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THE THERMAL OUTGASSING RATE OF MATERIALS USED IN VACUUM SYSTEMS

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Abstract

There are many rarely used materials in vacuum systems that are poorly investigated in terms of vacuum properties. For example, phosphors, scintillating materials, ferrites, various adhesives, etc. In addition, new organic materials are being developed with mechanical properties similar to those of conventional steel. The use of such materials is very promising in vacuum technology. This article presents the thermal degassing performance of several rarely used materials and promising materials for vacuum applications.

INTRODUCTION

At the moment, the outgassing properties of standard materials applying in high vacuum, such as stainless steel, copper and its alloys, aluminum and its alloys are studied well [1]. Nevertheless, it is often necessary to apply new substances, which are not widely spreaded in the vacuum technologies. For example, boron carbide is used for safety personnel and equipment from radiation induced fast neutrons; phosphors or scintillating materislas are used for beam diagnostics. Popularity of organic glasses, which mechanical properties are comparable with a steel is growing up. All these new materials are required the detailed investigations of their vacuum properties, such as outgassing rate, penetration for different gases, etc. before application in high-vacuum system.

Boron carbide is solved to be used in ITER (France) for a safety radiation of the personnel and equipment. Probably, the phosphor P43 will be applied in scintillating screens in Collector Ring (FAIR, Germany). Ultrahigh molecular weight polymethylmethacrylate can be employed in accelerating tubes in the industrial accelerators, although in this case the ultrahigh vacuum is not required.

SETUP AND EXPERIMENTAL METHOD

An experimental setup for measurement of outgassing rates shows on Fig. 1. Preliminary pumping of the system was carried out by a turbomolecular station, which included a turbomolecular pump TMP1 and oil-free membrane pump MP.

The pressure in the system was measured by the PA hot-cathode pressure gauge. Partial gas pressures were measured with the RGA quadrupole mass spectrometer from SRS (Stanford, United States).

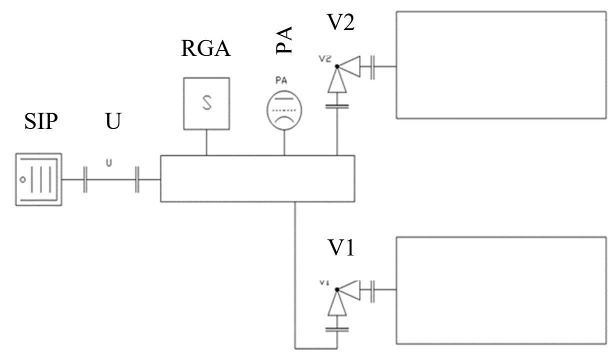

Figure 1: Experimental setup for outgassing rate measurements.

where SIP – sputter ion pump + TS (1000 l/s for H_2), RGA – mass-analyzer (up to 200 a.e.m.), PA – gauge with hot cathode (up to 10^{-12} Torr), U – pipe with known molecular conductivity, V1 and V2 – all metal gate valve.

The measurement method is based on the measurement of pressure drop on a vacuum element with known molecular conductivity (Dynamic Flow Method), as well as comparing the gas release of the empty test volume and the volume filled with samples. Measured background outgassing (stainless steel) was $3.3*10^{-11}$ Torr·l/s/cm^2 at 100 ºC,

$$q = \frac{(Po-Pc)U-Qss}{Asample} \quad (1)$$

where q – outgassing rate, Torr·l/s/cm^2; A_{sample} – total surface of samples, cm^2; P_o – pressure when valve V1(V2) is opened on the tested volume with samples, Torr; P_c – pressure when valve V1(V2) is closed on the tested volume with samples, Torr; Q_{ss} – gas flow of empty volume, Torr·l/s; U – molecular conductivity, l/s.

BORON CARBIDE

The outgassing test was conducted to confirm that the ceramic meets the requirements of the ITER Vacuum Handbook [2], i.e. outgassing rate is not more than 10^{-10} Torr·l/s/cm^2 for hydrogen and not more than

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10^{-12} Torr·l/s/cm^2 for all other impurities. Prior tests, samples were cleaned with isopropyl alcohol first of all, then ultrasonically treated into distilled water, and finally baked at 1000 ºC in vacuum. Baking cycle prior outgassing test was the following:

- Ramp up from room temperature to 320 ºC: 10 h,
- Steady temperature at 320 ºC for 24 h,
- Ramp back to room T: 10 h,
- Steady temperature at RT for 36 h,
- Ramp up to 100 ºC: 3 h,
- Steady Temperature at 100 ºCelsius for testing.

The area of ceramics was more than ten times bigger than the area of empty volume. After a year in a vacuum, the outgassing rate is strongly reduced: outgassing rate was reduced by three and half times, to 1.9·10^{-12} Torr·l/s/cm^{-2} (Table 1) [3-5]. Ceramic outgassing rate meets ITER Vacuum Handbook requirements with a large reserve.

Table 1: Measurement Results of Outgassing Rate from Boron Carbide

Time in vacuum	Outgassing rate [Torr·l/s/cm^2] at 100 ºC	Outgassing rate [Torr·l/s/cm^2] at RT
5 hours	7.52·10^{-12}	-
24 hours	7.26·10^{-12}	-
29 hours	7.5·10^{-12}	2,3·10^{-12}
8 months	3.0·10^{-12}	1.1·10-^{13}
12 months	2.22·10^{-12}	5.56·10^{-14}
20 months	2.09·10-^{12}	-
22 months	1.89·10^{-12}	-
23 months	1,87·10^{-12}	-

PHOSPHOR P43

Scintillating screens [6] for accurate bunch profile measurement will be used in Collector Ring. In according to Fig. 2 phosphor P43 is a one of material for scintillator uses with high light yield [7].

A chemical formula of P43 is Gd_2O_2S:Tb. The material deposition method is a sedimentation with extra adhesive material. The obtaining results were carried out within experiments without special cleaning, but before and after «in-situ» bakeout at 140 ºC for 24 hours. The results shows on Table 2.

The experiments reveals the presence of high peak of 32 a.m.u. (see Fig. 3) in the vacuum spectra. This phenomena have not answered yet and the question is still open. Now the setup is improved and only the phosphor will be heated. The tested volume will be kept at room temperature prior to the next experiments.

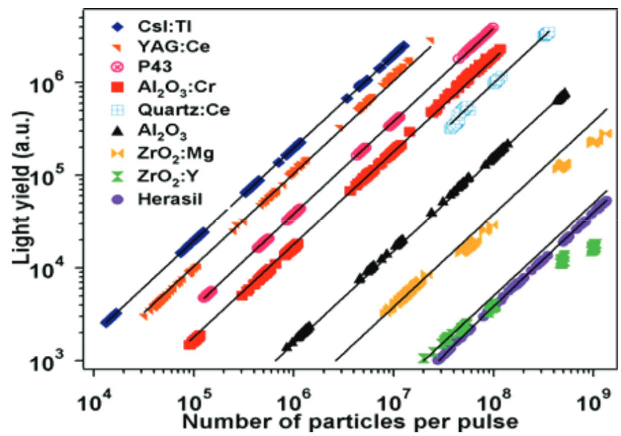

Figure 2: Light yield from various phosphors.

Table 2: Outgassing Rate from Phosphor P43 at Various Conditionings

Time in vacuum	Q, l*Torr/(s*cm^2)
Pumping down nº 1	
24 h	8,0 E-8
14 days	1,3 E-8
Pumping down nº 1. After bakeout nº 1 at 140 ºC	
24 h	1,0 E-10
Air vent. Pumping down nº 2	
24 h	9,4 E-9
48 h	4,6 E-9
Pumping down nº 2. After bakeout nº 2 at 140 ºC	
3 days	4,0 E-11
Air vent. Pumping down nº 3	
48 h	3,7 E-9
Pumping down nº 3. After bakeout nº 3 at 140 ºC	
24 h	3,7 E-11
7 days	1,0 E-11

ULTRAHIGH MOLECULAR WEIGHT POLYMETHYLMETHACRYLATE (UMW-PMMA)

There were used PMMA in the experiments, which subjected a purification by film evaporation method under reduced pressure with rotary vaporizer use.

Process of polymerization was carried out by electron flux from impulse linear accelerator ILU-6 (BINP, Russia).

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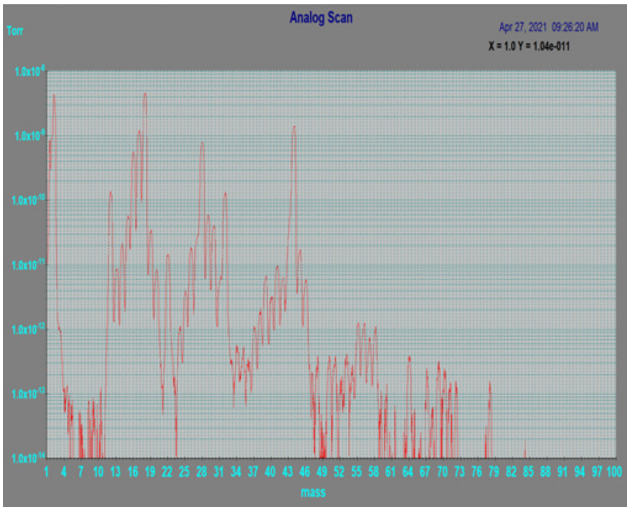

Figure 3: Spectrum residual gases after the third pumping down for 11 days and the third bakeout.

Samples nº 1 were obtained by bulk polymerization method at temperature of 60 ºC for 5 days. A molecular weight M_W~ $8 \cdot 10^6$ g/mole. Samples nº 1 were baked out at 110 ºC or not.

Sample nº 2 was obtained by frontal polymerization method at temperature of 60 ºC for 5 days. A molecular weight M_W~ 10^7 g/mole. Sample nº 2 was baked out at 110 ºC.

Both samples were mechanically treated and polished. After this, they were annealed at 110 ºC for 1 day. For comparison, «usual» PMMA has a molecular weight M_W~ 10^6 g/mole.

Table 3 shows the outgassing rate measurements from UMW-PMMA (sample nº 2) after various conditionings are presented and for stainless steel. «Usual» PMMA and UWM-PMMA (sample nº 1) didn't have any difference for outgassing rate.

CONCLUSIONS

1. The outgassing rate of boron carbide meets the requirements of the ITER Vacuum Handbook. This material is produced in large volumes that made it additionally attractive for use in the ITER. Nevertheless, extensive application of the boron carbide creates a significant gas load on the vacuum system that requires additional vacuum pumps.
2. The application of phosphor P43 is adequate choice. The vacuum level of $3 \cdot 10^{-9}$ Torr can be reached in Collector Ring, but presence of peak 32 a.m.u. requires additional discussions and investigations.
3. UMW-PMMA with frontal polymerization has the best outgassing rate versus «usual» PMMA, but it's necessary to investigate it on penetration for different gases.

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Table 3: Outgassing Rate from UMW-PMMA

Time in vacuum	Q, l*Torr/(s*cm²)	
	Stainless steel	UMW-PMMA Sample nº 2 (annealed)
1 day	2,3 E-10	7,8 E-7
2 days		4,4 E-7
3 days		3,3 E-7
4 days		2,7 E-7
7 days		2,0 E-7
8 days		1,6 E-7
11 days		1,2 E-7
		Bakeout at 120 ºC for 24 h
1 day		1,6 E-8
2 days		1,46 E-8
3 days		1,34 E-8
		Air vent
1 day		1,17 E-7
4 days		2,75 E-8

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