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Methods and equipment for the investigation of rheological properties of complex materials like convectional brick clays and ceramic reinforced composites

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Abstract

In the present work two special instruments are described and introduced which were developed for the rheological tests of materials like minerals, raw materials and semi-finished products of ceramic industry or complex materials like ceramic particles and fibre reinforced metal alloys and hetero-module, hetero-viscous and hetero-plastic materials with increased dynamic strength. The working principles of introduced 'rheotesters' are relatively simple and easy for use to determine rheological parameters like instantaneous elastic modulus, delayed elastic modulus or viscosity of damaged and undamaged material structures. The instruments give the opportunity to easily and quickly prepare the rheological model of tested materials.

Keywords: ceramics, composites, instrument, modulus of elasticity, rheology, viscosity
Kulcsszavak: kerámiák, kompozitok, vizsgáló berendezés, rugalmassági modulus, reológia, viszkozitás

1. Introduction

There are many scientific works can be found recently for the investigation of rheological properties of materials in nanoscale [1-4]. Nevertheless, it is quite difficult to determine in macro-scale the most important rheological parameters of complex materials like

- mined convectional brick clays,
- concrete mixtures reinforced with fibres,
- asphalt pavements and asphalt concretes,
- ceramic particles and ceramic fibre reinforced metallic matrix composites,
- hetero-module, hetero-viscous and hetero-plastic complex materials.

During the production of ceramics and ceramic reinforced composites it is obvious that the chemical and structural transformations in the materials are taking place as reactions in solid phase [5-10]. At a certain temperature and chemical or mineralogical composition the rate of these solid phase reactions are very high depending on the concentration of components and the volumes of their contact surfaces. Due to these, one of the most important technological goals during the production of convectional bricks, ceramic roof tiles, technical ceramics and ceramic reinforced composite materials is to give specific surface area as large as possible for the used raw materials during their crushing and comminuting. The achieved specific surface area of the components has very strong influence not only on forming processes and quality but on the required energy consumption of heat treatment or firing as well [11-15]. To get the necessary magnitude of specific surfaces of the raw materials the required energy consumption depends not only on their chemical and mineralogical

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composition and the working principle of crusher and mixture machines and equipment, but also on physical, mechanical and rheological parameters of the used materials.

The importance of rheological properties of raw materials during production of convectional bricks and ceramic roof tiles was first declared by *Hallmann* [16]. By his determination, most of the raw materials used in the building material industry can be described with the rheological equation of Eq. (1) as:

$$(\tau - \tau_0)^m = \eta(\dot{\varepsilon})^n \quad [\mathrm{Pa}] \tag{1}$$

where:
τ – the shear stress developed in the materials during processing (Pa);
τ_0 – the yield stress or static yield point of used materials (Pa);
η – the dynamic viscosity of materials during processing (Pas);
$\dot{\varepsilon}$- the shear rate developed in the material (s^{-1});
m, n – power law exponents.

Later, many authors have confirmed the above declaration of *Hallmann* during their investigations of rheological parameters of raw materials used for the production of different kinds of building materials. For example melted glasses, cement pastes with high water content, mortars, porcelain and china slurries for slip casting can be determined as:

$$\tau = \eta\dot{\varepsilon} \quad [\mathrm{Pa}] \tag{2}$$

It is obvious that Eq. (2) is generated from Eq. (1) under the following conditions:

$m = 1$, $n = 1$ and $t_0 = 0$ (3)

According to [17] the well prepared conventional brick clay during its extrusion can be characterized with the rheological equation of Eq. (4):

$$\tau - \tau_0 = \eta\dot{\varepsilon} \quad [\mathrm{Pa}] \tag{4}$$

The above equation is also generated from Eq. (1) at boundary conditions of:

$m = 1$ and $n = 1$ (5)

In the 1970s Russian scientists [18-21] have achieved remarkable results in the investigation of mined convectional brick clays with relative water content of $w_r \geq 15$ m%. From the beginning of the 1970s the researchers have more and more intensively investigated the physical, mechanical and rheological properties of clay minerals. For example [22] has shown that the convectional brick clays with water content of >12-15 m% have lost their mechanical strength and elastic behaviour and they have turned into plastic materials. On this basis [23] has supposed that the conventional clays with mined moisture condition (water content) are viscous-plastic materials which can be characterized with the rheological equation of Eq. (6) as:

$$\tau = \tau_0 + \eta\frac{dv}{dx} \quad [\mathrm{Pa}] \tag{6}$$

where η is the dynamic viscosity and dv/dx is the deformation rate gradient i.e. the shear rate during crushing the materials on high speed smooth rollers. This conception was later confirmed experimentally (*Fig. 1*) by [24].

Taking into consideration the rheological properties of mined convectional brick clays as non-Newtonian materials gave new opportunities to authors [25-31] to determine and optimize technological parameters of processing machines like crushers, extruders, etc. The basis of non-Newtonian rheological properties of ceramic raw materials and achieved results in theory of continuum mechanics gave also opportunities to develop mathematical methods for design smooth high speed rollers [32-35] and vacuum extruders for forming ceramic building materials and asbestos cement wall panels [36]. To examine the rheo-mechanical properties of elastic fibre reinforced viscous-plastic complex materials, new universal 'rotovisco' equipment was also developed [37].

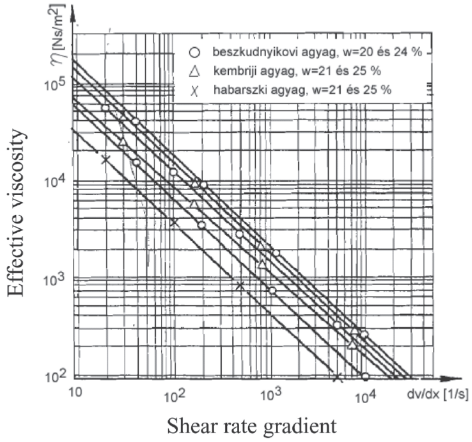


Fig. 1. *The dynamic (effective) viscosity of mined clay minerals as function of shear rates (taken from [29])*
1. ábra *Téglaipari agyagásványok effektív viszkozitása a deformációs gradiens függvényében (átvéve: [29])*

The knowledge of rheological properties and parameters of convectional brick clays is required to understand the physical and mechanical processes taking place during crushing, comminuting and forming of ceramic materials as well as to increase the efficiency of the machines used in ceramic technologies and industries [38-46]. For example [47] recommends the use of the rheological model of Eq. (7) to investigate and determine the physical and mechanical processes taking place in the materials in the working 'gaps' of pan mills:

$$\eta_g = a^n\eta_m \quad [\mathrm{Pas}] \tag{7}$$

where:
η_g – the dynamic viscosity of materials in the working gap of pan mill (Pas),
η_m – the measured dynamic viscosity of materials by the laboratory equipment (Pas),
a – coefficient; of which the value for 'Malyi-clay' is: 0.5-0.6,
n – power law exponent.

The value of the power law exponent can be determined as:

$$n = \frac{\lg\frac{\dot{\varepsilon}_g}{\dot{\varepsilon}_m}}{\lg 2} \tag{8}$$

where:
$\dot{\varepsilon}_g$ – the shear rate developed in the material during processing by industrial equipment (Pas),
$\dot{\varepsilon}_m$ – the shear rate developed in the material during rheological tests by laboratory equipment (Pas).

2. Traditional methods and equipment

There are many relatively simple methods and equipment available to determine and measure certain physical and mechanical properties of materials but it is quite difficult to measure and determine the physical, mechanical and rheological properties of complex materials like ceramic raw materials and ceramic particles or fibre reinforced composites [48-52]. The reason is that the instruments which are successfully working for rheological tests of materials in chemical, plastic, pharmaceutical, dairy and food industry cannot be used for tests of ceramic raw and semi-finished green materials because of their abrasivity, hardness, strength, etc. The advantages of these traditional rheotesters and instruments are their shortage in development the required shear stresses, mechanical pressures and/or temperatures or shear rates. Because of these for the rheological test of raw materials of ceramic and building materials industry are generally used so-called capillary viscometers (*Fig. 2*) with high value of mechanical pressure [39, 41, 53].

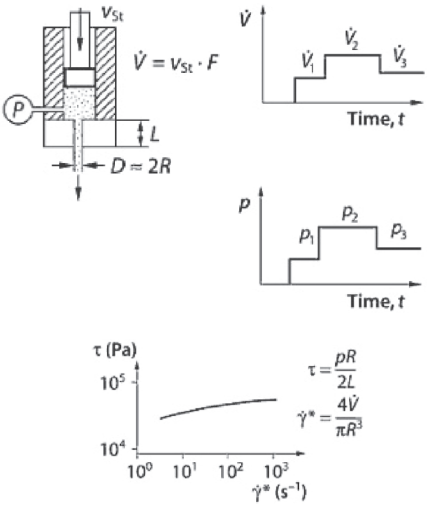


Fig. 2. The principle of capillary rheometers and the generated curves of volume flow (V(t)), pressure stress (p(t)) and shear stress

2. ábra A kapilláris reométer működési elve és az általa mért tömegáram (V(t)), nyomófeszültség (p(t)) és nyírófeszültség az idő függvényében

Using the capillary viscometer first necessary to determine the volume flow (V(t)) and saw pressure (p(t)) as function of time and further from these data can be determined the shear stress and shear rate in the materials passing through the capillary with *D*. So, from determined by experiment the values of shear stress as function of shear rate is:

$$\tau = f(\dot{\gamma}) \qquad [\mathrm{Pa}] \qquad (9)$$

The effective viscosity of materials can be determined as:

$$\eta_e = \frac{\frac{PR}{2L}}{\frac{4\dot{V}}{\pi R^3}} = \frac{\pi P R^4}{8 L \dot{V}} \qquad [\mathrm{Pas}] \qquad (10)$$

where:

L – the working length of capillary viscometer (m),

P – the mechanical pressure stress at the working length of capillary (Pa),

R – the radius of capillary (m),

$\dot{V}$ – the volume speed of tested material in the capillary (m^3/s).

The advantages of the capillary viscometer are the simplicity of construction, the optional value of pressure strength due to the hydraulic movement of the stamp in the cylinder, and the working ability at wide range of moisture or plasticizer. The main disadvantage of these kinds of rheometers that the pressure stress $\mathbf{p}_{ny}$ at the capillary is not permanent as its value during the measurement is changing as:

$$p_{ny} = p_b \cdot e^{-4\mu \frac{H}{D_0}} \qquad [\mathrm{Pa}] \qquad (11)$$

where:

μ – coefficient of external friction of tested materials at the walls of instrument,

D_0 – diameter of the saw stamp (m),

H – height of tested material in the cup of the viscometer (m),

p_b – pressure stress at the surface of the saw stamp (Pa).

During the rheological tests, the heights of the tested materials in the cup of viscometer are changing as shown in Eq. (12):

$$0 \le H \le H_0 \qquad [\mathrm{m}] \qquad (12)$$

The heights of the tested materials at a certain moment can be determined as:

$$H = H_0 - v_0 t \qquad [\mathrm{m}] \qquad (13)$$

where:

H_0 – the starting height of the tested material in the cup (m),

t – the time of measurement from the starting (s),

v_0 – the speed of the saw stamp in the cup (m/s).

3. The developed instruments and applied methods

The rheotester is reliable during the rheological investigation of ceramic raw materials, semi-finished products and ceramic particles or fibre reinforced metal matrix composites, it is relatively cheap and must satisfy the following requirements:

a. the instrument must be capable to measure and determine the rheological parameters of the frequently used raw materials and/or semi-finished products in wide range of technical and technological conditions in different sectors of silicate industry,

b. the instrument must be capable to develop the mechanical stresses and shear rates in the materials at the same level that happen during their passing through machines and mechanical equipment of technology lines,

c. the instrument must be capable to develop temperature used to be in technological process in the silicate industry and must be controllable inside of the materials during the whole testing process,

d. the instrument must be capable to measure also the compaction ratio, external friction coefficient and effective viscosity of tested materials under variation of mechanical stresses, temperature and shear rates,

e. the developed new construction must be simple, easy to produce and use,

f. the new instrument must be capable for quick testing of rheological parameters and the measured data must be reproducible.

So, the above requirements must also satisfy the following mathematical functional relationship for the compaction ratio:

$\Delta H/H= f(\sigma, H_\sigma, P, Q, T, w)$ [m] (14)

and for the external friction coefficient:

$\mu= F(\sigma, n, P, Q, T, w)$ (15)

as well as for effective viscosity:

$\eta_e = \Phi(\phi, \sigma, P, Q, T, w)$ [Pas] (16)

where:

σ – the normal mechanical pressure acting on the working surface of the tested material (Pa),
H_σ – the filling heights of material in the instrument (m),
P – the volume of used plasticizer (%),
Q – the chemical or oxide composition of the tested material,
T – the temperature of material during the measurement (°C),
w – the moisture content of tested material (%),
n – axis rotation rate (rpm),
γ – shear rate (s^{-1}).

To satisfy the above requirements a universal 'rotovisco' (*Fig. 3*) and a combined rheo-tribometer (*Fig. 4*) were developed partly in Russia and at Igrex Engineering Service Ltd. in Hungary.

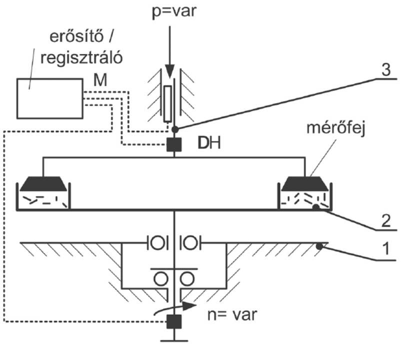


Fig. 3. Sketch of the developed universal 'rotovisco' laboratory instrument
1-rigid metallic frame, 2-rotatable ring-shaped measuring pot with tested materials, 3-unrotatable ring-shaped disk pressed down by a special hydraulic cylinder with variable pressure
3. ábra A kifejlesztett univerzális rotoviszkó elvi vázlata
1-rigid metallic frame, 2-rotatable ring-shaped measuring pot with tested materials, 3-unrotatable ring-shaped disk pressed down by a special hydraulic cylinder with variable pressure

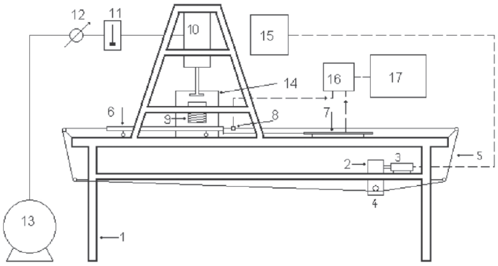


Fig. 4. Scheme of combined rheo-tribometer
1-instrument table, 2-small drive, 3-electric motor, 4-cable drum, 5-cableway, 6-batching car (with the shearing plate), 7-inductive displacement detector, 8-force-meter (spider), 9-heatable specimen holder, 10-pneumatic power cylinder, 11-magnetic valve, 12-pressure gauge, 13-compressor, 14-thermostat, 15-control unit, 16-data recorder (spider 8), 17-computer (capturing and processing data)
4. ábra A kifejlesztett kombinált reotribométer elvi vázlata
1-instrument table, 2-small drive, 3-electric motor, 4-cable drum, 5-cableway, 6-batching car (with the shearing plate), 7-inductive displacement detector, 8-force-meter (spider), 9-heatable specimen holder, 10-pneumatic power cylinder, 11-magnetic valve, 12-pressure gauge, 13-compressor, 14-thermostat, 15-control unit, 16-data recorder (spider 8), 17-computer (capturing and processing data)

The universal 'rotovisco' can be successfully used to the rheological tests of plasticized cement pastes and concretes reinforced with mineral fibres [51-54] as function of setting time and temperature [55]. The combined rheotribometer can be successfully used for rheological tests not only asphalt mixtures and pavements [56] but for different silicon-carbide composites [57-58] and for aluminum-titan alloys [59-60]. The complex rheological and mechanical test of complex materials like mined conventional brick clays also possible on this instrument [61]. The instrument measures the shear rates (), shear stress (and effective viscosity (η_e), during the rheological test and computes their values by Eqs. (17)-(18)-(19) as follows:

$$\dot{\gamma}_0 = \frac{\omega R_0}{H} \quad [s^{-1}] \qquad (17)$$

$$\tau = \frac{M}{A \cdot R_0} = \frac{M}{\pi(R^2 - r^2)R_0} \quad [Pa] \qquad (18)$$

$$\eta_e = \frac{H \cdot M}{\pi\omega(R^2 - r^2)R_0^2} \quad [Pas] \qquad (19)$$

where:

$\dot{\gamma}_0$ – the shear rate developed in the materials in the ring-shaped pot,
η_e – the effective viscosity of the material,
ω – the angular speed of ring-shaped container (s^{-1}),
A – the magnitude of sheared surface (m^2),
H – the height of the tested materials in the ring-shaped pot,
M – the value of the measured torque (Nm),
r – internal radius of ring-shaped pot (m),
R – external radius of ring-shaped pot (m),
R_0 – the radius belongs to the average volume speed of materials in the pot (m).

The working principle of the combined rheotribometer – patented in Hungary – is even simpler. The rheological parameters of tested materials also can be measured as function of chemical and mineralogical composition (Q), temperature (T), developed mechanical stress (σ), moisture content (w) and volume ratio of used plasticizer (P), etc. During the rheological tests the instrument measures and computes the values of shear rate (), shear stress (τ) and the effective viscosity (η_e) as:

$$\dot{\gamma}_0 = \frac{v}{H} \quad [s^{-1}] \qquad (20)$$

$$\tau = \frac{F}{A} \quad [Pa] \qquad (21)$$

$$\eta_0 = \frac{\tau}{\dot{\gamma}_0} = \frac{F \cdot H}{A \cdot v} \quad [Pas] \qquad (22)$$

where:

A – the magnitude of sheared surface (m^2),
F – the pulling force (N),
H – the working height of the tested materials (m),
V – the speed of batching car with the shearing plate (m/s).

Converting the batching car with shear plate into *Tolstoi's instrument* (*Fig. 5*), it is possible to get the deformation-times curves (*Fig. 6*) of the tested materials as function of their compositions, temperatures, and loading forces.

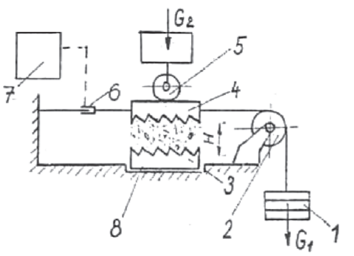
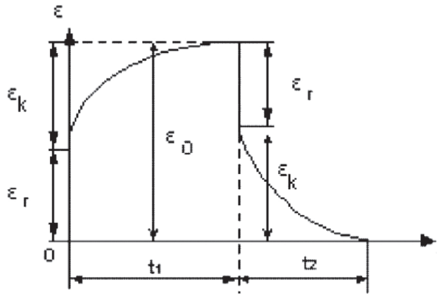
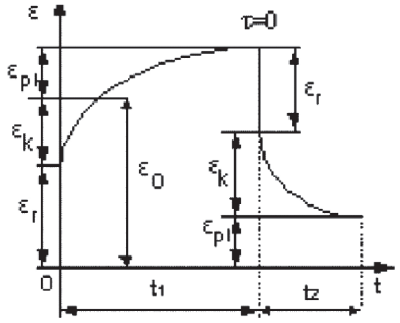


Fig. 5. Scheme of Tolstoi's instrument
1-loading force, 2-rope pulley, 3-lower tool, 4-upper tool, 5-hold-down roller, 6-displacement beacon, 7-printer, 8-material
5. ábra A Tolsztoj-féle készülék elvi vázlata
1-loading force, 2-rope pulley, 3-lower tool, 4-upper tool, 5-hold-down roller, 6-displacement beacon, 7-printer, 8-material



Below yield stress / Below yield stress



Over yield stress / Over yield stress

Fig. 6. Typical deformation of plasticized asbestos cement pastes as function of time
6. ábra A képlékeny nyers azbesztcement massza jellegzetes deformációja az idő függvényében

On the basis of the deformation-time curves created by combined rheotribometer it is possible to determine the instantaneous elastic modulus (E_1), the delayed elastic modulus (E_2) and viscosity of damaged (η_1) and undamaged material structures (η_2) as well as their static yield points (τ_0) for complex hetero-modulus, hetero-viscous-plastic materials like ceramic reinforced metal alloys and shield materials with extreme dynamic strength [62] in functional relationship as:

$E_1 = f(\tau, p, Q, T, w)$, [Pa] (23)
$E_2 = f(\tau, p, Q, T, w)$, [Pa] (24)
$\eta_1 = f(\tau, p, Q, T, w)$, [Pas] (25)
$\eta_2 = f(\tau, p, Q, T, w)$, [Pas] (26)
$\tau_0 = f(\tau, p, Q, T, w)$, [Pa] (27)

where:
τ – the shear stress developed in the tested material by loading force F_1 (Pa),
p – the pressure stress developed in the tested material by loading force F_2 (Pa),
Q – the mineralogical, chemical and grain structure of the tested material,
T – the temperature of the tested material during the measurement (^{0}C),
w – ratio of the moisture or quantity of used plasticizer in the tested material (m%).

On the basis of the deformation-time curves it is quite easy to determine and find the rheological parameters of tested materials and create their rheological models [63]; see for example *Figs. 7* and *8*.

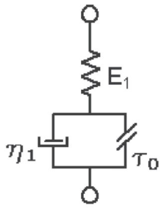


Fig.7. The typical rheological model of mined convectional brick clays used for production of ceramic roof tiles
7. ábra A tégla és a kerámia tetőcserép gyártásához használt bányanedves agyagásvány reomechanikai anyagmodellje

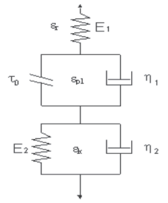


Fig. 8. The typical rheological model of asphalt pavements, asphalt concretes and elastic fibre reinforced cement pastes
8. ábra A lehűlt, megszilárdult útburkolati aszfaltkeverékek, valamint az ásványi szállal erősített friss beton reomechanikai anyagmodellje

4. Conclusions

The developed universal 'rotovisco' and combined rheotribometer fully satisfied the requirements explained in section 3. The instruments are not complicated and easy to use them to measure the most important rheological parameters of complex materials like convectional brick clays, asphalt pavements or other raw materials and semi-finished products of the building industry. By the requirements the instruments can be armed with special heating furnace of high temperature and they can be used for rheological test for complex materials like ceramic particles and ceramic fibre reinforced metal alloys and other hetero-modulus, hetero-viscous and hetero-plastic materials for safety and protection of transport equipment and flying objects.

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