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DOUBLE-VACUUM CRUCIBLE FOR TEMPERATURES UPTO 1600°C

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A new design of dual-vacuum crucible (Staudacher type) is described. The lower and upper part of the crucible is made of tantalum and titanium, respectively. The lower part is heated by a tantalum wire coil inserted into a helix furrow made in a thick-walled insulating cylinder. This cylinder was made from a machineable boron nitride ceramic. By means of this, the crucible may be safely heated in vacuum up to 1600°C.

Keywords: *boron nitride, crucible, extraction, noble gases, tantalum, titanium, UHV*

Introduction

Extraction of noble gases from mineral samples for mass spectrometric analysis is most often achieved by means a double vacuum crucible, e.g. as described by Staudacher *et al*. [1] and McDougall and Harrison [2]. This type of furnace consists of a cylindrical crucible made of tantalum, which is heated by a pair of semi-cylinders surrounding the crucible installed in the external vacuum, i.e. in an ultra-high-vacuum (UHV) chamber. This heating unit requires high electric power (low voltage but high current) to achieve temperatures up to 1500°C, although the heating semi-cylinders are screened by several metal cylinders in order to reduce heat loss due to radiation.

A similar system was constructed in our laboratory by Halas and Durakiewicz [3] several years ago, but the power supplied to the heater was significantly reduced by applying a zirconia (ZrO_2) ceramic as a thermal barrier. This ceramic has a very low thermal conductivity, but it is extremely sensitive to thermal shock. Also the heater made of tantalum wire (1.5 mm diameter, 1.0 m length) had the tendency to collapse at the highest temperature of 1500°C although ZrO_2 spacers were used. The drawbacks of using fragile ceramic and tantalum wire for the heater led us to an entirely new construction for the furnace. The zirconia was replaced by a machineable ceramic, boron nitride (BN), and the tantalum heating wire was installed into the ceramic body as described below. In this way we have obtained a highly useful furnace of low power consumption, which can be operated from room temperature up to 1600°C in a short time.

Construction

The materials used in the construction of the furnace are listed in Table 1 along with their physical properties. The crucible was made from a tantalum tube with an external diameter of 12.7 mm and a wall thickness of 0.5 mm. The bottom of the tube was made of a tantalum disc, 1.0 mm thick, which was electron-beam-welded. The length of the tantalum tube is 10 cm only and it comprises ca. 50% of the total length of the crucible. The upper part of the crucible, to which the tantalum tube was electron-beam-welded, is made from a titanium rod in such a manner that it can be connected to a water-cooled stainless CF 19 flange made in the upper part of the external vacuum chamber via a typical copper gasket, Fig. 1. While the part of the crucible made of tantalum is heated, the upper part made of titanium is exposed to vacuum and it conducts and radiates heat, which flows from the heated part of tantalum.

The heating tantalum wire, 1 mm in diameter, is perfectly insulated from the crucible cylinder by means of the machineable ceramic, BN. The wire was formed in the shape of a coil which was turned into helix furrow made in a thick wall BN cylinder as shown in Fig. 2. The bottom of the heater is closed by a piece of BN rod in which a Pt–Pt90Rh10 thermocouple was installed. The top of the heater is closed by a BN ring which fits to the Ta tube. All the BN pieces are inserted into molybdenum cylinder with a molybdenum ring at the bottom, which is connected by 2 steel springs to the upper part of the vacuum chamber, Fig. 1. The ends of the heating wire are connected to the power feedthroughs (5 mm

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Table 1 Selected physical properties of the materials* used in the construction of the crucible

Material	Melting point or upper continuous use temperature in UHV/°C	Thermal conductivity at room temperature/W m^{-1} K^{-1}	Linear coefficient of expansion at room temperature/$\cdot 10^{-6}$ K^{-1}
Titanium	1660	21.9	8.9
Tantalum	2996	57.5	6.6
Boron nitride	2500	15–50**	1.0***

*after Goodfellow Catalogue 2002; **depends on porosity; ***this value rises up to 30 at 1000°C.

diameter copper rods in CF 40 flange) at the bottom of the external vacuum chamber.

A general view of the UHV system used for safe heating of the crucible is shown in Fig. 3. A modern ion pump with light permanent magnet is connected to the chamber via a rectangular bellows-seated valve equipped with CF 40 flanges.

That temperature of the crucible is controlled by an ON/OFF digital controller, type BTC-9090 in our case, with precision of several degrees. The temperature indicated by the thermocouple can be tested by means of a pyrometer through a commercially available CF 16 viewport installed at the top of the crucible (Fig. 4). The viewport is protected by a home-made shutter comprising a single 0.5 mm thick nickel disc which can be rotated by a piece of a small external magnet. The disc axis is supported in the inner wall of a 20 mm thick Cu-gasket which fits to CF 19 flanges. The rotatable disc is opened during pyrometric measurement of temperature only, Fig. 5.

Fig. 1 The tantalum–titanium crucible with newly designed heating unit installed in the external vacuum chamber, a view through CF 100 access flange

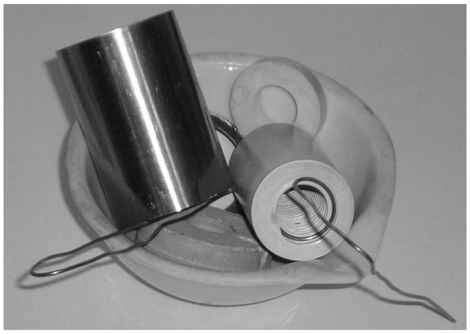

Fig. 2 The main parts of the heating unit: 1 mm tantalum wire coil is inserted into a furrow helix in the boron nitride cylinder

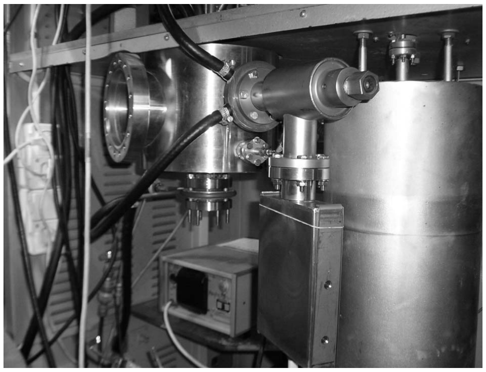

Fig. 3 A general view of the vacuum system. On the right is 25 L s^{-1} ion pump connected via a rectangular bellows-seated valve

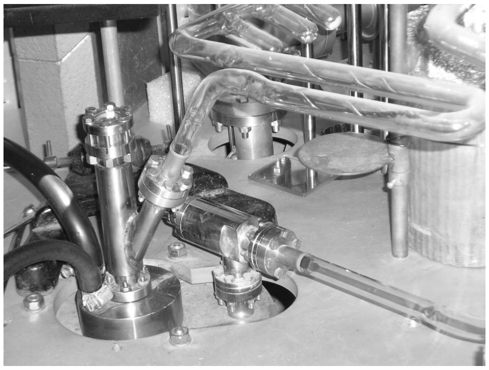

Fig. 4 A general view of the inner vacuum system for the extraction of noble gases

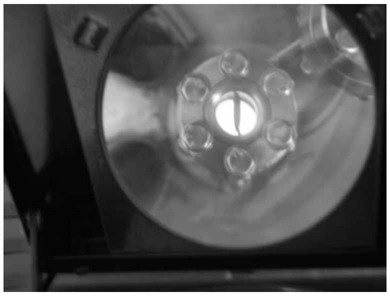

Fig. 5 A view through the viewport for pyrometric temperature determination of bottom of the crucible

Performance of the furnace

The maximum temperature obtained in our crucible is 1550°C, but higher temperature would be possible with use of a more effective pump to produce the external vacuum. In our case a small ion pump of 25 L s^{-1} speed was applied. With this pump the vacuum dropped to about 0.1 Pa at 1550°C, due to a strong degassing of the ceramics.

Our newly designed double-vacuum crucible is used for more than one year as a low-blank system for concentration determinations of noble gases in refractory minerals and glasses by means of static-vacuum mass spectrometry. The power consumption is always below 2.5 kW. The maximum A.C. voltage measured at the ports of the heating coil was 12 V when the total length of the heating wire was 1 m. It was noticed that this voltage varies nearly linearly with the measured temperature of the crucible. The temperature of 1000°C may be obtained over 10 min without any danger to the crucible components. Another 10 min is required to reach the maximum temperature. A low-rate water cooling is applied when the crucible is run above 1000°C, the following 3 flanges are cooled: at the top of the crucible, the power feedthrough and the ion-pump.

After one year of using of the reconstructed furnace we are still very satisfied of its performance. The only service which has to be done after 10–20 extractions is the removal of ‘ashes’ from the bottom of the crucible. This can be easily done after opening the viewport flange by means of a tube combined with an ordinary vacuum cleaner.

The low power consumption was achieved because heat loss was limited by applying the titanium endpiece of the crucible (the upper part of the crucible). Titanium has much lower thermal conductivity in comparison with tantalum, Table 1. Moreover, the use of titanium helps to obtain more clean noble gases due high absorption of reactive gases during cooling of the furnace after extraction.

References

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