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HERZOG

Benefits of high-frequency induction heating for borate fusion

Abstract

This paper provides a review on sample preparation by borate fusion and presents a comparison of different heating technologies. High-frequency induction fusion is a common method in industrial and research laboratories. Compared to instruments with gas or electrical resistance heating, induction systems offer substantial benefits including high sample throughput, flexible and rapid change of the fusion temperature as well as low maintenance and operating costs. Furthermore, recent developments allow a precise and accurate temperature control in induction heating which is comparable to the performance of resistance furnaces using thermocouples.

Key words

• **Borate fusion** • **Bead One HF** • **HAG-HF** • **Induction furnaces** • **XRF**

Introduction

Borate fusion is a standard sample preparation method widely used for quality control in industrial laboratories and scientific research. Sample preparation by fusion eliminates particle size effects and matrix interference. In addition, fused glass beads exhibit high homogeneity and structural consistency. Accordingly, it has been demonstrated for specific applications that borate fusion results in better accuracy and precision compared to other procedures like, e.g., preparation of pressed pellets [1].

Various commercial equipment is available on the market which mainly differs in degree of automation and heating technology. Most instruments perform the fusion process automatically. The process includes heating of the melt, pouring into the casting dish and cooling it down to a glass bead. In contrast, preceding and subsequent preparatory steps such as sample and flux dosing as well as cleaning of the platinum ware are usually carried out manually by the operator. Only a few fully automatic systems such as the HAG-HF (Herzog, Germany) cover the entire process from dosing to fusion and cleaning.

Three different furnace technologies are regularly used in the fusion instruments, with each technology having its own special characteristics:

1. Gas furnaces

2. Electrical resistances furnaces

3. High frequency induction furnaces

In this application note, we will briefly summarize the technical features of each type of furnace, highlight their advantages and disadvantages, and compare them with each other.

Gas furnaces

Instruments using gas as a heating medium often have low initial investment costs. Still, the expenses for gas consumption and measures to be taken for safe operation should be factored into the investment calculation. In addition, the fusion temperature in these units is often not monitored and controlled. Instead, the target heat is only regulated by gas flow rate through the burner. Therefore, it is often unknown what the actual fusion temperature is. Accordingly, these systems have a temperature inaccuracy, which is in the range of 20 °C [2]. In addition, the instruments are more susceptible to technical defects and malfunctions. For example, the closure of a nozzle can mean that the intended fusion temperature is not reached. Such temperature inaccuracies may cause significant analytical errors, especially with samples containing highly volatile elements such as SO3.

In addition, in instruments with several gas furnaces in series, the center positions might be at a higher temperature than on the outside as they also receive heat from both adjacent burners. This condition may adversely affect the reproducibility of the sample preparation and increase the analytical bias.

Electrical resistance furnaces

Electric resistance furnaces offer a higher level of safety and better temperature control compared to gas furnaces. In the case of equipment with multiple furnaces, it is mandatory that the temperature in each furnace chamber is individually controlled and regulated using its own controller. Otherwise, temperature differences of up to 30 °C between each furnace may occur [3].

Temperature inertia is a significant disadvantage of electric resistance furnaces. All instruments must be heated up a considerable amount of time in advance to get them to the required temperature and ready for operation. Many devices have an automatic start mechanism integrated into their control system so that they are ready for use at the start of work. The temperature inertia leads to greater limitations when running a fusion program with two or more temperature set points. Particularly reducing the temperature can take a long time due to the effective furnace insulation causing a low cooling rate. A further unfavorable side effect of this is an overall reduction in sample throughput of this instrument. Another drawback of electrical resistance furnaces is that it is not feasible to adequately oxidize samples with reducing phases to avoid damage to the platinum crucible by formation of eutectic alloys.

High-frequency induction furnaces

In high-frequency induction furnaces the actual fusion temperature is measured in real time by an infrared (IR) pyrometer pointing at the platinum crucible. As a result, the temperature set point is reached almost immediately and can be kept precisely within the target range even in the event of disturbing influences. In fusion instruments from Herzog, the precision of the temperature control is about 5 °C after performing a temperature calibration in the crucible [4, 5]. As an option of Herzog's fully automatic HAG-HF systems, a special crucible alignment mechanism can be applied. In this case the temperature precision improves further to values less than 2 °C [6]. This makes the HAG-HF especially suitable for applications requiring high temperature precision and accuracy like for samples containing volatile elements.

In automatic systems such as the HAG-HF, up to 8 heating units are in operation simultaneously. Each unit is controlled by its own control loop system. Therefore, the temperature is not influenced by adjacent fusion processes. This ensures highly accurate and precise temperature control in each individual induction unit (Figure 1).

Figure 1: *Automatic high-frequency fusion system model HAG-HF (Herzog, Germany).*

The temperature set points in induction fusion machines are reached almost instantaneously. Figure 2 shows an example of a heating profiles as assessed in the semi-automatic induction fusion instrument Bead One HF (Herzog, Germany, Figure 3) compared to an electrical resistance furnace.

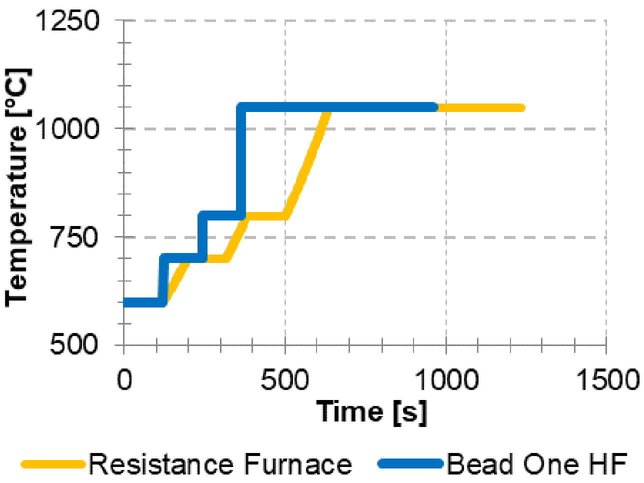

Figure 2: *Heating profile of the Bead One HF in comparison to a resistance furnace. It is also possible for the Bead One HF to set ramps with different slopes in order to slow down the heating.*

Figure 3: *Semi-automatic high-frequency fusion system model Bead One HF (Herzog, Germany).*

The fusion program involves an initial oxidation step. Using a stepwise increase to the next temperature level the Bead One HF completes the whole fusion program within in approximately 950 s. By contrast, the electrical resistance furnace passes through each temperature level at a limited ramp rate due to its temperature inertia. Consequently, the entire process takes about 1300 s, which is about one third longer than with the Bead One HF. Cooling the electrical resistance furnace to the initial temperature of 600 °C to prepare the next sample takes a further substantial amount of time. Therefore, the sample throughput of high-frequency induction systems is usually significantly higher than with electrical resistance furnaces. As a matter of course, high-frequency induction instruments cannot only increase the temperature in steps, but also run precise ramps if required for sample preparation.

In the semi-automatic induction fusion system Bead One HF (Figure 3), the operator places the crucible with the sample/flux mixture and the casting dish into the fusion position. After choosing the fusion program on the HMI panel, the Bead One HF automatically performs the fusion including homogenization of the melt and pouring into the casting dish which is heated up by an induction coil. The glass bead is then cooled down in a controlled manner to prevent the formation of cracks. After approximately 10 to 15 min total fusion duration, the glass bead can be manually removed and analyzed by XRF or LIBS.

In the fully automatic fusion system HAG-HF (Figure 2) both sample and flux are automatically dosed into the crucible and mixed before the fusion process is performed. After completion of the fusion, the platinum ware is automatically cleaned in an ultrasonic bath, rinsed with distilled water, and made available for reuse. The glass beads are automatically transferred from the dish onto a transport belt and then into a magazine or directly into a XRF spectrometer for analysis.

Conclusion

High frequency induction furnaces offer many advantages including:

- Flexible and rapid change of the fusion temperature
- Highly precise and accurate temperature control
- Oxidation is more efficient as it does not happen in reducing atmosphere
- Limited wear of induction heating parts
- Independent temperature control for each heating unit without interference from adjacent fusion processes
- No occupational hazards in the working environment due to gas

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