The oxygen concentration in the sample was too low to detect a site preference for this ion, if any.

Table II gives the calculated bond lengths. The Y–$(O,N)_3$ bonds and the Y–$(O,N)_4$ bonds were considerably longer than the Y–$(O,N)_2$ bond, which was parallel to the c-axis. This agrees with the results obtained by XRD [2]. The site preference observed for Si and Al does not lead to clear changes in (Si,Al)–(O,N) bond lengths. The $(Si,Al)_1$–(O,N) bonds, which have more Si character than the $(Si,Al)_2$–(O,N) bonds, were not systematically shorter as would have been expected on the basis of the atomic radii. In fact, the mean bond length was 0.1757 nm, which is between the values of 0.1784 nm observed in α-Si_3N_4 and 0.1739 nm in β-Si_3N_4.

TABLE II Calculated bond lengths

Bond	Length (nm)
Y–(Si,Al)	0.279 90
Y–$(N,O)_2^a$	0.232 41
Y–$(N,O)_3^a$	0.268 57
Y–$(N,O)_3^b$	0.268 58
Y–$(N,O)_4^a$	0.266 30
Y–$(N,O)_4^b$	0.266 29
$(Si,Al)_1$–$(N,O)_2$	0.179 10
$(Si,Al)_1$–$(N,O)_3^a$	0.175 68
$(Si,Al)_1$–$(N,O)_3^f$	0.173 85
$(Si,Al)_1$–$(N,O)_4^b$	0.178 59
$(Si,Al)_2$–$(N,O)_1^a$	0.171 05
$(Si,Al)_2$–$(N,O)_3^b$	0.174 42
$(Si,Al)_2$–$(N,O)_4^a$	0.172 81
$(Si,Al)_2$–$(N,O)_4^d$	0.180 09

Symmetry operations: (a) x, y, z; (b) $-y, x-y, z$; (c) $y-x, -x, z$; (d) $y, x, \frac{1}{2}+z$; (e) $-x, y-x, \frac{1}{2}+z$; and (f) $x-y, -y, \frac{1}{2}+z$.

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