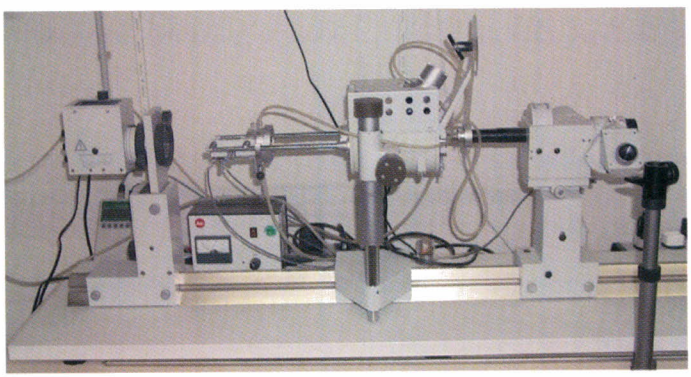
GLAZE CHARACTERISATION USING THE HOT STAGE MICROSCOPE: A PRACTICAL APPROACH

S. Link, M. Engels

FGK Forschungsinstitut für Anorganische Werkstoffe -Glas/Keramik- GmbH, Hoehr-Grenzhausen, Germany



ABSTRACT

The present communication discusses the use of the hot stage microscope in the ceramic industry for the characterisation of ceramic glazes, as developed in the international technological co-operation between FGK in Höhr-Grenzhausen (D) and TNO TPD in Eindhoven (NL).

The image analysis of the glaze sample as function of temperature, using state of the art equipment and adequate software is illustrated.

Parameters like height of sample, contact angle, sample surface area, and the contact area between body (carrier) and glaze are plotted against temperature. The change in the height and sample area is used to discuss the melting and sintering mechanism of ceramic glazes used in the ceramic industry. The changes in contact angle and contact area are related to the wetting ability of the glaze.

The graphs generated are used to define characteristic points, which qualify glaze characteristics and glaze behaviour: sintering point, deformation point, half sphere point and flow point. The background and definition of these points, and the limitations regarding accuracy and use of these points are discussed, as well as the use of the data to estimate practical parameters like glaze sealing temperature. This parameter has a significant influence on the enclosure of bubbles in the glaze, which can lead to bubble defects. While this characteristic point cannot be measured with the hot stage microscope, the sealing temperature is validated by using gradient firing.

With this combination between hot stage microscopy and the gradient firing, a reliable system was created.

Examples illustrate the use of the hot stage evaluation: the curve characteristics for different glaze systems are related to the behaviour of the glaze during firing. The effects of viscosity, surface tension and the wetting ability of the glaze on the body, as derived from the hot stage evaluation, are discussed.

The definition of related glaze characteristics, using the hot stage data, is demonstrated using the approach according to Scholze to determine glaze viscosity. The viscosity of different glaze systems is calculated as a function of temperature, using a set of three characteristic points with empirically defined viscosity values.

Examples of the evaluation of different glazes for porcelain, tile and sanitary ware are illustrated by the practical implementation of the result for a porcelain glaze, e.g. for glaze comparison and for troubleshooting purposes, e.g. for reducing pinhole defects.

1. OBJECTIVES

The main objective is the validated use of the Hot Stage microscope, interpretation methods and additional measurement to support:

- Full control of bubble formation during the firing process.
- Development of manufacturing technology to produce glazes with zero large bubbles.
- Zero large bubbles means: Bubbles with a diameter of less than 30 to 50 μm, and 100 μm from the surface. Such bubbles are not seen visually by the naked eye.
- Reduction of the reject rate in industrial manufacturing.

The approach has been successfully tested in a European Growth Project in the 6th Framework Programme "Bubble Control in Glazes for Ceramic Products BUCOGLACER".

2. EXPERIMENTAL

The glaze melting behaviour is measured using the Hot Stage Microscope under controlled conditions, by image analysis and definition of characteristic points in sintering / melting behaviour. Reference methods for these measurements are:

ISO 540

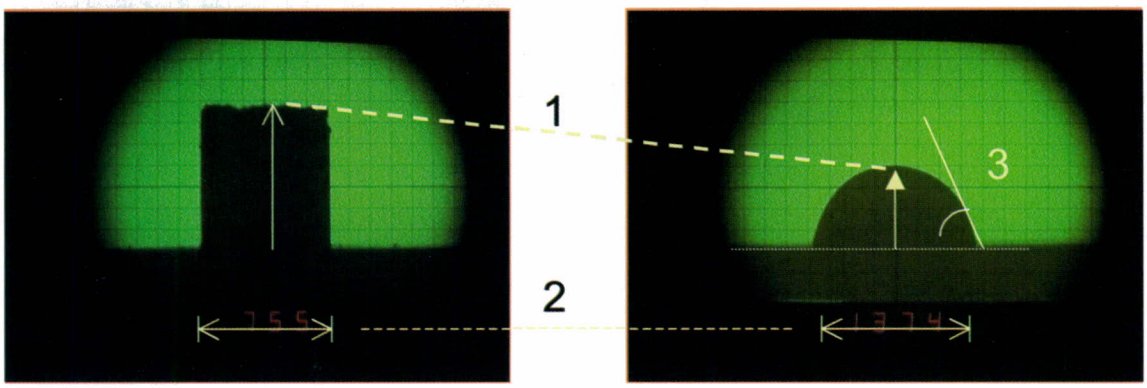
Solid mineral fuels; Determination of the fusibility of ash -high temperature tube method-.

DIN 51 730

Bestimmung des Asche-Schmelzverhaltens.

Although similar, these standards are different regarding the definition and use of the characteristic points, which can be determined when observing the melting and sintering behaviour. These differences can also be found in the

literature references, and have been used to define characteristic points of use for the intended use. In most points these two Standards are confirmed, but they do not exactly agree. The literature also contains different points of view for correct measuring and for the description of the characteristic points.



The characteristic points are defined using the measurement of

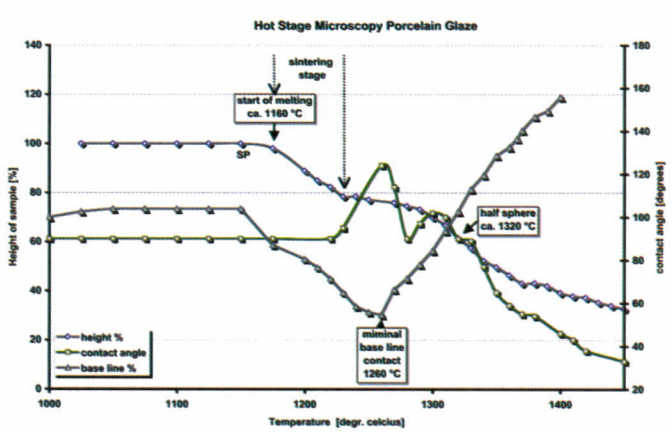
1. Height.
2. Base contact line.
3. Contact angle.
4. Area (using image analysis software or balance and drawing a zwetsch-curve) as a function of temperature.

The characteristic Points of Hot Stage Microscopy in combination with the sealing test after gradient firing can be used for:

- First: Evaluating glaze melting behaviour.
- Second: Calculating glaze viscosity as f (T) to support glaze optimisation / troubleshooting activities.

In addition the gradient firing of glazed ceramic slabs can be used to define the sealing temperature, using permeability tests.

3. EXAMPLES



If the interval **Sintering Point** to **Sealing Temperature** is long.
- Closure when glaze sintering has progressed, so enclosure is limited.

If the interval **Sintering Point** to **Sealing Temperature** is short.
- Closure when glaze has many intergranular space, so many enclosures (bubbles).

If the interval **Sealing Temperature** to **Half Sphere Point** is long,
- limited flow (viscosity), so enclosed bubbles will not escape easily.

If the interval **Sealing Temperature** to **Half Sphere Point** is short,
- More flow, so the enclosed bubbles will escape more easily.

Example Evaluation

SP - MP

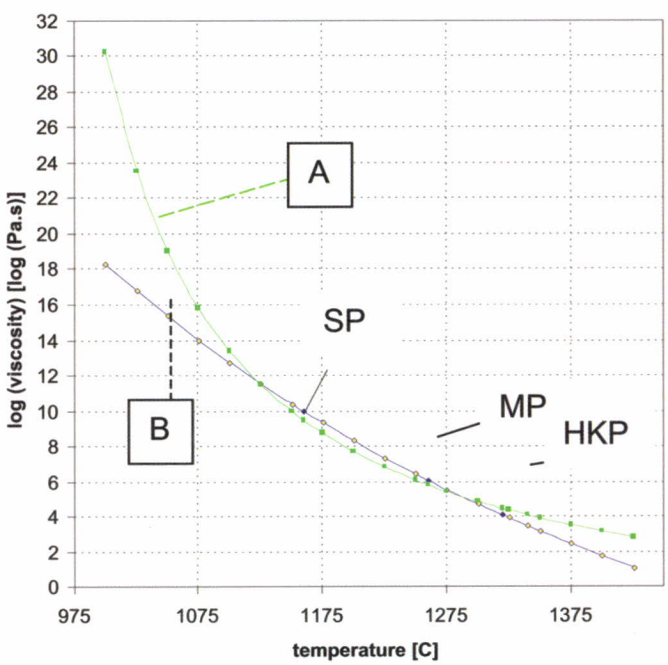
Glaze A has lower viscosity, so more and bigger bubble growth.

MP - HKP

Glaze A has higher viscosity, so more bubbles enclosed stationary.

Conclusion

Glaze A has some big bubbles and Glaze B has more little bubbles under surface



4. CONCLUSION

- The hot stage measurements can be used to evaluate glaze sintering / melting behaviour in production directly in combination with gradient firing to define the sealing temperature.

- Hot Stage characteristic points can be used to evaluate glaze viscosity.

- The Definition of characteristic points should be clear for discussion and agreement between supplier, development and production! Under these circumstances:

  - Hot stage evaluation can be used for quality control, to optimise glazes and support troubleshooting activities.

  - Image analysis software facilitates direct glaze evaluation and interpretation for practical use.