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THERMAL ANALYSIS OF FORMATION OF SIALONS BY CARBOTHERMAL REDUCTION OF CLAYS

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Abstract

The formation at high temperatures of phases such as β′, *X*, and O′-sialon by carbothermal reduction of clays has been studied using simultaneous thermal analysis (STA) and thermomechanical analysis (TMA). Two reducing agents, silicon carbide and carbon, were used, with either kaolinite or bentonite. The progress of reaction was followed by interrupted and continuous thermal analysis procedures using STA and TMA and the changes correlated with the phases observed by X-ray diffraction at different stages. The conversion of kaolinite to β′-sialon is characterised by the dehydration and transformation of kaolinite independent of the nitriding atmosphere below 1400°C. At higher temperatures nitriding commences. Similar behaviour is observed with bentonite transforming to lower substitution sialon phases. The weight changes are correlated with oxidation/reduction reactions and mechanisms are suggested to explain the observed phenomena. The data obtained will permit the optimization of thermal cycles for batch production of sialon powders from natural minerals.

Keywords: carbothermal reduction, clays, formation of sialons, thermal analysis

Introduction

Industry requires high quality ceramics at low cost and such products, made from nitride bonded engineering ceramics for example, can be used in a variety of applications where conventional metallic materials are inadequate. However fabrication of low cost, reliable ceramic products has proved to be problematic due to difficulties in processing and in achieving adequate nitride phases for bonding.

Production of sialon from naturally occurring aluminosilicates by carbothermal reduction has been investigated in several previous studies. Lee and Cutler, [1] were the first to report the use of kaolinite and carbon in nitrogen gas to produce sialon solid solution at temperatures of 1300–1400°C. In a more complex system, Sugahara *et al.* [2] synthesized β′-sialon from montmorillonite. The reducing agent was polyacrylonitrile, intercalated into the lamellar structure of the montmorillonite. The resulting material was heated in nitrogen to various temperatures over 1150°C and the products determined by X-ray diffraction

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analysis. The mechanism of formation of β'-sialon by carbothermal reduction of kaolinite was investigated by Higgins and Hendry [3] using powdered coal as the reducing agent. They concluded that the reaction to produce β'-sialon from carbon and kaolinite takes 8 h to completion at 1400°C and 4 h to completion at 1450°C. The process occurs in three stages: thermal decomposition of kaolinite to mullite and free silica, followed by reduction of free silica to silicon carbide, then reaction of carbon, silicon carbide and mullite with nitrogen gas to give sialon. The first two stages are independent of atmosphere, but the presence of excess nitrogen during the third stage is critical to ensure complete nitridation; otherwise β'-sialon is not produced.

Reactions involving kaolinite have been shown to depend strongly on pellet size, the presence of iron [1], nitrogen flow rate [4], surface area of the powders and impurities in the raw components [5] whilst reactions with bentonite depend on the temperature, source of carbon and atmosphere [2, 6].

In most of the previous studies listed above, formation of sialon has been studied using large samples (10–20 g) in horizontal or vertical furnaces, using evolved gas analysis during the experiment and X-ray diffraction analysis of the cooled sample to infer reaction pathways. It is the aim of this work to use simultaneous thermal analysis and thermomechanical analysis to study sialon formation in situ to enable quantification of thermal effects as they occur during reduction and subsequent nitridation. Two reducing agents, carbon and silicon carbide were used with two clays, kaolinite and bentonite to compare the high temperature phases formed in a nitrogen atmosphere.

The data obtained will be used to optimize processing routes for sialon powders fabricated from naturally occurring minerals, and designed for subsequent use in the manufacture of nitride or nitride-bonded engineering ceramics.

Experimental techniques

Materials

Kaolin Supreme and bentonite were supplied by E. C. C. International Ltd. Silicon carbide was supplied by Goodfellow Metals Ltd and the carbon powder

Table 1 Purity and particle size of materials

Material	Supplier	Purity / %	Average particle size / μm
Kaolinite	ECC	97.5	2
Bentonite	ECC	94.3	2
Silicon carbide	GOODFELLOW	99.0	5
Carbon	ALDRICH	99.0	8

was from Aldrich Chemical Company Ltd. The particle sizes and purity are listed in Table 1.

The gases used during thermal analysis were all supplied by BOC Ltd.: Nitrogen 99.99% pure; Air 99.99% pure; and Argon 99.99% pure.

Powder preparation

Appropriate amounts of clay mineral and reducing agent to make 5, 10, 20 and 30 vol% of reducing agent in the clay were weighed out into 300 ml plastic bottles. The powders were milled for 12 h using alumina milling balls and isopropyl alcohol as a wetting agent on a Pascal rotating machine at 1425 rpm. The mixtures were then dried using a drying lamp and dry milled for about 30 min. The dried mixtures were stored over silica gel in desiccator jars.

Thermal analysis

Thermal analysis was carried out using equipment from Polymer Laboratories, models STA-1640 and TMA-1500. Samples were heated from ambient temperature to the temperature specified by the heating programme at 20 deg·min^{-1} in nitrogen, argon or air flowing at 50 ml/min. The average sample size for STA was 20 mg of powder in alumina crucibles. The average thickness of TMA pressed powder samples was 3 mm.

X-ray diffraction

Powder X-ray diffraction analysis data were obtained with a Philips diffractometer and a Hagg-Guinier camera. CuK$_\alpha$ radiation and a potassium chloride standard were used.

Results

Effect of atmosphere on heating curves for kaolinite and bentonite

Kaolinite

Figure 1 shows the results obtained when pure kaolinite was heated in nitrogen, air, and argon at 20 deg·min^{-1} up to 1400°C. The weight loss between 400 and 600°C is due to loss of structural water and is the same for each gas within the limits of experimental error (14.0±0.8%). The peak areas per milligram of sample of the corresponding endotherms on the DTA curve are also the same within the limits of experimental error (2%). The exothermic peaks at 1000°C and above correspond to the formation of crystalline phases, the components of which depend on the types and amounts of impurities in the clay. The kaolinite used in this work was 97.5% pure and thus only mullite and cristobalite were the crystalline phases detected by X-ray diffraction analysis. The products were the same in all three gases. The greatest peak area per milligram of sample was for argon. Thereafter three second order effects were observed, occurring at lower temperatures for argon, higher for nitrogen and highest for air. Mullite formation is assisted by heating kaolinite in an inert gas [7] and this is confirmed by the present observations.

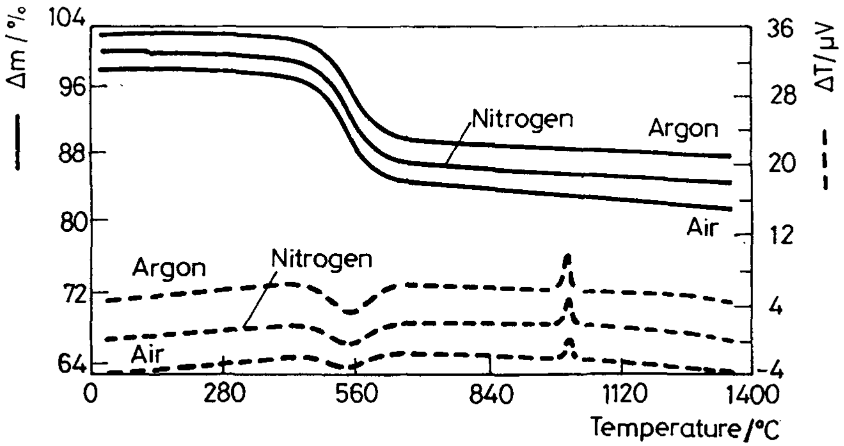

Fig. 1 Comparison of STA heating curves of kaolinite in nitrogen, air and argon

Thus the essential features of heating kaolinite can be summarised as follows:

$2SiO_2{\cdot}Al_2O_3{\cdot}2H_2O{\equiv}Si_2Al_2O_5(OH)_4$ kaolinite / low temp.
$Si_2Al_2O_5(OH)_4 \rightarrow Si_2Al_2O_7$ $(+\ 2H_2O)$ metakaolin / >600°C.
$3Si_2Al_2O_7 \rightarrow Si_2Al_6O_{13} + SiO_2$ mull + crist. / >1000°C.

Gworek and Towers [8] performed a similar set of experiments with a sample of kaolinite which had orthoclase present as an impurity. They found that the kaolinite structure existed up to the endothermic reaction, above which temperature no structure could be identified until γ-alumina appeared at 990°C when the exothermic reaction began. γ-alumina was the only crystalline structure detected up to 1170°C, above which only the mullite structure was detected. They found no evidence of silica or α-alumina but further investigations revealed that the presence of silica prevented the transformation of γ → α–alumina and needle shaped mullite crystal formation was assisted by heating in a reducing atmosphere. These results have since been confirmed by other workers as well as the present study in which the exothermic clay transformation is affected by impurities in the clay matrix and the furnace atmosphere.

Bentonite

Figure 2 shows the results obtained when bentonite is heated in air, argon and nitrogen to 1400°C. The weight losses under 200°C are due to loss of absorbed water and the weight loss between 500 and 700°C is due to loss of structural water (5.02±0.34%). The DTA endotherms (15.95±0.32 μV/mg) corresponding to the loss of structural water are similar within the limits of experimental error. Around 860°C there is an "S"-shaped thermal event corresponding to the final destruction of the anhydrous structure with various phases recrystallizing thereafter. These phases and the number of exotherms observed depend on the degree of isomorphous substitution and impurities present in the mineral [9]. The products identified by X-ray diffraction analysis were mullite and cristobalite for all three atmospheres. The greatest peak area per milligram for the exotherm occurring about 1000°C was observed in air (–9.33) followed by nitrogen (–7.76) then argon (–5.49). A sample heated to 1000°C in nitrogen, then X-rayed showed only cristobalite present, but at 1200°C mullite and cristobalite were detected. The peak at 1055°C is caused by the nucleation of mullite [9], and as the amount of mullite formed in bentonite is much less than that in kaolinite, the peak areas in Fig. 2 are much smaller than in Fig. 1 and considerably broader.

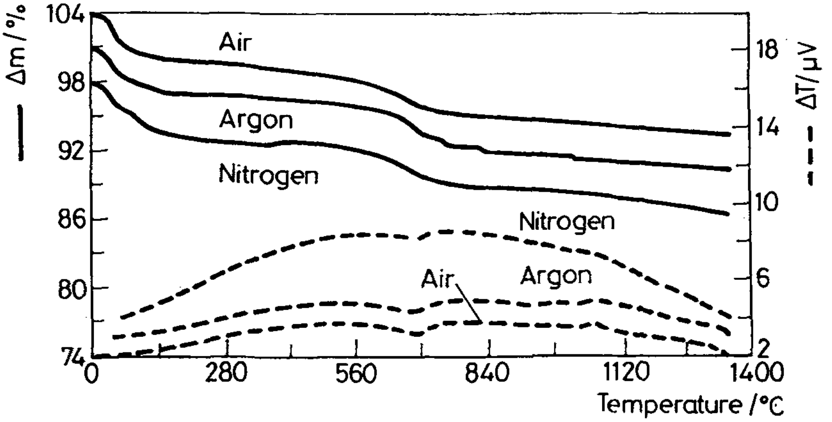

Fig. 2 Comparison of STA heating curves of bentonite in nitrogen, air and argon

The essential features of decomposition are therefore the same as for kaolinite but with different proportions of the final phases.

These results show that the products formed up to 1400°C are not dependent on furnace atmosphere. Differences in the main exothermic peak area were observed, but there was no evidence that a particular gas either promoted or inhibited the main exotherm for both clays corresponding to mullite nucleation.

Effect of reducing agent on clays up to 1400°C

Tables 2 and 3 show the products obtained in nitrogen and argon for carbon and silicon carbide added to kaolinite and bentonite. Up to 1400°C, no reaction was detected on the STA apparatus and no reduction products were observed by X-ray diffraction analysis. Addition of either reducing agent to kaolinite raised the onset, peak and final temperatures of the main exothermic peak in both atmospheres and carbon added to kaolinite in nitrogen increased the temperature of the second order effect at 1250°C, thought to be the nucleation of mullite crystals with needle-like morphology. Addition of either reducing agent to bentonite in nitrogen raised the temperature of the main exothermic peak but in argon addition of either reducing agent lowered the temperatures of the main exothermic peak. Addition of either reducing agent to bentonite in both atmospheres lowered the temperature of the second order effect at 1250°C.

Table 2 Products of reducing agents added to kaolinite

Additive	Atmosphere	Products
Carbon	Nitrogen	Mullite, Cristobalite
Carbon	Argon	Mullite, Cristobalite
Silicon carbide	Nitrogen	Mullite, Silicon carbide, Cristobalite
Silicon carbide	Argon	Mullite, Silicon carbide, Cristobalite

Table 3 Products of reducing agents added to bentonite

Additive	Atmosphere	Products
Carbon	Nitrogen	Mullite, Cristobalite
Carbon	Argon	Mullite, Cristobalite
Silicon carbide	Nitrogen	Mullite, Silicon carbide, Cristobalite
Silicon carbide	Argon	Mullite, Silicon carbide, Cristobalite

Identification of precursors and end products

Figure 3 shows the results of heating a sample of kaolinite with 30 volume percent carbon powder added in a flowing nitrogen atmosphere. Initially, X-ray diffraction analysis detected kaolinite of a high crystallinity. Loss of structural water was observed between 400 and 600°C, resulting in meta-kaolin which showed only four lines on X-ray diffraction analysis. These lines became weaker as samples were heated to higher temperatures until at 1200°C, X-ray diffraction analysis showed mullite and cristobalite. These phases persisted on heating to 1500°C and holding at that temperature for one hour. The weight loss above 1400°C is due to carbon reacting with the oxygen in the clay. During the isotherm at 1500°C, there is a weight gain indicating absorption of nitrogen, but the amount is less than 3% and the amount of sialon phases produced is below the limit of detection by X-ray diffraction analysis.

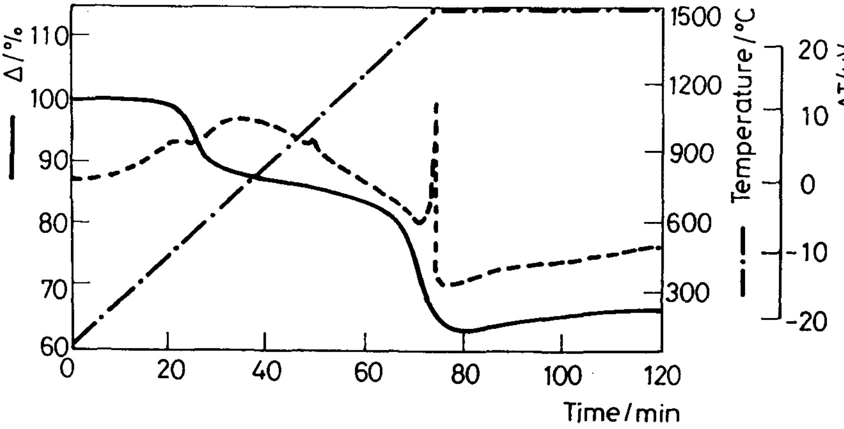

Fig. 3 STA curve of kaolinite with reductant (30 vol. % carbon) in flowing nitrogen gas

Figure 4 is the curve obtained for 30 volume percent silicon carbide in kaolinite. Initially, X-ray diffraction analysis detected well-crystallised kaolinite and silicon carbide. Loss of structural water was again observed between 400°C and 600°C, resulting in meta-kaolin and silicon carbide. As before, the meta-kaolin became weaker as the temperature increased until at 1200°C, phases detected were mullite, cristobalite and silicon carbide. These phases persisted up to 1400°C, with no additional weight loss (Fig. 4). At the end of the isotherm at 1500°C, phases detected were mullite, β'-sialon of z value 2, cristobalite and silicon carbide. Sialon was obtained for this powder system because the silicon carbide reduction is a slower reaction than that with carbon which gives more opportunity for nitrogen to react with the sample without reductant loss. That is, in Fig. 3 there is considerable loss of CO and SiO by reaction of C and SiO_2 below 1500°C with the result that there is only a negligible extent of nitridation. In Fig. 4 no reduction loss occurs before 1500°C at which temperature nitridation has also commenced. The additional Si from SiC gives a lower z value β' as shown schematically in Fig. 8. This behaviour can be modelled by a series of possible chemical reactions as set out below.

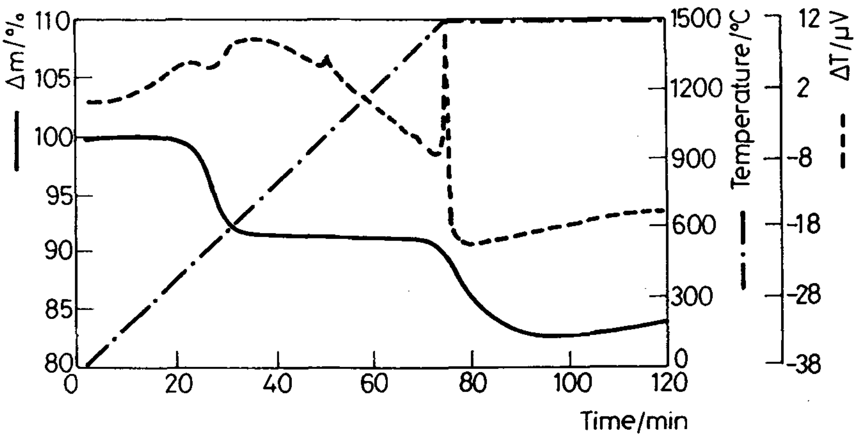

Fig. 4 Simultaneous TG and DTA of kaolinite with reductant (30 vol. % silicon carbide) in flowing nitrogen atmosphere

The first stage of the process of carbon reduction (Fig. 3) does not involve carbon directly but is the water loss reaction at 400–600°C (which may remove some carbon by the water-gas reaction but is ignored at present).

For 30 volume % in the mix

$$\underset{\text{kaolinite}}{Si_2Al_2O_5(OH)_4} + 7C \rightarrow \underset{\text{meta-kaolin}}{Si_2Al_2O_7} + 2H_2O(g) + 7C \qquad (1)$$

The weight loss for this reaction is 10.5% as is shown in Fig. 3. The next stage – above 1050°C – is decomposition of meta-kaolin to mullite and silica although crystallization of these phases as mullite and cristobalite does not occur until higher temperatures.

$$3Si_2Al_2O_7 + 21C \rightarrow Si_2Al_6O_{13} + 4SiO_2 + 21C \qquad (2)$$

No weight change is involved although it is clear from Fig. 3 that an increasing weight loss occurs above 1200°C and may be accounted as,

$$Si_2Al_6O_{13} + 4SiO_2 + 21C \rightarrow Si_2Al_2O_{13} + 4SiC + 8CO + 9C \qquad (3)$$

if it is assumed that mullite is stable and that silica, either in glassy form or as cristobalite reacts with carbon. The weight loss for this reaction is 24.5% which is again in line with Fig. 3. It is unlikely that all of the silica forms silicon carbide as in Fig. 3 since a small weight gain occurs at 1500°C indicating some nitrogen pick-up from the gas. No nitride phase was detected by X-ray diffraction however which indicates less than 2% by volume of that phase.

A similar analysis can be applied to the case of 30 volume % silicon carbide in Fig. 4 where the water loss is 10% and no further loss occurs until the 1500°C isothermal stage. If the reaction for reduction nitridation is then written in terms of the observed phases at 1500°C – β′ ($z = 2$), mullite and cristobalite – the following may be assumed

$$Si_2Al_6O_{13} + 4SiO_2 + 3.4SiC + 3.4N_2(g) \rightarrow$$

$$3.4CO(g) + 3.64SiO_2 + 0.62Si_2Al_6O_{13} + 1.13Si_4Al_2O_2N_6$$

if all of the SiC is consumed. The imported point from this reaction is that no weight change is involved and this is the case regardless of the proportion of silicon carbide used for reduction. The weight loss and subsequent weight gain in Fig. 4 must therefore be due to side reactions to produce silicon monoxide and nitridation of the powder to β′ ($z = 2$).

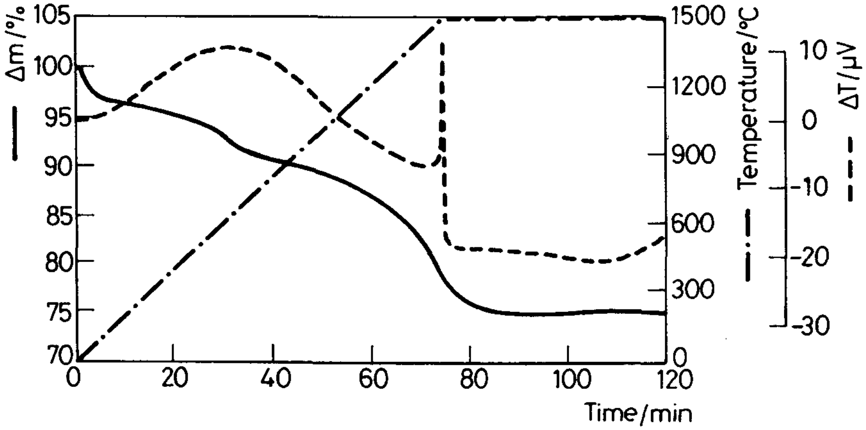

Fig. 5 Simultaneous TG and DTA of bentonite with reductant (30 vol % carbon) in flowing nitrogen gas

Figure 5 shows the results when 30 volume percent carbon in bentonite is heated in nitrogen. Initially, a mixture of montmorillonite and cristobalite was detected by X-ray diffraction analysis. Loss of structural water was observed between 500 and 700°C, resulting in anhydrous montmorillonite and cristobalite. At 1200°C, mullite and cristobalite were detected and these phases persisted up to 1400°C. After an isotherm at 1500°C for one hour, the phase detected was *X*-phase sialon. Analysis of the weight losses can again be reconciled as was shown above for kaolinite and a similar continuing weight loss occurs from about 900°C to 1500°C due to the reactivity of the carbon/clay mix. The appearance of X-phase as the only crystalline phase may then be reconciled with Fig. 3, where no crystalline nitrogen-containing phases were formed, by reference to Fig. 8. A small, extent of nitridation of kaolinite compositions should give mullite/glass predominantly with a small proportion of X-phase which in Fig. 3 is too low to detect. A similar extent of reduction of bentonite compositions places the phase mixture in the glass/X-phase region or in the low O′ compositions of the O′/glass/X-phase field, which is consistent with X-phase being the only crystalline phase present in significant amounts.

Figure 6 shows the curves obtained when 30 volume percent silicon carbide in bentonite was heated in nitrogen. Initially, the phases present were montmorillonite, cristobalite and silicon carbide. Loss of water was observed between 500 and 700°C (as before, weight loss is consistent with calculations) and results in anhydrous montmorillonite, cristobalite and silicon carbide. At 1200°C, mullite, cristobalite and silicon carbide were the phases present and no weight loss occurs. These phases persisted up to 1500°C during an isotherm of one hour at that temperature. No nitride phase was observed in this case although a small weight gain (3%) occurred at isothermal hold.

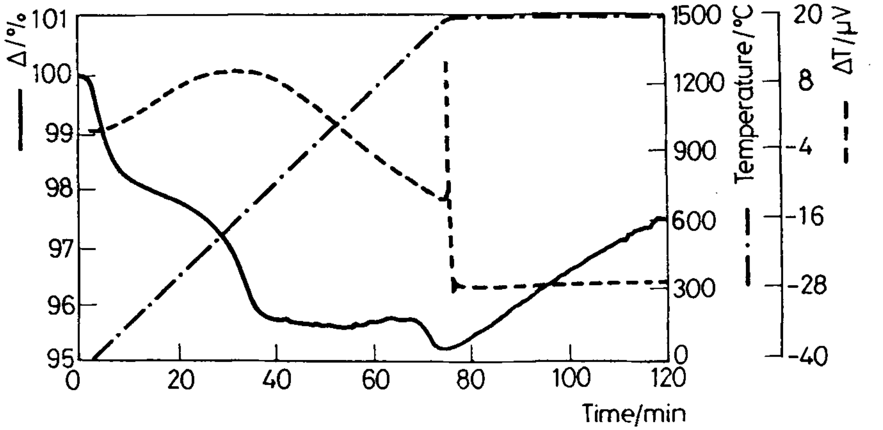

Fig. 6 Simultaneous TG and DTA of bentonite with reductant (30 vol % silicon carbide) in flowing nitrogen gas

Thermomechanical analysis

Thermomechanical analysis was carried out on pressed samples of each of the four powder systems used in the previous section. Samples were heated in nitrogen to 1500°C at 20 deg·min^{-1}, with an isotherm at 1500°C for one hour, and cooled. The load used was 20 g. Figure 7 shows the results obtained for the four reduction systems.

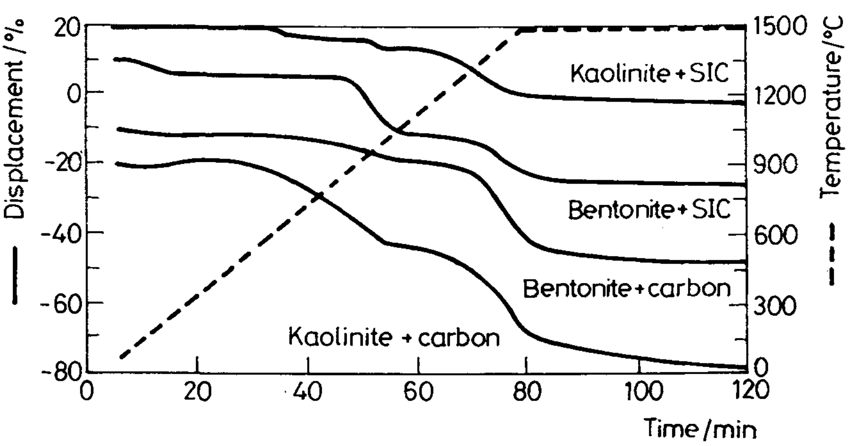

Fig. 7 Thermomechanical analysis of the four mixtures pressed into pellets

Carbon in kaolinite showed a contraction starting at 400°C, corresponding to loss of structural water leading to collapse of the layer structure. At 980°C there was an inflection, corresponding to the exothermic peak on the DTA curve. Onset of significant sintering was detected at about 1200°C. Overall, the sample contracted in length by 60% and the end products identified by X-ray diffraction analysis were mullite and β′-sialon of z value 3.

Silicon carbide in kaolinite underwent a contraction due to loss of structural water, starting at 400°C, as above. There was an inflection at about 1000°C again corresponding to the exothermic peak on the DTA curve and the onset of sintering was detected at about 1200°C. The overall reduction in length was 22% and the end products of the reduction were mullite, β′-sialon of z value 3 and silicon carbide. The contraction of the carbon in bentonite sample (Fig. 7) started at 500°C. This was due to loss of structural water and lasted until 900°C, at which temperature there was an inflection, corresponding to the main exothermic peak on the DTA signal attributed to final destruction of the montmorillonite matrix [9]. Onset of sintering was again detected at about 1200°C. The overall reduction in length of the sample was 40% and the products were X phase and O′-sialon. In the case of silicon carbide in bentonite, contraction started at 850%. Onset of sintering was detected at 1200°C and the total reduction in length was 30%. The end products were silicon carbide and cristobalite.

The general observations from this set of experiments are that for the four reduction systems, the onset of sintering is about 1200°C and the samples with silicon carbide contracted less than the carbon samples. Both kaolinite samples displayed a point of inflection at 1000°C, corresponding to the exotherm on the differential temperature curve of the STA. In both cases mullite and β′-sialon of

z value 3 were detected by X-ray diffraction analysis. Both bentonite samples displayed a point of inflection at about 900°C, corresponding to the exotherm observed on the differential temperature curve of the STA.

Table 4 compares the products obtained from STA and TMA using the same temperature programme. The differences observed are first of all due to the experimental conditions required for each technique: in STA the sample is held in an open alumina crucible, with the effect that there is only a small surface area for the atmosphere to diffuse into (16 mm^2), but in TMA the sample is supported at the top and the bottom, leaving a surface area of 56 mm^2 for nitriding reaction with the gas. So, for carbon in kaolinite, for example, the products of STA were mullite and cristobalite, whilst the products from TMA were mullite and β'-sialon of z value 3, due to the increased reaction surface.

Table 4 Comparison of products from STA and TMA

Reducing system	High temperature phases detected by XRD	
	STA	TMA
Carbon and kaolinite	Mullite, Cristobalite	Mullite, β'-sialon (z = 3)
Silicon carbide and kaolinite	Mullite, Cristobalite, β'-sialon (z = 2), Silicon carbide	Mullite, β'-sialon (z = 3) Silicon carbide
Carbon and bentonite	X–phase	X–phase O'-sialon
Silicon carbide and bentonite	Mullite, Cristobalite, Silicon carbide	Cristobalite, Silicon carbide

Thus in order to describe fully the mechanisms of reaction in each system – clay and reductant – it is necessary to compound all of the information from STA, TMA and X-ray diffraction. There will clearly remain certain inhomogeneities in the analysis but in a full-scale reactor the pellet or granule sizes are approximately the size of the STA and TMA pellets used in the present work and therefore give a reasonable insight into behaviour during reduction.

(i) Carbon/kaolinite. The high reactivity of carbon with the decomposed clay results in carbonaceous weight losses which can be modelled according to simple chemical reactions to produce β' (z = 3) but with low yields due to weight loss. The pellets are however readily sinterable as shown by TMA under low loads.

(ii) Silicon carbide/kaolinite. The additional stability of silicon carbide relative to carbon means that the onset of reduction and nitriding is delayed to higher temperatures and the silicon from the carbide contributes to a lower z-value of β′ as shown in Fig. 8. The shrinkage of the pellets above 1200°C is the same as for carbon reduction (Fig. 7) but overall it is lower due to the suppression of low temperature weight loss.

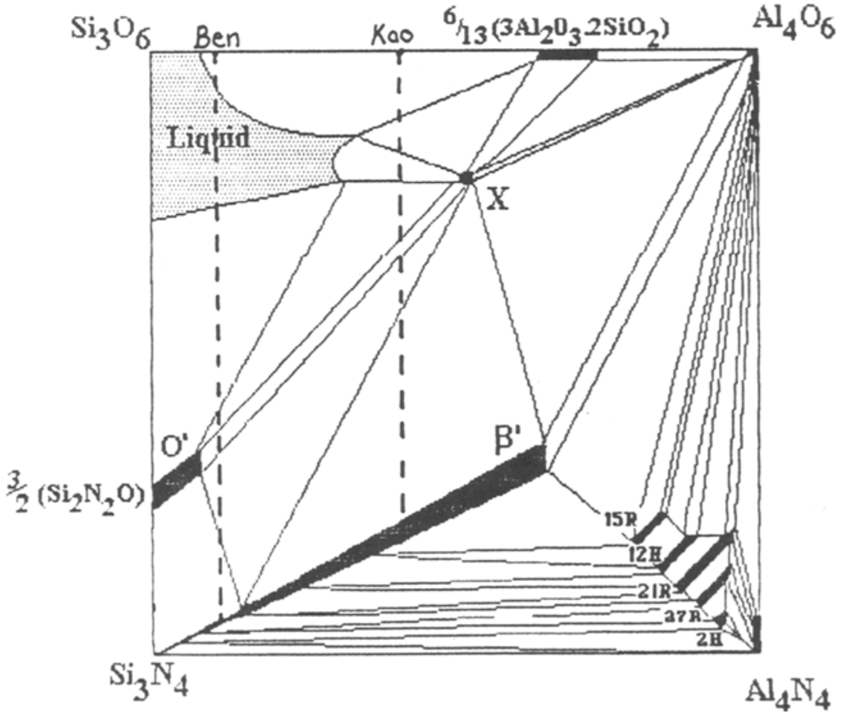

Fig. 8 Sialon phase diagram [11]

(iii) Carbon/bentonite. This clay was chosen to give a low z-value sialon material but the maximum amount of carbon used (30 volume percent), although theoretically sufficient, is too low in practice for complete conversion due to side reactions (low temperature weight loss). The nitride phase formed is therefore in the O′ and X-phase region, presumably accompanied by a silicate glass from the dissociated clay and impurities.

(iv) Silicon carbide/bentonite. Under neither set of conditions (Table 4) did nitride phase form for this reduction process. The reactivity of the carbide which is only effective at high temperatures is clearly insufficient in one hour to cause nitridation. Higgins and Hendry [3] noted however that practical process times are up to eight hours duration at 1500°C.

Conclusions

The following general conclusions may be drawn from the study. The sialon phase diagram and associated chemical reaction calculations give a reliable guide to the behaviour of clay materials during reduction/nitriding reactions although during heating and in the early reaction stages, as studied here, there will be inhomogeneity due to mass transport control from and to the gas.

Carbon is a more effective reductant than silicon carbide and as a result the reduction reactions commence at lower temperatures. However this also leads to higher weight losses with carbon and potential loss of nitride product yield due to formation of, for example, silicon monoxide. In this regard reduction reactions are independent of the gas atmosphere below approximately 1400°C as proposed by Higgins and Hendry [3] but no evidence was found for formation of silicon carbide as an intermediate step in carbon reduction as claimed by the same authors.

The use of thermal analysis for sialon formation provides information on rate of reaction, onset temperature of reactions, instantaneous weight loss and gas evolution during firing [10]. The results presented therefore allow a more detailed understanding of the processes taking place during reduction and nitriding of clays and facilitate the choice of reductant combined with acceptable heating rates as material is fed to the hot zone of the reactor. With carbon reduction the use of increased heating rates will prevent low temperature loss, while with silicon carbide the heating rate is not critical but longer times may be necessary for reactions particularly in the formation of low z-value β'-sialon where a maximum extent of reduction is required.

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Zusammenfassung — Mittels simultaner Thermoanalyse (STA) und thermomechanischer Analyse (TMA) wurde bei hohen Temperaturen die Bildung von Phasen wie β', X' und O' Sialon durch carbothermische Reduktion von Tonerden untersucht. Bei Kaolinit und Bentonit wurden zwei Reduktionsmittel benutzt: Siliziumcarbid und Kohlenstoff. Das Fortschreiten der Reaktion wurde mittels unterbrochener und fortlaufender thermoanalytischer Verfahren (Einsatz von STA und TMA) verfolgt und die Veränderungen mit den durch Röntgendiffraktion in verschiedenen Stufen beobachteten Phasen in Korrelation gesetzt.

Der Umsatz von Kaolinit zu β'-Sialon ist – unabhängig von der Nitrieratmosphäre – unter 1400°C durch die Dehydratation und die Umwandlung von Kaolinit gekennzeichnet. Bei höheren Temperaturen beginnt die Nitrierung. Ein ähnliches Verhalten wird für Bentonit bei der Umwandlung in niedersubstituierte Sialonphasen beobachtet. Die Gewichtsänderungen stehen mit Oxidations/Reduktionsreaktionen in Korrelation und es werden Mechanismen zur Deutung der beobachteten Erscheinungen vorgeschlagen.

Die erhaltenen Angaben gestatten die Optimierung der Thermocyclen für die Fertigung von Sialonpulvern aus natürlichen Mineralien.