



The role of microstrain on the thermostructural behaviour of industrial kaolin deformed by ball milling at low mechanical load

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ABSTRACT

The high-temperature structural behaviour of kaolin deformed by compaction and shear in a ball mill was investigated by extending the temperature range to 1500 °C. The deformation was induced for the first time with steps of low mechanical load through a specifically built planetary ball milling working in a controlled thermodynamic environment (25 °C and at a vacuum of 0.13 Pa). The investigated kaolin was made of about 65% of a well-ordered kaolinite, 10% of dickite and 25% of quartz. The mechanical energy transfer to the material was measured via the microstrain $\langle \varepsilon^2 \rangle^{1/2}$ because we wanted to investigate the details of the evolution of the kaolin modification as a function of the microstrain.

At the very early stage of milling (up to 1 h), no strain was accumulated in the kaolinite structure which, however, presented lamination, layer flattening and texturing. Further milling induced a progressive reduction of the stacking layer coherence and an increase of the microstrain, in both cases as a non linear function of the deformation time.

The thermo-structural behaviour of kaolin was investigated by TG-DTA in a helium atmosphere at 10 °C/min of heating rate. In the medium temperature range (400–1000 °C), the mechanical milling affected the dehydroxylation reaction of kaolinite by a significant anticipation of about 300 °C of the temperature range, which usually occurs between 400 and 800 °C. Other reactions related to the formation of a Si-spinel and other intermediate phases were observed. The mechanical deformation severely influenced of the high-temperature reactions related to mullite and cristobalite formation. All thermal reactions linearly correlate to the microstrain $\langle \varepsilon^2 \rangle^{1/2}$ accumulated in the kaolinite structure. The reported data are of particular usefulness in industrial applications involving grinding or milling of kaolin.

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1. Introduction

Kaolinite is widely used in many applications, such as cement and ceramic processing, as a filler in polymer-nanocomposite production, in paints, paper and rubber (see for a review Murray et al. 1993). A lot of physical and chemical properties of kaolinite are related to its surface reactivity which may be improved by particle size reduction obtained by industrial grinding and/or specific micronisation processes. Industrial grinding represents a high cost-effective process because the majority of the mechanical energy is wasted by the grinding devices and only a few percentage is effectively used for the material comminution and deformation (Wills and Napier-Munn 2006).

For these means, the influence of the mechanical deformation on the kaolinite structure and its main physico-chemical properties has been widely investigated (Aglietti et al. 1986b; Frost et al., 2001a, 2001b; Juhász 1980; Juhász and Opoczky 1990; Mako et al. 2006).

However, the deformation was induced by using various types of grinding devices that rendered the comparison of the results difficult. In addition, in some cases the structural properties of kaolinite were investigated after large amounts of mechanical energy load had been transferred to the material, which had caused fast amorphisation in short time periods (Frost et al. 2001a; Kristof et al. 1993; Mako et al. 2006).

Furthermore, the effect of mechanical deformation on the thermal behaviour of kaolinite was investigated only up to about 1000 °C and was generally ascribed to a decrease in the dehydroxylation temperature reaction and an expectation of medium-high temperature phase recrystallisation (Aglietti et al. 1986a; Frost et al. 2001a; Gonzalez Garcia et al. 1991; Horvath et al. 2003; Miller and Oulton 1970; Suraj et al. 1997).

Further, some authors (Gonzalez Garcia et al. 1991; Mako et al. 2006; Reynolds and Bish 2002) have investigated the role of the crystallinity degree of kaolinite in the grinding process, whereas others have shown that a high content of quartz (<50%) contributed to the grinding favouring the amorphisation of the kaolinite (Mako et al. 2001).

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The literature does not provide details of the evolution of the thermo-structural behaviour of kaolinite induced by mechanical deformation performed in a controlled environment. Furthermore, the thermo-structural behaviour at very high temperature (1500 °C) close to the melting point has not so far been investigated.

A salient feature of our study is the use of low mechanical loads which provide a better understanding of the fine details of the structural transformation minimising amorphisation. We used the microstrain $\langle \varepsilon^2 \rangle^{1/2}$ to monitor the mechanical energy transferred to the material as this parameter is best suited for future comparison of results obtained with different experimental set-ups, as already implemented for talc (Dellisanti and Valdrè 2010).

Furthermore, for the first time, the thermal behaviour of deformed kaolinite was investigated to 1500 °C by TG-DTG and DTA to understand the mechanism of the high temperature phase transformations related to the microstrain $\langle \varepsilon^2 \rangle^{1/2}$ which may be useful for technological purposes.

The deformation of kaolinite was induced by using a specifically built planetary ball mill working in controlled environmental conditions at room temperature of 25 °C and in a vacuum of 0.13 Pa, as already implemented on other layer silicates (Christidis et al. 2005; Dellisanti et al. 2006; Dellisanti and Valdrè 2010; Dellisanti et al. 2009).

A detailed study of the high temperature behaviour up to 1400 °C of kaolinite was performed only on undeformed kaolinite (Bellotto et al. 1995; Chakraborty 2003; Gualtieri and Bellotto 1998; Gualtieri et al. 1995; Michot et al. 2008).

2. Materials and methods

A commercial powdered kaolin containing about 65% of a well-ordered kaolinite (Hinckley index of 1.3), 10% of dickite and 25% of quartz, was used for the investigation. Microchemistry was checked by X-ray microanalysis (Gatti et al. 1996). Kaolin was mechanically deformed via a planetary ball mill (Pulverisette 5, Fritsch, Germany) which was home-modified for work at room temperature (25 °C) and under medium/high-vacuum of 0.13 Pa, using specifically built air-cooled mechanical jar-reactors. This system has already been described and successfully applied for the mechanical deformation of other compounds and minerals such as smectites, talc, pure metals (Ni, Fe) and Zr-alloys and it was successful to control the crystallite size and minimise the amount of amorphous material produced during milling (Bonetti et al. 1998; Dellisanti and Valdrè 2005; Valdrè et al. 1999). The mechanical apparatus induces simultaneous compaction and shear to the material. Kaolin was treated for 1, 5, 10 and 20 h and the samples hereinafter called 0-h (untreated), 1-h, 5-h, 10-h and 20-h respectively. The effect of the ball impact to the structure of kaolinite was so low that no strain accumulated up to 1 h of milling. The work conditions were controlled by adjusting the following parameters: type of vials and balls, spinning velocity, ball weight and size and vial environment.

Mineralogical and crystallographical data were obtained by X-ray powder diffraction (XRD) by using a PANalytical X'Pert Pro system, equipped with PIXCel detector with graphite monochromator, under the following conditions: CuK α radiation; power supply 40 kV/30 mA; divergence and detector slits both of 1°; angular range 5–65° (2 θ) at 0.02° (2 θ) step size and counting time of 1 s/step. Analyses were carried out on quasi-randomly oriented powder specimen prepared by filling a side-entry aluminium holder.

XRD quantitative analysis was performed by using X Powder Plus (Martin 2004), which uses methods of non-linear least square for full profile diffractograms. The quantitative analysis is achieved with a modification of the normalised RIR (Reference Intensity Ratio) method (Chung 1974; Martin 2004).

Mineralogical parameters were determined by processing the XRD patterns by the Winfit programme (Krumm 1996) taking into

account the instrumental broadening. Hinckley index (HI), used to characterise the crystallinity degree of kaolinite, was measured by using (110) and (111) reflections peaks following the method proposed by Hinckley (1963).

The crystallite thickness, given as coherent scattering domain (CSD) along c-axis, and the corresponding microstrain $\langle \varepsilon^2 \rangle^{1/2}$ were determined by using the MudMaster® programme (Eberl et al. 1996) based on the Bertaut–Warren–Averbach (BWA) method as described by Drits et al. (1998). The microstrain, measured in nm units, represents the “averaged” crystal deformation calculated along the c-direction (00 l). We have applied the BWA method to three family reflections 001, 002 and 003. The BWA method is based on the analysis of the Fourier coefficients resolved for the microstrain contribution to the diffracted peaks. However, a detailed mathematical description of the BWA method is beyond the scope of the present work and the interested reader may refer to specific publications (Drits et al. 1998; Klug and Alexander 1974).

Some XRD analyses were performed also on samples treated at a constant temperature of 1050 °C overnight (about 14 h) and on samples heated up to 1500 °C at 10 °C/min followed by a quenching at a rate of about 50 °C/s by using a cold helium gas at RT, flowing at 1 ml/s.

TG-DTG-DTA thermal behaviour was studied in helium atmosphere by using a Setaram Labsys™ double furnace apparatus (temperature range 50–1500 °C; heating rate 10 °C/min; platinum crucibles; calcined Al₂O₃ as reference substance; flow rate of helium 0.27 ml/s; temperature accuracy about ± 1 °C). BaCO₃ was used as a calibration standard for DTA.

Infra-red spectra were obtained by using a Nicolet Avatar 360 Fourier Transform Infra Red (FT-IR) spectrometer, using a spectral resolution of 4 cm⁻¹ and 64 accumulations per spectrum. A mixture of about 1 mg of kaolin and 200 mg of KBr was prepared to obtain pellets of 13 mm diameter under a pressure of 8 metric tonnes/cm². Before analysis the pellets were heated overnight at 110 °C to remove absorbed water. Mineral bands were assigned according to Farmer (1974), Farmer and Russell (1964) and Madejova and Komadel (2001).

The particle size distribution was determined by a Fritsch Laser Particle Sizer Analysette 22 which uses Mie patterns obtained by laser light scattering with the particles dispersed in water. The measuring range was 0.15–500 μ m (equivalent spherical diameter). The results are given as volume frequency in percent vs. particles size.

3. Results

3.1. Coherent scattering domain, microstrain, FTIR and particle size distribution

Fig. 1 shows the XRD patterns of kaolin before and after a number of hours of mechanical milling. Fig. 2 shows the coherent scattering domain (CSD) and microstrain $\langle \varepsilon^2 \rangle^{1/2}$ along c-axis of kaolinite, derived from the spectra of Fig. 1 using the Bertaut–Warren–Averbach method.

At the very early stages of milling (up to 1 h) the CSD remained constant and no microstrain accumulated in the kaolinite structure.

Significant changes occurred at 5 and 10 h of milling, with a progressive intensity decrease and simultaneous broadening of the (00 l) basal diffraction peaks. Consequently, a reduction of CSD and an increase of the microstrain $\langle \varepsilon^2 \rangle^{1/2}$ was measured (Figs. 1, 2). Prolonged milling up to 20 h did not induce any further structural modification. CSD and microstrain of kaolinite reached a level of saturation (Figs. 1, 2). Slight amorphisation of kaolinite maybe present in the 20-h sample as suggested by a low bump background between 15 and 30 2 θ ° (see the inset of Fig. 1).

FTIR analysis (Fig. 3) is consistent with the previous results. As already investigated by various authors (see for instance, Frost and Van der Gaast 1997), Kaolinite presents three FTIR peaks relative to

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