

## Development of Young's Modulus of Illitic Clay during Heating up to 1100 °C

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Young's modulus of green illite from Füzéradvány (Hungary) was measured in-situ in the temperature interval 20 °C–1100 °C and auxiliary analyses DTA, TG and TDA, XRD and EGA, were also performed. It was found that a removal of the physically bound water (20 °C–250 °C) sets illite crystals closer, which leads to a significant increase of Young's modulus from its initial value of 7 GPa to the maximum 12 GPa at 300 °C. Young's modulus slightly decreases in the temperature interval of dehydroxylation of illite (300 °C–800 °C), and which then reaches up to 45 GPa at 1100 °C, increasing exponentially as a consequence of the sintering above 800 °C.

**Keywords:** illite; Young's modulus; physically bound water; dehydroxylation; sintering.

### 1. INTRODUCTION

Illite is a significant rock forming mineral that is a main component of illitic clay and soil. Its structure consists of the repetition of tetrahedron–octahedron–tetrahedron (T-O-T) sheets in one layer. The interlayer space is occupied mainly by potassium cations, which are responsible for the absence of swelling. Additionally the variable amounts of water molecules lie between the T-O-T layers [1–4].

Illitic clays, also called ball clays, are very often used in the manufacturing of traditional ceramics [5–9] and geopolymers [10]. Illite is usually mixed not only with other clays but also with microscopic crystals of carbonates, feldspars, micas and quartz and is very rarely found separately in nature.

Almost pure illite was found in the Füzéradvány location in North-Eastern Hungary [11, 12]. This illite was previously studied in [3–5, 11, 13, 14] and in other works. It was specified that this illite is of 1M polytype [3, 4]. According to [11] the illite contains 48.51 mass % of SiO<sub>2</sub>, 31.26 mass % of Al<sub>2</sub>O<sub>3</sub>, 7.85 mass % of K<sub>2</sub>O and ~ 4.3 mass % of Fe, Ti, Ca, Mg, Na and S oxides. Loss on ignition is ~ 6.9 mass %. The SEM images show individual grains with highly altered or damaged rim structures. In general, mostly hexagonal grains are separated by fractures with variable widths between 1 and 10 μm. The presence of a minor amount of quartz was seen in the CL images and was also confirmed by XRD [15].

Generally, during heating up to 1200 °C, illite goes through several changes. The first of which is a loss of physically bound water which takes place between room temperature and 300 °C [16, 17]. Because H<sub>2</sub>O is bound to T-O-T clay minerals in different ways, the loss of the

physically bound water occurs in overlapping temperature intervals. For illite, two DTA endothermic minima were observed in this temperature region [16].

The next process is dihydroxylation, which begins at ~ 450 °C [2, 13, 16–18] and is accompanied with mass loss of 5.2–5.6 mass % [17] and no shrinking [17–19]. It is still not possible to reach a unique definition because there are several possible models of the mechanism reaction of the illite dehydroxylation [13]. An Arrhenius plot showed two slopes which gave two values of the activation energy 697 kJ mol<sup>-1</sup> (for higher temperatures) and 231 kJ mol<sup>-1</sup> (for lower temperatures) [13].

Illite peaks in XRD pattern are present during heating up to ~ 950 °C when begin to diminish and disappear at ~ 1050 °C [9, 14, 18]. The high-temperature reactions are accompanied with steep contraction [14, 19] and new phases are formed. A quantitative XRD analysis gave a mineralogical composition of the Füzéradvány body fired at 1080 °C: quartz (1.9–7.9 %), Ca-plagioclase (1.2 %), γ-alumina (9.8–11.9 %), mullite (4.5–5.8 %), K-feldspar (1.2–2.1 %), and amorphous phase (75.4–77.1 %) [14].

Elastic properties of illite determined by some authors are listed in Table 1 compiled from data given in [20–24]. All data in Table 1 were measured at room temperature using green illite samples. The values, with the exception of [23, 24], are calculated for illite without pores.

From the ceramic point of view, some part of illite in the kaolin-based ceramic mixture increases a content of the glassy phase as a consequence of the fluxing action of K<sub>2</sub>O [5, 6] and decreases the firing temperature [25]. Increasing content of illite in such mixtures results in a decrease of mullite, cristobalite and quartz in the fired products [5]. Illite continues to expand up to the sintering and the shrinkage starts at the same time as vitrification. In this temperature range, the mechanical strength rapidly increases and the material becomes plastic. This is a reason why illite (or illitic clay) is preferred for a fast firing [19].

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**Table 1.** Isotropic elastic modulus [GPa] and Poisson's ratio of illite

	Bulk modulus (K)	Shear modulus (G)	Young's modulus (E)	Poisson's ratio ( $\mu$ )
[20]	60.1	25.3	66.6 <sup>1</sup>	0.316 <sup>2</sup>
[21]	60.2	25.4	66.8 <sup>2</sup>	0.315 <sup>2</sup>
[22]	62.2	25.7	67.8 <sup>1</sup>	0.319 <sup>2</sup>
[23]	6-12 <sup>3</sup>	4-6 <sup>3</sup>	10-15 <sup>1</sup>	-
[24]	-	-	10-15 <sup>1</sup>	-

<sup>1</sup> calculated from  $K$  and  $G$  as  $E = 9KG/(3K + G)$ ,  
<sup>2</sup> calculated from  $G$  and  $E$  as  $\mu = (E/2G) - 1$ ,  
<sup>3</sup> for clay generally.

As follows from the references [1–4, 13, 18], the main focus of the previous research was the structure of illite, its reactions to dehydroxylation and high temperature. The mechanical properties of illite during heating remain unknown. The aim of this work is experimental study of the elastic properties of illite measured in-situ during heating.

## 2. EXPERIMENTAL

### Samples

Samples made from raw illite, supplied from the mine in Füzérvány, were used for thermal analyses DTA, TG, TDA and mf-TMA (modulated-force thermomechanical analysis) during heating. The composition of illitic clay, as given by a supplier [25], is shown in Table 2.

**Table 2.** The chemical composition of illite from Füzérvány, Hungary (in mass %)

SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	L.O.I.
58.0	24.0	0.6	0.05	0.38	1.70	7.85	0.10	7.3

The raw illite, which was in pieces of 0.2–3 cm, was firstly crushed and then sieved to obtain a powder with particles not larger than 0.1 mm. Then 750 g of the powder were mixed with 250 g of the distilled water to obtain a plastic mass. Cylindrical samples were made from this mass with a laboratory extruder. After open air free drying, the samples contained ~ 3 mass % of the physically bound water and their diameter was ~ 11 mm.

A heating rate (5 °C min<sup>-1</sup>) was the same for all thermal analyses. The green samples were heated in the air from room temperature up to 1100 °C, which is the presumptive highest firing temperature used in industries for building clay ceramics.

### Modulated-force thermomechanical analysis (mf-TMA)

Modulated-force thermomechanical analysis, based on free vibration of the sample in a fundamental flexural mode, was used for a measurement of Young's modulus on the cylindrical samples with initial dimensions  $\varnothing 11 \times 120$  mm during heating from 20 °C to 1100 °C with a rate of 5 °C min<sup>-1</sup>. The vibrations were excited with an electromagnetic impactor and detected with a microphone. A resonant frequency of the vibrations was obtained using the fast Fourier transformation. The resonant frequency  $f$ ,

the length  $l$ , diameter  $d$ , and the mass  $m$  of the sample were used for calculating  $E$  according to [26, 27]. Since the ratio length/diameter = 10.9 < 20, the value of  $E$  must be multiplied with a correction coefficient  $T_{1c} = 1.04402$  (calculated for a circular cross-section and Poisson's ratio  $\mu = 0.3$ ). Young's modulus at the temperature  $t$  is

$$E = 1.6774 \frac{m_0 l_0^3 [1 + \Delta m(t)/m_0]}{d_0^4 [1 + \Delta l(t)/l_0]} f^2(t), \quad (1)$$

where  $\Delta l(t)/l_0$ ,  $\Delta m(t)/m_0$ , are relative linear thermal expansion and relative mass change of the sample measured by dilatometer and TG analyzer respectively. Deriving Eq. (1), the equal relative expansion of the sample in both directions, axial and radial, was presumed. The values  $l_0$ ,  $d_0$ ,  $m_0$  are the initial length, diameter, and mass of the sample at room temperature, respectively, and  $f(t)$  is the resonant frequency at the temperature  $t$ .

### Differential thermal analysis (DTA), thermogravimetry (TG) and thermodilatometric analysis (TDA)

As follows from Eq. (1), the determination of  $E$  requires measuring the actual dimensions and mass of the sample during heating. This task is done by TD and TG analyses. Given the relative expansion  $\Delta l(t)/l_0$ , as measured by the dilatometer, and the initial dimensions  $l_0$ ,  $d_0$ , the true length  $l(t)$  and diameter  $d(t)$  of the sample were calculated. Similarly, the true mass  $m(t)$  was determined from the relative mass change  $\Delta m(t)/m_0$  measured with TGA/DTA analyzer Derivatograph 1000 [28].

To reduce the temperature shift between TG, DTA, TDA and mf-TMA curves, the samples for these analyses had the same cross-section. The sample dimensions were  $\varnothing 11 \times 40$  mm for TDA and  $\varnothing 11 \times 20$  mm for TG and DTA. A reference compact sample for DTA had the dimensions of  $\varnothing 11 \times 20$  mm and was made from pressed Al<sub>2</sub>O<sub>3</sub> powder.

### X-ray diffractometry (XRD) analysis

The XRD analysis of the powder samples was performed with the diffractometer BRUKER D8 Advance with Cu anticathode ( $\lambda_{\alpha_1} = 1.5406$  Å), accelerating voltage 40 kV and beam current 40 mA. Data was obtained with the BRUKER LynxEye detector. The Rietveld method was used for a quantitative XRD analysis.

### Evolved gas analysis (EGA)

The evolved gas analysis was performed on a powder sample (50 mg) using Setaram LabSys 1600 thermoanalyzer coupled with Pfeiffer Omnistar Mass Spectrometer in the atmosphere 79 % of Ar + 21 % of O<sub>2</sub> with flow rate of 60 ml min<sup>-1</sup>.

### Porosity

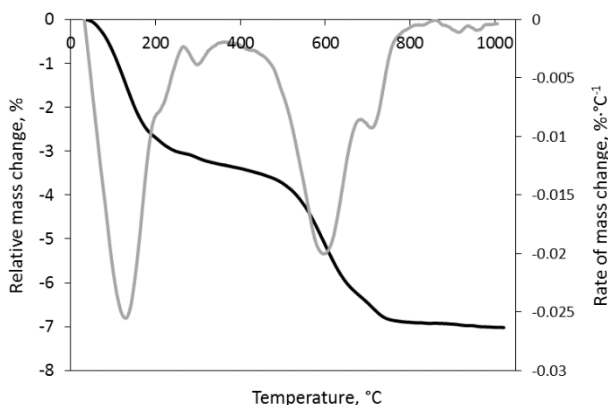
The porosity was calculated with the help of experimentally determined bulk density and matrix density. The bulk density was obtained by means of size and weight measurements of cylindrical samples. The matrix density was measured by the helium pycnometry (Pycnomatic ATC, Porotec).

### 3. RESULTS AND DISCUSSION

The results of the quantitative XRD analysis conducted at room temperature for the green sample and the samples fired at the temperatures 650 °C, 750 °C and 1100 °C are given in Table 3. The green sample contains illite as a main phase with an addition of 12 mass % of quartz and small amounts of montmorillonite and K-feldspar orthoclase. As the firing temperature increases, the amount of illite decreases, while the amount of muscovite and amorphous phase increases. In the sample fired at 1100 °C, no phyllosilicates are observed. The sum of parts of illite, muscovite and amorphous phase is approximately constant (80 mass % for the green sample, 80 mass % for the sample fired at 650 °C, 82 mass % for the sample fired at 750 °C and 79 mass % for the sample fired at 1100 °C). The transformation of illite at higher temperatures can be a source of muscovite [29]. The cause of the amorphous phase in the samples fired at 650 °C and 750 °C is probably also due to thermal changes of illite/muscovite. The amorphous phase in the sample fired at 1100 °C is a glassy phase. K-feldspar (orthoclase and sanidine) withstands thermal treatment and does not create the glassy phase. Mullite is also created at the highest firing temperature.

**Table 3.** Mineral composition of the samples in mass %

	green	650 °C	750 °C	1100 °C
Illite	80	69	34	0
Montmorillonite	4	3	4	0
Muscovite	0	7	35	0
Quartz	12	12	11	11
Orthoclase	4	5	4	2
Sanidine	0	0	0	4
Mullite	0	0	0	4
Amorphous	0	4	13	79



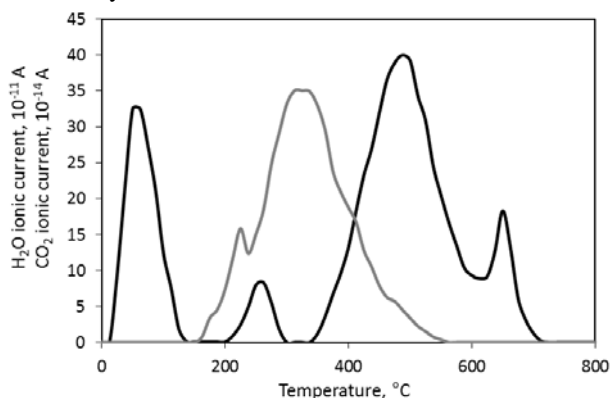
**Fig. 1.** Thermogravimetry of illite, TG (black line) and DTG (gray line)

The results of thermogravimetric analysis (Fig. 1.) show three steps of the decrease of the sample mass. The first step is a result of the physically bound H<sub>2</sub>O escaping (between 20 °C and 300 °C) which occurs in two overlapping processes. The escaping of the physically bound water occurs in two overlaying processes. The first

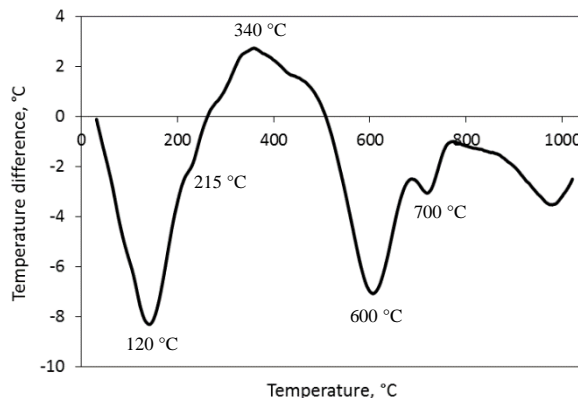
of them, which is more intensive, reaches the maximum rate at ~ 120 °C and the second one at ~ 215 °C. According to [2], a mass loss with small minimum at ~ 300 °C in the DTG curve is related to dehydration when H<sub>2</sub>O molecules located in K-free sites of the illite interlayers are removed.

The second step is a release of the constituent water (between 400 °C and 800 °C). A dehydroxylation of illite runs in two steps which reach the maximum rate at the temperatures ~ 600 °C and ~ 700 °C being in accordance with the results showed in [13].

The results of TG are supported with the mass spectrometry (EGA) where an emission of H<sub>2</sub>O and CO<sub>2</sub> were measured, see Fig. 2. EGA showed that H<sub>2</sub>O is emitted at three clearly separate temperature intervals. The first two, which are connected with the physically bound water, are also registered in DTG and DTA curve where two overlaid processes are expressed with one intensive minimum at ~ 150 °C and one shoulder at ~ 230 °C. The dehydroxylation of illite is registered by EGA between 350 °C and 700 °C as a two-step process which is an agreement with results of TG and DTA. According to EGA, the emission of the small amount of CO<sub>2</sub> is also present and is caused by the burning of the organic matter in illite clay.



**Fig. 2.** Mass spectrometric results for escaping gases H<sub>2</sub>O (black line) and CO<sub>2</sub> (gray line)

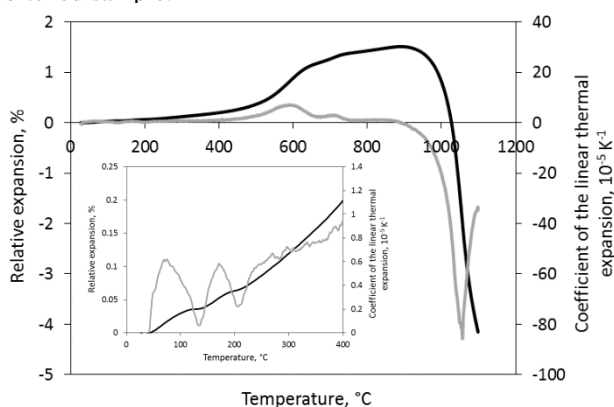


**Fig. 3.** DTA curve of illite

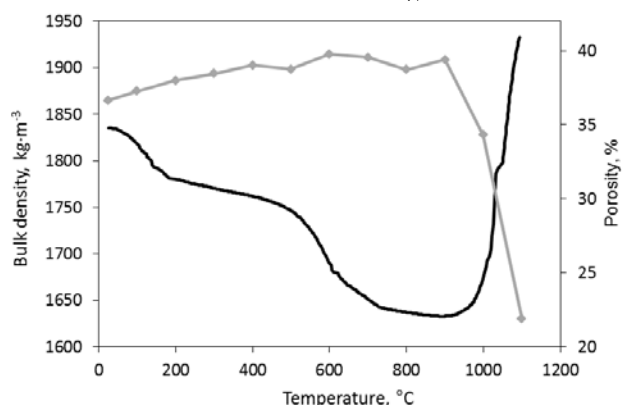
Since these processes are linked with the absorption of heat, they are represented three clear endotherms in the DTA curve with minima at 120 °C, 600 °C and 700 °C and two shoulders at ~ 215 °C and ~ 300 °C, see Fig. 3. Besides that, there is an absorption of heat above 800 °C which results in a change of metacillite into a disordered

structure and also a vitrification of the sample. An exotherm with maxima at  $\sim 340$  °C in DTA curve is caused by the thermooxidation of organic matter.

The results of TDA in the temperature interval between 20 °C and 1100 °C are depicted in Fig. 4, where the interval between 20 °C and 400 °C is enlarged. The sample slightly expands its volume from room temperature up to  $\sim 500$  °C (coefficient of the linear thermal expansion CLTE is  $\sim 1 \times 10^{-5} \text{ K}^{-1}$ ) where the dehydroxylation starts. During the dehydroxylation, the expansion becomes faster and after its completion, the expanding continues with CLTE  $\sim 1 \times 10^{-5} \text{ K}^{-1}$  up to the temperature of 900 °C. TDA also confirms two-step dehydroxylation of illite via two maxima of the CLTE, the first of them is at  $\sim 600$  °C and the second is at  $\sim 700$  °C. This correlates with TG and DTA. Above 900 °C, the TDA curve begins to bend downwards as a result of the sintering and vitrification [19]. Besides that, above 800 °C metacillite began to change into a disorder structure. At  $\sim 1100$  °C, the layer structure of metacillite had been destroyed, and amorphous  $\text{SiO}_2$  and mullite crystallite appeared [18]. These structural changes also led to the contraction of the sample. The TDA curve of the fired sample, Fig. 4, confirms a small amount of quartz via small step around 570 °C. The presence of the glassy phase exhibits itself as a contraction above 1000 °C caused by the press of the dilatometer's push-rod on the softened sample.



**Fig. 4.** Relative expansion (black line) and coefficient of the linear thermal expansion (gray line) of illite. The inset shows the interval 20 – 400 °C in greater details



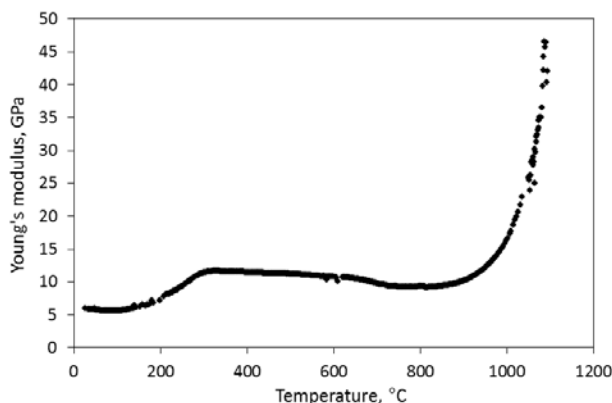
**Fig. 5.** Bulk density (black line) and porosity of illite (gray line)

Since  $E$  is proportional to a bulk density, which follows from Eq. (1), we derived this quantity from results of TG and TDA (Fig. 5.). The bulk density is determined

with the mass loss up to 700 °C, which reaches  $\sim 7$  mass % while the thermal expansion is only 1.4 %. Above 700 °C, the sample mass becomes constant and the bulk density is determined through the thermal contraction only. The contraction is caused by the sintering as noted above.

We also measured the porosity. The obtained results in Fig. 5 show that the porosity of the samples increases up to the firing temperature of 600 °C. Then a mild decrease of the porosity occurs for the samples fired at 700 °C and 800 °C followed by steep decrease of the porosity of samples fired at 1000 °C and 1100 °C, the decrease in the porosity being 17 %.

The relationship between  $E$ , which was calculated by Eq. (1), and the temperature  $t$  is depicted in Fig. 6. The first interval, which characterizes a dehydration at low temperatures up to  $\sim 300$  °C, is typical for samples made from clay materials. We obtained similar pictures for samples made from green electroporcelain mixtures, as well as from kaolin and illitic brick clays [30–32].  $E$  of the samples, which had an equilibrium moisture 1–2 mass% after drying for a long time in the open air, is very sensitive to the loss of this water when the  $\text{H}_2\text{O}$  molecules escape from faces of the illite crystals. This process makes contacts between the crystals tighter which leads to an improvement of the elastic properties. The crystals are held together by ionic forces which are stronger when the crystals are drawn into intimate contact by the departing water films [33]. When we compare the results of mf-TMA and TDA in this low-temperature interval, we find that  $E$  of the sample increases while its volume also increases contrary to theoretical expectation which states that  $E$  is inversely proportional to the thermal expansion. We can conclude that the elastic behavior is much more influenced by properties of the boundaries between crystals than their interiors.



**Fig. 6.** Young's modulus of illite

No phase change takes place between the end of dehydration and the start of dehydroxylation. The linear decrease of  $E$  is caused by the weakening of the interatomic forces because the temperatures are too low for sintering. There is a good correlation with TDA results in the same temperature interval.

Above 500 °C, the dehydroxylation of illite that begins is accompanied with mass change (Fig. 1.) and thermal expansion (Fig. 4.). Dehydroxylation is reflected in  $E$  as a decrease of its values (Fig. 6.). It can be caused by the microporosity which results from simultaneous

increase of the volume of the sample and a decrease of its mass. It also leads to lesser values of the bulk density above 500 °C (Fig. 5.). But  $E$  does not decrease as dramatically as the bulk density does between 500 °C and 700 °C. In spite of the expected weakening of the metaillite crystals,  $E$  decreases only slightly. It also refers to a main role of the contacts between crystals which seems to be more important than mechanical properties of the crystals. And, finally,  $E$  exponentially increases due to sintering above 800 °C. This fact is also confirmed with TDA results.

#### 4. CONCLUSIONS

Young's modulus of green illite from Füzérradvány (Hungary) was measured in the temperature interval 20 °C–1100 °C and auxiliary analyses DTA, TG and TDA, XRD and EGA, were also performed. We found that:

- A removal of the physically bound water (20 °C–250 °C) sets illite crystals closer, which leads to a significant increase of Young's modulus.
- In the next section, between the end of the escape of the physically bound water and the start of the intensive sintering (300 °C–800 °C), the Young's modulus slightly decreases. Dehydroxylation does not significantly influence the development of Young's modulus.
- Young's modulus exponentially increases above 800 °C as a consequence of the sintering.

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