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Viscosity and thermal evolution of density and wetting angle of a commercial glaze by means of hot stage microscopy

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Abstract

In the present work we attempt to describe the results of the thermal behavior analysis of a commercial glaze obtained in a thermal microscope developed by the research group. At the same time, the density and the wetting angle of the glaze with an alumina surface was evaluated. Particularly the sintering temperature, the sphere, semi-sphere and fusion temperatures were also determined. These present characteristic viscosities. Afterward, by means of the de Vogel-Fulcher-Tammann equation, the viscosity as a function of temperature was estimated.

In the first part the developed equipment is described together with the image analysis carried out for the in-situ cylindrical samples (2,5 mm of height and diameter). The thermal cycle was up to 1200ºC with a heating rate of 5ºC/min. The developed equipment presents the following characteristics: some problems with the temperature evaluation, it was slightly affected by air currents and difficulties for achieving an adequate parallelism. On the other hand, the potentialities of the equipment for development and study of the thermal behavior of glasses was established.

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Keywords: Glass, hot stage microscopy, viscosity, wetting angle.

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1. Introduction

A glass is a molten inorganic product that has cooled down to a rigid state without experiencing crystallization (Shelby (2005)). Considering only their main technical properties, common glass can be described as an amorphous inorganic product, consisting predominantly of silica (a typical soda-lime glass is formed by approximately 70 wt. % of SiO_2, the rest is mostly Na_2O and CaO), hard, brittle, transparent, high chemical resistant and deformable at high temperature.

Traditional porous ceramics are often seal off through the application of a glass cover that is usually applied in powder form (for immersion or vaporization of aqueous dispersions) and is then melted in a second heat treatment (Matthes (1990)). If the vitreous coat fits properly to the ceramic body it is known as glazed ceramic. In addition, this cover is usually of high chemical and mechanical resistance. This cover is often modified by the addition of colorants (oxides or inorganic pigments), other modifiers or opacifiers.

Ceramic glazes are manufactured from glass frits obtained by casting melts of homogeneous inorganic mixes of mainly siliceous composition accompanied by alumina and alkaline and alkaline-earth oxides (Matthes (1990)).

The thermal behaviour of the glazed ceramic is of technological importance. Thermal expansion coefficient is of particular interest, whose detaching forms crackle defects; so as wettability, whose detaching generates nodulization defects. Knowledge of the complete viscosity curves allows knowing temperatures of technological interest, such as workability temperature, to which glass can be shaped by the action of an external force; the practical melting temperature, allowing the rise of gases generated by reaction in the production of glass in economically viable times, among other temperatures of practical interest (Shelby (2005)). The high temperatures viscosity of glaze is also important because its detaching produces the coating to drip and uneven coating thickness. Another important property is the glass transition temperature, which is generally defined as the point that separates the behaviour of the glass between a solid and an undercooled liquid. The knowledge of this temperature is useful in practice, since that annealing of glass is done near glass transition. Annealing is a process of essential importance that prevents cracks that occur as a result of internal stresses generated during cooling of glass.

The viscosity of a glass has an exponential dependence with temperature. Some glasses have Arrhenius type behaviour, associated with an activated mechanism for viscous flow. But in the majority of the cases, the activation energy of viscous flow is not constant and varies with temperature. For this reason, the most commonly used model to describe this dependence is the Vogel-Fulcher-Tamman model (VFT) (Fulcher (1925)), which adjusts very well the experimental data, although care must be taken to calculate viscosity near glass transition temperature by using this model, since the viscosity is overestimated in the vicinity of this point.

High temperatures measurements of viscosity (η) of glass involve high complexity and expensive equipment, such as high temperature rotational viscometers, with platinum rotors and crucibles (Shelby (2005)). Another employed method is the fiber elongation (Shelby (2005)), in which viscosity measurement is based on the deformation speed of a glass fiber by applying a constant traction force, compared with a standard glass fibber of known viscosity. There is however a cheaper alternative which is based on determining the temperatures at which a small piece of glass made up from compact glass powder is deformed and acquires certain geometries, which have fixed viscosities. This method also allows obtaining information with an efficient use of time, since only one experiment needs to be carried out.

Scholze (1962) was the first to define characteristics geometries that adopts a pill of compact glass powder when subjected to a heating cycle. In this way, characteristics points were defined, such as first shrinkage, maximum shrinkage, deformation, sphere and flow; and in turn assigned fixed values of viscosity. Pascual et al. (2001) established a method for calculating the viscosities of the mentioned characteristic geometries and compared them with values obtained by a high-temperature viscometer. In a later work, more precise viscosity values of the same geometries were obtained, and, in addition, the sphere point viscosity was determined which had not been taken into account before (Pascual et al. (2005)).

In the present work, we were able to establish the temperatures of the defined geometries of fixed viscosities calculated in the mentioned work of Pascual et. al. (2005); in addition to other relevant measures, by means of a hot stage microscope (HSM) developed in CETMIC, using MatLab® for image processing. The viscosity-temperature points were adjusted to the VFT model to obtain the complete curve of viscosity as a function of temperature.