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A novel approach for optimizing the temperature precision of induction fusion devices

Abstract

Borate fusion applications involving the determination of volatile elements such as sulfur require a highly precise temperature control of the fusion instrument. In high-frequency induction systems, temperature is measured by IR pyrometers pointed to the outer wall of the platinum- gold crucible. In a first step we examined whether consecutive measurement on different spots on the crucible had an influence on the temperature variability. We found that rotation of the crucible by steps of 10 ° caused an increased temperature variability with a standard deviation (SD) of ± 6.9 °C. In a second step, we used the HAG-HF system to automatically rotate two different crucibles A and B in such a way that the IR pyrometer always measured at the same spot. This led to an improvement of the temperature variability with a SD of ± 1.8 °C (crucible A) and ± 1.1 °C (crucible B). Hence, the novel approach further improves the already excellent temperature precision of the Herzog induction fusion systems by a factor of approximately 4.

Key words

• **Borate fusion** • **Induction heating** • **Temperature control** • **HAG-HF** • **Infrared pyrometer**

Introduction

Borate fusion is a method in which almost all oxidic and inorganic materials can be prepared for analysis by, e.g., X-Ray fluorescence (XRF) or Laser Induced Breakdown Spectroscopy (LIBS). The sample material is mixed with an excess of fusion flux such as lithium tetraborate. The glass forming flux breaks up the chemical compounds in the sample. This process eliminates matrix and grain size effects thus increasing analytical accuracy and precision.

For this study, we used fusion systems which are based on high-frequency induction technology. This technique causes a direct heating of the platinum-gold crucible containing the sample/ flux mixture. The heat is then released inward from the crucible wall causing the material to melt.

The target temperature is monitored in real time by an infrared pyrometer (IR pyrometer) pointed at the outer wall of the pyrometer. Continuous temperature measurement makes it possible to keep the temperature constant within a very narrow range. Furthermore, it is possible to immediately compensate for disturbing external influences that could interfere with the temperature stability.

In order to achieve the highest possible temperature accuracy during the fusion process it is critical to adjust the IR pyrometer exactly to the emissivity of the platinum-gold crucible [1, 2]. The emissivity describes the ability of a material to emit thermal radiation and depends on various factors. First and foremost, the emissivity of the platinum-gold crucible is determined by its surface properties such as material composition, roughness or oxidation [3].

Platinum-gold crucibles used in daily laboratory routine usually have an inhomogeneous surface quality due to wear and tear such as scratches. This means that the emissivity may vary depending on which spot on the crucible surface is being measured by the IR pyrometer. Since the crucible is positioned in a crucible holder and rests on a brim during fusion, the temperature is always measured at the same crucible height. Therefore, only the rotation of the crucible can result in measurement on spots with different emissivity during consecutive fusion processes.

Rotation of the crucible between two consecutive fusion processes may lead to different actual melt temperatures even if target temperature and sample composition are the same for both fusion processes. This is due to the emissivity differences at the two different crucible spots measured by the IR pyrometer. The nominal temperature values read out by the IR pyrometer are nevertheless identical since the measurement system is based on identical emissivity values for the entire crucible.

Even slight differences in the melt temperature can possibly lead to a significant bias in the analytical reproducibility. Highly accurate and consistent temperatures in borate fusion is particularly important when volatile elements are present within the sample. Sulfate sulfur, for example, shows an exponentially increasing volatilization rate above 1000 °C [4]. Consequently, special attention must be paid to reproducible temperature conditions in borate fusion applications which are usually performed in a temperature range above 1000 °C.

In this application note we first investigate the influence of crucible rotation on the temperature reproducibility. Second, we present a novel method to automatically detect the rotation and align the crucible in order to improve the reproducibility of temperature measurement by the IR pyrometer.

Methods

The experiments were performed with high-frequency induction fusion machines from Herzog, Germany. We used the semi-automatic fusion device model Bead One HF and the fully-automatic fusion system HAG-HF with integrated material and flux dosing as well as cleaning of platinum ware.

In the first step, we assessed the impact of the crucible rotation on the temperature measurement by the IR pyrometer. For this purpose, an emissivity value of 0.5 was assumed for the entire crucible. The corresponding emissivity parameter on the IR pyrometer was set to this value. Furthermore, the crucible was heated by using a constant power value of the high-frequency generator instead of a target temperature value. This chosen procedure is different to the routine process where the fusion takes place at a constant target temperature which is achieved by variably adjusting the high-frequency power. However, the advantage of that procedure is that heating at constant power causes differences in crucible emissivity to result in different pyrometer temperatures.

We measured the temperature values over an arc of 170 ° on the crucible circumference. For this purpose, the empty crucible was placed into the Bead One HF and heated at constant power for 120 s while the temperature data was recorded using the PrepMaster Analytics Software (Herzog, Germany). After each measurement, the crucible was manually rotated in 10 ° increments resulting in 17 different measurements per experimental cycle. We performed two complete cycles.

In the second step, we examined whether automatic correction of the crucible rotation has an impact on the temperature reproducibility. This investigation was carried out at the automatic HAG-HF-system. Initially, an engraving was applied to the outside of the base of the crucible, which allows the crucible rotation to be clearly identified by an automatic camera system. In each trial, the crucible was fed into the system unaligned. Before the robot placed the crucible on the fusion position of the HAG-HF, the rotation angle of the crucible was automatically detected by the camera system. This allows the robot to perform an adjustment movement so that the crucible was always aligned to the same rotational angle and the IR pyrometer was always pointed at the same spot for all temperature measurements. We used two different crucibles (A and B) to assess the influence of different crucible weights and surface qualities. For each crucible we performed 10 trials without and 10 trials with automatic compensation of the crucible rotation resulting in a total of 40 trials.

Results

As shown in Figure 1 the rotation of the crucible had a significant influence on the temperature as assessed by the IR pyrometer. Within the measured crucible area, the temperature varied between 1085 and 1121 °C (mean 1109 °C) with a mean ± standard deviation (SD) of ± 6.9 °C. The temperature values at the respective rotation angles were similar in the two experimental cycles performed. This indicates that the measured temperature differences arose from different emissivities caused by differences in the crucible surface quality.

Figure 2 shows the impact of the automatic rotation detection and crucible alignment on reproducibility of the temperature measurement. In crucible A, the standard deviation was 7.8 °C (mean 1144 °C) for trials without crucible alignment. Crucible alignment led to a significant reduction of the temperature variability with a standard deviation of 1.8 °C (mean 1127 °C). In crucible B, the standard deviation for trials without crucible orientation was 4.4 °C (mean 1044 °C) whereas crucible alignment reduced the standard deviation to 1.1 °C (mean 1046 °C).

Conclusion

The result of this study demonstrates that alignment of the crucible rotation to the IR pyrometer is an effective approach to further improve the temperature reproducibility during the fusion process. Accordingly, adjustment of the crucible rotation is sufficient to achieve a minimum variability in the fusion temperature.

The crucible feeding, the handling in the fusion

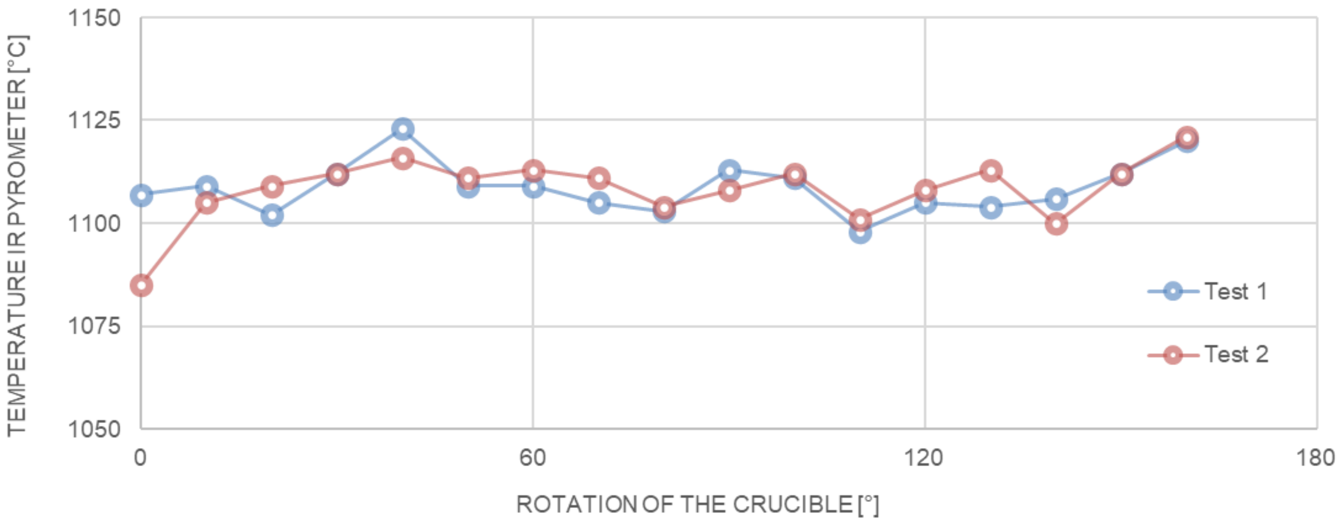

Figure 1: *Results of the first test. Pyrometer temperature (crucible temperature) was measured each time when the crucible was rotated by 10 ° increments.*

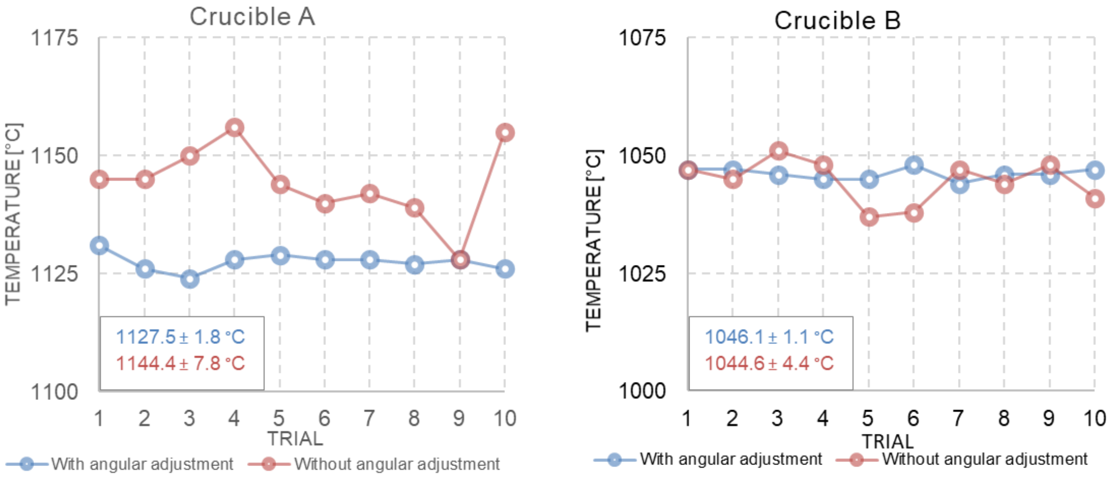

Figure 2: *Results of the second test using crucible A and B. Display of IR pyrometer temperature for crucible handling with and without angular adjustment.*

device, sample mixing or cleaning of the platinum ware inevitably results in unpredictable rotation of the crucible. This means that the measuring spot of the IR pyrometer changes with each fusion process. The detection and adjustment of rotations ensures that the IR pyrometers is always pointed at the same spot and the emissivity is identical for each fusion process. Our results show that this approach further improves the already excellent temperature precision of the Herzog fusion systems by a factor of about 4.

The method is applicable to both manual and automatic fusion equipment. For manual instruments such as the Bead One HF, the operator must ensure accurate alignment of the crucible with the pyrometer. A mark on the crucible, such as a notch in the brim, can be helpful to identify the correct orientation. In automatic systems such as the HAG-HF, the crucible orientation is reliably detected with the aid of an engraving applied to the crucible base.

The high positioning accuracy of the six-axis robot and the automatic centering of the crucible by the gripper are additional factors that provide excellent conditions for the precise temperature control in fully automatic fusion systems. The automatic adjustment of the crucible rotation, in addition to features such as high-precision sample and flux dosing, is another component that enables fusion processes to be performed under perfectly reproducible conditions.

References

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