J Mater Sci (2007) 42:8745–8752
DOI 10.1007/s10853-006-0192-7

A study of the influence of muscovite on the thermal transformations of kaolinite from room temperature up to 1,100 °C

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Received: 20 September 2005 / Accepted: 10 April 2006 / Published online: 18 July 2007

Abstract Global thermal transformation of kaolinite–muscovite mixtures containing at most 25 mass % of muscovite was studied up to 1,100 °C. Quantitative and qualitative DTA analysis reveals a great intereaction between the two phylosilicates above 900 °C, which is characterized by an enhancement of spinodal demixion contribution during the structural reorganization of metakaolinite. The corresponding enthalpy variation are respectively –32 ± 1 kJ/mol and –15 ± 1 kJ/mol of kaolinite for pure kaolinite and for the mixture containing 15 mass % of muscovite. This interaction is mainly correlated with the diffusion of potassium ions from muscovite platelets into metakaolinite structure. The resulting consequence is an enhancement of mullite crystallization and grain growth at 1,050 °C (earlier than in the case of pure kaolinite mineral).

Introduction

Kaolin based materials represent a large part of silicate ceramic production in the world. Their final properties depend on the composition of starting materials [1, 2] and therefore, one should master the thermal evolution of the major constituents and the effect of minor components or impurities on these transformations, in order to give out high quality and high performance products.

Many studies have been conducted on the thermal transformations of kaolinite [3–11] and muscovite [12–14] minerals using various characterization methods. It is obvious that these phyllosilicates start their transformation by a dehydroxylation reaction associated to an endothermic peak on differentiel thermal analysis (DTA) curves. This transformation occurs in the range 400–650 °C and 800–900 °C respectively for kaolinite and muscovite minerals. The resultant metakaolinite has a poorly crystalline structure (2-dimensional order and few -OH groups) compare to the dehydroxylated (or high temperature) muscovite which remains in a well crystalline state. In fact, structural data for muscovite were obtained by X-ray study and thermal analysis [13–18]. The high temperature or dehydroxylated muscovite exhibits distorded octahedral layers with 6 to 4 or 5-fold Al, leading to a rearrangement of the adjacent tetrahedral layers. Trere is also an increase of the celle volume mainly due to a larger c parameter (from 20.0–20.1 Å to 20.2–20.3 Å, depending on authors). Towards 1,000 °C, metakaolinite exhibits a structural reorganization expressed through an exothermic peak on the DTA curve. Some authors associated this exothermic phenomenon to the nucleation and growth of mullite while other authors correlated it with the demixion mechanism involved in the reorganization process. This exotherm is therefore a result of a spontaneous formation of—alumina and/or mullite, the kinetic of this transformation sequence process being highly dependent on the nature of starting kaolinite and the thermal history [19–22]. Near 1,250 °C a small exothermic effect appears and is assigned to the simultaneous crystallization of cristobalite and growth of secondary mullite [23–26]. Recent studies [27, 28] seem to indicate two exothermic peaks

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in the range 1,000–1,400 °C. The proposed mullitization process path involves two simultaneous mechanisms There is an early crystallization by nucleation in aluminosilicate phase (1,160 °C) associated with the Al–Si spinel polymorphycal transformation to mullite (1,240 °C). In the case of dehydroxylated muscovite there is not such controversy. In fact, it is assessed that orthoclase is formed below 1140°C without any signature on DTA curves. This orthoclase melts incongruently at 1,140 °C (endothermic DTA peak), leading to a mixture of mullite, leucite and a liquid phase. When starting muscovite contains more than 3 mass % of iron oxide, corundum can be formed instead of mullite [29].

Considering the fact that kaolin is often associated with variable quantities of muscovite mica, since most kaolin deposits result from the decomposition of potassic mica, we find it interesting to conduct a study on the interaction of these two minerals from room temperature to 1,100 °C. For this purpose, kaolin-muscovite mixes containing at most 25 mass % of muscovite, were used in order to remain in the range of compositions used in ceramic industry.

Materials and experimental procedure

Kaolinite and muscovite mineral were provided through two raw materials of very high purity, respectively kaolin KGa-1b from the Clay Mineral Society (University of Missouri, USA) and potassic mica (MBH) from Bihar (India). Their chemical and mineralogical compositions, calculated from spectrometric analysis using ICP-AES (Inductively Coupled Plasma) and from X-ray powder diffraction analysis (XRPD), are listed in Table 1 and are in good correlation with those given in literature [30, 31]. The experiments were carried out on the as-received raw materials and on kaolin-muscovite mixtures containing 5, 10, 15, 20 and 25 mass % of muscovite mica. In order to optimized the homogeneity of kaolin-muscovite mixtures, the raw materials were grounded and sieved to 40 µm prior to mixing. This latter operation was achieved in distilled water with adequate relative proportions of raw materials under gentle ball-milling.

Thermal analysis were performed using a Setsys 2400 apparatus from SETARAM equipped with a DSC-TGA 1500 head system. For the whole samples, heat treatment were performed from room temperature up to 1,500 °C using six different heating rates (3, 5, 8, 10, 15 and 20 °C/mn) and alumina treated at 1,500 °C serves as reference material. Each experiment was carried out on 100 mg of materials under ambient atmospheric pressure (about 1.013×10^5 Pa). Two methods were used for quantitative analysis. The first one consist in calculating the peak surface area; actually, the application of relation (1) lead to the enthalpy evaluation after a calibration of the apparatus with reference materials from the ICTAC list [32] in order to determine the thermal exchange coefficient K.

$$A = \frac{Q}{K} m \tag{1}$$

In this equation, A is the peak surface area, Q represents the heat exchanged during the transformation considered, m designs the mass of reactive compound and K is the thermal exchange coefficient which is function of the DTA device.

For our study, potassium sulfate (K_2SO_4) and strontium carbonate ($SrCO_3$) have been selected as reference materials for the calibration of the kaolinite dehydroxylation and metakaolinite structural reorganization peaks respectively. This choice is justify by the fact that these materials are subject to allotropic transformations respectively at 586 and 925 °C. In every surface area calculations, the baseline line has been supposed linear from the starting point to the end point because no derivation or shift of the baseline has been observed. This procedure has not been applied for the study of the dehydroxylation peak of muscovite

Table 1 Raw materials composition (mass %, with a precision of 0.001%)

	Kaolin Kga-1b		Potassic mica from BIHAR	
	Chemical	Mineralogical	Chemical	Mineralogical
SiO_2	44.2		45.40	
Al_2O_3	39.6	Kaolinite = 96	37.01	
Fe_2O_3	0.2		1.10	
TiO_2	1.4	Quartz < 1	–	
MgO	0.03		0.1	Muscovite ≈ 99
Na_2O	0.01	Anatase > 1	0.18	
K_2O	0.05		11.8	
P_2O_5	0.03	Gibbsite > 2	–	
Loss of ignition at 1,000 °C	13.78		4.3	
Other	0.7		0.01	

since it was not possible to determine with accuracy the baseline on the DTA curves of our mixtures. The use of this method allows to reach the enthalpy variation occurring during a given transformation.

The second method, developed by Kissinger [33], is based on the heating-rate-dependence of the temperature of the peak maximum. The relation used in this case is given below as Eq. 2.

$$\frac{E\beta}{RT_{\mathrm{m}}^{2}} = A\ \exp\left(-\frac{E}{RT_{\mathrm{m}}}\right) \qquad (2)$$

where β represents the heating rate, E is the apparent activation energy, T_{m} represents the temperature of the peak maximum, R is the perfect gas constant and A is the pre-exponential constant. If the activation energy remains constant, the plot of $\ln(\beta/T_{\mathrm{m}}^{2}) = f(1/T_{\mathrm{m}})$ gives a straight line with a slope equal to $-E/R$. Therefore, one can deduced the apparent activation energy of the transformation studied.

XRPD analysis was performed on the final products using the CuK$_{\alpha 1}$ monochromatic radiation, on a Siemens D5000 Bragg-Brentanno type diffractometer. Data were collected in the range $3 \leq 2\theta \leq 45°$ in step scan mode with a step of 0.03° and a counting time of 2.5 s/step. Microstructure observation was carried out on a Stereoscan 260 scanning electronic microscope (SEM) equipped with a PGT-Prism detector for energy dispersive spectrometry (EDS) analysis.

Results

The DTA and thermogravimetric analysis (TGA) curves obtained for the raw materials are shown respectively on Fig. 1a, b. The resulting global mass loss, respectively 13.78 and 4.3 mass % for kaolin KGa-1b and muscovite MBH, are coherent with the chemical and mineralogical analysis given on Table 1. Also, the observed transformation domains are also in agreement with those listed in literature [34]. These results confirm the high purity of the starting materials (96 mass % of kaolinite mineral in kaolin KGa-1b and almost 99 mass % of muscovite mineral in potassic mica from Bihar). Since this study is conducted from room temperature to 1,100 °C, the obtained results were sorted into two transformation domains: from room temperature to 900 °C and from 900 °C to 1,100 °C.

From room temperature to 900 °C

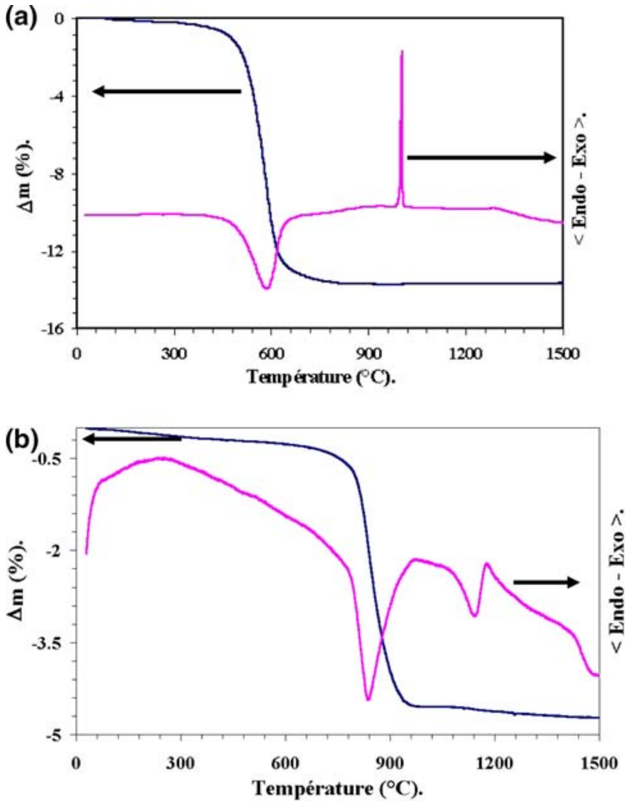

Fig. 1 DTA and TGA curves of kaolinite (**a**) and muscovite (**b**) raw materials for a heating rate of 10 °C/mn.

In this temperature range, there are two distinct dehydroxylation reactions as exhibited on Fig. 2a, b respectively for kaolinite and muscovite. In the two cases, there are no significant changes of the endotherms with increasing muscovite content. The peak intensities exhibited a linear dependence with respect to the amount of reacting minerals (kaolinite and muscovite). These observations indicate no remarkable interaction between the two minerals until the end of muscovite dehydroxylation (900 °C).

The application of quantitative analysis to the dehydroxylation process of kaolinite leads to the enthalpy variation values contained in Table 2, with a mean value of about 200 ± 7 kJ/mol. There is a good correlation with literature [18, 35] since the values found there varies from 170 kJ/mol to 200 kJ/mol.

In the case of muscovite dehydroxylation which occurs between 800 °C and 900 °C, the application of the Kissinger method leads to an apparent activation energy of 405 ± 25 kJ/mol of muscovite. This result is rather coherent with the muscovite dehydroxylation enthalpy variation reported in literature [18], which varies from 226 kJ/mol to 377 kJ/mol of muscovite depending on the experimental conditions. A similar calculation was performed for kaolinite dehydroxylation between 450 °C and 700 °C, and the corresponding apparent activation energy was 175 ± 10 kJ/mol.

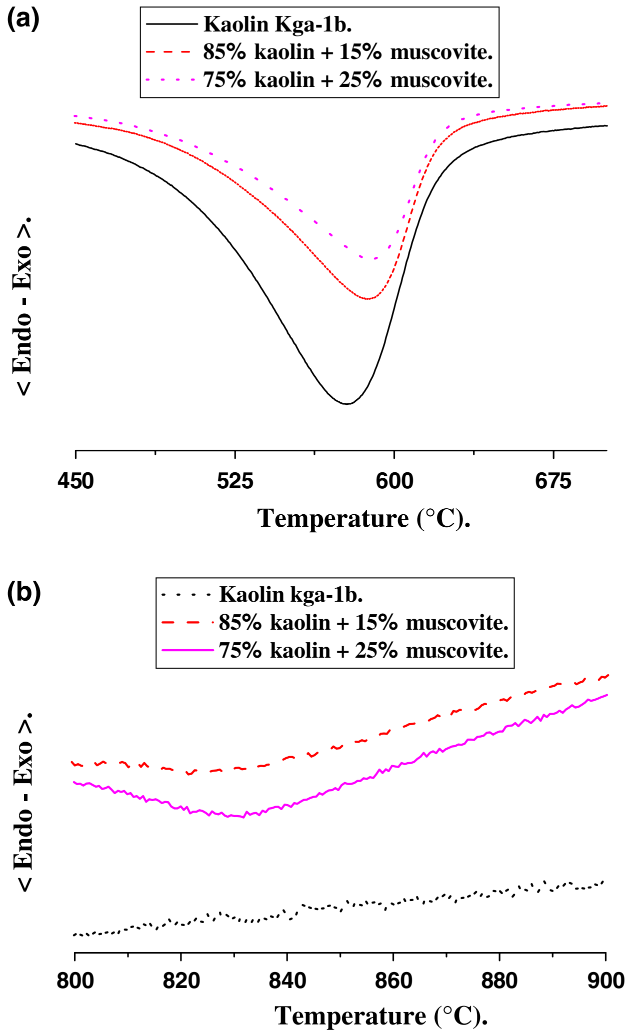

Fig. 2 Evolution of the endothermic peaks of kaolinite dehydroxylation (**a**) and muscovite dehydroxylation (**b**) with muscovite content in our mixes (heating rate of 10 °C/mn)

The latter result is coherent with values reported in literature [35] (in the range 170–200 kJ/mol).

Table 2 Enthalpy variations evaluated by peak surface area for kaolin kga-1b dehydroxylation and structural reorganization

Heating rate (°C/mn)	$\Delta H_{\text{Dehydroxylation}}$ (±7 kJ/mol kaolinite)	$\Delta H_{\text{Reorganization}}$ (±1 kJ/mol kaolinite)
20	194	–36
10	200	–35
5	202	–30
3	207	–31

Table 3 Kaolinite dehydroxylation and structural reorganization enthalpy variations evaluated by peak surface area for the different kaolinite–muscovite mixture (heating rate = 10 °C/mn)

Muscovite content (mass %)	$\Delta H_{\text{Dehydroxylation}}$ (±1 kJ/mol kaolinite)	$\Delta H_{\text{Reorganization}}$ (±1 kJ/mol kaolinite)
0	200	–35
5	197	–16
10	199	–15
15	199	–15
20	197	–15
25	199	–15

Table 4 Apparent activation energy values obtained by Kissinger method

Kaolin (mass %)	100	95	90	85	80	75
Muscovite (mass %)	0	5	10	15	20	25
$E_{\text{reorganization}}$ (±50 kJ/mol kaolinite)	900	1050	960	1020	1040	1045

For kaolinite–muscovite mixtures, it was possible to evaluated the enthalpy variation associated to kaolinite dehydroxylation. Results obtained are reported in Table 3. The enthalpy variation values are almost constant despite the increasing muscovite content. As a matter of fact, the association of the quantitative and qualitative analysis leads to the conclusion that there is no evidence of any interaction between kaolinite and muscovite in this temperature range (Table 4).

Evolution between 900 °C and 1,100 °C

The only phenomenon detected here by DTA is an exothermic transformation related to the structural reorganization of metakaolinite phase. As shown on Fig. 3, this

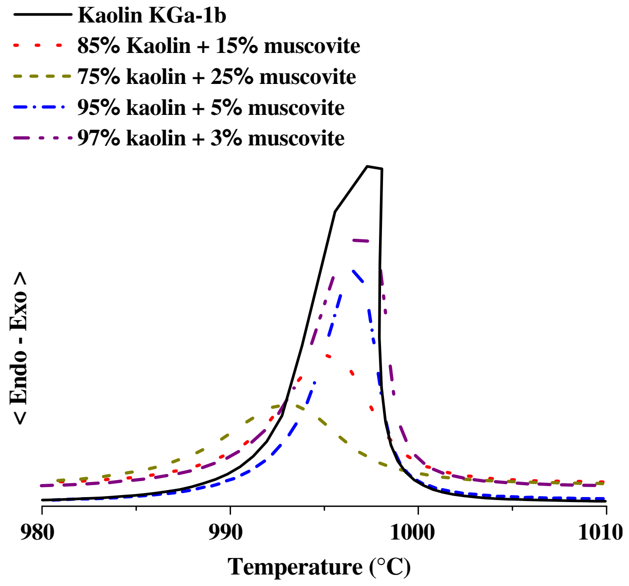

Fig. 3 Influence of muscovite on the exothermic peak associated to the structural reorganization of metakaolinite(heating rate of 10 °C/mn)

exothermic peak is strongly affected by the presence of muscovite even in small quantities. Its intensity does no longer exhibit a linear dependence with respect to the amount of reacting metakaolinite. But we also noticed that this decrease in peak intensity was not proportional to the muscovite content of the studied samples. Besides, there is a slight displacement of the exothermic peak towards lower temperatures (Fig. 4). According, to these observations, one can suggest that the two phyllosicates are likely to intereact at temperatures greater than 900 °C. Thus in this temperature range, the interaction betwween kaolinite and muscovite leads to a significant modification of the exothermic phenomenon.

The application of quantitative analysis leads to heat released values presented in Table 2. For pure kaolin KGa-1b, the enthalpy variation related to structural reorganization is about –32 ± 1 kJ/mol of kaolinite. These results deduced from peak surface area calculations are in good agreement with those proposed in literature [35], ranging from 0 kJ/mol to –32 kJ/mol of kaolinite, depending on the impurities and on the crystallinity of the starting kaolinite and also on the experimental conditions.

For Kga-1b-MBH mixtures, it appears that the addition of only 5 mass % of muscovite leads to a change in the enthalpy variation associated to the exothermic phenomenon from –32 kJ/mol to –16 kJ/mol. Further increase of muscovite content does not change significantly the latter value which remains around –15 ± 1 kJ/mol. Thus, it implies that the interaction between kaolinite and muscovite is effective during the structural reorganization of metakaolinite, this effect being only due to the presence of muscovite instead of the amount of muscovite. Therefore, muscovite seems to act as a modifier in the transformation process of metakaolinite even in small proportion. A similar enthalpy variation have been observed in iron doped kaolinite [36] where the enthalpy released varies from –32 to –29 kJ/mol of kaolinite with iron addition, after what further increase of iron content does not have any additional effect.

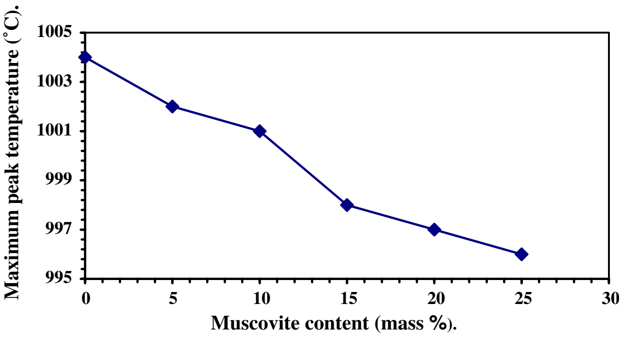

Fig. 4 Maximum peak displacement (for the structural reorganization transformation) with muscovite content at a heating rate of 10 °C/mn

Discussion

It is evident from the obtained result that there is a strong influence of muscovite mineral on the structural reorganization of metakaolinite. But there still an uncertainty concerning the approximate temperature at which this interaction begins. Actually, only one element permits to differentiate qualitatively kaolinite from muscovite, and this element is potassium. Therefore, it was interesting to follow the probable diffusion of potassium ions in metakaolinite. For this purpose, specific samples were elaborated, they consist of muscovite platelets sandwiched between two kaolin layers (Fig. 5a). The elaboration of these samples implies the preparation of two adequate suspensions of kaolin KGa-1b and muscovite MBH. After a good homogenization, one half of the kaolin suspension was slip-casted in a plaster mold, a few minutes later, the muscovite suspension was poured over the kaolin layer and one minute later, the second-half of kaolin suspension was casted over the muscovite layer. The whole sample was air-dried then oven-dried at 120 °C for about 48 h. The obtained product was heat treated at several temperatures for 2 h. EDS analysis was performed on polished surfaces of the samples heated at 900, 925 and 990 °C and the results are shown on Fig. 5. After 2 h at 900 °C (Fig. 5c), there is no evidence for potassium diffusion in metakaolinite. But after 2 h at 925 and 990 °C, potassium is detected in metakaolinite (see Fig. 5d, e).

These observations comfort the results of qualitative and quantitative analysis by DTA, pointing out the fact that the interaction between the two phyllosilicates begins above 900 °C and is enhanced with temperature increase. This behavior can be explained by the fact that below 900 °C, kaolinite and muscovite are subjected to dehydroxylation processes. After theses dehydroxylation processes, the structures are weaken, mainly in the case of metakaolinite. Otherwise, XRPD study conducted on Kga-1b-MBH mixtures has evidenced that in high temperature muscovite, the potassium ions despite their presence in the structure are not strongly linked [37]. Combining to this point the higher diffusion coefficient of potassium ions compare to those of the others constitutive ions (Si^{4+}, Al^{3+}), one can easily understand the diffusion of potassium in metakaolinite layers above 900 °C.

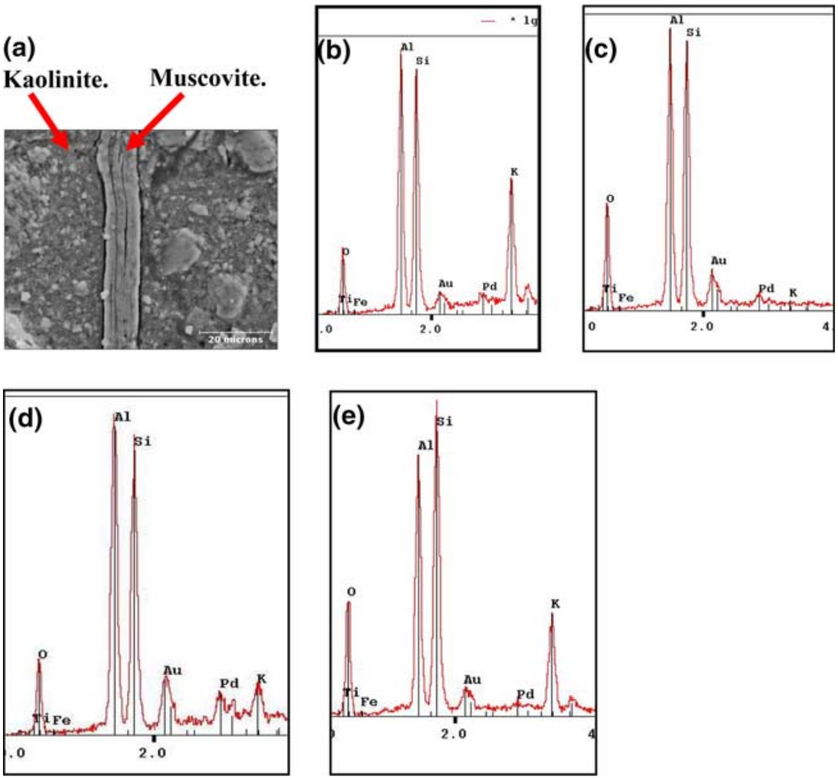

Fig. 5 SEM observation of samples heated at 900 °C for 2 h and analyzed by EDS (**a**) and qualitative EDS analysis near kaolinite–muscovite interface in muscovite zone (**b**) and in metakaolinite region at 2 μm from the interface (**c**, **d**, **e**) are respectively the EDS analysis of metakoalinite zone at 4 μm from the interface of a sample for a heat treatment at 925 °C and at 10 μm from the interface on a sample heated at 990 °C for 2 h

Is there any correlation between these diffusion processes and the enthalpy variations observed during the structural reorganization of metakaolinite? In order to propose an answer to this question, one should considered the case of structural reorganization in kaolin minerals without impurities. It has been shown that this exothermic transformation proceeds through an immiscibility phase separation mechanism [38], which gives rise to interconnected alumina-rich and silica-rich regions from which mullite crystallizes. This mechanism consist of an evolution through a spinodal demixion (athermic), followed by a spontaneous cooperative demixion of the whole compound (strongly exothermic) leading to a release of energy which is measured by DTA or DSC experiment. Thus, considering the immiscibility zone pointed out by us [39] on a vertical section of the ternary diagram SiO_2–Al_2O_3–K_2O, composition zone of the studied mixtures, it is obvious to supposed that the transformation process of these mixtures is similar to that of kaolinite minerals. Therefore, the main difference will be the presence of potassium in metakaolinite structure. Since metakaolinite is a quasi-amorphous compound, it can be treated as a silicate glass in which potassium ions act as modifiers by bridging Al–O bonds. In fact, the whole structure is destabilized and the diffusion of the other positive ions (Al^{3+} and Si^{4+}) is enhanced, leading to a more important contribution of the spinodal demixion compare to cooperative demixion contribution in the reorganization process. As a matter of fact, there should be less energy release (cooperative demixion) than in the case of pure metakaolinite as observed in this study.

Besides the decrease of the energy released during structural reorganization, because of potassium diffusion, we also noticed an early crystallization of mullite in KGa-1b-MBH mixtures through XRPD analysis. As shown on Fig. 6, characteristic peaks of mullite are observed at 1,050 °C in kaolinite–muscovite mixes whereas in the pure kaolin and muscovite minerals,

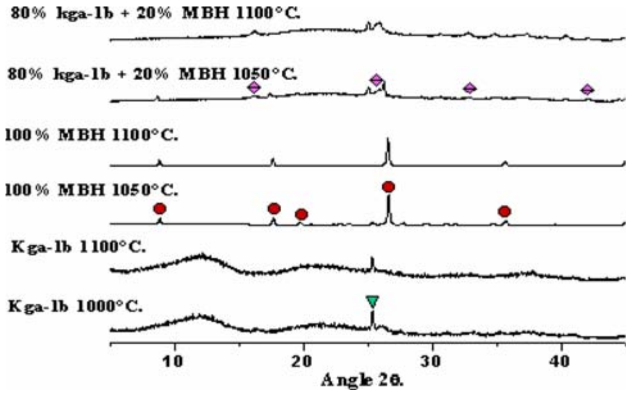

Fig. 6 XRPD diagrams of raw materials and kaolinite–muscovite mixes heated between 1,000 and 1,100 °C for 2 h. (▼) Anatase; (●) Muscovite; (◆) Mullite

one should reach 1,150 °C in order to detect mullite peaks. These observations are in agreement with those of Brindley and Udagawa [40, 41]. They evidenced that mullite developps at lower temperature (viz 1,000 °C) with muscovite increase in kaolinite–muscovite-quartz mixtures. A SEM observation has been performed at kaolinite–muscovite interface after a heat treatment of 2 h at 1,050 °C followed by a chemical etching with fluorhydric acid (Fig. 7). At this interface, mullite needles appear clearly and they seem to grow along three preferential directions. This enhancement of mullite crystallization and growth in KGa-1b-MBH mixtures can also be correlated to the observed diffusion of potassium ions in metakaolinite structure. In fact, the mean size of the starting kaolin material is about 2 µm (Fig. 8) and therefore is smaller than the diffusion distances observed by EDS analysis (respectively 4 µm and 14 µm around the interface at 925 and 990 °C). Thus, it is obvious that the diffusion of potassium ions occurs almost in the whole kaolinite platelets, inducing a perturbation in the system through the enhancement of Al^{3+} and Si^{4+} diffusion, which favors the release of less energy and the early nucleation and growth of mullite.

Conclusion

Kaolinite–muscovite associations behavior has been investigated from room temperature to 1,100 °C in order to observed the effect of muscovite on the thermal transformations of kaolinite. For this purpose, qualitative and quantitative DTA associated with EDS and XRPD analysis were used. The evaluation of the energies involved in the various reactions was performed using the classic peak surface area calculation and the Kissinger theory.

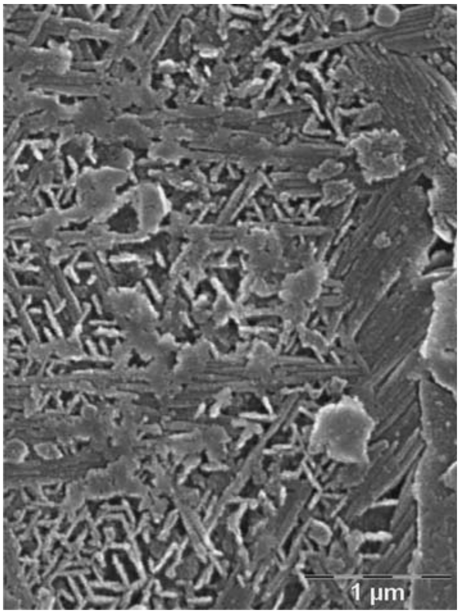

Fig. 7 SEM observation of kaolinite–muscovite interface after a heat treatment of 2 h at 1,050 °C and a chemical etching

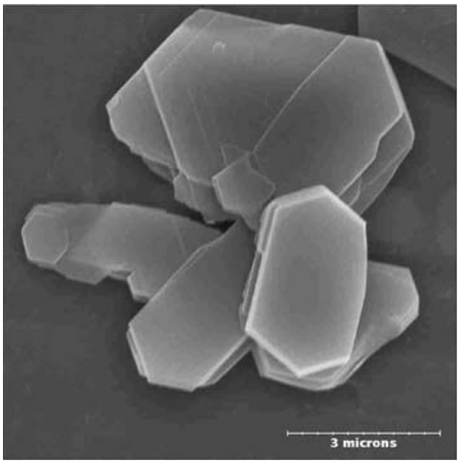

Fig. 8 SEM image of the starting kaolin powder

The results obtained up to 900 °C were in good agreement with literature and indicate the absence of interaction between the two clay minerals. This conclusion is also supported by EDS analysis which shows no potassium diffusion in metakaolinite layers.

Between 900 °C and 1,100 °C, the main transformation is the structural reorganization of metakaolinite characterized by an exothermic peak around 980–1,000 °C. Qualitatively, there is a slight displacement of the exothermic phenomenon to lower temperature and a great decrease of peak intensity with increasing muscovite content. Enthalpy variation values obtained from peak surface area measurements are in good agreement with literature and tend to decrease in presence of muscovite. Further increase of muscovite content does not lead to more significant changes. These observations suggest the existence of interaction between metakaolinite and muscovite above 900 °C as confirm by EDS analysis results. In fact, there is a diffusion of potassium ions in metakaolinite layers. Therefore, the part of spinodal demixion mechanism is increased through the enhancement of the diffusion of aluminum and silicon ions in the system. It results in a release of less energy associated to the part of cooperative demixion mechanism during the structural reorganization of our mixtures than in the case of pure kaolinite mineral. The other consequence of potassium diffusion into metakaolinite structure is the enhancement of an early (1,050 °C) mullite crystallization and grain growth.

Acknowledgements We are grateful to Pr. Yvon Jacques of LEM (Laboratoire d'Environnement et de Minéralurgie) laboratory of Nancy university for providing muscovite raw material.

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