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Investigation of preparation of Ca-α-Sialon powders via artificial neural networks analysis

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Abstract

Artificial neural networks (ANNs) have been applied in target forecasting since their promising performances in the areas of control and pattern recognition. This paper aims to develop a methodological approach for estimating the relative contents of Ca-α-Sialon by using ANNs. Ca-α-Sialon powders were prepared by the reaction nitridation method using Si, Al, $CaCO_3$ and Al_2O_3 as main starting materials. The effects of processing parameters such as reaction temperature, contents of Y_2O_3, TiO_2, and Fe_2O_3 additives, and α-Si_3N_4 seeds on the synthesis of Ca-α-Sialon were investigated in detail. The results showed that Ca-α-Sialon can be prepared after 8–20 h at 1550 °C, and that the addition of Y_2O_3 is more effective for promoting the Ca-α-Sialon formation than TiO_2 and Fe_2O_3. The addition of α-Si_3N_4 seeds plays a positive role in promoting the Ca-α-Sialon formation. A back propagation ANN was used to establish a model to predict the reaction extents (relative contents of Ca-α-Sialon) corresponding to various processing conditions. The results from the reliability verification tests matched well with those predicted by the ANNs model, which proves the effectiveness of the proposed methodology.

Keywords: Ca-α-Sialon; Powders; Reaction nitridation; Back propagation; Artificial neural networks

1. Introduction

Artificial neural networks (ANNs) models are built on networks of processing units called neurons that are arranged in layers and connected to one another by restricted links with associated weights. Recently, the ANNs technique has found widespread applications in establishing linear and non-linear relationships between various processing parameters and experimental results because of its unique advantages, including: (1) noise-insensitivity provides an accurate prediction in the presence of uncertain data and measurement errors, (2) learning and adaptivity allow the system to update (modify) its internal structure in response to changing environment, (3) generalization enables application of the model to unlearned data, and (4) high parallelism implies fast processing and hardware failure-tolerance [1,2]. So far, ANNs have been successfully used in many different fields for modeling and predicting the relationship between the input parameters and output target.

Sialons are Si_3N_4-based solid solutions, where silicon and nitrogen are partially replaced by aluminum and oxygen [3]. They exist in two polymorphic forms, α (having a formula of $Me_mSi_{12-(m+n)}Al_{(m+n)}O_nN_{16-n}$, where $m+n$(Si–N) bonds are replaced by m(Al–N) and n(Al–O), and M is a metal ion typically Li, Mg, Ca, Y and Ln (where $Z \geq 58$) and β (with a formula of $Si_{6-z}Al_zO_zN_{8-z}$, where z ranges from 0 to 4.3.), with the same hexagonal crystals but different stacking sequences along c axes. Sialon based materials are ideal for the development of some high-technology ceramics used especially in metallurgical, chemical, and mechanical industries owing to their outstanding performance such as good thermal shock resistance, high hot strength and nonwetability to slag and molten steel. Previously, we have reported a series of investigations on the preparation of Sialon and Sialon-based composites by a reaction nitridation or a carbothermal reduction nitridation process [4–5]. Sialon based materials exhibited

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some fascinating high temperature mechanical properties and promising potential applications for high-temperature ceramics [4–9].

Conventionally, Sialon is produced by sintering of a Si_3N_4–AlN–M_xO_y powder mixture at a high temperature via a liquid sintering process [3]. This process usually requires high sintering temperature (1700–2000 °C) and highly pure nitride powders as starting materials [10], which suffers from several drawbacks such as high cost, energy consumption, rigid agglomeration of as-synthesized powder (a post-synthesis grinding step is needed to pulverize the products), etc. Thus, how to synthesize α-Sialon at a relatively low cost under a low temperature is of great interest.

In this work, Ca-α-Sialon powders were prepared by the reaction nitridation method using Si, Al, $CaCO_3$ and Al_2O_3 as main starting materials. The effects of reaction temperature, contents of Y_2O_3, TiO_2, and Fe_2O_3 additives, and α-Si_3N_4 seeds on the Ca-α-Sialon synthesis were also investigated in detail. The preparation of Ca-α-Sialon varies significantly with the processing parameters, e.g. the reaction temperature and soaking time, the contents of Y_2O_3, TiO_2, and Fe_2O_3 additives, and α-Si_3N_4 crystal seeds. Since these parameters vary throughout a rather wide range, a detailed investigation on the preparation of Ca-α-Sialon by using nitridation reaction method is a time-consuming and cost-demanding task. At this point an approach based on ANNs can be useful, since ANNs can model relationships between processing parameters and output targets without requirement of knowledge of the exact formula among them. Thus, a back propagation (BP) ANNs method was used to establish a model between the relative contents (RCS) of Ca-α-Sialon in final products and the corresponding processing parameters. The ANNs were initially trained by using the BP and an accuracy of approximately 99.999% was achieved, and in the second step, the trained ANNs model was used to predict the RCS of Ca-α-Sialon in several samples. The results from the reliability verification tests showed that the calculated results matched well with the tested ones.

2. Experimental procedure

Raw materials used to prepare Ca-α-Sialon ($CaSi_9Al_3ON_{15}$) included α-Al_2O_3 ($\geq$ 98 wt%, $d_{50} \approx 5$ μm), silicon ($\geq$ 98 wt%, $d_{50} \approx 43$ μm), aluminum ($\geq$ 98 wt%, $d_{50} \approx 64$ μm), $CaCO_3$ (analytical reagent grade), α-Si_3N_4, Y_2O_3 (analytical reagent grade), TiO_2 (analytical reagent grade), and Fe_2O_3 (analytical reagent grade) powders. The preparation reaction of Ca-α-Sialon by the reaction nitridation method is as follows,

$$Al_2O_3(s)+9Si(s)+Al(s)+CaO(s)+15/2N_2(g) = CaSi_9Al_3ON_{15}(s)+3/2O_2(g) \quad (1)$$

α-Si_3N_4 seeds, and Y_2O_3, TiO_2, and Fe_2O_3 additives were added in the starting material batches to promote the formation of Ca-α-Sialon, and the corresponding four series of samples were designated as SS, YS, TS and FS, respectively. The raw materials were mixed in the requisite ratios in a ball mill for 1 h and then fired in a nitriding furnace at 1550 °C for 8, 12, and 20 h, respectively, in a flowing N_2 atmosphere (N_2 purity = 99.9999 wt%). The detailed compositions and preparation conditions are shown in Table 1.

Phases and crystalline structure of α-Sialon in the fired samples were identified by using an X-ray diffractometer (XRD) (Philips X'Pert PRO diffractometer). Patterns were recorded between 10° and 80° (2θ) at 40 kV and 40 mA using Ni filtered Cu Kα radiation (λ = 0.154187 nm). And the relative contents of α-Sialon and β-Sialon were evaluated based on the α-(102), α-(210), β-(101) and β-(210) peak heights according to the following formula proposed by Gazzar and Messier [11],

$$\alpha(\%) = [I_{\alpha(102)} + I_{\alpha(210)}]/[I_{\alpha(102)} + I_{\alpha(210)} + I_{\beta(101)} + I_{\beta(210)}] \quad (2)$$

This equation is applicable for pure α- and β-Si_3N_4 and also assumed to be applicable for Sialon materials because of the

Table 1
Starting compositions of the specimens.

Sample	Composition (wt%)					Remarks
	Al+Si+$CaCO_3$+Al_2O_3	Y_2O_3	TiO_2	Fe_2O_3	α-Si_3N_4	
YS-0	100	0	0	0	0	Effects of Y_2O_3 additive
YS-2	100	2	0	0	0	
YS-3	100	3	0	0	0	
YS-4	100	4	0	0	0	
TS-0	100	0	0	0	0	Effects of TiO_2 additive
TS-2	100	0	2	0	0	
TS-3	100	0	3	0	0	
TS-4	100	0	4	0	0	
FS-0	100	0	0	0	0	Effects of Fe_2O_3 additive
FS-2	100	0	0	2	0	
FS-3	100	0	0	3	0	
FS-4	100	0	0	4	0	
SS-0	100	3	0	0	0	Effects of α-Si_3N_4 seeds
SS-1	100	3	0	0	1	
SS-3	100	3	0	0	3	
SS-5	100	3	0	0	5	

similarity in their crystal structures. To calculate the α-Sialon content, only α and β-Sialon phases were taken into account, and minor amounts of amorphous phases and secondary phases were neglected. Microstructures of the samples, after carbon coating, were examined by using a scanning electron microscopy (Model, JSM-5610LV, JEOL, JAPAN, 20 kV).

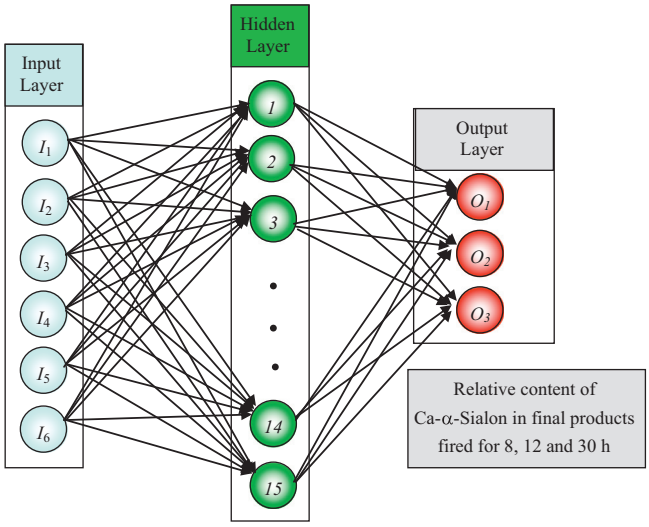

Fig. 1. Illustration of a three-layered neural network with 6 input layers, 15 hidden layers and 3 output layers.

3. Artificial neural networks (ANNs) analysis

The back propagation (BP) learning algorithm was used in this study. The networks have an input layer, a hidden layer and an output layer, as schematically shown in Fig.1, which include 6, 15 and 3 neurons respectively. The 6 input neurons in the input layer (input vector ($I_1 \ldots I_6$)), correspond to the amounts of Si and Al starting materials, the contents of Y_2O_3, Fe_2O_3 and TiO_2 additives, and the amounts of α-Si_3N_4 seeds. The 3 object output neurons in the output layer correspond to RCS of Ca-α-Siaon in final products annealed at 1550 °C for 8, 12, and 20 h, respectively.

To train and test the neural networks, the experimental data are divided into two sets: (1) The training set (26 cases, specimens 1#–26# in Table 2), which is used for training until the network has learned the relationship between the inputs and the output; (2) the test set (3 cases, specimens 27#–29# in Table 2) which verifies the generalization ability of the ANNs as an independent data set. Input–output pairs are presented to the networks and weights are adjusted to minimize the errors between the networks outputs and actual values. Once training

Table 2
Relative contents of Ca-α-Siaon in final products nitrided at 1550 °C for various hours.

Sample	Al_2O_3	CaO	Si	Al	Y_2O_3	Fe_2O_3	TiO_2	α-Si_3N_4	RCS (%)		
									8 h	12 h	20 h
1#	22.3	16.5	63.5	6.2	3	0	0	0	22.3	17.5	0
2#	22.3	16.5	57.7	6.8	3	0	0	0	30.9	21.6	0
3#	22.3	16.5	63.5	6.8	3	0	0	0	16.9	22.9	0
4#	22.3	16.5	57.7	6.2	3	0	0	1	22.6	32.2	24.5
5#	22.3	16.5	57.7	6.2	3	0	0	3	24.8	34.9	23.9
6#	22.3	16.5	57.7	6.2	3	0	0	5	29.8	42.3	40.1
7#	22.3	16.5	57.7	6.2	0	3	0	0	21.4	27.3	18.3
8#	22.3	16.5	63.5	6.2	0	3	0	0	38.7	42.3	0
9#	22.3	16.5	57.7	6.8	0	3	0	0	29.4	17.1	0
10#	22.3	16.5	63.5	6.8	0	3	0	0	30.4	17.1	0
11#	22.3	16.5	57.7	6.2	0	3	0	1	22.1	31.6	20.4
12#	22.3	16.5	57.7	6.2	0	3	0	3	22.3	34.8	29.6
13#	22.3	16.5	57.7	6.2	0	3	0	5	25.6	40.1	31.5
14#	22.3	16.5	57.7	6.2	0	0	3	0	26.5	60.3	23.4
15#	22.3	16.5	63.5	6.2	0	0	3	0	0	0	0
16#	22.3	16.5	57.7	6.8	0	0	3	0	16.4	25.9	17.6
17#	22.3	16.5	63.5	6.8	0	0	3	0	0	0	0
18#	22.3	16.5	57.7	6.2	0	0	3	1	0	10.5	11.2
19#	22.3	16.5	57.7	6.2	0	0	3	3	15.6	21.8	20.9
20#	22.3	16.5	57.7	6.2	0	0	3	5	17.8	28.2	25.6
21#	22.3	16.5	57.7	6.2	2	0	0	0	15.7	18.2	17.6
22#	22.3	16.5	57.7	6.2	4	0	0	0	35.8	65.6	40
23#	22.3	16.5	57.7	6.2	0	2	0	0	0	13.5	0
24#	22.3	16.5	57.7	6.2	0	4	0	0	18.2	20	19.5
25#	22.3	16.5	57.7	6.2	0	0	2	0	13.5	12.3	15.2
26#	22.3	16.5	57.7	6.2	0	0	4	0	14.6	15.4	12.8
27#	22.3	16.5	57.7	6.2	3	0	0	0	18.8	30	21.7
28#	22.3	16.5	63.5	6.2	0	3	0	3	17.4	19.3	15.6
29#	22.3	16.5	63.5	6.8	0	0	3	3	35.2	64.7	26.1

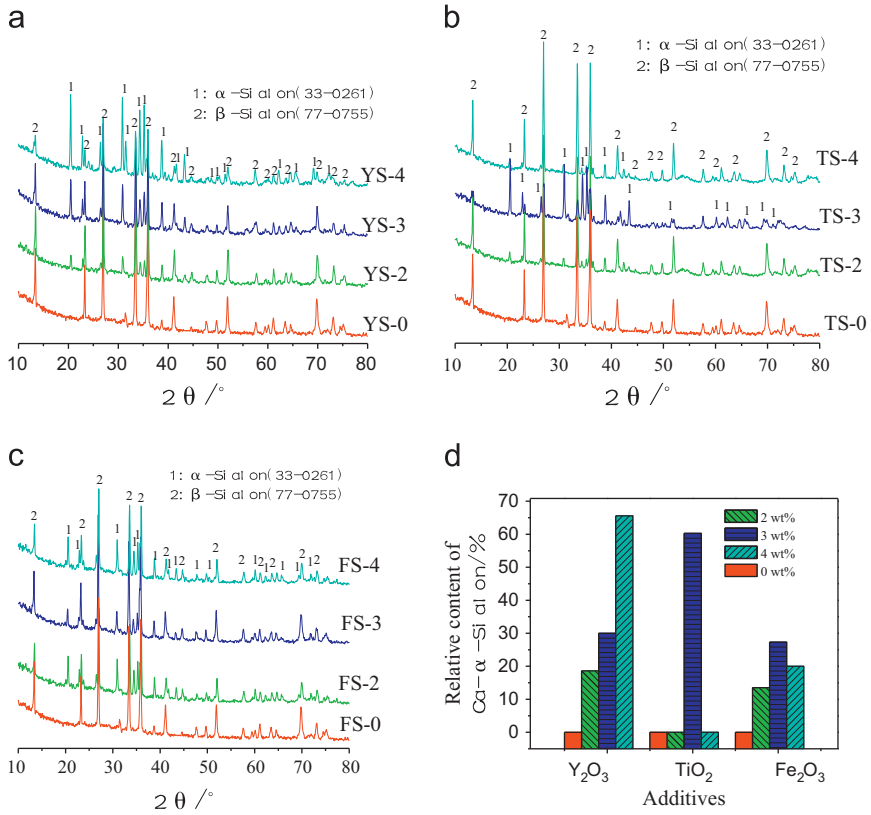

Fig. 2. XRD patterns and the RCS of Ca-α-Sialon in final products nitrided at 1550 °C for 12 h with Y_2O_3, TiO_2 and Fe_2O_3 as additives. (a) XRD of YS series samples, (b) XRD of TS series samples, (c) XRD of FS series samples and (d) Relative content of Ca-α-Sialon in YS, TS, and FS series samples.

is completed, predictions from a new set of data may be done using the already trained networks. The mathematical background, the procedures for training and testing the ANNs and account of its history can be found in Ref. [12].

The Neural Networks Toolbox of MATLAB 6.5 was used to form the ANNs. The tan-sigmoid transfer function was used for the input layer and the hidden layer, and log-sigmoid transfer function used for the output layer. The back propagation networks training function updates weight and bias values according to the Levenberg–Marquardt optimization [13]. Mean squared error (MSE) determining the networks performance is calculated according to

$$\mathrm{MSE} = \frac{1}{N}\sum_{i=1}^{N}(y_i - y_k)^2 \quad (3)$$

where y_i is the predicted value of the i^{th} item, y_k is the target value of the i^{th} item and N is the number of training cycle.

4. Results and discussion

4.1. Effects of Y_2O_3, TiO_2 and Fe_2O_3 additions on the Ca-α-Siaon formation

XRD patterns of YS (Fig. 2a), TS (Fig. 2b) and FS (Fig. 2c) series samples nitrided at 1550 °C for 12 h are shown in Fig. 2. Pure Ca-α-Sialon powders were hard to be synthesized via the present route, and β-Sialon phases were found in all the samples (Fig. 2a–c). Furthermore, no α-Sialon was detected

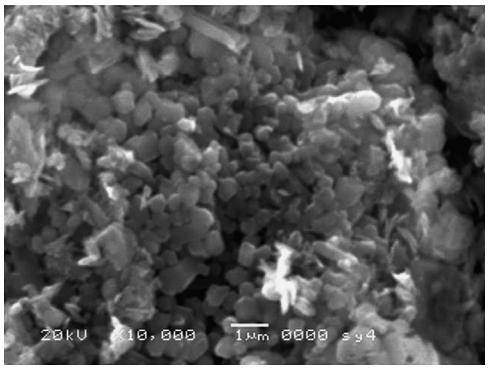

Fig. 3. SEM micrograph of YS-4 specimen nitrided at 1550 °C for 12 h.

in the samples without additives, whereas its formation became much more evident in the samples containing Y_2O_3, TiO_2 and Fe_2O_3 additives, indicating that the use of these additives is very important for the preparation of Ca-α-Sialon via the present reaction nitridation method. Among these additives, Y_2O_3 shows the best and TiO_2 the second best promoting-effect in the preparation of Ca-α-Sialon, whereas Fe_2O_3 shows the least (Fig. 2d). The optimal amounts of Y_2O_3, TiO_2 and Fe_2O_3, in terms of Fig. 2d, are 4 wt%, 3 wt% and 3 wt%, respectively. Among all the nitrided samples, the highest relative content of α-Sialon is about 66% in YS-4.

The improvements in the yield of Ca-α-Sialon due to Y_2O_3 could be ascribed to two main factors. First, the addition of Y_2O_3 could lead to a lower temperature for liquid formation in

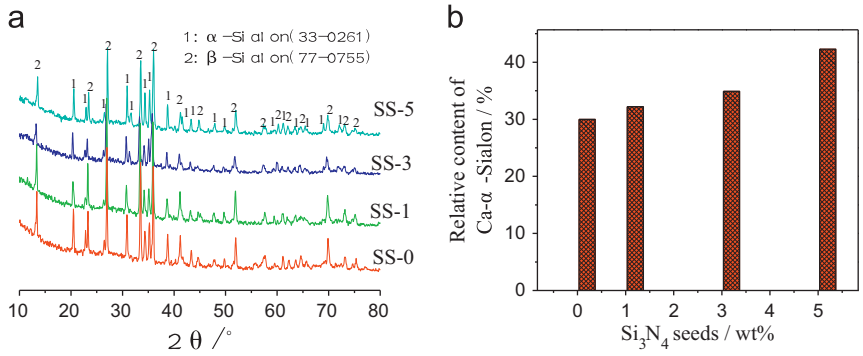

Fig. 4. XRD patterns and the RCS of Ca-α-Sialon in final products nitrided at 1550 °C for 12 h with α-Si_3N_4 as crystal seeds. (a) XRD and (b) Relative content of Ca-α-Sialon.

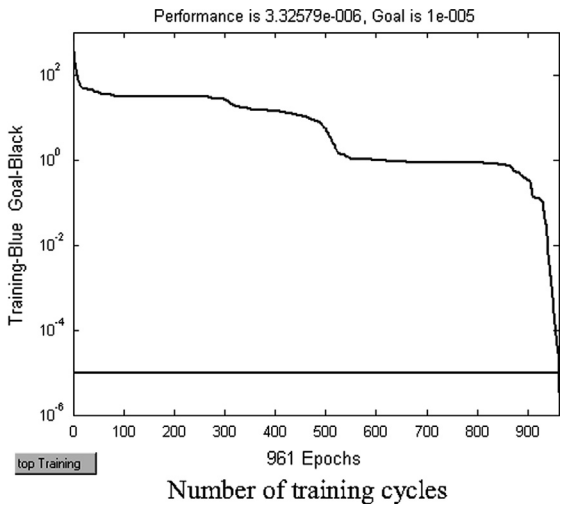

Fig. 5. Calculation performance of present ANNs model.

the system, as well as an increased amount of liquid phase, thus promoting the formation of α-Sialon. Second, the added Y_2O_3 could enter into the lattice of α-Sialon, stabilizing the latter's structure, which is not the case with the additions of TiO_2 and Fe_2O_3. It was reported that Fe_2O_3 can only facilitate the Si nitridation at relatively low temperatures [14]. As for the second best additive, TiO_2, it can dissolve in Al_2O_3 to form a TiO_2–Al_2O_3 solid-solution which results in the distortion of the Al_2O_3 lattices, increasing the reactivity of the starting Al_2O_3 powder [15], and thus promoting the Ca-α-Sialon formation. An SEM image of prepared YS-4 sample is shown in Fig. 3, revealing that the microstructure is mainly composed of 1–2 μm equiaxed grains and elongated grains with an aspect ratio of about 3–10.

4.2. Effects of Si_3N_4 seeds on the Ca-α-Siaon formation

XRD patterns and relative contents of Ca-α-Sialon in α-Si_3N_4 seeded samples after 12 h nitridation at 1550 °C are shown in Fig. 4. It can be seen that addition of α-Si_3N_4 crystal seeds can also promote the Ca-α-Sialon formation. As α-Sialon is a solid-solution of α-Si_3N_4 and Al_2O_3, it can be considered

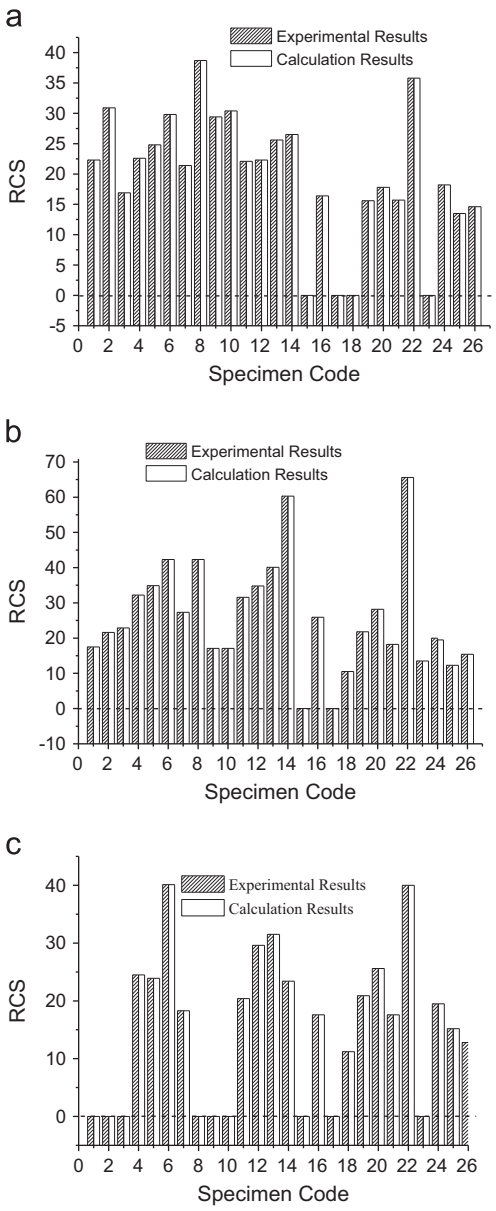

Fig. 6. Correlation between experimental RCS of Ca-α-Sialon in final products and calculated RCS values by ANNs model. (a) 1550°C/8 h, (b) 1550°C/12 h and (c) 1550°C/20 h.

that addition of α-Si_3N_4 seeds have favored the formation of α-Si_3N_4 from the nitridation of Si, thus promoting the Ca-α-Sialon formation. The role played by the added α-Si_3N_4 seeds can be further explained according to the theory of heterogeneous nucleation [16]. The presence of the seeds acts as substrates for heterogeneous nucleation of α-Si_3N_4 via the nitridation of Si, accelerating the α-Si_3N_4 formation because of the lower energy barrier. Based on the heterogeneous nucleation theory it can be suggested that the added α-Si_3N_4 acting as nucleation seeds could decrease the potential barrier for nucleation and thus accelerate nucleation and growth of the α-Si_3N_4 at a relatively low temperature, and consequently promote the Ca-α-Sialon formation.

4.3. Artificial neural network methodology for evaluation of Ca-α-Sialon formation

Properties forecasting plays a very prominent role in materials science. Numerous investigations addressing this problem have generally employed statistical methods, such as regression or autoregressive and moving average. However, properties forecasting is very complicated owing to the influence of various processing parameters. The aim of using the artificial neural networks (ANNs) in present paper is to establish a model between RCS of Ca-α-Sialon in the samples and the corresponding processing parameters, so as to predict the former in the final products. After 961 training cycles, the level of error was satisfactory and further calculation had no significant effect on error reduction, the error evolution during the ANNs calculation illustrating in Fig. 5 clearly indicates this. During the training process, the maximum mean relative error was found to be as low as 0.001%. The comparison of the simulation results by ANNs training and the experimental results of sample 1 to 26# are shown in Fig. 6. The excellent matches between the simulation results and the experimental results illustrating in Fig. 6, indicate that the ANNs model can well learn the fuzzy relation between inputs and outputs for the training data set. The relationship between the calculated RCS and the experimental RCS can be expressed as follows,

$$y = \mathrm{iw}\{3,2\}\mathrm{iw}\{2,1\}\tan\{\mathrm{iw}\{1,1\}\tan(x + b\{1\}) + b\{2\}\} + b\{3\} \quad (4)$$

where y is the calculated RCS by ANNs; x is the experimental RCS; iw{1,1}: weight to layer 1 from input; iw{2,1}: weight to layer 2; iw{3,2}: weight to layer 3; b{1}: Bias to layer 1; b{2}: Bias to layer 2; b{3}: Bias to layer 3. The values of iw{1,1}, iw{2,1}, iw{3,2}, b{1}, b{2}, and b{3} for ANN calculation are showed in Table 3.

Table 3
The values of iw{1,1}, iw{2,1}, iw{3,2}, b{1}, b{2}, and b{3}for ANN calculation.

Weight/ Bias	Calculated values during the training process of ANN
iw{1,1}	=[−0.20474 −0.27659 1.2108 −0.85148 0.29568 −6.9638; −0.11028 −1.2205 −4.5458 −4.9791 0.51038 −0.31208; −0.046797 −7.9244 3.2211 8.6123 10.2135 5.4498; 0.88109 0.53671 −2.4886 −1.2898 −0.81039 6.535; −0.23033 4.4184 −2.0935 2.2542 2.5473 −0.8503; −0.86398 0.57076 8.7358 −0.50706 −0.31232 −0.33893]
iw{2,1}	=[−0.84363 −5.4935 −7.6367 1.681 −1.5268 −1.9556; 5.1729 5.4275 4.7559 2.7981 −2.7566 7.0428; 5.0498 −7.4107 −8.1415 2.7561 −6.3159 5.9793; 5.78 4.4154 −9.8883 −0.85683 1.088 0.40358; 0.3482 −20.5441 6.9063 1.8936 3.8138 14.9328; 1.1654 −6.7547 10.5655 −4.0962 4.7013 0.56646; −2.2183 −2.1349 4.7874 −0.7925 2.4265 8.5773; 4.9276 2.8036 −1.8013 −3.9283 −3.3527 4.9781; −0.12409 −4.4299 2.5133 −6.482 4.7291 3.9578; 2.5541 9.016 −0.67038 −3.0864 2.8201 15.646; 0.646 0.71201 1.2172 3.6943 0.73903 1.2132]
iw{3,2}	=[−6.0761 −3.7133 −0.82477 7.4844 7.5636 26.8076 −33.2205 8.138 43.4484 −8.8265 −18.2559 4.7817 5.0264 −31.2487 1.9414; 5.9454 −20.4545 −14.8838 −15.0373 −0.11145 −0.36293 2.8073 21.1486 −15.45 27.118 −9.2538 −7.8099 −5.5071 14.1871 −5.0435; 2.1614 −13.1608 −1.6475 5.2098 −19.4957 17.0953 0.84926 3.2592 11.3197 5.9522 −0.94946 −4.1573 1.4527 −2.8502 −38.1895]
b{1}	=[11.7199; 29.2837; 25.8243; −23.4217; −4.3277; 47.5199]
b{2}	=[5.6588; −4.1259; 10.95; 0.61973; −0.59683; 2.142; 1.6793; −1.4487; 8.2926; 5.4667; −1.5556; −2.5178; −7.7218; −9.6285; −4.2786]
b{3}	=[3.6751; 7.442; 1.8121]

Table 4
Comparison between calculated results via ANN networks and experimental results.

Sample	Soaking time (h)	Experimental RCS of α-Siaon	Calculated RCS of α-Siaon	Absolute error	Relative error (%)
27#	8	18.8	19.6	0.8	4.3
	12	30	28.3	−1.7	−5.7
	20	21.7	21.9	0.2	0.9
28#	8	17.4	18.1	0.7	4.0
	12	19.3	18.9	−0.4	−2.1
	20	15.6	16.1	0.5	3.2
29#	8	35.2	35.3	0.1	0.3
	12	64.7	65.2	0.5	0.8
	20	26.1	24.9	−1.2	−4.6

To confirm the validity of the constructed ANNs model so as to predict the RSC of Ca-α-Sialon in some certain samples, the corresponding processing parameters (the amounts of starting materials of Si and Al, the contents of Y_2O_3, Fe_2O_3 and TiO_2 additives, and the amounts of α-Si_3N_4 crystal seeds) of samples 26–29# were input in the ANNs model to obtain the simulated RCS of Ca-α-Sialon. The comparison between the simulated RCS of Ca-α-Sialon by using the trained networks and the experimental RCS of Ca-α-Sialon is shown in Table 4. The relative errors between predicted values and measured results are found to be in the range of 0.3–4.6%, and the mean relative error during simulation and prediction is found to be as low as 3.4% for those samples, which shows that the prediction ability of the networks is quite satisfactory.

5. Conclusion

Ca-α-Sialon powders were prepared at 1550 °C by reaction nitridation method with Si, Al, $CaCO_3$ and Al_2O_3 as starting materials. For the preparation of Ca-α-Sialon powders, the use of Y_2O_3 and TiO_2 additive is much more effective than that of Fe_2O_3 in promoting the Ca-α-Sialon formation. A back propagation artificial neural networks model with a 6-15-3 (nodes number of input layer-hidden layer-output layer) configuration was developed to predict the relative content of α-Sialon in the final products prepared by the nitridation reaction method. The results calculated by ANNs match well with the experimental ones, and the average relative error is as low as 0.001%. It can be concluded that the work presented in this paper could be used as a guideline for further research on the applicability and the development of artificial intelligence techniques for preparation of Ca-α-Sialon by the nitridation reaction method.

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