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A PRACTICAL SUBLIMATION SOURCE FOR LARGE-SCALE CHROMIUM GETTERING IN FUSION DEVICES*

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ABSTRACT

This paper describes the technique of chromium gettering with a large-scale sublimation source which resembles in its design the VARIAN Ti-Ball™. It consists of a hollow chromium sphere with a diameter of approximately 3 cm and an incandescent filament for radiation heating from inside the ball. While the fabrication of the source is described in a companion paper, we discuss here the gettering technique. The experimental arrangement consists of an UHV system instrumented for total and partial pressure measurements, a film thickness monitor, thermocouples, an optical pyrometer, and appropriate instrumentation to measure the heating power. The results show the temperature and corresponding sublimation rate of the Cr-Ball as function of input power. In addition, an example of the total pumping speed of a gettered surface is shown.

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I. Introduction

Gettering the inner wall of the plasma vessel has improved the performance of many plasma devices. The perferred getter material has been titanium because of well developed techniques and commercially available sublimation sources. Titanium gettering controls gaseous impurities such as O_2, CO and N_2 in fusion devices, but also pumps the plasma fuel, i.e., hydrogen isotopes. While impurities are mainly pumped by surface adsorption, hydrogen pumping consists of surface adsorption with subsequent bulk diffusion. Thus, as a function of applied sublimation cycles, the film grows thicker and large hydrogen inventories are built up in the device. This precludes the use of titanium getters in fusion experiments involving tritium operation since the tritium inventory must be kept as low as possible.

Recent investigations have shown that chromium gettering provides a promising alternate to titanium gettering [1,2]. While titanium pumps large quantities of hydrogen, the pumping capacity of chromium for hydrogen is only about one monolayer adsorbed on the surface without any evidence of bulk diffusion. Sticking coefficients as function of chromium surface coverage for various gases of interest in fusion research have been measured and found to be similar to titanium. As a result of these investigations, chromium has been proposed as a getter material for fusion applications.

This paper describes the technique of chromium gettering with a large-scale sublimation source as required for fusion applications. The new source resembles in its design the VARIAN Ti-Ball™ and consists of a hollow chromium cylinder with hemi-spherical ends approximately 3 cm in diameter and 4 cm in length and an incandescent filament for radiation heating from inside the ball. Construction details are given elsewhere [3].

In the experiments to be discussed in this paper, measurements were made of the input power required to heat the Cr-ball to temperatures which produced the same sublimation rates as the Ti-balls™ currently used in fusion machines.

II. Experimental Apparatus

The experiment was contained in a stainless steel bell jar as shown in Fig. 1 and was pumped by either a 400 ℓ/s turbomolecular pump or a 60 ℓ/s ion pump depending upon the specific experiment being conducted. The sublimation source (Ti or Cr) was mounted at the center of the chamber in order to provide the most homogeneous deposition possible from these sources. A nude ionization gauge to provide total gas pressure measurements and a quadrupole mass spectrometer to provide gas analysis data were installed with 90° elbows to avoid line-of-sight paths with the source. A viewport, to allow optical temperature measurements, was mounted at the end of a 50 cm tube with a shutter at the opposite end to minimize depositions on the window. A thermocouple was attached to the sublimation source

to calibrate the optical pyrometer with respect to emissivity of the Cr-ball and transmission of the viewpoint. A leak valve was installed to provide a constant leak rate for measuring the total pumping speed of the deposited getter film. A 60V/60A d.c. power supply was used to heat the sources.

III. Sublimation Rate Measurements

Measurements at various temperatures and corresponding sublimation rates were made on the Cr-ball and compared to the corresponding Ti-ball^{TM} data which served as a baseline. Vapor pressure data for chromium and titanium may be found in the literature [4]. The relation between vapor pressure and sublimation rate is easily obtained assuming that at any given vapor pressure the sublimation rate R_{subl} is in equilibrium with the adsorption rate at the surface:

$$R_{subl}(T) = \alpha \frac{n\bar{v}}{4} \tag{1}$$

where α, n and $\bar{v}$ are the sticking coefficient, density and average velocity respectively. The sticking coefficient of metals is commonly assumed to be unity. Substituting $n = p/kT$ and $\bar{v} = \sqrt{8kT/\pi m}$, we obtain the sublimation rate

$$R_{subl}(T) = 3.5 \cdot 10^{22} \frac{p(T)}{\sqrt{MT}}, \tag{2}$$

where M is the molecular weight, p(T) the pressure in Torr and T the absolute temperature. The calculated sublimation rates, R_{subl}, in

units of atoms/cm^{2}s, as a function of temperature for titanium and chromium are shown in Fig. 2 as solid lines.

Experimental values of the sublimation rate, R_{subl} were determined by measuring the deposition rate on a film thickness monitor (oscillating quartz crystal) at a distance Y from the Cr-ball. Assuming spherical isotropy of the chromium source, the measured deposition rate was integrated over the sphere with radius Y to obtain the total deposition rate from the Cr-ball. Relating this to the surface area of the Cr-ball, which is 38 cm^2, finally yields the specific sublimation rate in atoms/cm^{2}s.

Temperature measurements were made with an optical pyrometer and a thermocouple. After contact failures with spot-welded thermocouples on the chromium source, a sheathed thermocouple was placed in a well drilled into the chromium. This technique was also unsuccessful because of inadequate thermal contact which produced errors of ~ 20% at 1000 °C. Due to these problems, the temperature measurements were finally performed utilizing an optical pyrometer. Since optical pyrometry requires a knowledge of the emissivity of the object whose temperature is being measured, a near-blackbody cavity consisting of a 0.5 mm diameter hole 2mm in depth, was made in each sublimation source[5]. The temperature within the hole was measured with a micro pyrometer having a spatial resolution of 0.1 mm at the hole. The data obtained in this manner was used without additional corrections.

Measured values of the sublimation rate, R_{subl}, as a function of temperature are shown in Fig. 2 for chromium and titanium. The solid circles represent values measured from the Ti-ball™ source utilizing the VARIAN power supply at a setting of 0.1 gm/hr, the sublimation rate usually used in ISX-B.

Figure 3 shows the measured values of R_{subl} as a function of d. c. input power for the Cr-ball and Ti-ball™.

IV. Performance of the Getter Film

Mass scans taken before and after chromium gettering are shown in Fig. 4. In this example, the system had been up to air and contained an unusually high percentage of water vapor. A typical pump-down, with a chromium getter cycle, is shown in Fig. 5 where the total pressure is shown as a function of time. In this example the vacuum chamber was pumped to a quasi-equilibrium pressure of 1.0×10^{-7} torr by the 60 ℓ/s ion pump. A chromium getter cycle was then initiated which lasted for 3.5 minutes. During this time the pressure rose to 8×10^{-7} torr due to outgassing of the source and surrounding areas and then decreased as the pumping speed exceeded the outgassing rate. When the getter source was turned off, a second steep pressure drop was observed and at the end of a 20 minute period the total pressure was reduced to 8×10^{-9} torr. The average specific pumping speed of the getter was in this case 0.7 ℓ/s cm^2 with water vapor the major component of the residual gas. Since the sticking coefficient and therefore the pumping speed of any getter material depends upon many variables including surface contamination, roughness, and temperature, measured values may vary

significantly. Sticking coefficients also depend greatly on the gas species being pumped. The initial sticking coefficient for methane on chromium is less than 10^{-3} and inert gases are not pumped except by "burial" during the getter cycle. On the other hand, we have measured values of near unity for the initial sticking coefficient of oxygen on chromium. We have also observed that the pumping capacity of chromium as well as titanium getter surfaces for oxygen is essentially unaltered after the surface has been saturated with either deuterium or nitrogen [6].

V. Summary

Several Cr-balls have been lab-tested and few problems have been encountered. Above input power levels of 700 watts, short circuits in the filaments have been observed and a filament failed while operating at 1 kW. Although large crystallites are formed in the chromium, there have been no problems in thermal cycling from zero power to full operating power. Large outgassing rates have been observed from uncooled vacuum chamber walls in a small system while heating the Cr-ball. Therefore, if this source is installed in a chamber where the gettering surface is close-by, the surface should be water-cooled.

For convenience, the Cr-ball may be maintained at stand-by power (~ 150 watts) if desired. In this case approximately 3 minutes after application of the power required (~ 410 watts) to reach a temperature of 1150°C, a sublimation rate of 0.1 gm/hr should be established.

References

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Figure Captions

Fig. 1. Experimental arrangement: (1) sublimation Source (Ti or Cr), (2) thermocouple, (3) quartz crystal film deposition monitor, (4) leak valve, (5) viewport, (6) optical pyrometer, (7) shutter, (8) quadrupole mass spectrometer, (9) nude ionization gauge, (10) stainless steel vacuum chamber.

Fig. 2. Measured sublimation rates in atoms/cm^2s as a function of temperature for chromium and titanium. The solid lines are calculated from vapor pressure curves. The data for titanium represented by solid circles were obtained by setting the VARIAN Ti-ball^{TM} power supply at 0.1 gm/hr.

Fig. 3. Measured sublimation rates in gm/cm^2s as a function of input power to the filament of the chromium and titanium sources.

Fig. 4. Mass scan "before" and "after" chromium gettering.

Fig. 5. Total pressure as a function of time before, during, and after chromium getter cycle.

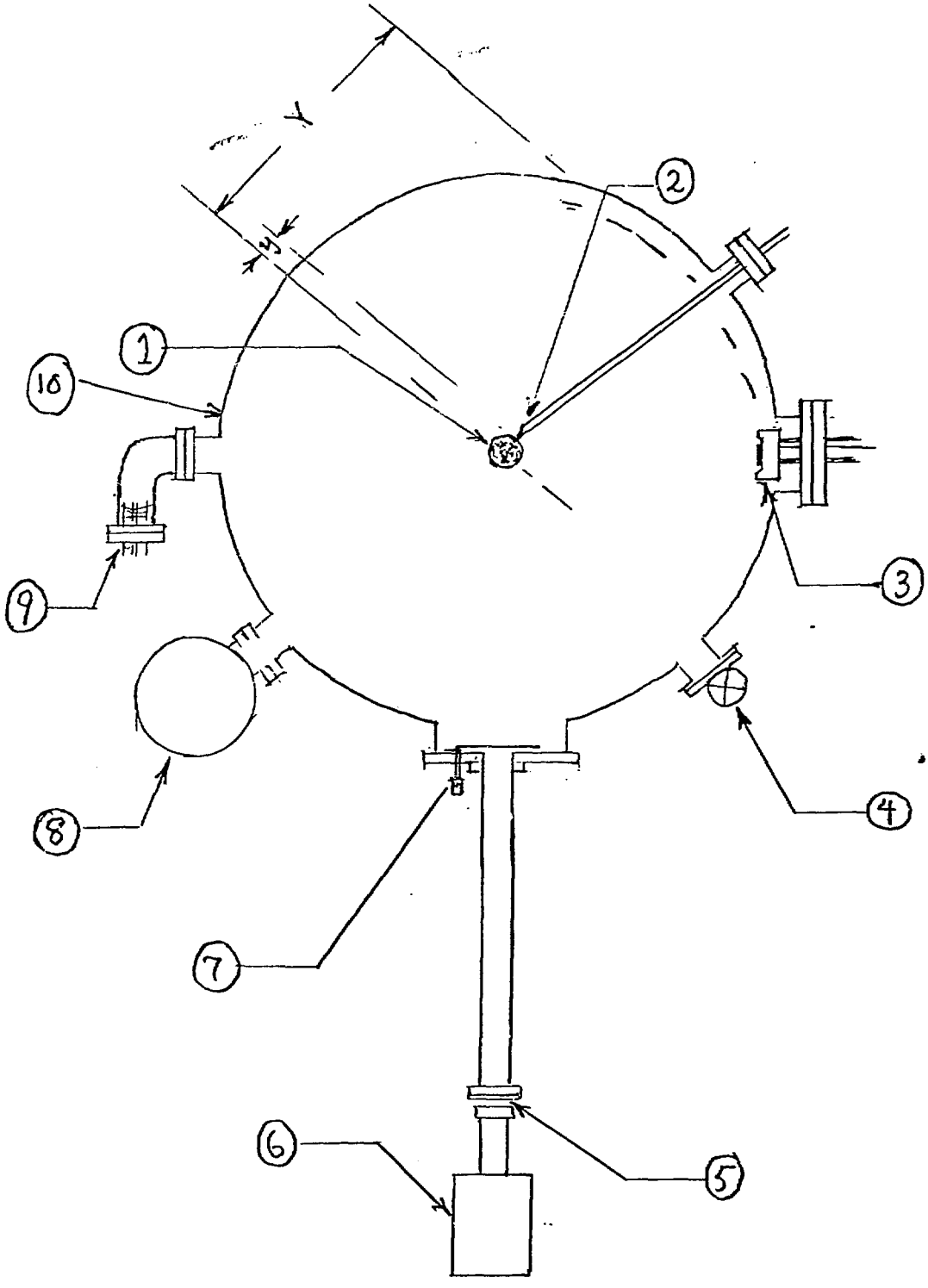

Fig. 1