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Investigation of the phase composition and stability of the α-SiAlONs by the Rietveld method

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Abstract

Different compositions of Y- and Nd-α-SiAlONs were sintered at 1825 °C and analysed by the XRD method. The Rietveld technique was used for characterisation of the diffraction pattern and the x value of α-SiAlON, which has a composition of $R_x^{+v}Si_{12-(m+n)}Al_{m+n}O_nN_{16-n}$ (R = Y, Nd). It was shown that the method yields reliable data for the content of the rare earth cations in the α-SiAlON phase, even in mixtures of α-SiAlONs with β- or α-Si_3N_4, but cannot be used for the determination of the Al and/or O contents of the α-SiAlONs. On the basis of these data, the stability regions of Y- and Nd-α-SiAlONs were established. Furthermore, it was found that the solubility area of the α-SiAlONs depends on the size of the additives. It was also shown that the solubility area of the Y-α-SiAlON is extended to lower X values than those suggested in the literature.

Keywords: Sialons; Rietveld method; X-ray methods

1. Introduction

In the last few years research in the field of SiAlON materials has been focused on α-SiAlON phases with the composition $R_x^{+v}Si_{12-(m+n)}Al_{m+n}O_nN_{16-n}$ ($x = m/v$, where v represents the valence state of the cation R and R is a large cation, typically Li, Mg, Ca, Y or Ln with $Z \geqslant 60$).[1] The increasing interest is caused by the observation of the formation of needle-like microstructures and the resultant increase in the fracture toughness of the materials. The aim of this research is to improve the fracture toughness up to the level of β-Si_3N_4 materials while retaining the hardness at a level of 30–50% higher than that found in β-Si_3N_4 materials.

In recent years there have been several investigations in the field of α-SiAlONs regarding phase relationships and the stability area of the α-SiAlONs with different rare earth cations.[2–16] Solubility limits have been determined by measuring the unit cell dimensions of the Y-α-SiAlON phase, $Y_x^{+v}Si_{12-(m+n)}Al_{m+n}O_nN_{16-n}$ with $m = 3x$, and have thus been reported as $1.0 \leqslant m \leqslant 2.4$ and $n \leqslant 1.7$ by Sun et al. and as $1.0 \leqslant m \leqslant 2.5$ and $0.5 \leqslant n \leqslant 1.24$ by Stutz et al.[6,13] These investigations were made in the classical way by determining the phases by X-ray diffraction and in some cases by additional analysis of the lattice parameter. However the lattice parameters of the α-SiAlON depend on the n and m values; therefore, these compositions cannot easily be determined by measuring the lattice parameters.[15] The interaction with the sintering atmosphere can lead to a shift in the composition, which makes the interpretation of the results more difficult (weight losses of 1–2% are quite typical). The data of Rosenflanz and Chen are given in Fig. 1. It was shown that the stability area of the α-SiAlONs depends both on the size of the rare earth ion and on the temperature. Increasing the size of the rare earth and decreasing the temperature leads to reduced solubility of Al and O in the α-SiAlON, i.e. to lower maximum n values. This behaviour could explain the observed reverse α→β-SiAlON transformation.[17] The change in the minimum x value in the α-SiAlON was not observed up to now.

Methodological development of the interpretation of X-ray diffraction data has resulted in improved Rietveld analysis programs, which are being increasingly used for phase analysis. The Rietveld method is based on the calculation of a theoretical diffraction pattern and the fitting of the calculated diagram to the experimental

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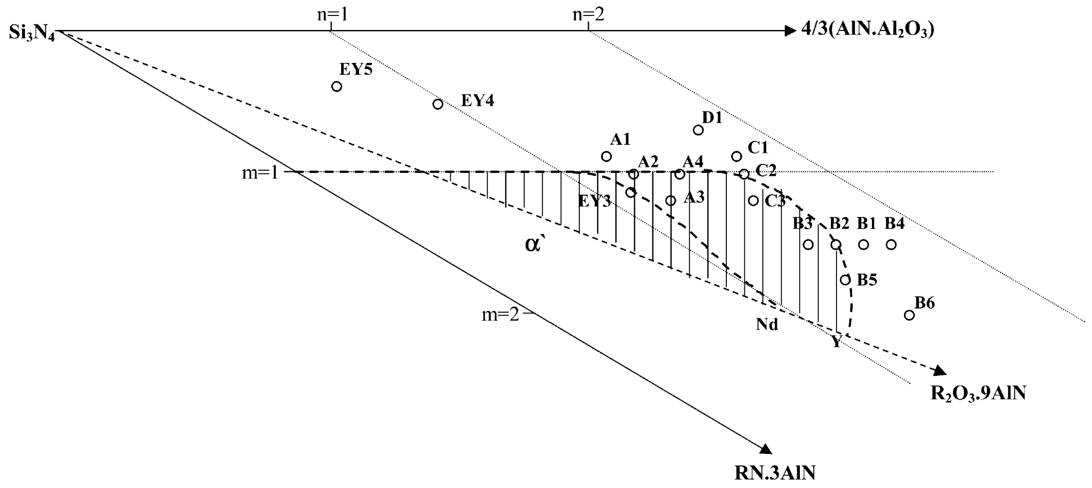

Fig. 1. Composition of samples in respect to the α-SiAlON homogeneity range (the data about the homogeneity region were taken from Rosenflanz[16]).

one.[18] For this, the structures of the phases must be known. The diffraction pattern of α-SiAlON with different R cations must strongly depend on the amount of the cation which is incorporated into the structure. Using this fact, it should be possible to determine the formula or at least the x and m values of the α-SiAlON precisely, even in mixtures with other phases.

The aim of this paper is to show that the Rietveld method can be used successfully for the analysis of the α-SiAlON phase composition in different ceramic materials. In an additional study, the changes in the phase relationships at different temperatures was studied.[20]

2. Experimental

Different compositions of α-SiAlON were prepared using the starting powders α-Si_3N_4 (UBE-10, containing 1.6% oxygen), AlN (Tokuyama, containing 1% oxygen), Al_2O_3 (99.99%, AKP50), Y_2O_3 (99.99% HC Starck) and Nd_2O_3 (99.99% Rhone-Poulenc). The oxygen contents of the nitride powders were taken into account for calculation of the compositions. The starting powders were weighed and milled in water-free isopropanol alcohol for 4 h in an agate jar using agate milling media. The mixed powders were dried and pressed into solid cylinders and were then isostatically pressed under a pressure of 200 MPa. The starting compositions are given in Table 1. In Fig. 1, the compositions are shown with respect to the homogeneity range of the α-SiAlONs determined by Rosenflanz.[16]

Sintering was carried out in a gas-pressure sintering furnace (GPS) in a nitrogen atmosphere (50 bar) at 1825 °C for 3 h with a heating rate of 10 °C/min up to 1825 °C. After sintering, the furnace was cooled down to room temperature at a cooling rate of 20 °C/min.

The density was measured by the Archimedes method. The results of sintering are given in Table 1. Sintering was carried out in a RBSN crucible. The weight losses were lower than 1%.

After sintering, the surface was ground (at least 4 mm were removed) and the phase composition was measured by X-ray diffraction. The XRD measurements were carried out with CuK_α radiation, a Ni filter or a graphite monochromator and the X-ray diffractometer XRD 7 (Seifert FPM). Quantitative XRD analysis and determination of the atomic positions and the occupation factors were performed using the program REFINE+ + (Seifert FPM).[19] These results are given in Table 1. The refinement of the α-SiAlON structure was performed using the α-Si_3N_4 structure as a basis.[21] The results for different compositions are given in Table 2. R_{wp} and R_{phase} (sum of the squares of the residuals weighted by the relative contribution of each phase to the intensity at the data point; this value is more sensitive than the R_{wp} parameter to the fitting of the phase) values were determined to control the quality of the calculations.[18,19] The R_{wp} and R_{phase} were in the range of 5–6% only, containing small amounts of the polytype 21R samples the R_{wp} value increases up to 7–8% without significant change of the R_{phase}. This is caused due to the fact, that the polytype 21R was not considered during calculations (no reliable data for the structure). Some of the samples were pulverised and mixed with Si as an internal standard for more accurate determination of the lattice parameters.

For the determination of the α-SiAlON content and the x and m values in samples containing β-Si_3N_4 or residual α-Si_3N_4 in addition to α-SiAlON, it was necessary to quantify the error due to overlapping of the peaks. For this purpose, two series of mixtures of sample EY 3a and β-Si_3N_4 (laboratory production) or α-Si_3N_4 (UBE-E10) powders were used.

Table 1
Composition, density and results of the X-ray diffraction measurement of samples sintered at 1825 °C

No	Composition values			Density (g/cm^3)	Δw^{b}%	Phase composition	x-Measured	β'-phase z value
	m	n	x					
YA1	0.86	1.26	0.286	2.141	0.86	$98.7\alpha'$-$1.3\beta'$	0.302	–
YA2	1.0	1.26	0.333	2.281	0.61	$100\alpha'$	0.363	–
YA3	1.16	1.26	0.386	2.379	0.71	$100\alpha'$	0.404	–
YA4	1.0	1.45	0.333	2.361	1.40	$100\alpha'$	0.349	–
YB1	1.54	1.67	0.513	3.308	0.45	$100\alpha'$-21R(vw)	0.478	–
YB2	1.54	1.54	0.513	3.295	0.43	$100\alpha'$-21R(vw)	0.495	–
YB3	1.54	1.44	0.513	3.171	0.36	$100\alpha'$	0.51	-
YB4	1.54	1.8	0.513	3.318	0.55	$100\alpha'$-21R(w)	0.474	–
YB5	1.8	1.4	0.6	3.276	0.32	$100\alpha'$	0.595	–
YB6	2.0	1.5	0.667	3.297	0.21	$97.8\alpha'$-M(w)-YAG(vw)	0.625	–
YC1	0.86	1.73	0.287	3.225	0.48	$94.3\alpha'$-$5.7\beta'$	0.293	0.85
YC2	1.0	1.62	0.333	2.713	0.63	$100\alpha'$	0.338	–
YC3	1.16	1.54	0.387	2.685	0.46	$100\alpha'$	0.407	–
YD1	0.7	1.8	0.233	3.210	0.62	$73.4\alpha'$-$24.3\beta'$-12H(w)	0.308	0.90
EY5	0.44	0.76	0.147	1.766	0.41	$58.7\alpha'$-$41.3\beta'$	0.240	0.35
EY4	0.59	0.98	0.197	1.980	0.79	$69.9\alpha'$-$30.1\beta'$	0.454	0.46
EY3	1.12	1.17	0.373	2.240	0.65	$100\alpha'$	0.362	–
EY3a[a]	1.12	1.17	0.373	2.240	0.65	$100\alpha'$	0.362	–
NA1	0.86	1.26	0.286	2.516	0.63	$93.6\alpha'$-$6.4\beta'$	0.301	–
NA2	1.0	1.26	0.333	2.767	0.51	$100\alpha'$-21R(w)	0.336	–
NA3	1.16	1.26	0.386	2.929	0.61	$100\alpha'$-21R(w)	0.384	–
NA4	1.0	1.45	0.333	3.359	0.58	$94\alpha'$-$3.2\beta'$-21R(w)	0.316	–
NB1	1.54	1.67	0.513	3.467	0.69	$100\alpha'$-21R(w)	0.410	–
NB2	1.54	1.54	0.513	3.445	0.51	$100\alpha'$-21R(w)	0.422	–
NB3	1.54	1.44	0.513	3.427	0.56	$100\alpha'$-21R(w)	0.421	–
NC1	0.86	1.73	0.287	3.337	0.73	$58.6\alpha'$-$38.3\beta'$-21R(w)	0.316	0.57
NC2	1.0	1.62	0.333	3.363	0.57	$84.7\alpha'$-$13.2\beta'$-21R(w)	0.309	0.52
NC3	1.16	1.54	0.387	3.386	0.64	$100\alpha'$-21R(w)	0.339	–
ND1	0.7	1.8	0.233	3.315	0.70	$37.3\alpha'$-$58.7\beta'$-21R(w)	0.330	0.58

12H, $SiAl_5O_2N_5$; 21R, $SiAl_6O_2N_6$; YAG, $Al_5Y_3O_{12}$; mellilite, M, $Y_2Si_3N_4O_3$.

[a] Contain 1.3% additional Y_2O_3.

[b] Weight loss during sintering.

3. Results and discussion

3.1. XRD measurement and structure determination

The calculated data for the α-SiAlON structure are given in Table 2 and illustrated in Fig. 2. XRD analysis of samples YA 2 and NA 2 was repeated three times. It was found that the deviations in the atomic parameters were lower than the accuracy of the structural refinement. The obtained values are given in Table 2. For the estimation of the accuracy of the occupation factor for Nd and Y which corresponds to $x/2$ in the SiAlON composition $R_xSi_{12-(m+n)}Al_{m+n}O_nN_{16-n}$ (R = Y, Nd) was fixed at values differ from the determined before values and the refinement of the structure was carried out again with this fixed values. The deviations of the observed R_{phase} values from that determined without fixing the occupation factor of the R cation are given in Fig. 3. The measurements show that the R_{phase} values depend strongly on the occupation factor of the rare earth cation and have a strong minimum at the real x value. This confirms that the method can successfully be used for calculation of the x and m values in the α-SiAlONs. The mean determined error of the x values is less than ±0.03. The same procedure was carried out for the ratios of Al/Si and O/N. The variation of the determined R_{phase} values is only 0.1–0.2% for Al/Si ratios between 0 and 0.6. Therefore the method cannot be used to determine the n value in α-SiAlON.

The determined x values show no systematic change due to the changing of the Al/Si and O/N ratios, indicating the low sensitivity of the method for determining the Al/Si and O/N ratios. The determined x values for samples which are situated inside the homogeneity region of the α-SiAlON show a good agreement between measured and calculated values (Table 1,

Table 2
Results of the refinement of the structures of different α-SiAlON compositions by the Rietveld method

Parameter		NA 2	YB 5	YA 2	YA 3
		Nd-α-SiAlON	Y-α-SiAlON	Y-α-SiAlON	Y-α-SiAlON
Space group		159	159	159	159
a, *nm*		0.78137	0.78381	0.78106	0.78158
c, *nm*		0.56935	0.57183	0.56935	0.56990
R (Nd, Y)	*z*	0.695	0.693	0.691	0.694
(position 2b)	B, nm^2	0.0085	0.00508	0.00403	0.00651
	occupation factor	0.17	0.296	0.170	0.195
(Si, Al)1 (position 6c)	*x*	0.511	0.508	0.511	0.511
	y	0.428	0.427	0.427	0.427
	z	0.660	0.660	0.657	0.660
	B, nm^2	0.010	0.009	0.012	0.011
(Si, Al)2					
(position 6c)	*x*	0.170	0.169	0.170	0.170
	y	0.917	0.917	0.916	0.916
	z	0.4498	0.452	0.448	0.451
	B, nm^2	0.0045	0.0072	0.0049	0.0055
(N, O)1 (position 6c)	*x*	0.612	0.607	0.611	0.611
	y	0.956	0.947	0.952	0.952
	z	0.443	0.439	0.446	0.448
	B, nm^2	0.0076	0	0.0089	0.006
(N, O)2 (position 6c)	*x*	0.320	0.321	0.322	0.320
	y	0.00634	0.00663	0.00691	0.00555
	z	0.707	0.704	0.704	0.708
	B, nm^2	0.00628	0.00876	0.00858	0.00815
(N, O)3 (position 2b)	*z*	0.107	0.0989	0.103	0.104
	B, nm^2	0	0	0.006	0.006
(N, O)4 (position 2a)	*z*	0.452	0.449	0.437	0.432
	B, nm^2	0	0.01	0.0066	0.0030
x determined from the occupation factor		0.34	0.592	0.34	0.39
x determined from the starting composition		0.333	0.6	0.333	0.386
n value using the lattice parameter and Eq. (1)		1.15	1.09	1.07	1.07

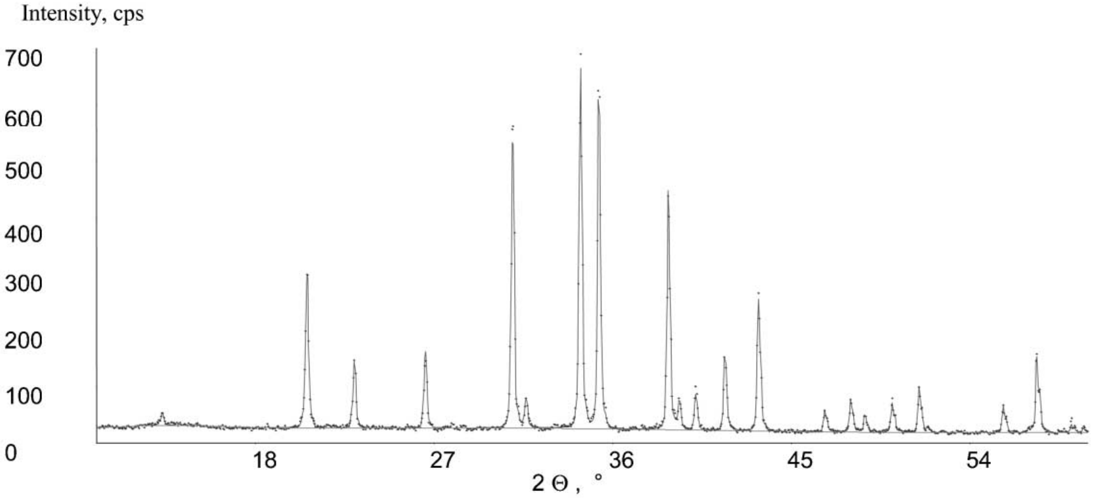

Fig. 2. Measured and calculated XRD patterns for composition YC 2.

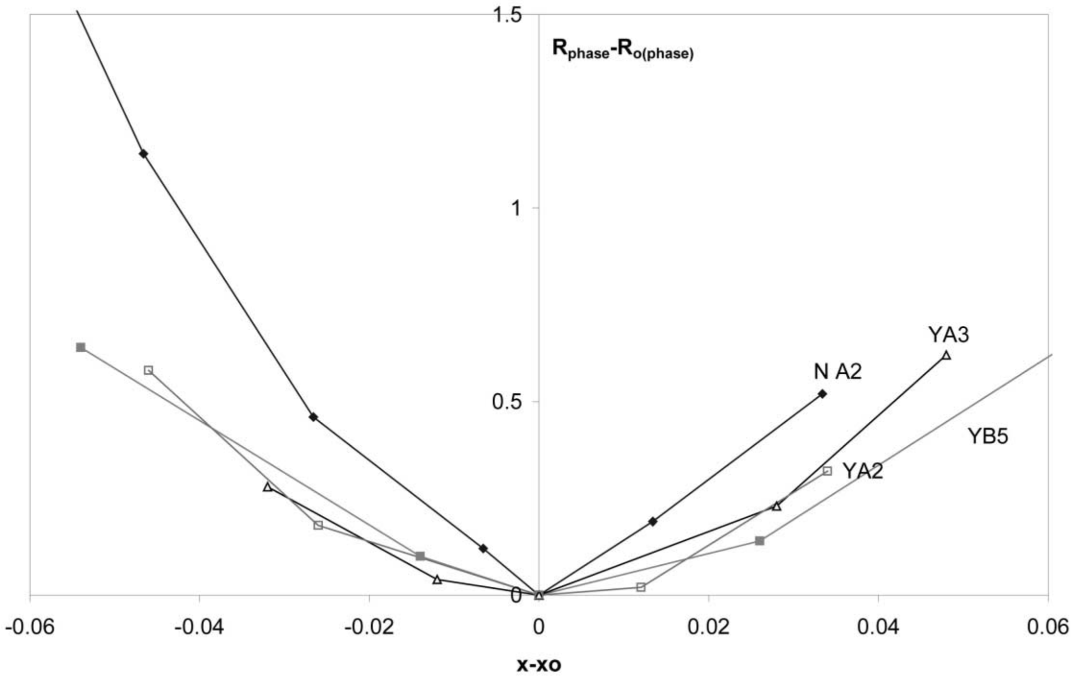

Fig. 3. Change in the R_{phase} values with deviation of the×value from the calculated real composition (corresponding to the minimum R_{phase} value) for different samples [x value for NA2: x (NA2) = 0.33; x (YA2) = 0.36; x (YA3) = 0.40; x (YB5) = 0.595].

Fig. 4). This is an additional evidence for the validity of the determined values. The x values do not systematically depend on the calculated B values.

In the literature exist different equations connecting the n- and m-values of the α-SiAlON composition with the lattice parameter.[6,23] These equations

$$a = 7.752 + 0.036m + 0.02n;$$
$$c = 5.620 + 0.031m + 0.04n \qquad (1)^{23}$$

and

$$a = 7.752 + 0.045m + 0.009n;$$
$$c = 5.620 + 0.048m + 0.009n \qquad (2)^{6}$$

gives strong differences for the dependencies on the n value. The equation after[23] seems to be more accurate for the determination of the n values. Nevertheless, taking into account the accuracy of the parameters of the equation (≈10%[24]) and the error of the determination of m (≈5%) the n values calculated using this equation can diver by 10 to 20% from the real data.[24] The lattice parameter determined by the Rietveld method has not the needed accuracy, therefore, additionally the lattice parameter were determined with Si as an internal standard. The calculated n values based on the so determined lattice parameter gives slightly lower values even for the compositions which show only α-SiAlON. This can be connected on the one hand with the shift in the oxygen content due to weight losses during sintering on the other hand with the uncertainty of the equations explained above. Therefore the n values calculated in this manner must be used with care.

Different mixtures of α-SiAlON and β-Si_3N_4 or α-Si_3N_4 were analysed to investigate the influence of the content of these phases on the x values of the α-SiAlON. The results shown in Figs. 5 and 6 show that a β-Si_3N_4 or α-Si_3N_4 content of up to 50% has no influence on the measured x values of the α-SiAlON phase. Nevertheless, the results clearly show that this method can also be used successfully for the determination of mixtures of α-SiAlON with higher amounts of α- or ß-Si_3N_4. The influence of the 21 R or 12 H polytyps on the calculation of the composition of the α-SiAlON was not investigated in detail. In the samples these phases had

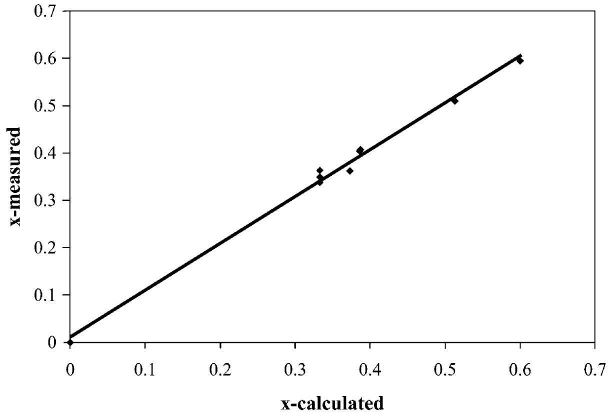

Fig. 4. Relationship between the x value determined by the XRD method and that calculated by the weight fractions of the components for pure α-SiAlON samples.

normally a very low concentration (<10%). Additionally the most intensive peaks of these phases can be well separated from the α-SiAlON peaks. Therefore an increased error of the determination of the α-SiAlON composition is not likely.

For example values of $R_{\mathrm{phase}} = 5.8\%$ and $R_{\mathrm{wp}} = 9.8\%$ were obtained for the sample YB4 containing the 21R polytype. Whereas the values for the sample YB3 with only α-SiAlON were $R_{\mathrm{phase}} = 5.7\%$ and $R_{\mathrm{wp}} = 8.0\%$.

3.2. Use of the method for determining the homogeneity region of Y-α-SiAlON

The samples, whose compositions are given in Table 1 and Fig. 1, were analysed to confirm the homogeneity region of α-SiAlON. The samples were chosen in the region where the limits of the homogeneity area were expected (Fig. 1). Different groups of samples were chosen. These groups were YA, the minimum x value when the n value is 1.26; YB, with high m and n values; and YC, with intermediate m and n values and some additional compositions with very low n and different m values. The XRD results for samples YA 2 and YA 3 showed α-SiAlON as the only crystalline phase, whereas for sample YA 1 they reveal α-SiAlON and trace amounts of β-SiAlON as crystalline phases. The minimum x value was measured as 0.30 ± 0.01. This corresponds to an m value of 0.9, which is slightly lower than the value from the literature.[16]

The results obtained for sample EY 5, which had lower m and n values than series A (see Fig. 1), showed a low x value ($x = 0.24$). Similar results were found for

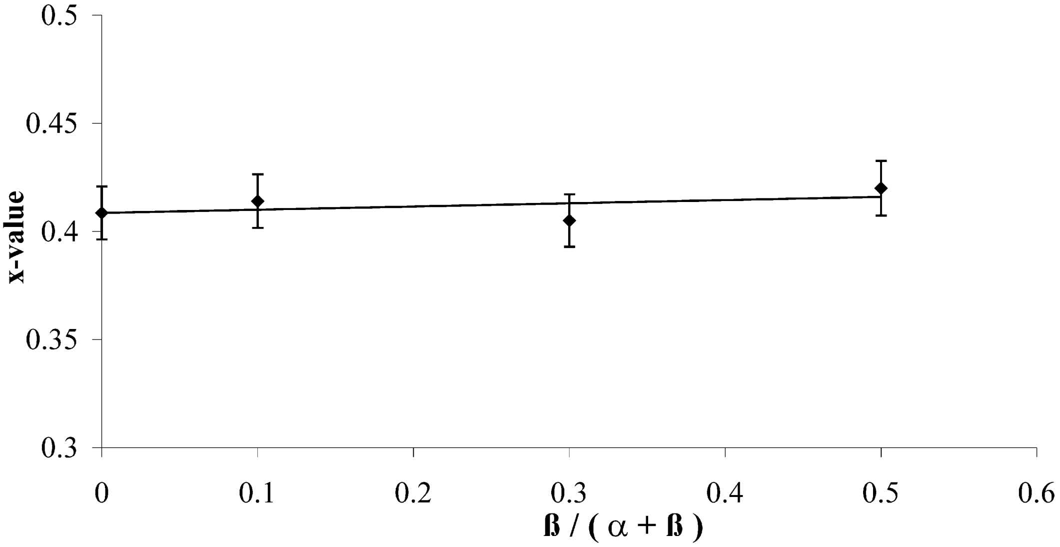

Fig. 5. Change in the measured x values with changing ratio of β-Si_3N_4/(β-Si_3N_4 + α-SiAlON) (mixture of sample EY 3a and β-Si_3N_4 powder).

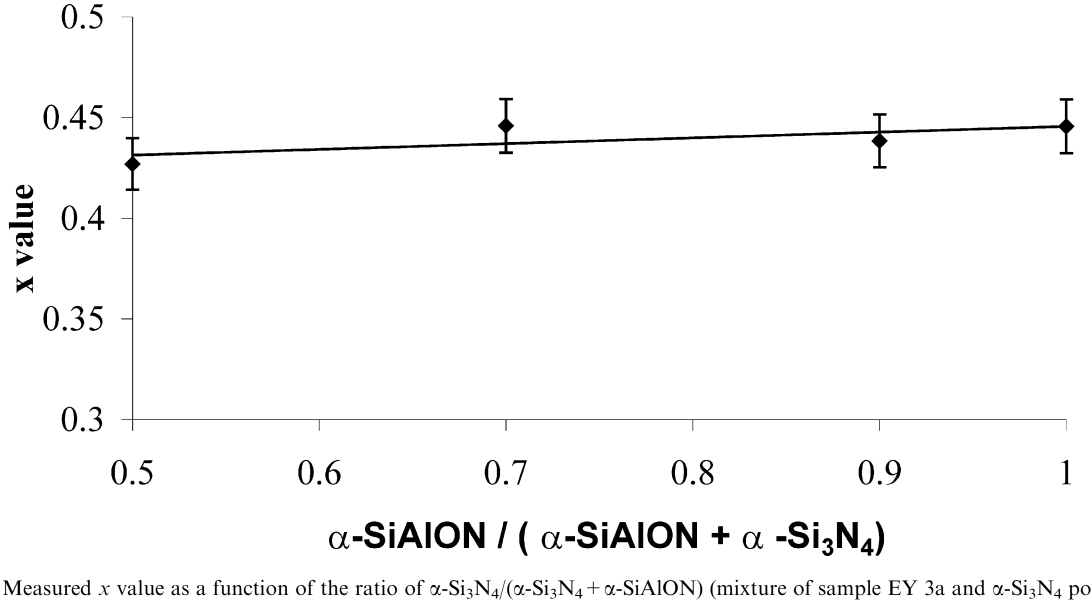

Fig. 6. Measured x value as a function of the ratio of α-Si_3N_4/(α-Si_3N_4 + α-SiAlON) (mixture of sample EY 3a and α-Si_3N_4 powder).

other compositions.[20] This corresponds to an m value of 0.72, which is much lower than the lowest value expected from the literature and also from the data in series A. The measured z value for the β-SiAlON in this sample was 0.35, corresponding to $n = 0.70$. Using the phase rules it was possible to obtain the n value of the corresponding composition of the α-SiAlON, which was calculated as 0.76–0.8 from these data. The composition and the resulting boundary are shown in Fig. 7. α-SiAlON (61%) and β-SiAlON 39% were calculated using the determined m, n and z values. These values are in good agreement with the results obtained using XRD.

The lower x value can be explained in terms of the stability area for α-SiAlON, which is larger at higher temperatures than that observed in the literature.[16,22] The x values of YA 1 and YC 1 and YD1 confirmed this approach. Although sample YC 1 contains β-SiAlON after sintering, the observed x value corresponds to that in the starting composition. In addition, the z value of the formed β-SiAlONs and the composition of the α-SiAlON phase have nearly the same Al/Si ratio as that found in the starting composition. If it is assumed that at high temperatures, compositions YA 1 and YC 1 consist of only α-SiAlON, with the x value corresponding to that in the starting composition, then during cooling only small grains should decompose or the bigger grains should dissolve at the surface only and form the observed β-SiAlON. Due to the low diffusion coefficient inside the grains, the x value will remain the same as it was at high temperatures. That is why we found a discrepancy between the x value and the phase composition in the samples near the solubility limit. Evidence that a higher stability area of α-SiAlON ($m < 1$) can exist was found by Rosenflanz.[16] using microprobe analysis. For the Y-α-SiAlON, an x value of 0.29 ($m = 0.87$) for samples heat treated at 1950 °C was found.

In samples in which no stable liquid phase and only a very small amount of transient liquid exist during heat treatment (low n and m values), the formation of the equilibrium phases can be difficult, due to the low diffusion coefficients of the solids. The low density of these samples is also an indication for this. This can be the reason for the high x values observed in sample EY 4. Investigations of the kinetics have shown that at intermediate temperatures α-SiAlONs with x values that are higher than equilibrium x values could be formed.[20] A second possible reason can be that at low temperatures the stability region of the α-SiAlON is much lower than at high temperatures and the α-SiAlON compositions formed at low temperatures correspond to the boundary of the stability area.

Further experiments involving long-term isothermal treatment of samples in the two-phase region at different temperatures as well as investigations of the kinetics of α-SiAlON formation are necessary to confirm one of these two possibilities.

According to the phase composition analysis, the stability area of the Y-α-SiAlON can be drawn as shown in Fig. 7. The minimum m and maximum n values of the α-SiAlON which are in equilibrium with β-SiAlON were $m = 0.93 \pm 0.03$ and n 1.6–1.7 taking into account the starting composition of the samples and the phases observed. The calculated n values from the Eq. (1) shows lower values. Therefore further investigations are necessary. The microprobe analysis by Rosenflanz showed similar results, with $m = 0.87$ and $n = 1.61$ for samples sintered at 1950 °C.

At higher m values, the oxygen and Al solubilities are lower. For the samples in the series YB with $m = 1.54$, the maximum n value must be close to 1.54. When the compositions shift from the solubility limit of α-SiAlON, the observed x values decrease. This can be explained by the observed secondary 21R phase and the

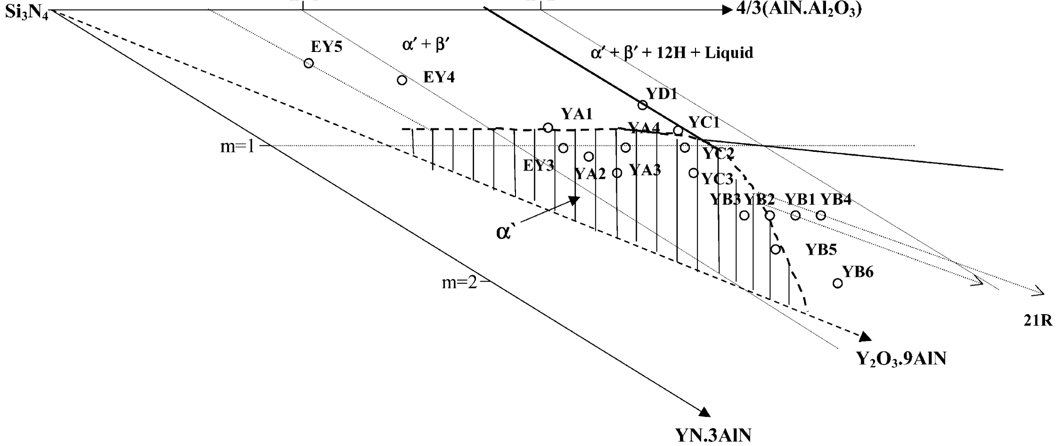

Fig. 7. Homogeneity area of Nd-α-SiAlON at 1825 °C.

lever rule (Fig. 6). These data are in agreement with the phase diagram obtained by Rosenflanz and Chen.

The maximum solubility at the line Si_3N_4–$Y_2O_3{\cdot}9AlN$, shown in Fig. 7, was taken from Rosenflanz and Chen.[16]

3.3. Use of the method for determining the homogeneity region of Nd-α-SiAlON

The observed values for the Nd-α-SiAlON compositions are given in Table 1. The results are significantly different from those of the Y system.

Samples NA 1, NC 1 and NC 2 contain much more β-SiAlON than the corresponding Y samples. The maximum z value for β-SiAlON in equilibrium with the Nd-α-SiAlON is much lower than for the Y sample (0.57 for NC 1 and 0.85 for YC 1). In addition, the NC 1 sample contains the 21R phase, which was not observed in the corresponding Y-containing sample.

The measured x value for the Nd-α-SiAlON, which was 0.32 ± 0.01 for NC 1, is slightly higher than for the YC 1, which was 0.29. These data suggest that this composition is really outside the homogeneity area of the α-SiAlON, even at high temperatures. These results are in good agreement with the results of Rosenflanz[16] (compare Figs. 1 and 8). Similar differences can be found for the samples in series B. All of the samples contain beside α-SiAlON the 21R phase. The x values in the samples of the series NB are significantly lower than the values corresponding to the overall composition of the samples. This suggests that these samples are really outside of the stability area. The measured x values in the neodymium system are lower than in the samples with the same composition in the yttrium-containing system. This indicates that the solubility limit of the α-SiAlON in this direction is much lower than in the corresponding yttrium-containing system. Using the observed phase content and the lever rule, the maximum n value can be estimated as 1.0.

The maximum n value which is in equilibrium with the β-SiAlON ($z = 0.58 \pm 0.03$) could be estimated as 1.1–1.5; the corresponding m value was 0.96 ± 0.05.

This is also in good agreement with the values obtained by Rosenflanz for α-SiAlON, $n = 1.1$ and $m = 0.97$ after sintering at 1950 °C.[16] The resulting possible homogeneity area for Nd-α-SiAlON system is given in Fig. 8.

4. Conclusions

Methodological development of the interpretation of X-ray diffraction data results in an improvement in the knowledge about stability regions and phase relationships in various α-SiAlON systems. The diffraction patterns of the α-SiAlON with different R cations were analysed by the Rietveld method. It was shown that by using this method, the amount of the cation R incorporated in the α-SiAlON structure can be calculated with a high accuracy. The Rietveld method gives reliable data for the content of the rare earth cations, i.e. the m value, but cannot be used for the determination of the Al or O contents of the α-SiAlONs, i.e the n value. The method can also be used in mixtures of α-SiAlON and α- or β-Si_3N_4.

This method was also applied to determine the phase relationships in R_2O_3–Si_3N_4–AlN–Al_2O_3, where R = Y or Nd. For the Y-α-SiAlON region at a high temperature (1825 °C), the minimum m value was determined as 0.72 ± 0.03. Hence the stability range of Y-α-SiAlON at higher temperatures is much larger than that shown in the literature. According to the Rietveld method results, the minimum m and maximum n values for Y-α-SiAlON in equilibrium with β-SiAlON are $m = 0.93 \pm 0.3$ and

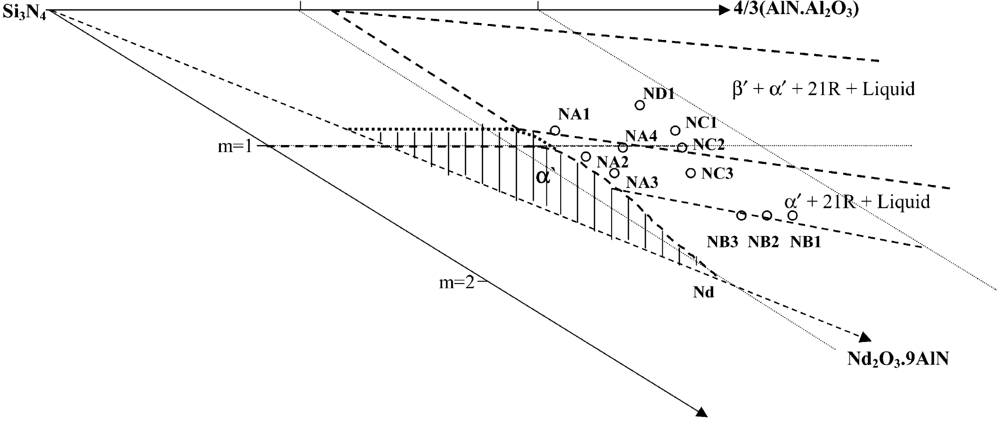

Fig. 8. Homogeneity area of Y-α-SiAlON at 1825 °C.

$n = 1.6$–1.7. For the Nd-α-SiAlON system, it was shown that the solubility limits for Nd-α-SiAlON are much lower than for Y-α-SiAlON. The maximum n value which is in equilibrium with β-SiAlON ($z = 0.58 \pm 0.03$) could be estimated as 1.1–1.5; the corresponding m value was 0.93 ± 0.05.

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