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Plasma sprayed coatings for RF wave absorption

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Abstract

High requirements for fusion reactor materials and for experimental fusion devices have led within the fusion community to the development and testing of various coatings of the surfaces of in-vessel components and biological shields for microwave heating systems. Based on contacts with ITER, W7X and the Spanish Stellarator TJ-II, IPP Prague has initiated a development, production and test program on various low-*Z* materials. This paper reports on the production, development and properties of B_4C, Si and Al_2O_3 coatings sprayed by water stabilized plasma. Main focus is on their radio frequency wave reflection properties. Further characterization includes the coating structure by microscopy, phase composition by X-ray diffraction and oxygen content measurement by atomic absorption spectroscopy. The results are discussed with respect to processing conditions as well as potential application.

1. Introduction

Complex requirements on materials for fusion reactors [1] have led to development of composite materials, e.g., thermally sprayed coatings. Typical applications are plasma facing components inside the reactor vessel. These have to withstand high heat loads (both continuous and transient) [2], irradiation and particle bombardment and should be compatible with the plasma. Other applications call for specific dielectric properties, e.g., different types of probes and their holders, limiters, antennas for the excitation of radio frequency (RF) waves in plasma and transmission lines of RF energy. RF waves of different frequency and power level are used in such devices for a number of purposes: diagnostics, plasma heating, non-inductive excitation of toroidal current (RF current drive), etc. It is clear that the high frequency properties of the materials used have to be known.

At the Institute of Plasma Physics, Prague, CZ, plasma sprayed coatings are being developed for several fusion related applications. One of them is a microwave absorbing layer for the ECRH waveguide of the TJ-II device in Madrid. The coatings are to serve as an additional shielding for biological protection. Thus, absorption of microwaves, as well as other electrical properties, were the focus of this study. Coatings from three different materials were sprayed by water stabilized plasma (WSP), some of them underwent various post-treatment. Their properties were characterized and compared in view of their potential application.

2. Experimental

2.1. Preparation of the coatings

2.1.1. Spraying

Three types of coatings were sprayed and tested, boron carbide, silicon and alumina, all on 2 mm stainless

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steel substrates. Boron carbide was suggested by the Ciemat scientists, and is also a candidate material for plasma facing components in certain fusion devices. Silicon, as an alternative coating material for plasma facing components, was selected because of its easier sprayability and favorable high-temperature properties. Alumina was selected as a reference material, because of long time experience with its spraying in our laboratory and positive experience with its use in Tokamak Castor. The coatings were sprayed by the WSP torch [3] at different conditions. An overview of the samples is given in the left half of Table 1.

2.1.2. Post-treatment

Two types of post-treatment were applied. B_4C coatings were soaked in ethanol and water, to dissolve the oxides. This was done in connection with the main scope if the project, since oxygen content is important for plasma facing components. Both Al_2O_3 and B_4C coatings were covered with a thin layer of Aquadag colloidal graphite, to further reduce microwave reflection, based on previous experience with similar coatings in Tokamak Castor. The thickness of the graphite layer was a few μm, comparable to the surface roughness of the plasma sprayed coating.

2.2. Reflectometric measurement of RF characteristics of the coatings

Reflection of microwaves incident on the material interface is the most important characteristics from the point of view of the material shielding properties. How much power is reflected depends on material properties (described by the complex permitivity involving the dielectric constant as its real part as well as RF losses given by $\mathrm{tg}\,\delta$ as its imaginary part) and also on the geometry (first of all on the thickness of the layer resulting in different degrees of multiple reflections effect, especially in the case of coatings with metallic substrate, and generally also on the angle of the wave incidence). The value of the reflected power is characterized by a power reflection coefficient $R = (E_{ref}/E_{inc})^2$, where E_{ref} and E_{inc} are the amplitudes of the reflected and incident wave, respectively.

Representative measurements of the wave reflection coefficient have been done in a closed waveguide system (microwave bridge) shown in Fig. 1, where both the incident and reflected waves can be exactly measured. This bridge is formed by a hybrid T-junction MT in its center with four arms: 1 (H arm) – measuring one, 2 – feeding one with microwave generator (35 GHz, 200 mW),

Table 1
Parameters of the samples (No. – sample number, t_c – coating thickness, d – feeding distance, l – spraying distance, T – preheat temperature) and results of the reflectometric measurements (R_p – power reflection coefficient, ε – dielectric constant and $\mathrm{tg}\,\delta$ – loss tangent of the samples before and after the two types of post-treatment)

No.	Sprayed material	t_c (mm)	d (mm)	l (mm)	T (°C)	As-sprayed			After oxide dissolution	Coated with graphite
						R	ε	$\mathrm{tg}\,\delta$ ($\times 10^3$)	R	R
1	B_4C	0.45	26	340	110	0.64	1.70	2.70		0.58
2	B_4C	0.41	28	340	300	0.50	1.70	2.70		
3	B_4C	0.41	28	340	300	0.82			0.67	
4	B_4C	0.20	24	340	110	0.51	1.66	1.97		
5	B_4C	0.20	24	340	110	0.54			0.81	
6	B_4C	0.15	28	340	200	0.18	1.90	2.87		
7	B_4C	0.15	28	340	200	0.15			0.66	
8	Si	0.24	34	340		0.68	1.65	3.05		
9	Si	0.24	34	340		0.71	1.65	3.05		0.50
10	Si	0.23	34	380		0.66	1.65	3.40		
11	Si	0.21	34	340		0.67	1.65	3.05		
12	Al_2O_3	0.60	30	260		0.88				0.14
13	Al_2O_3	0.55	30	290		0.83				0.28
14	Al_2O_3	0.50	30	320		0.82				0.28
15	Gray Al_2O_3	1.20				0.72				0.028
16	Gray Al_2O_3	0.70	25	320		0.58				0.064
17	Gray Al_2O_3	0.60	35	230		0.83				0.096
18	Stainless					0.95				0.50

The spraying parameters (d, l, T) are expressed only when varied on purpose; otherwise, baseline parameters for a given material were used. Substrate (no. 18) properties are included for comparison.

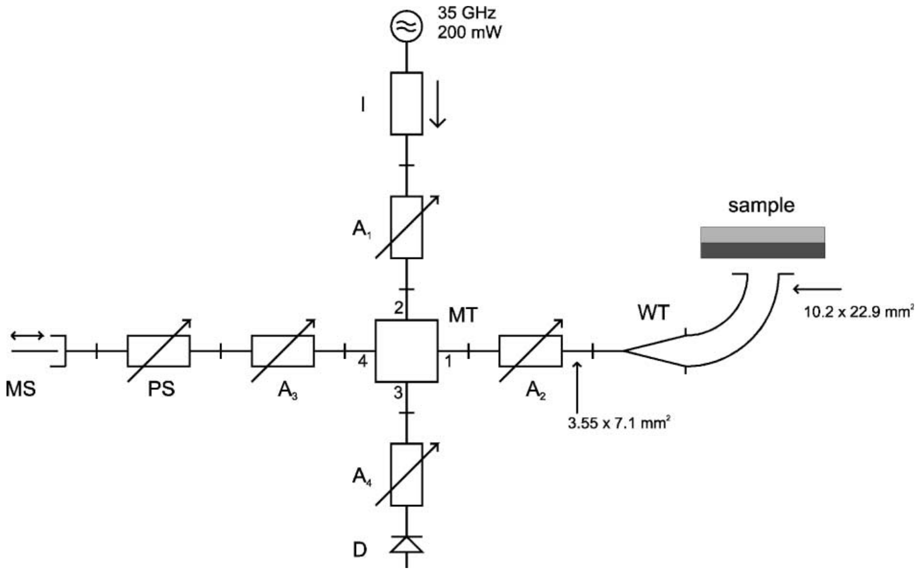

Fig. 1. Schematic diagram of a microwave interferometer on the reflected wave for the measurements of impedance and complex permitivity of dielectric material. I – ferrite isolator, A – variable attenuator, PS – phase shifter, MS – movable short, MT – magic tee (hybrid tee junction), WT – waveguide transition, D – detecting diode.

3 – detection one with diode D, 4 (E arm) – reference one, terminated by a waveguide movable short MS. Letter A in the scheme denotes attenuators, PS a phase shifter and I a waveguide isolator. To investigate a larger surface of the coatings, the waveguide dimensions of the measuring arm have been increased from standard 8 mm waveguide (7.1×3.55 mm^2) to 3 cm one (22.9×10.1 mm^2) using a tapered waveguide transition WT. The samples with the coatings have been fixed to the end of this measuring arm 1. Note that losses of the probing wave by lateral radiation through the sample-waveguide crevice have been found to be negligible.

For the determination of the power reflection coefficient R only, the measurement of the voltage-standing wave ratio S_V of the standing wave pattern, formed by interference of the reference wave (coming from the arm 4) and the reflected wave (coming from the arm 1) in the waveguide of the bridge detection arm 3, is sufficient. Specifically, there exists a simple relation between both quantities:

$$R = [(S_V - 1)/(S_V + 1)]^2.$$

Note that $S_V = (U_{max}/U_{min})^{1/2}$, where U_{max} and U_{min} are maximum and minimum signals detected by the diode D (the diode has a quadratic characteristics, i.e., the signal U is proportional to E^2), which can be easily found if the movable short MS is shifted a sufficient distance. The values of the power reflection coefficient R obtained in such way are given in the right half of Table 1.

In addition, using a precise attenuator A_3 and movable short MS, microwave bridge shown in Fig. 1 enables us to measure not only the amplitude of the reflected wave, but the phase of this wave as well (first of all phase change φ if the sample is inserted). By careful balancing of the effect of the sample in comparison with a perfect conducting metallic plate, the changes of the phase φ (radian) (measured by the movable waveguide short MS) and the attenuation of the reflected wave A [dB] (measured by the precise attenuator A_3) can be determined. Under assumption of low loss material and neglecting the effect of multiple reflections due to the small thickness t of the samples compared to the probing wavelength used, it may be shown that the dielectric constant ε and loss tangent $\mathrm{tg}\,\delta$ of the sprayed material are related to φ and A [4]

$$\varepsilon = (\varphi\lambda_0/2\pi t)^2 \quad \text{and} \quad \mathrm{tg}\,\delta = -(A/4.34\varphi).$$

Here t is the thickness of the dielectric layer, λ_0 – vacuum wavelength of the probing wave (8.6 mm in our case). Some values of ε and $\mathrm{tg}\,\delta$ obtained are given in Table 1 also.

2.3. Further characterization

The morphology of the coatings was observed using a Cambridge CAMSCAN-4DV scanning electron microscope.

The phase composition of the B_4C coatings was assessed by XRD. The measurements were performed on a Siemens D-500 diffractometer with Cu-Kα radiation, in the angular range 10°–90° of 2θ. The intensities of the most intense peaks of B, B_4C and B_2O_3 were calculated.

The B_4C samples were soaked in a fixed amount of ethanol and water to dissolve the oxide (B_2O_3 exists in two modifications, crystalline form is soluble in water, while the amorphous form is soluble in ethanol [5]). In the solution, the boron concentration was determined by atomic absorption spectrometry (AAS) on a Varian SPECTR AA 880 instrument, using a flame atomization technique with acetylene-N_2O at the wavelength of 249.8 nm. From this, the percentage of B_2O_3 in the coating was calculated.

3. Results and discussion

3.1. Reflection of microwaves

Table 1 summarizes the results of the measurement of power reflection coefficient (R), standing wave ration (S_V), dielectric constant (ε) and loss tangent (tg δ) of the coated samples.

The B_4C coatings achieved the wave reflection as low as 15%. A significant variation of R with spraying conditions is observed. Varying the spraying conditions generally results in changes of pore structure and composition. The removal of oxides by dissolution in ethanol and water increases the reflection significantly. Si coatings showed power reflection around 70%, without much variation between different samples.

As for the Al_2O_3 coatings, the average reflection is higher than in the case of B_4C. However, covering these coatings with colloidal graphite caused a very significant decrease of reflection (i.e., a better absorption effect is achieved), in some cases to several percents only. This combination of coatings with graphite layer yields the best absorbing results (it should be noted that these measurements were performed under the most stringent conditions – perpendicular incidence; under off-normal angles, the absorption would be even higher). The major factor in this substantial improvement of the absorption properties are probably the high porosity and the surface roughness of Al_2O_3 coatings (see Fig. 2). As the colloidal graphite (a conductive medium) fills the irregular pores of the Al_2O_3 coating (a dielectric), it creates an effectively rough surface, compared to the uncoated one. Note that addition of graphite layer to the B_4C and Si samples resulted in decreasing reflection as well, but not as significantly as in the Al_2O_3 case.

3.2. Other properties

The dielectric constants of B_4C and Si coatings are significantly smaller than those of bulk counterparts – 5 [6] and 11.4 [7], respectively. This is a consequence of the coatings' porous structure. AAS results show that the B_4C coatings contain 0.5–4 wt% B_2O_3, mostly in amorphous form. The oxide amount decreases with substrate preheating. A possible explanation is that B_2O_3 in the coating evaporates at high temperatures on the coating surface. However, no clear correlation between oxide content and reflectivity was observed. Presence of crystalline B_2O_3 and elemental boron was detected by XRD [8].

4. Conclusions

Dielectric properties, including the power reflection coefficient, were measured in the microwave range at 35 GHz on plasma sprayed B_4C, Si and Al_2O_3 coatings. A variation with the spraying conditions and post-treatment was observed. The R value of the B_4C coatings ranged from 0.15 to 0.82. The lowest reflection was

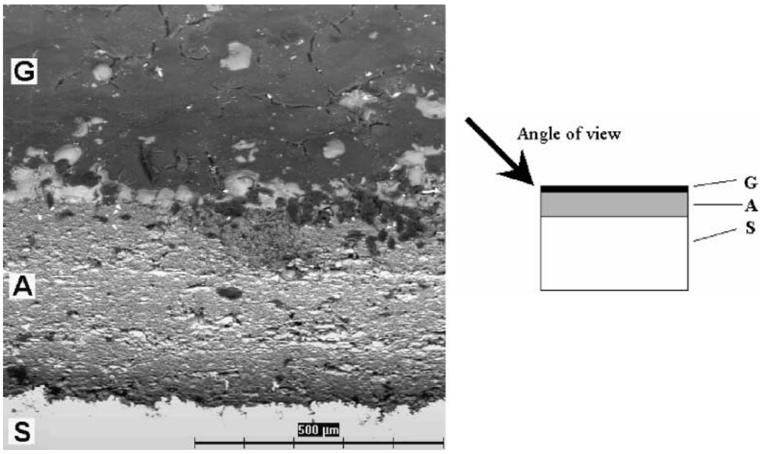

Fig. 2. Morphology of the surface of the colloidal graphite layer (G) and a cross-section of the Al_2O_3 coating (A) on the substrate (S), viewed at 45° in backscattered electrons. A few cracks are present in the graphite layer; numerous pores can be seen in the Al_2O_3 layer.

obtained on Al_2O_3 coatings covered with colloidal graphite as low as 0.03. This can be interesting from the point of view of use in RF equipment during channeling of large power in thermonuclear devices.

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