Use of Platinum Ware in XRF

Introduction

Crucibles and casting dishes are needed to produce fused beads with the VITRIOX® ELECTRIC and VITRIOX® GAS fusion machines. A special platinum and gold alloy with a composition of 95% Pt and 5% Au is commonly used. This alloy is characterized by mechanical durability and, due to the gold content, low material adhesion. In addition, this alloy is also available as FKS platinum, i.e., by adding a trace of zirconium oxide, the service life can be further increased. FLUXANA also offers quartz crucibles (Can only be used in the VITRIOX® ELECTRIC) as an alternative to platinum crucibles.

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Type of platinum ware used in XRF

Crucible:

The standard crucible has a volume of 20 ml and is used for most fusions. For some special applications, e. g. ferro-alloys and slags, a larger crucible or a crucible with bumps are used to guarantee a successful fusion. Another special case is the quartz crucible. It´s a development by FLUXANA that allows the fusion of samples that would damage platinum ware (e.g. aluminium, carbides, nitrides). Quartz crucibles can only be used once and can only be used on the VITRIOX® ELECTRIC, not on the VITRIOX® GAS.

Mould (or casting dish):

Moulds come in different sizes depending on the XRF spectrometer in which the fused beads are analysed. Common diameters range from 29 to 39 mm.

Lids:

By using lids that are mounted onto the crucible, volatile elements can be analysed using the VITRIOX® ELECTRIC. This allows the user to analyse for example fluorine and chlorine in a cement sample. Without the use of lids, these elements would evaporate during fusion.

Recycling of platinum ware:

After some time, the platinum ware begins to wear down. Damage to the surface and sagging of the casting dishes are common signs of the end to the life-time of the platinum ware. The platinum ware then needs to be recycled. FLUXANA offers this recycling service without any risk or downtime for the customers.

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Here are some examples for signs of wear which indicate that the platinum should be recycled soon:

Crucible:

- Heavily increased time needed for cleaning the crucible after fusion
- Increasing amount of leftover sample in the crucible after fusion (see Fig. 1 upper left)
- Strongly roughened crucible bottom (see Fig. 1 upper right)
- Deformation of and big scratches on the crucible
- Heavily attacked platinum with or without holes (Fig. 1 bottom)

Figure 1: different types of damage to the crucible

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Mould:

- Deformation of and big scratches on the mould
- Fused bead is sticking to the mould
- Strongly roughened mould surface (see Fig. 2 left)
- Bottom of the mould is no longer straight but forms a bump (see Fig. 2 right)

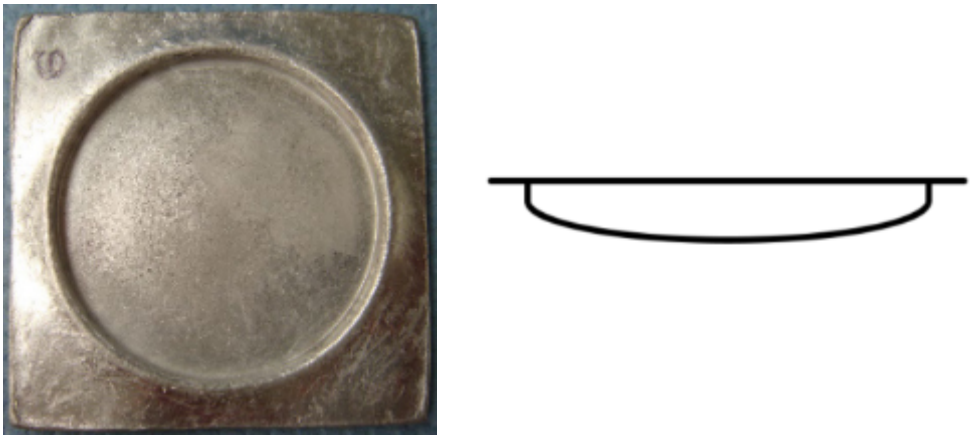

Figure 2: Strongly roughened mould surface (left), mould bottom forms a bump (right)

Available Platinum Ware:

Both crucibles and casting dishes are required to conduct the fusion. Both are available in different sizes and models.

Platinum Ware for VITRIOX® ELECTRIC

Item No.	Description	Inner/Outer Diameter in mm	Height mm	Bottom mm	Weight g
FS-VIT01	Crucible VIT.E	20 / 34	38	0.5	45
- FS-VID1	Lids for FS-VIT01	-	-	-	8
FS-VIT02	Crucible VIT.E Viscous	22.9 / 39.1	36	0.5	54
FS-VIT03	Crucible VIT.E Ferro	22.9 / 39.1	36	0.5	55
- FS-VID02	Lids for FS-VIT02/03	-	-	-	11
FS-VIA4012	Mould VIT.E	29 / 31	3.8	0.8	30
FS-VIA4011	Mould VIT.E	32 / 34	3.8	0.8	30
FS-VIA4010	Mould VIT.E	34 / 36	3.8	0.8	46
FS-VIA4009	Mould VIT.E	39 / 41	3.8	0.8	46
FS-VIA4015	Mould VIT.E	39 / 41	3.8	1.4	80

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Platinum Ware for VITRIOX® GAS

Item No.	Description	Inner/Outer Diameter in mm	Height mm	Bottom mm	Weight g
FS-OT866	Crucible VIT.GAS	20 / 34	38	0.5	45
FS-OT867	Crucible VIT.GAS	22.5 / 36	38	0.5	54
FS-OA434	Mould VIT.GAS	29 / 31	3.8	0.8	31
FS-OA877	Mould VIT.GAS	32 / 34	3.8	0.8	31
FS-OA438	Mould VIT.GAS	34 / 36	3.8	0.8	45
FS-OA439	Mould VIT.GAS	39 / 41	3.8	0.8	46

Table 1 Overview of platinum ware for VITRIOX® ELECTRIC and VITRIOX® GAS

Standard Applications in Fusion

For standard applications, 1 g sample and 8 g flux (e.g., FX-X65) are weighed into the crucible. The VITRIOX® ELECTRIC and GAS fusion machines have preset fusion programs so that the crucible with the sample and the corresponding casting dish must simply be placed into the machine and then the fusion is carried out automatically. When completed, it is only necessary to remove the finished fused bead from the casting dish and measure it in the X-ray fluorescence instrument.

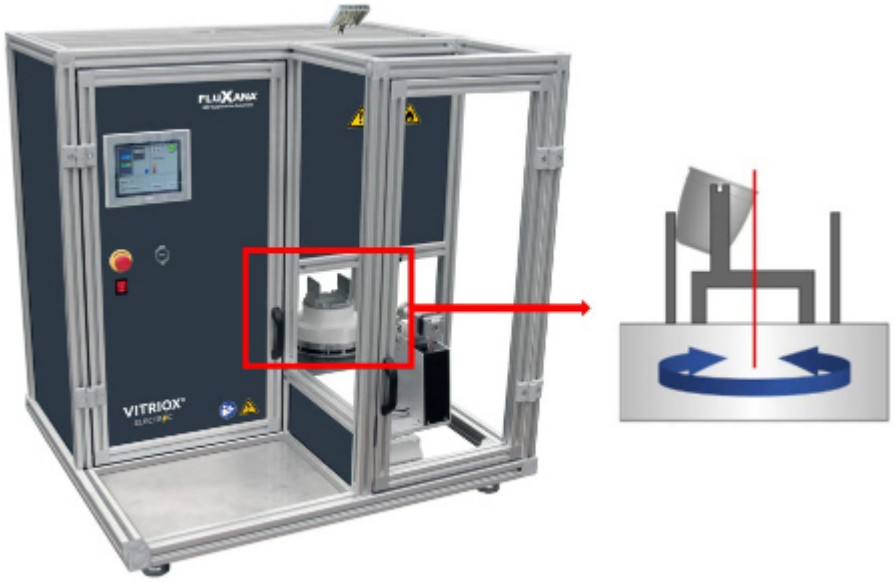

Figure 3: Rotation stirring principle in the VITRIOX® ELECTRIC

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What makes the VITRIOX® ELECTRIC so special is the 3D stirring principle (Fig. 1). The crucible is rotated with high speed outside the axis of rotation. Acceleration and deceleration lead to the stirring effect as shown in Fig. 2. The stirring of the VITRIOX® GAS work in a similar fashion.

Figure 4: Application of the rotation stirring principle in the crucibles with the VITRIOX® ELECTRIC

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Cleaning of the platinum ware after fusion.

Used platinum ware needs to be cleaned in an ultrasonic bath:

Figure 5: ultra sonic bath for the cleaning of platinum

Tools

Item No.	Description
FS-OZ327	Crucible tongs, platinum shoes, length 230 mm, ca. 5 g Pt
VU-US1.9kit	Ultrasonic bath with heating, Vol. ca. 1.9 l for cleaning

Table 2: Tong and ultrasonic bath are important tools for the cleaning of platinum ware after fusion

For more information on the use of the ultrasonic bath see this video: Link

The following solutions and settings can be used when cleaning:

- 10% citric acid in a beaker at 50°C in an ultrasonic bath
- Faster: 20% citric acid in a beaker at 80°C in an ultrasonic bath
- More effective, but also more aggressive: 10% HCl in a beaker

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After the platinum ware was cleaned in the ultrasonic bath, it must be washed with distilled water and dried.

Note: The ultrasonic bath must never be filled directly with acids. When using acids, a beaker with a rubber retaining ring and white plastic lid must be used.

Safety instructions when cleaning with an ultrasonic bath

- After inserting the glass, the water level in the ultrasonic bath must not fall below the mark. However, to avoid overflowing, the bath must not be overfilled either. The inserted beaker must float freely in the basin without touching the bottom.
- The filling for the ultrasonic bath should consist only of water with a few drops of dishwashing liquid.
- Solids in the ultrasonic bath and contact between the bottom of the beaker and the bottom of the ultrasonic bath support pitting and thus total damage.
- When using 10% hydrochloric acid, the most effective cleaning agent, it is important to be sure that none of the acid drips into the bath when removing the ware from the ultrasonic bath. In this case, the water in the ultrasonic bath must be replaced immediately; otherwise the bottom of the ultrasonic bath would be destroyed in a short amount of time.
- Acid drops that get onto the white plastic lid must be removed immediately (e.g. paper towel). Do not rinse with water while it is lying on the ultrasonic bath. If rinsing, then only under running water in the sink!
- During extensive use, cleaning of platinum ware becomes more difficult, as a small amount of gold is dissolved out of the surface with every fusion.
- Always wear personal protective clothing when working with HCL acid! Lab coat, protective gloves and face protection are mandatory.

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Special Applications with the VITRIOX® ELECTRIC and VITRIOX® GAS: Ferroalloys and Slag

In addition to oxidic materials that can be simply mixed with a flux and then fused, there are other materials, such as ferro-alloys or slag with metallic components that require an extra oxidizing agent to prevent damage to the platinum ware.

Using a VITRIOX® ELECTRIC or GAS in combination with an obligatory pre-oxidization step, sample, oxidizing agent and flux can be weighed directly into the crucible for many of these materials. The FS-VIT03 (VITRIOX® ELECTRIC) and FX-OT867 (VTIRIOX® GAS) crucibles were developed for this application because more volume for the oxidation reaction is required in the crucible (see Fig. 3).

Figure 6: FS-VIT03 and FS-OT867 crucible compared to a standard FS-VIT01 crucible.

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Special Applications with the VITRIOX® ELECTRIC: High Viscosity Fusions, e.g., Sand or Phosphate

Fusion with the VITRIOX® ELECTRIC is characterized by its high repeatability for XRF analysis. It is superior to all other commercially available fusion machines. The reason for this is the unique, patented stirring principle. Basically, the efficiency of the stirring depends on the viscosity of the fusion. If this is too high, the contents of the crucible cannot be homogenized. FLUXANA developed the special FS-VIT02 crucible to overcome this problem. This crucible is equipped with dents and a champagne bottle shaped bottom. The changes in the surface force the fusion to be well mixed. This new stirring principle was successfully developed during a comprehensive bachelor thesis (see Fig. 7). At this time, the minimum stirring speed required to achieve a homogeneous fusion with sufficient precision was also examined. As a result, the diagram below shows that the new FS-VIT02 crucible requires a much lower stirring speed than the standard FS-VIT01 crucible, thus making it especially suitable for high viscosity fusions.

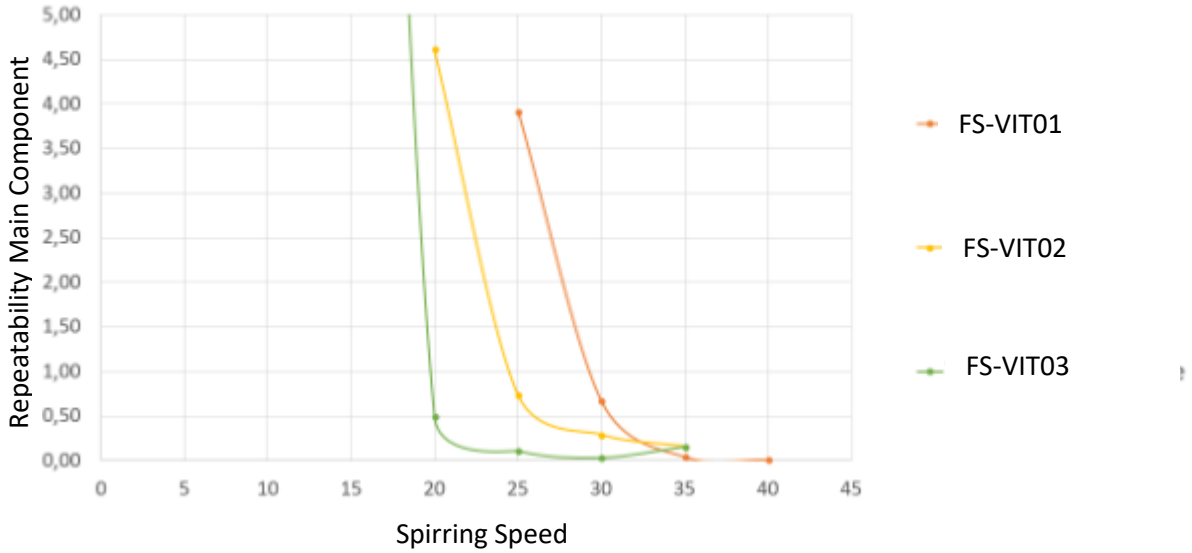

Figure 7: Stirring speed depending on the precision for different crucible forms.

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Special Applications with the VITRIOX® ELECTRIC: Using Quartz Crucibles

It happens again and again that the fusion of samples containing so-called platinum poisons (sulfides, metals especially aluminum, carbides, nitrides) leads to considerable damage to the platinum/gold crucibles used. The use of quartz crucibles as an alternative to platinum crucibles is described in a separate whitepaper [2].

Literature

[1] Rainer Schramm, XRF Practical and Easy, 2nd Edition, FLUXANA (2017).

[2] Whitepaper: The Use of Quartz Crucibles in Place of Platinum

[3] Whitepaper: Influence of the Stirring Mechanism on the Precision of Fusion

[4] www.fluxana.com