Borate fusion of “platinum poisons”- Automatic coating of the crucible with a protective layer

Abstract

Here, we present an approach for automatic formation of a protection layer in Pt/Au crucibles by using the fusion machines Bead One HF and HAG-HF. The rocking mechanism of the machines is applied to form a protective layer of lithium tetraborate covering the entire circumference of the inner crucible wall. The protection layer is extremely stable and allows fusion of “platinum poisons” like, e.g., silicon metal, ferroalloys, copper, molybdenum, sponge iron or sintered material. Furthermore, this method supersedes manual handling of hot liquid acids thus significantly enhancing occupational safety.

Key words

• **Borate Fusion** • **Bead One HF** • **Platinum poison** • **Cu / Mo** • **Sponge Iron** • **Metallic Si**

Introduction

There are several elements which form low-melting eutectics with platinum causing detrimental effects on crucibles used for fusion. They are commonly designated “platinum poisons”. For example, the eutectic melting temperature of platinum in combination with metallic silicon is around 830 °C. This temperature is well below the fusion temperature thus resulting in serious harm to the platinum equipment (LUPTON et al. 1997). Figure 1 shows circumscribed melting of a platinum crucible after being in contact with SiC at high temperatures. An efficient workaround to this problem is coating of the inner crucible wall with a protective layer to shield the platinum (RUTHERFORD 1995).

Copper is a further harmful element. While not being considered a platinum poison in the narrower sense, special precautions should be taken during the fusion process. Such, reducing conditions have to be avoided and the copper must be completely oxidized (LUPTON et al. 1997). Here again, forming a protection layer from flux is a reasonable measure to prevent rapid failure of the platinum equipment. After layer formation, the crucible can be filled with oxidizing agent, sample material and the rest of the flux. Then the sample is oxidized at temperatures below the melting point of the flux (930 °C).

Figure 1: Melting of a platinum crucible after contact with SiC at high temperatures (LUPTON et al. 1995).

Formation of the protective layer

A portion of the lithium tetraborate used for fusion is taken to form a protection layer within the crucible. Out of 10 g lithium tetraborate for a 39 mm diameter bead, 6.5 g is used for the protection layer. The material is filled into a $Pt_{95}Au_5$ crucible and subsequently molten in the Bead One HF high frequency induction furnace. In order to decrease the viscosity of the liquid, a quick melting step is performed at high temperatures of 1200 °C. Once the flux is completely molten, the very effective swirling mechanism for the crucible is started. After 10 s, the heating is shut down while swirling continues in order to press the material up against the inner walls. After 60 s, the mechanism is stopped. At this time, the lithium tetraborate already stiffened to solid glass. Now, the protected crucible surface is ready for inserting the oxidizing agent (white) and the sample to be oxidized (brown). In Figure 2 and 3 the protection layer within the platinum crucibles can be clearly depicted. In Figure 2, we used a new crucible. The polished inner surface led to an irregular upper rim of the protective layer. In Figure 3, the inner surface of the elder crucible is rougher, resulting in a sharp upper rim. Please note that in neither of them cracking or crystallization of the protective layer is apparent. Both layers show the same protective effect.

Figure 2: Protection wall within a new crucible with a polished inside. The protection wall is half as high as the crucible. The oxidizing agent (white) and the sample material (brown) can safely be oxidized without contact to the platinum.

Figure 3: Protection wall within a used crucible. The protection wall is half as high as the crucible but more sharply delineated compared to Figure 2.

Conclusion

The formation of a protection layer is a standard procedure for dealing with platinum poisons (PETIN et al., 1985). The Bead One HF provides the fully-automatic formation of the protection layer which brings significant benefits to the laboratory. First, occupational safety is increased since the operator is no longer exposed to hot and hazardous liquids. Second, automatic preparation improves reliability and reproducibility of the process. Third, there is no need for special training to perform the operation and the personnel's workforce is released for more efficient and responsible tasks.

The method is also applicable to the automatic fusion system HAG-HF. This enables the implementation of applications with high sample throughput requiring the formation of a protection layer.

The automatic protection layer formation is a unique feature of HERZOG high-frequency fusion systems as it is based on three-dimensional rocking movements of the crucible. Most other commercial fusion systems offer rotation of the crucible only around one axis. However, this is not sufficient for building a layer covering the entire circumference of the crucible.

References

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Germany

HERZOG Maschinenfabrik
GmbH & Co.KG
Auf dem Gehren 1
49086 Osnabrück
Germany
Phone +49 541 93320
info@herzog-maschinenfabrik.de
www.herzog-maschinenfabrik.de

USA

HERZOG Automation Corp.
16600 Sprague Road, Suite 400
Cleveland, Ohio 44130
USA
Phone +1 440 891 9777
info@herzogautomation.com
www.herzogautomation.com

Japan

HERZOG Japan Co., Ltd.
3-7, Komagome 2-chome
Toshima-ku
Tokio 170-0003
Japan
Phone +81 3 5907 1771
info@herzog.co.jp
www.herzog.co.jp

China

HERZOG (Shanghai) Automation
Equipment Co., Ltd.
Section A2, 2/F, Building 6
No. 473, West Fute 1st Road,
Waigaoqiao F.T.Z., Shagnhai,
200131
P.R.China
Phone +86 21 50375915
info@herzog-automation.com.cn
www.herzog-automation.com.cn