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BORON CARBIDE CERAMICS AS NEUTRON SHIELDING FOR ITER PORT-PLUGS

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Abstract

During the work on integrating diagnostics into the ITER ports, a problem arose with providing the required radiation protection. It was proposed to place a large amount of neutron-absorbing material, boron carbide, in the vacuum chamber in the port plug. Intensive studies of the chemical composition, thermal and vacuum properties of various B_4C ceramics from different manufacturers have been carried out. Sintered ceramics in the form of blocks with holes were selected. A specification for the production of ceramics for ITER ports was agreed upon. Experiments on irradiation of ceramics with neutrons at the VITA accelerator have been carried out. The degradation of mechanical properties after prolonged fast neutron irradiation has been investigated. Serial production of sintered ceramics has been started. The ITER Organization has approved the End of manufacturing reports of serial batches of ceramics.

1. INTRODUCTION

The task of ITER diagnostic ports is to place equipment in them and ensure its operability, including ensuring protection of diagnostics from various loads, including neutron ones. Since the port usually contains several different diagnostic and auxiliary systems, typically developed in different countries, the placement of equipment and its protection is called integration. [1],[2]. Neutron protection should not only reduce the neutron flux during a plasma discharge (which is necessary for the normal operation of various detectors), but also ensure a low level of port activation so that personnel can carry out work in the port after the discharge stops. Basic requirements: shutdown dose rate (SDDR) near the port plug no more than 100 microSieverts/hour after 10^6 seconds (12 days) and no more than 10 microSieverts/hour after 24 hours in the port cell [1] at a distance of 30 cm from the bioshield, Fig. 1.

The neutron yield in the ITER plasma at a power of 700 MW should be $2.48 \cdot 10^{20}$ neutrons/s with a neutron energy of 14 MeV, which creates a neutron flux to the first wall of about 10^{14} neutrons/cm$^2 \cdot$s [3]. The difficulty lies in the fact that both the entire port plug and individual Diagnostic Shield Module (DSM) are subject to strict design restrictions on dry weight: 45 tons of the entire equatorial port (EPP) plug and 10 tons of each DMS in it, which does not allow the use of classical iron-water protection. [2]. For neutron calculations in 2010, based on the conceptual design review (CDR) of the generic structure of the equatorial port plug (GEPP), it was a solid layered cube of steel and water [4]. There were no through holes for diagnostics (the real ports will contain cutouts), so the neutron calculations showed compliance with the design requirements.

Calculations in 2012 of the integrated EPP #1 in sufficient detail showed that if the DMSs consists only of steel and water and is limited by weight and there are cutouts in the first wall, then the SDDR in the near-port space exceeds the requirement of 100 μSv/h by 2 orders of magnitude [5]. After it became clear that the neutron protection of the port could not be provided without filler, the task arose to choose the optimal material. Neutron calculations were performed on the attenuation of a monoenergetic neutron flux with three energies (1 keV, 1 MeV, 14 MeV) when passing through a very simple DMS model with a filler [6]. A large number of materials were compared with each other. DSM with a lot of water inside showed better protection than with boron carbide. B_4C took the 4th place among the reviewed materials in terms of protection effectiveness at a fixed weight of

DMS. In fact, it is impossible to use a significant amount of water to protect ports due to additional requirements for ports (ensuring turbulent flow in Baking mode) and the specifics of French legislation (pressure nuclear equipment order ESPN) [7].

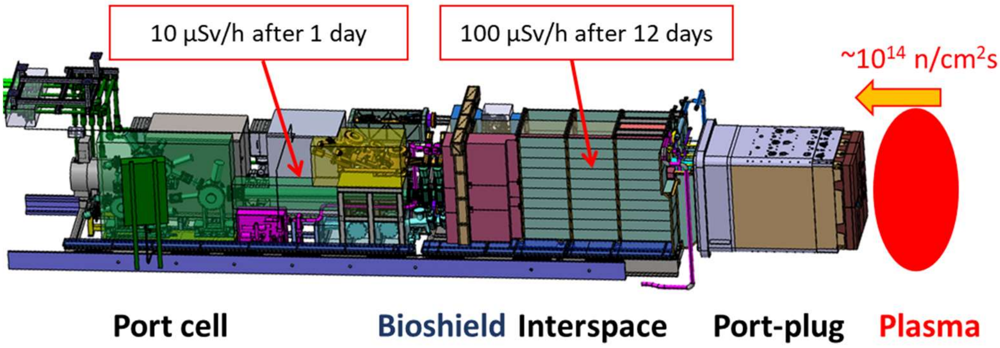

FIG. 1. Diagram of the ITER equatorial diagnostic port #11, approximate neutron fluxes at diagnostic first wall, and dose requirements after reactor shutdown in different zones. The plug port is located in a vacuum, its back plate is the boundary of the vacuum and a physical barrier to tritium retention.

As a result, in 2014 it was concluded that boron carbide is the best material that can be used in ITER ports, but only if vacuum tests confirm its suitability. [6]. The choice of boron carbide is quite clear: due to its low atomic weight and high neutron capture cross-section, boron carbide is an effective flux absorber for both fast and slow neutrons. Boron carbide is also chemically inert and thermally stable.

In 2013, the BINP, together with RFDA and ITER Organization (IO), started the integration of the diagnostic ports (EP11 [8], Upper ports (UP) 02 and 08 [9]). The task for the port integrators was to find and coordinate a way to use B_4C, obtain ceramic samples from manufacturers, conduct comprehensive studies, prove the possibility of using ceramics in ITER diagnostic ports, and conduct all the necessary approvals to start purchasing ceramics.

The first proposal in the BINP on the use of boron carbide in EPP #11 was to pour boron carbide powder into steel boxes with cutouts for diagnostic systems in the port. A similar proposal was considered in the report of the ITER Organization [6], in the same way boron carbide is used in the absorbing elements of fission reactors. The proposal to pour boron carbide in the form of tablets into the cutouts of the all–metal frame of the DMS was made by American colleagues, and later by European integrators. In both cases (powder or tablets) there are two problems: how to ensure heat removal from boron carbide heated by neutrons and gamma rays during discharge (and B_4C heating in the vacuum chamber heating mode) and compliance with ITER vacuum requirements. It turned out that it is strongly discouraged to create trapped volumes in the ITER vacuum chamber [10], so this idea had to be abandoned (Fusion for Energy and IDOM have a special opinion on this issue).

After the ban on the use of B_4C powder, BINP proposed using boron carbide ceramics in the form of ceramic tiles that are tightly packed in steel boxes. This form of ceramic blocks is the cheapest way to make them. The use of ceramic products (tiles, tablets) made of boron carbide as a filler has a number of advantages over the use of powdered boron carbide. Such advantages include a small specific surface area and porosity, increased density close to crystalline, low outgassing rate, shape stability and less dust formation under thermomechanical loads, and high thermal conductivity.

During the joint development of a modular design for boron carbide diagnostic ports with the ITER Organization, it was proposed to use ceramics in the form of blocks with holes that are placed on the trays (Fig. 2) [2],[11]. The blocks are fixed in cassettes by steel rods with steel plate springs mounted on them, Fig. 2. Calculations have shown that with the help of boron carbide ceramics (and other improvements), ITER radiation protection requirements can be met [12].

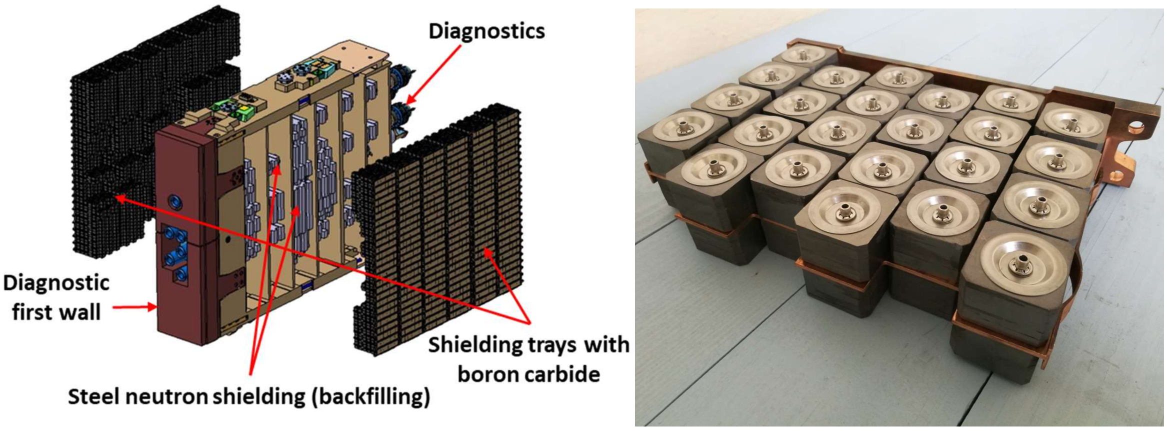

FIG. 2. Radiation protection scheme of the Diagnostic Shield Module (DSM) #2 at ITER Equatorial Port No. 11 (left) and BINP made full scale mockup of shielding tray with B_4C hot-pressed blocks (right).

2. STUDIES OF CHEMICAL COMPOSITION AND THERMAL PROPERTIES OF VARIOUS BORON CARBIDE CERAMICS

An important question that needed to be solved was what type of ceramics to use? The properties and price of ceramics based on boron carbide significantly depend on the technology of its manufacture. There are several main types of boron carbide ceramics [13],[14],[15]:

1) Hot pressed. Ceramics turn out to be very expensive, in addition, productivity is low. The ceramic density is 2.52 g/cm^3, very close to the theoretical maximum. Modulus of elasticity >450 GPa.

2) Reaction bonded. The method is relatively inexpensive and high-performance, based on cold (automatic) pressing of blanks followed by their high-temperature (up to 1500 ˚C) impregnation with molten silicon, during the reaction with which a high-density material is formed, but in this case there is a noticeable proportion of silicon impurities in ceramics, which reduces the boron content. The density is about 2.64 g/cm3, which is higher than that of hot-pressed ceramics, which is critical for ITER ports. The modulus of elasticity is 400 GPa.

3) Sintered (Sintering). Cold (automatic) pressing and sintering in the furnace (~2200 ˚C). This method makes it possible to produce ceramics in large quantities and is much cheaper than hot pressing. With this method, porosity of about 5% is possible. The density is 2.3-2.4 g/cm^3, depending on the production technology. The modulus of elasticity is ~360 GPa.

Numerous studies have been carried out to select a suitable boron carbide ceramic and demonstrate that it meets the stringent requirements of IO [13],[14],[15]. The chemical composition of sintered, hot-pressed and reaction-bonded ceramics from different manufacturers was investigated [13],[14],[15]. The amount of impurities in sintered and hot-pressed ceramics of boron carbide did not exceed 1%, which meets the requirements of ITER [13],[14]. Hot-pressed ceramics have less porosity, but they are much more expensive and the speed of production is limited. Sintered ceramics made by Virial Ltd. were chosen for the ports developed by the BINP [2].

A detailed specification ITER_D_457TBH for the manufacture of ceramics has been agreed with the ITER Organization [16]. The specification specified the requirements for the chemical composition, thermal and vacuum properties of ceramics, as well as the manufacturer's quality assurance requirements. The requirements for the thermal properties of ceramics are given in Table 1. BINP also conducts research on the resistance of boron carbide ceramics to pulsed heating. [17],[18]. The thermal properties of sintered boron carbide ceramics at various temperatures were measured and found to meet the specification [16]. The measurement results are shown in Table 1.

TABLE 1. Thermal properties of ceramics according to the specification [16] and measurement results for sintered ceramics manufactured by Virial Ltd.:

Temperature	25 ˚C	100 ˚C	500 ˚C
Specific Heat (Cp) in J·kg^{-1}·K^{-1}	900-1050	1100-1310	1800-2000
Measurements, ± 10%	1026	1308	2001
Mean Linear Thermal Expansion Coefficient, in 10^{-6}K^{-1}	-	3.10-3.30	4.50-4.78

Measurements, ± 5%	-	3.12	4.78
Thermal Conductivity (K) in $W \cdot m^{-1} \cdot K^{-1}$	27.0-42.0	21.0-30.9	15.0-21.0
Measurements, ± 10%	32.71	30.81	19.85

3. VACUUM PROPERTIES OF VARIOUS BORON CARBIDE CERAMICS

Since the conceptual design stage of ITER diagnostic ports was not supposed to use ceramics, and in the port design it is planned to use a large number of ceramics, it requires careful confirmation of the possibility of using a large number of ceramics in a ITER vacuum vessel. I.e., the ceramic should not contain dangerous contaminants, and shall meet the requirements of the ITER Vacuum Handbook (IVH). In particular, IVH requires that all materials in the ITER vacuum chamber have the outgassing rate below $1 \cdot 10^{-7}$ $Pa \cdot m^3 \cdot s^{-1} \cdot m^{-2}$ for hydrogen at 100 °C and below $1 \cdot 10^{-9}$ $Pa \cdot m^3 \cdot s^{-1} \cdot m^{-2}$ for impurities [10].

A procedure for vacuum tests of ceramics was agreed upon with the ITER Organization, including pre-cleaning, heating samples in vacuum to 200-240 ˚C (which simulates the conditions of the Baking regime in ITER), cooling the samples, reheating them to 100 ˚C and measuring [14],[15]. The first experiments showed that some types of ceramics can meet the requirements of IVH [13]. Previously, the outgassing rate of B_4C ceramics in a vacuum at a temperature of 100 ˚C was not measured.

Tray-mounted ceramic blocks will fill all available space inside the ports (Fig.1). In Equatorial Port 11 alone, 40 thousand ceramic blocks with a total surface area of 407 m^2 will be installed [14],[15]. Due to the large area of ceramics in the port plug and, consequently, the large possible gas release, additional vacuum tests had to be carried out in order to reliably confirm the possibility of using sintered ceramics [2],[15],[19]. We used 638 samples of 55×55×5 mm in size, with a total area of 4.56 m^2 [2],[15]. The tests demonstrated that after 24 hours of heating in vacuum, the ceramics met the IVH [10] and manufacturing specifications for ceramics [16]. And after 30 months in a vacuum, the outgassing decreases to $2.1 \cdot 10^{-9}$ $Pa \cdot m^3 \cdot s^{-1} \cdot m^{-2}$ [2].

After discussion with the ITER vacuum group, it was decided to significantly limit the outgassing of ceramics in the approved specifications [16]. There are very strict requirements for ceramics: the outgassing rate is limited to $1 \cdot 10^{-8}$ $Pa \cdot m^3 \cdot s^{-1} \cdot m^{-2}$ for hydrogen at 100 °C and below $1 \cdot 10^{-10}$ $Pa \cdot m^3 \cdot s^{-1} \cdot m^{-2}$ for impurities. These limits are 10 times lower than the requirements for any other material in the ITER vacuum chamber. Earlier tests have shown that sintered ceramics manufactured by Virial Ltd. meet these requirements. The results of the vacuum tests performed with the supplied ceramic blocks are presented in section 5.

4. NEUTRON ACTIVATION OF CERAMICS, DEGRADATION AFTER FAST NEUTRON IRRADIATION

At the BINP for the development of boron neutron capture therapy of malignant tumors, an accelerator source of epithermal neutrons was proposed and created [20]. Experiments on irradiation of ceramics with fast [14] and slow neutrons [15] were carried out at this neutron source. Slow neutron experiments allowed to measure the manganese content in the samples (0.0001% in sintered Virial ceramics) [15]. The first fast neutron experiments (average energy 5.68 MeV, fluence $3.6 \cdot 10^{12}$ neutrons/cm^2) were performed to evaluate the activation of ceramics [14]. The activity of the ceramic samples was 50 μSv/h after the end of irradiation and 0.14 μSv/h (natural background level) after 3 days.

The spectrum of the induced activity of the samples was measured with a SEG-1KP-IFTP gamma-ray spectrometer based on a semiconductor detector made of highly pure germanium. In Fig. 3 shows the spectrum of sintered boron carbide ceramics measured half an hour after the stopping of fast neutron irradiation [15]. The impurities shown in Fig. 3 have a very low content in ceramics (for Mn, it is indicated above), but due to the sensitivity of the activation method, they are easily detected.

Then, a long-term fast neutron test with a fluence of 10^{14} n/cm^2 was performed to evaluate the degradation of the ceramic properties. After irradiation, it was found that not a single sample was cracked. Mechanical strength decreased by 15% (Table 2).

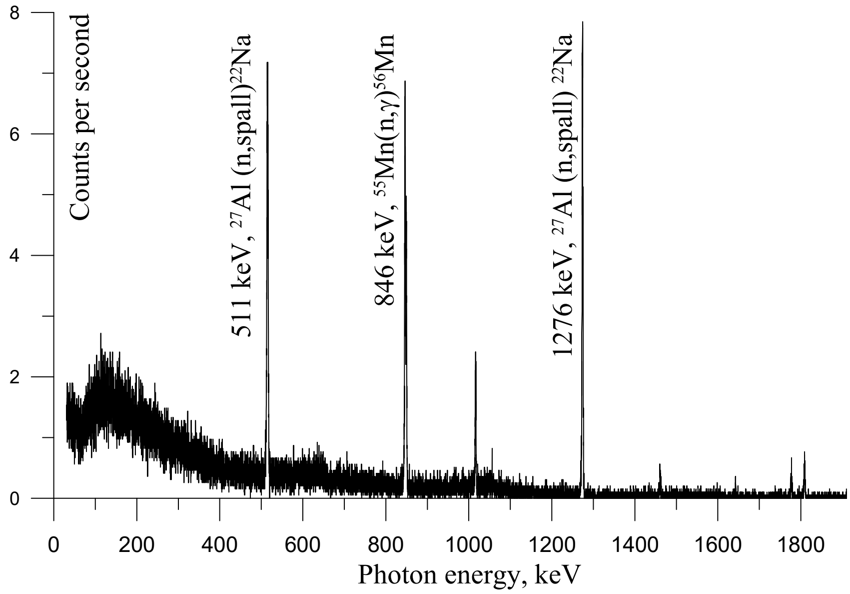

FIG. 3. Spectrum of gamma rays emitted by sintered boron carbide ceramics (Virial) after fast neutron irradiation. Each bright line shows the reaction in which it is born.

Neutron calculations have shown that the typical neutron flux inside the EPP #11 DMS will be $10^8÷10^{11}$ n/cm^2·s [2],[12], which, with a discharge duration of 400 seconds, will give a fluence of $4·10^{11}÷4·10^{13}$ neutrons/cm^2 in one shot. Thus, the fluence at 10^{14} n/cm^2 roughly corresponds to 2 to 200 DT ITER discharges, depending on the location of the ceramic block inside the EPP DMS.

TABLE 2. Degradation of mechanical properties of sintered boron carbide ceramics produced by Virial Ltd. after irradiation by fast neutrons with fluence 10^{14} n/cm^2:

Properties	After irradiation	Initial ceramics
Density, g/cm^3	2.14±0,02	2.26±0,01
Young's modulus E, GPa	365±14	420±7
Bending strength σ, MPa	190±13	220±11

The ITER organization calculated the rate of formation of helium and tritium in EPP#11 (Equatorial Port Plug) elements, which showed that in the first column of ceramics the amount of helium was 10^{18} atoms/ cm^3 [21], and in the last column, 10^{14} atoms/cm^3 [2],[12],[21]. With a boron atom density of 10^{23}/cm^3, the boron burnout in the ITER experiments (at the end of the SA-2 scenario [21]) can be considered negligible [2].

For optical diagnostics placed in diagnostic ports, the issue of dust absence is important. Dust can appear when ceramic blocks crack under the influence of a neutron flux. As shown by the experiments conducted on VITA at 10^{14} n/cm^2 fluence, not a single sample cracked. To evaluate the swelling of ceramics under the influence of neutrons, one can use the results of irradiation of boron carbide in a fast neutron reactor [22]. The swelling coefficient was estimated at 3.5% for 10^{28} captures/m^3 [22]. As mentioned above, neutron calculations have shown that up to 10^{18} helium atoms/cm^3 can form in ceramics at the end of the SA-2 scenario [21], which makes it possible to estimate the swelling coefficient at 0.00035%, i.e. very small.

5. SERIAL PRODUCTION OF B_4C CERAMICS ACCORDING TO THE REQUIREMENTS OF ITER ORGANIZATION

The specification for the manufacture of ceramics requires the manufacturer to prepare a series of documents (Quality Plan, Manufacturing Program, Manufacturing and Inspection Plan, and others) and coordinate them with the ITER Organization before the start of production. Virial Ltd. has started serial deliveries of sintered ceramics for installation in ITER ports. The manufacturer prepared all necessary documents, which were approved by the IO, and successfully passed the audits.

A cleaning procedure was agreed upon by the manufacturer of ceramic blocks before packaging in accordance with ITER requirements: washing of blocks in ethyl alcohol; washing in an ultrasonic bath in distilled water, annealing in a vacuum furnace at 1000 ºC for at least 4 hours.

After annealing and cooling of the furnace, the ceramics are packed under vacuum in individual MET/PE bags (Fig.4). After this procedure, the ceramics, after being removed from the packages without additional cleaning, can be immediately installed in the tray for installation in ITER ports.

FIG.4. After the cleaning procedure, each ceramic block is placed in a sealed MET-PE bag and vacuumed by the manufacturer.

Vacuum tests of the first delivery batch were carried out by the BINP. The measured spectrum of gases is shown in Fig. 5. Outgassing rate was $8.25\cdot10^{-9}$ $Pa\cdot m^3\cdot s^{-1}\cdot m^{-2}$, ceramics meet all ITER requirements. The IO approved the End of manufacturing report of the first delivery batch [23]. Then the ITER Organization approved the Reports on the production of all batches for the equatorial port #11 (more than 40 thousand blocks) [24].

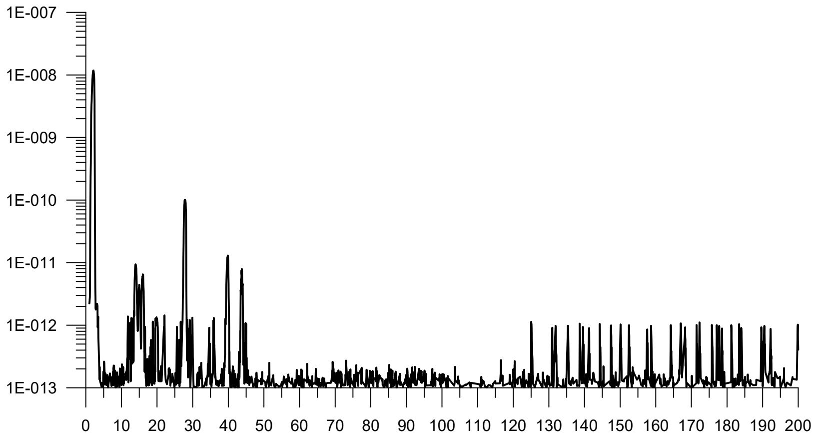

FIG.5. Mass spectrum of gasses measured with a quadrupole mass spectrometer for first delivery batch Virial-sintered boron carbide ceramics at 100 °C. On the vertical axis: the partial pressure in Torr; on the horizontal axis: the atomic mass. Masses (a.u.m.) and components of the six highest peaks: 2 H_2, 14 N, 16 O, 28 CO, 40 Ar, 44 CO_2.

6. CONCLUSION

A detailed study of the chemical composition, thermal, vacuum and mechanical properties of various carbide ceramics has been carried out. Long-term research has shown the possibility of using boron carbide ceramics in the ITER vacuum chamber for neutron protection of diagnostic ports. The ITER organization has agreed to place a large number of ceramics in a vacuum chamber. Serial production of sintered ceramic blocks has been launched for diagnostic equatorial port #11, upper ports #02, 07 and 08. More than 50 thousand ceramic blocks have already been produced.

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