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Coating materials for fusion application in China

G.-N. Luo [a,*], Q. Li [a], M. Liu [b], X.B. Zheng [c], J.L. Chen [a], Q.G. Guo [d], X. Liu [e]

[a] Institute of Plasma Physics, Chinese Academy of Sciences, Hefei 230031, China
[b] Guangzhou Research Institute of Nonferrous Metals, Guangzhou 510651, China
[c] Shanghai Institute of Ceramics, Chinese Academy of Sciences, Shanghai 200051, China
[d] Shan'xi Institute of Coal Chemistry, Chinese Academy of Sciences, Taiyuan 030001, China
[e] Southwest Institute of Physics, Chengdu 610041, China

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ABSTRACT

Thick SiC coatings of ∼100 μm on graphite tiles, prepared by chemical vapor infiltration of Si into the tiles and the following reactions between Si and C, are used as plasma facing material (PFM) on HT-7 superconducting tokamak and Experimental Advanced Superconducting Tokamak (EAST). With increase in the heating and driving power in EAST, the present plasma facing component (PFC) of the SiC/C tiles bolted to heat sink will be replaced by W coatings on actively cooled Cu heat sink, prepared by vacuum plasma spraying (VPS) adopting different interlayer. The VPS-W/Cu PFC with built-in cooling channels were prepared and mounted into the HT-7 acting as a movable limiter. Behavior of heat load onto the limiter and the material was studied. The Cu coatings on the Inconel 625 tubes were successfully prepared by high velocity air–fuel (HVAF) thermal spraying, being used as the liquid nitrogen (LN2) shields of the in-vessel cryopump for divertor pumping in EAST.

1. Introduction

In order to reduce chemical erosion and hydrogen isotope retention of the carbon based materials (CBM), thick SiC coatings of ∼100 μm on doped graphite named GBST1308 (1% B4C, 2.5% Si, 7.5% Ti) have been developed by Shan'xi Institute of Coal Chemistry, Chinese Academy of Sciences (ASICC) and Institute of Plasma Physics, Chinese Academy of Sciences (ASIPP) by chemical vapor infiltration of Si into the graphite tiles and the following reactions between Si and C [1–6]. And the SiC/C tiles are now being used as PFM on the HT-7 limiters and the whole EAST plasma-facing surface. With increase in the heating and driving power in EAST, the heat load onto divertor will increase significantly, and the W coating on actively cooled heat sink will be a better choice for PFC [7]. W coatings on carbon substrate and Cu heat sink have been developed at Guangzhou Research Institute of Nonferrous Metals (GZRINM) and Shanghai Institute of Ceramics, Chinese Academy of Sciences (ASSIC), in collaborations with ASIPP and Southwest Institute of Physics (SWIP) by vacuum plasma spraying (VPS) with different kinds of interlayers [8–11]. For efficient divertor pumping, an in-vessel cryopump has been assembled beside the outer target of lower divertor in EAST, in which two liquid nitrogen (LN2) cryostats, made of Inconel 625 tubes, were covered by complicated Cu stripe coatings to improve the poor thermal conductance of the Inconel tubes. The Cu coatings were successfully prepared on the 3D machined tubes by high velocity air–fuel (HVAF) thermal spraying at GZRINM. Other coatings under preliminary R&D include B_4C and Al_2O_3, for the potential use as protection layer of low hybrid current driving (LHCD) antenna and barrier layer of tritium permeation, respectively.

2. The SiC coatings

The research for the doped graphite with thick gradient distributed low-Z carbide coatings (SiC, B_4C) is a promising way to reduce erosion. In order to develop new grade of CBM and SiC coatings, intensive collaboration activities were conducted in recent years between ASIPP and ASICC [1–3]. One kind of doped graphite named GBST1308 (1% B_4C, 2.5% Si, 7.5% Ti) has been developed, showing characteristics of high density, low open porosity, high strength and high thermal conductivity. The SiC coatings on the doped graphite GBST1308 have also been developed by a technique of chemical vapor reaction (CVR) combined with chemical vapor infiltration (CVI) which provides gradient SiC coating by the infiltration of reaction gas through the open pores. This process yields sufficient resistance against exfoliation. Fig. 1 shows the SEM images of the SiC coating. It was observed that the thickness of the SiC coating is ∼100 μm and the size of the crystal is ∼20 μm. Dependence of thermal diffusivity and thermal conductivity on temperature was measured. Due to the formation of SiC coating on the surface of doped graphite, thermal diffusivity showed a decrease, and the gap between with and without coatings became more and more less with the rise of temperature. It was observed that

* Corresponding author. Tel./fax: +86 551 5592525.
E-mail address: gnluo@ipp.ac.cn (G.-N. Luo).

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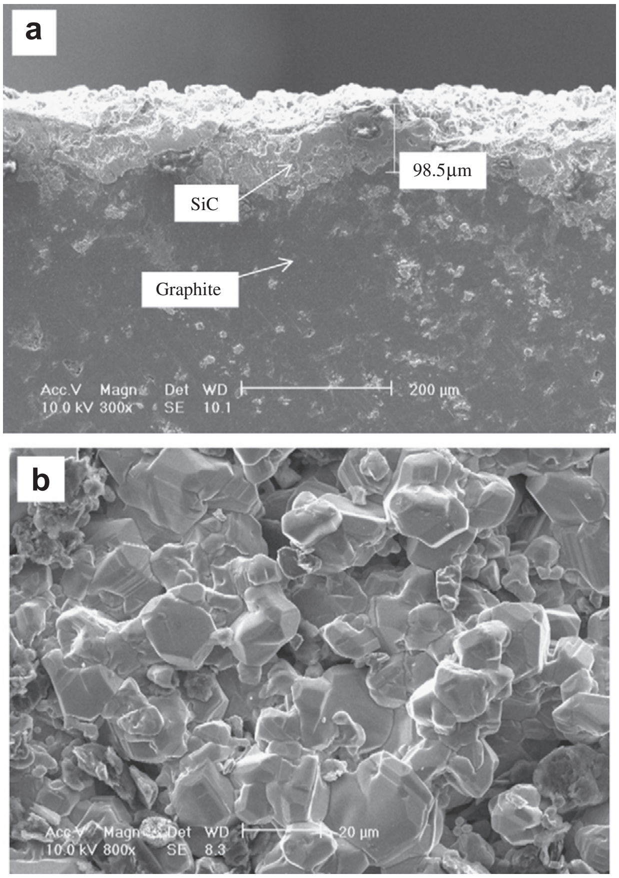

Fig. 1. SEM images for (a) cross-section and (b) surface of SiC coatings.

thermal conductivity of SiC coated samples show a weak dependence on temperature, which makes it one promising candidate as high heat flux component [4]. Thermal shock resistance of SiC coated doped graphite was carried out by water-quenching test with the critical temperature difference (DT) of 1000 °C. The SiC coatings showed very strong adhesion with carbon substrate. Only some microcracks can be observed and no spallation occurred for the SiC coating [4]. Steady state high heat flux and pulsed power irradiation experiments were performed in a well diagnosed e-beam device at ASIPP. It has been found from the laboratory experiments that thick SiC gradient coatings exhibit superior surface characteristics and satisfactory thermal shock resistance. In the last campaign of year 2000, a smaller and discrete molybdenum limiter was switched to a larger and fully circular SiC coated graphite limiter with actively water-cooling, as shown in Fig. 2a. From the first campaign of year 2004, a new belt limiter plus up and down toroidal limiters with a total area of 2.5 m^2, have been installed in the HT-7 device with a bolting connection between the coated tiles and the actively cooled heat sink, as shown in Fig. 2b [2,3]. The problems of strong hard X-ray emission from the limiter and metal impurities in the plasma with high-power LHCD have been alleviated after installing the new graphite limiter. Higher plasma parameters have been achieved, while a similar discharge with the previous Mo limiter was very difficult to obtain. All these results show that the new graphite with SiC coating as the limiter material is satisfactory for long pulse plasma discharges in the HT-7 tokamak [5]. Contrary to carbon based materials, SiC coatings behaved much lower chemical and physical sputtering, capability of oxygen gettering, and lower hydrogen recycling. This is important for impurity control during long pulse discharges with evidence of lower Zeff. After installation a new carbon limiter, the value of Zeff

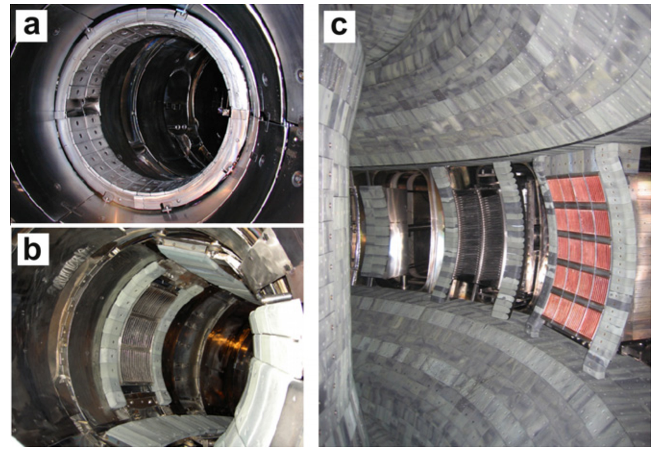

Fig. 2. Pictures of (a) a fully circular poloidal limiter and (b) a belt limiter on the high field side plus up and down toroidal limiter in HT-7 device, and (c) the in-vessel wall armed with SiC coated graphite in EAST device.

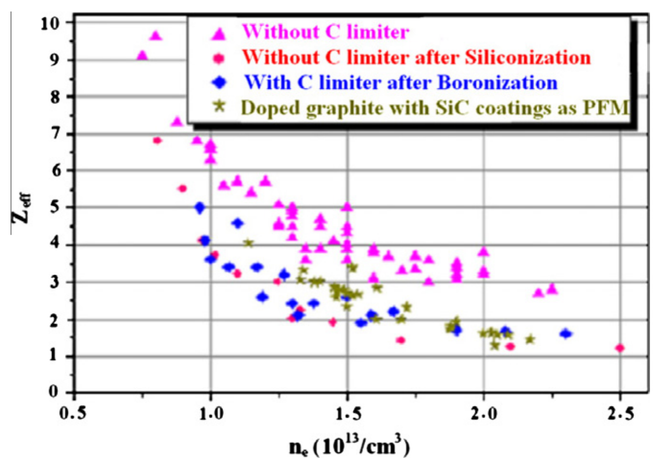

Fig. 3. The effects of HT-7 limiter materials on Zeff [2,3].

is similar to the case of boronization, only slightly higher than siliconization, as shown in Fig. 3 [2,3]. After exposure to HT-7 plasma of two operation campaigns, it was shown that the erosion of the coating on GBST1308 was two times less than for the uncoated, widely used GBST1308 and five times less than pure graphite. XPS analysis shows that the surface of the carbon tile absorbs a large amount of oxygen and forms a stable SiO_2 [6]. The GBST1308 tiles coated with SiC, bolted to the active cooling heat sink, is now used as the only PFM for the EAST device since the campaign of year 2008, as shown in Fig. 2c. Significant improvements of the plasma performance were observed and stable DN discharges over 60 s have been achieved in 2009 Spring campaign.

3. VPS-W coatings

Tungsten (W) offers a better choice as the PFM for the future fusion reactor, mainly attributed to its highest melting point of all metals, high threshold energy for physical sputtering, no chemical reaction with deuterium, good thermal conductivity, and high strength at elevated temperatures [12]. Tungsten coatings on C and Cu substrates attracts much attention, as the former has the advantage of full compatibility with the current support structure and the latter can provide simultaneously the joining of the W armour with the heat sink to make up plasma facing components

[10]. In previous works, VPS-W coatings of several microns on carbon and copper substrates have been developed. Recently, the research on thick VPS-W coating of ~1 mm on CuCrZr heat sink was carried out for EAST tungsten divertor project. With increase in the heating and driving power in EAST, the heat load onto divertor will increase significantly and the present PFC of SiC/C tiles bolted to Cu heat sink will not be able to withstand it. In the future 3–5 years, the graphite tile PFC will be gradually replaced by VPS-W coated CuCrZr PFC with built-in cooling channels [7]. Before installing the VPS-W/Cu PFCs to the EAST divertor, it is necessary to investigate the quality of VPS-W/Cu mock-ups by high heat flux test and tokamaks plasma irradiation.

3.1. VPS-W coatings on carbon substrate

VPS-W coatings on CFC tiles have been used in current fusion devices, such as ASDEX-Upgrade, with a multi-layered W and Re interface pre-deposited by physical vapor deposition (PVD) [13,14]. Due to its high thermal conductivity and thermal expansion close to the graphite substrate, silicon at much lower cost is considered as the substitute for rhenium, and multilayered Si and W structure pre-deposited by PVD as interface of W/C coating is a new attempt. Even though Si reacts with C (substrate materials) and SiC is formed at high temperature, the coefficient of thermal expansion of SiC is very close to that of tungsten, and a stable SiC phase might act as a diffusion barrier of carbon elements. As shown in Fig. 4, VPS-W coated graphite with a multilayered interface consisting of three layers of PVD-W and three layers of PVD-Si were developed by SWIP recently [10], and each layer is 1–2 μm thick and total thickness of the interface is about 12 μm. VPS-W coating is 0.3 mm thick with a porosity of about 6%. Bonding strength between W coating and substrate is larger than 40 MPa. The thermal conductivity of VPS-W coating was measured by laser flash method, and the results showed that the thermal conductivity of W coating is about half of the pure tungsten made by powder metallurgy. The thermal fatigue properties of VPS-W coating were evaluated by cyclic heat load tests. The results indicate that the VPS-W coated graphite can endure 1000 cycles without visible failure at an absorbed power density of 4.8 MW/m^2 and 5 s pulse duration.

3.2. VPS-W coatings on copper substrate

VPS-W coatings on Cu substrate have also been developed in SWIP [10]. The VPS-W coating has thickness of 0.3 mm and porosity of about 6%. Bonding strength between W coating and copper is larger than 40 MPa. In order to reduce thermal stress, the W and Cu transition interface was prepared before spraying pure tungsten coating. W/Cu ratio was increased from 0 to 100% gradually, and the total thickness of this transition layer is about 150 μm. VPS-W coated Cu can endure 1000 cycles without visible failure at an absorbed power density of 4.8 MW/m^2 and 5 s pluse duration.

ASIPP has cooperated with Shanghai Institute of Ceramics to develop VPS-W coatings on Cu. In order to strengthen the adhesion of the coating with the substrate and reduce thermal stress, some interlays were used, such as W/Cu gradient layer, Ti layer and NiCrAl layer. The low porosity of 7.6% and the narrow pore size distribution of 0.08–1 μm were obtained in the VPS-W coatings. The oxygen content of the VPS-W coatings was measured to be about 0.35 wt% by means of EDS. The thermal conductivity of 59.3 W/m/K and bonding strength of 44.8 MPa were achieved. From the high heat flux tests one concludes that VPS-W coating with W/Cu gradient layer kept the lower temperature, which was about 1100 °C at 10 MW/m^2 incident heat flux [11,15].

The W coating on Cu substrate was also prepared using a VPS device at GZRINM [8]. Ar and H_2 were used as plasma source gas with the flow rates of 40–60 l/min and 5–7 l/min, respectively. Powder carrier gas was Ar with a flow rate of 3–5 l/min. The spray distance was 200–300 mm. In order to reduce thermal stresses, a W/Cu gradient interlayer of 0.2–0.3 mm was prepared before spraying pure tungsten coating. With at least five steps of changing the feeding ratio of Cu and W powders, the layer composition was gradually varied from pure Cu to pure W. Thick pure W coatings of >1 mm were then deposited on the interlayer. Fig. 5 shows typical SEM micrographs of the VPS-W coatings with the lamellar graded W/Cu transition layer. The coatings have a porosity <10%

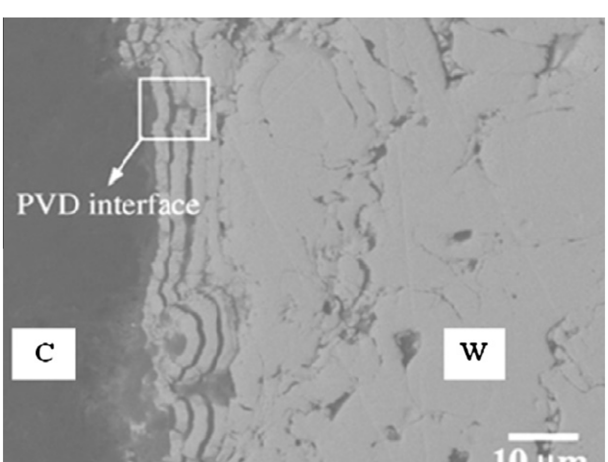

Fig. 4. Cross-section morphology of VPS-W/C with multi-layer W and Si PVD interface [7].

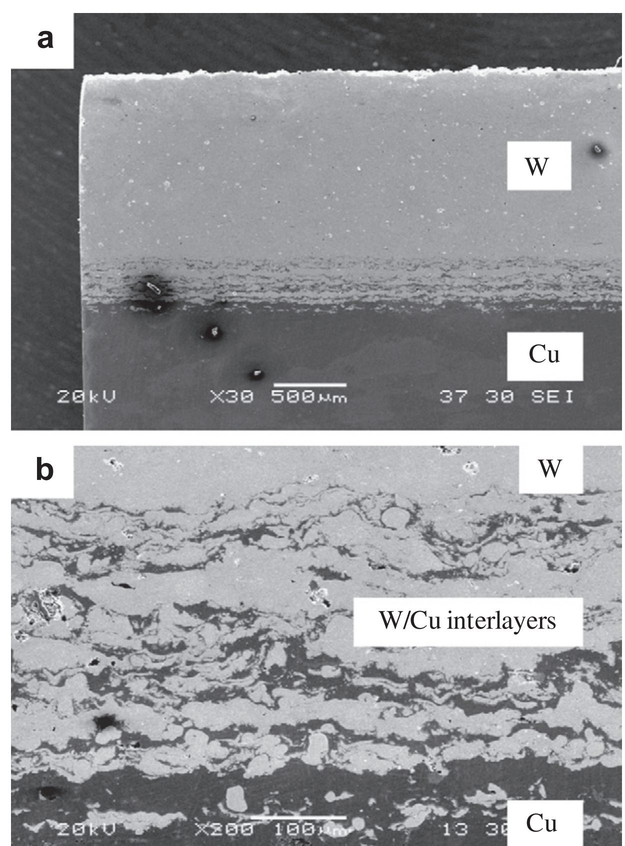

Fig. 5. Cross-section images of VPS-W/Cu sample showing gradient W/Cu interlayers [5].

and an oxygen content of 0.4 wt%. The thermal conductivity of the coating was measured by laser flash method and its value reached to 80 W/m/K. E-beam irradiation of 20 cycles was done in all and each cycle lasted 100 s with heat load of 9.6 MW/m^2. The pressure of cooling water of 0.4 MPa and the flow rate of 2m^3/h were maintained, leading to a flowing velocity of 3.5 m/s in the cooling channels. After the tests, no formation of cracks was observed within the gradient interlayer, and no obvious evidence showing propagation of the as-deposited cracks at the coating surface.

An actively cooled VPS-W/Cu movabe limiter (ML) prepared at GZRINM has been installed into HT-7 for testing the integrity of the W/Cu PFC and studying the plasma-tungsten interactions [7,8]. The VPS-W/Cu ML consists of a W coating of 1 mm on Cu heat sink (150 mm × 50 mm × 40 mm) with two built-in cooling channels of 10 mm in diameter, as shown in Fig. 6. The ML was exposed to the Ohmic plasma discharges with plasma current I_P~140 kA and durations ~0.7 s, and long pulse plasmas with durations ~60 s, driven by LHCD of ~130 kW. During the experiment, the ML was inserted into the plasmas at various radial locations with $r<a$, where a = 270 mm, the minor radius determined by the fixed graphite limiters. To estimate the heat flux to the ML, an IR camera was placed at the lower port, aiming directly at the ML, to monitor its surface temperature. For a typical Ohmic discharge with plasma current I_P~140 kA, line average density ~1.5 × 10^{19} /m^3, electron temperature ~500 eV, and pulse duration of ~0.7 s, the maximum temperature is about 65 °C at $r = a$. The temperature increases as the ML moves deep into the plasma, with the maximum temperature reaching 200 °C at r = 256 mm. The temperatures at the ML surface are much higher in the LHCD plasmas, as compared to those in the OH discharges. The temperature of the round edges in the ML reaches to 700 °C. After 20 Ohmic plasmas and 20 LHCD plasmas irradiation, there was no indication of debonding between coating and substrate and no obvious evidence showing propagation of the as-deposited cracks at the coating surface. A simple model was developed to understand heat transport to the ML surface based on the so-called cosine model and taking the "funnel effect" into account [16]. For typical Ohmic discharges with duration of 0.7 s, the simulated temperature distribution by ANSYS is consistent with the IR measurements, using the calculated heat fluxes of 0.8 MW/m^2 from the simple heat transport model. For the LHCD discharges, the maximum temperature and heat flux on the ML surface reached ~700 °C and 7 MW/m^2, respectively.

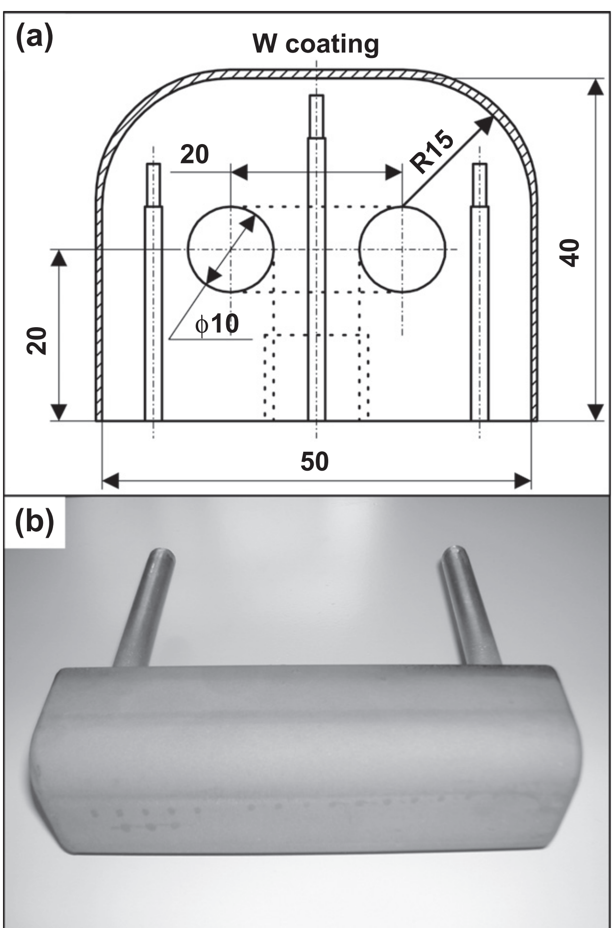

Fig. 6. The VPS-W/Cu PFC for the movable limiter, (a) cross-section drawing in units of millimeter; (b) picture showing W coated surfaces and SS tubes for cooling water [4].

4. HVAF-Cu coating

A round tubular in-vessel cryopump of 4 m plus in diameter has been fabricated and installed beside the outer target of lower divertor and has operated reliably for efficient divertor pumping in the EAST spring campaign this year, showing capability of pumping D_2 at a rate higher than 70 m^3/s at pressure level of 10^{-3} Pa. The configuration of the cryopump is similar to the cryopumps on DIII-D [17]. The pump consists of three concentric Inconel 625 tubes, comprising the liquid helium tube, an inner nitrogen shield and an outer nitrogen shield. And two non-concentric liquid nitrogen 625 tubes were insert-welded to the inner nitrogen shield and the outer nitrogen shield, respectively.

In order to increase the thermal conductance of the nitrogen shields, the two shield tubes were coated with Cu strips by high velocity air–fuel (HVAF) thermal spraying. The HVAF technique has been widely used mainly due to its high flame velocity and moderate temperature. The moderate flame temperature is especially suitable for deposition of coating materials with low melting temperature. An Intelli-Jet HVAF system made in USA was used for spraying the copper strip coatings on 625 tubes at GZRINM. The Cu powders of an average particle size of 38 μm were used for thermal spraying the Cu coating. Ethene was used as a fuel gas with a flow rate of 73 psi (first level) and 43 psi (second level), and the flow rate of the air was 85 psi. The spray distance was 350–400 mm, and the powder feed rate was 157 g/min. Fig. 7 shows the picture of the Cu strip coatings being sprayed by the HVAF technique. The thickness of the Cu coatings is ~0.4 mm with a NiCr interlayer of tens of μm, and the broadness of the copper strips is 25 mm. The thermal conductivity of the copper coatings gets to 150–200 W/m/K, and the hardness of the copper coating reaches to HB160–200. The bonding strength of the coating to the substrate was mea-

Fig. 7. Picture of HVAF thermal spraying of Cu coating on Inconel 625 tube used in EAST in-vessel cryopump.

sured to ~55 MPa. There were no indication of cracking and debonding of the copper coatings. The temperature of the substrate during spaying was <150 °C. No oxidation of copper coatings and Inconel 625 tubes was observed during thermal spaying process. Thermal shocking test was performed with 20 thermal cycles between 80 K (LN2) and 500 K (baking). The samples showed good adhesion and performance, and there were no failure of cracking and debonding of the copper coatings.

5. Conclusions

The SiC coated doped graphite GBST1308 tiles developed by a technique of CVR combined with CVI are used as the plasma facing materials on the HT-7 and EAST devices. And significant improvements of the plasma performance were observed. The W coatings on carbon substrate and Cu heat sink have been developed by VPS adopting different kinds of interlayers. The VPS-W/Cu PFC with built-in cooling channels was mounted into the HT-7 acting as a directly-cooled movable limiter. There was no indication of damage on the surfaces after more than 20 Ohmic plasmas and 20 long pulse LHCD pasmas irradiation. The two liquid nitrogen (LN2) cryostats of the in-vessel cryopump in EAST were covered by copper stripe coatings on the Inconel 625 tubes, using the high velocity air–fuel (HVAF) thermal spraying.

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