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Crucible calibration for improved temperature accuracy in the induction fusion process

Abstract

Induction heating units from Herzog allow the online temperature measurement of the platinum ware by an infrared pyrometer. Here we present a new approach for automatic temperature calibration of the platinum/ gold crucible (patent pending). This method leads to a significant enhancement of temperature accuracy and repeatability which are critical parameters for accurate and precise analysis results.

Key words

• Fusion• Induction heating • Crucible • Calibration • Infrared pyrometer

Introduction

Borate fusion by induction heating offers significant benefits compared to gas-flame burners or electrically heated muffle furnaces. Induction systems are extremely fast and flexible enabling a quasi-instantaneous adjustment of the target temperature in the range between 300 and 1400°C. This has been proven particularly useful for applications and laboratories requiring short sample preparation times and variable temperature settings. Furthermore, induction heating systems allow the direct and continuous temperature measurement of platinum/ gold crucibles and casting dishes by an infrared pyrometer. This makes it possible to reach the preset temperature within seconds and keep it stable and unaffected by external interference factors.

In order to achieve an absolutely accurate measurement of the crucible temperature, the emissivity of each individual crucible has to be taken into account. Emissivity is the measure of an object's ability to emit infrared energy as an indicator for the temperature of the object. The optical system of the pyrometer receives the infrared radiation and focuses it on a detector. The detector converts the radiation into electrical signals which are displayed as temperature values. In order to take a high-precision temperature measurement, the pyrometer should be specifically adjusted to the emissivity of the object being measured.

Typically, crucibles vary in material structure, elemental composition, weight, surface shininess and other features. This may cause small variations in the emissivity of each crucible possibly leading to slightly different temperature reading by the pyrometer. A further impact factor systematically affecting crucible emissivity is the temperature level used for borate fusion. Generally, higher temperatures increase the emissivity of the crucible while lower temperatures lead to a decline.

Here, we present a simple and quick approach for automatic adaptation of the pyrometer to the emissivity of each individual crucible. This crucible calibration method (patent pending) compensates for material-related and temperature-dependent variation of the crucible in order to enhance temperature accuracy and repeatability.

Method

The crucible calibration is performed by melting a defined amount of Lithium tetraborate. The whole process is straightforward and carried out fully-automatically by the PLC of the fusion machine. During the calibration, the PLC simultaneously records the pyrometer reading and the temperature within the melt at four different energy steps of the high frequency generator. Based on these reference values for pyrometer and melt temperature, the system automatically determines the exact emissivity values for each crucible at each temperature level. In all subsequent fusion operations involving this crucible, the PLC automatically adjusts the correct emissivity value of the pyrometer.

The reference temperature values within the melt are obtained using a special calibration device ("calibrator", Figure 1). The calibrator inserts the thermocouple (type K) into the melt and ensures that it is always placed in the same position for absolutely reproducible measurements. The calibrator can use various thermocouples types according to the demands on measurement accuracy. In fully-automatic fusion systems (HAG-HF), the thermocouple is automatically inserted into the melt. In semi-automatic machines (Bead One HF), the HMI panel guides through the process and prompts the operator to insert the calibrator. This can be done from outside the machine without any contact to the heating unit. In most cases, the calibration is performed only once and takes less than 10 minutes.

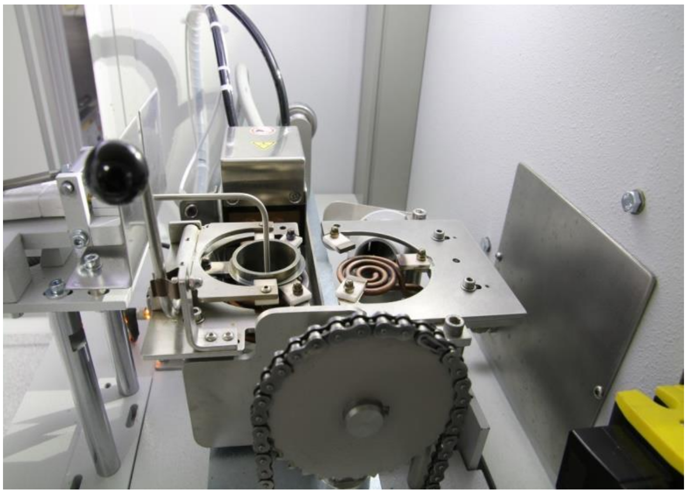

Figure 1: *Calibration device in the semi-automatic Bead One HF fusion machine*

Results

The calibration results in an exact adjustment of the infrared pyrometer to the emissivity of the crucible. This is made possible by the direct measurement of the real temperature within the melt and comparison to the pyrometer temperature. Based on the difference between melt and pyrometer temperature the system computes the emissivity of the crucible.

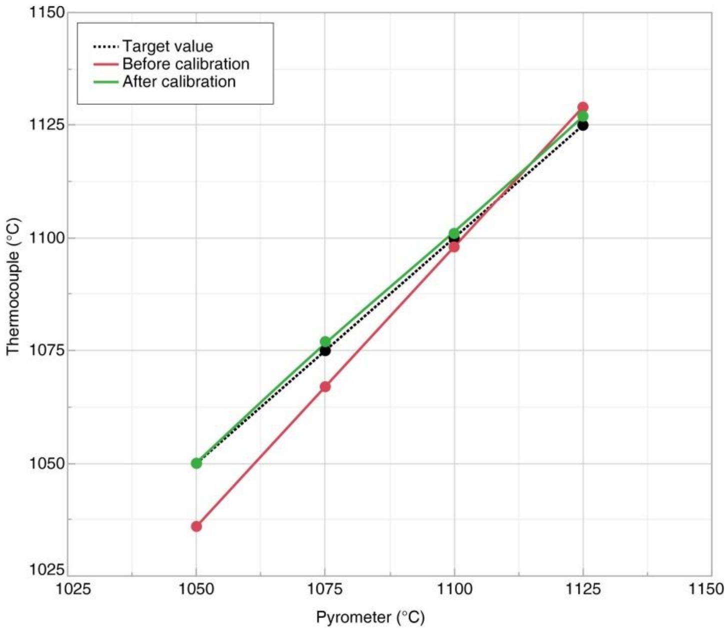

Figure 2: *Effect of the crucible calibration on the temperature accuracy as measured by thermocouple within the melt.*

After calibration and emissivity adaptation, the pyrometer reading corresponds to the actual temperature within the melt. Figure 2 illustrates exemplarily this effect. The red line of the graph shows the state before calibration. The maximum difference between pyrometer and melt temperature is 14°C at a target temperature of 1050°C. After calibration (green line), the maximum difference decreases to 2°C pyrometer. This is within limits of accuracy of the thermocouple element meaning that the pyrometer temperature is virtually identical to the temperature within the melt.

The temperature range which is covered by the calibration is sufficient for almost all fusion applications. It might be necessary to execute a calibration for another temperature range below the flux melting temperature, e.g., in order to oxidize sample material. This can also be done quickly and easy using the calibration routine of the fusion machines.

Discussion

A fundamental prerequisite for achieving reproducible analysis results is to ensure reproducible sample preparation conditions. According to cautious estimates, about 30% of the total analytical error is due to insufficient and poorly controlled sample preparation.

The crucible temperature is a particularly critical parameter during preparation of glass beads. Deviations from the target temperature, upward and downward, have impact on the volatilization of the flux, viscosity of the melt, loss of volatile elements and other factors. Repeatability of the temperature is one of the keys to success for producing and analyzing glass beads.

More important than the absolute temperature chosen for fusion is the precise and repeatable temperature control during the process.

The induction technology by Herzog provides full control over the fusion process and allows online monitoring of the crucible and dish temperature as a crucial parameter. The crucible calibration represents a further improvement resulting in better reproducibility of the sample preparation process. It gives the operator a rigorous temperature control independent from the platinum ware used for fusion. The method is very easy, quick and reliable. It is available for the fully-automatic HAG-HF and the semiautomatic Bead One HF benchtop instrument.