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**INTERNATIONAL NUCLEAR DATA COMMITTEE**

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**2<sup>nd</sup> (Final) IAEA Research Co-ordination Meeting on  
“Plasma-material Interaction Data for Mixed Plasma Facing  
Materials in Fusion Reactors”**

**October 16-17, 2000, IAEA Headquarters  
Vienna, Austria**

**SUMMARY REPORT**

**Prepared by: R.E.H. Clark**

**November 2001**

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**IAEA NUCLEAR DATA SECTION, WAGRAMERSTRASSE 5, A-1400 VIENNA**



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## **Abstract**

The proceedings and conclusions of the 2nd Research Co-ordination Meeting on “Plasma-Material Interaction Data for Mixed Plasma Facing Materials in Fusion Reactors”, held on October 16 and 17, 2000 at the IAEA Headquarters in Vienna, are briefly described. This report includes a summary of the presentations made by the meeting participants and a review of the accomplishments of the Co-ordinated Research Project (CRP). In addition, short summaries from the participants are included indicating the specific research completed in support of this CRP.

Reproduced by the IAEA in Austria  
November, 2001

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## 1. Introduction

The 2<sup>nd</sup> and final Research Co-ordination Meeting (RCM) of the participants of the IAEA Co-ordinated Research Project (CRP) on “Plasma-material interaction data for mixed plasma facing materials in fusion reactors” was held on October 16-17, 2000, at the IAEA Headquarters in Vienna, Austria. The topic of this CRP is of high importance to the research effort in magnetic fusion energy. The topic of mixed materials comes about from the requirements imposed by a number of factors.

The selection of the armor materials for the plasma-facing components is a compromise between multiple requirements derived from the unique features of a burning fusion plasma environment. The factors that affect the selection come primarily from the requirements of plasma performance (e.g., minimize impurity contamination and the resulting radiation losses in the confined plasma), engineering integrity, component lifetime (e.g., withstand thermal stresses, acceptable erosion), and safety (e.g., minimize tritium and radioactive dust inventories). Challenging trade-offs in material choice for plasma-facing components were identified in the internationally coordinated R&D program supporting the Engineering Design Activities of the ITER project. The increase in plasma duration (e.g., pulse length and cumulative run time), together with the increase of the plasma energy, represent the largest changes in operational conditions of a next-step long-pulse DT fusion reactor, compared to today's fusion facilities. This will give rise to important plasma-physics effects and plasma-material interactions that will critically influence performance, operation and safety. For example, divertor carbon erosion will increase from being barely measurable at a micron scale in today's tokamaks to a scale of several cm in the next-experimental fusion device, e.g., ITER. Tritium co-deposited with carbon will strongly affect the operation of machines with carbon plasma-facing components. Particle flows in the divertor plasma affect erosion and re-deposition, and a quantitative understanding of the connection is a subject of current research. The current designs of next-step devices (e.g., ITER-FEAT, FIRE) employ more than one plasma-facing material selected for their suitability to regions of the vessel with different power and particle flux characteristics. In the ITER-FEAT design, the low-Z material Be will cover the large areas of the vessel wall, the high-Z material W will protect the divertor and baffle regions. Carbon is retained for those parts of the divertor where the SOL strikes the vertical target and energy deposition from disruptions and ELMs will occur, mainly because of its good power handling, thermal shock resistance and the fact that it does not melt or change its shape even under extreme temperature excursions. However, maintaining carbon in the design has a strong impact on the control of the T-inventory and efficient in-situ techniques are required to recover the tritium retained in the co-deposited layers. The use of different materials requires the compatibility of the materials in co-deposited layers throughout the machine. These design requirements are the motivation for the current CRP on the plasma interactions with mixed materials and the associated RCMs.

The objectives of this RCM were:

- a) to review the results of the work done within the individual CRP projects in the period between the 1<sup>st</sup> and 2<sup>nd</sup> RCM;
- b) to summarise the overall accomplishments of the CRP over its entire duration;

- c) to agree on a method of submitting final reports of work accomplished and their publication;
- d) to agree on a plan for assessing and submitting cross section data generated from this CRP for inclusion in the IAEA database.

The meeting was attended by most of the principal scientific investigators of the individual CRP projects with the following adjustments. L. Errea attended in place of A. Riera. V. Ulanov is no longer participating in the CRP and has been replaced by E. Zhukovskaya. M. Guseva attended in place of Yu.V. Martynenko. R. Anderl attended in place of G. Longhurst. M. Akiba was unable to attend. Regretfully, K. Ashida passed away prior to the meeting. He was very much missed by all the participants. K. Watanabe attended to present final results from his collaborations with K. Ashida. The list of meeting participants is attached as an Appendix 1.

## 2. Brief Meeting Proceedings

The meeting began with a welcoming address by Mr. D. Muir, Head of the Nuclear Data Section. The Meeting Agenda was adopted without change (see Appendix 2). The meeting then continued with four sessions, the first three devoted to reports of the latest work from each individual project, and the fourth session devoted to formulating conclusions for the CRP and discussion of publication of final results and assessment and transmittal of cross section data for inclusion in the IAEA database.

The first session of the meeting was chaired by K. Watanabe and included results presented by R. Anderl, V. Alimov, and E. Zhukovskaya.

The first speaker of the session, **R. Anderl** from the Idaho National Engineering and Environmental Laboratory, presented his recent work on deuterium retention in tungsten and tungsten carbide. Dr. Anderl first summarized previous work on deuterium implantation and use of thermal desorption spectroscopy (TDS) for measurement of retained deuterium. He then presented results from his recent work on retention of deuterium in different forms of tungsten and the investigation of the influence of carbon on deuterium uptake in tungsten materials. He presented thorough descriptions of the various samples used and the ion implantation system employed. He then presented results from TDS spectra for a number of experiments covering a variety of conditions. Finally, he summarized his principle findings including the differences in deuterium retention from different fabrication processes and the affect of temperature on the retention process. His summary report is included as an attachment.

**V.Kh Alimov** of the Institute of Physical Chemistry, Russian Academy of Sciences, was the second speaker of the first session. Dr. Alimov presented a summary of his recent work on deuterium retention in tungsten-carbon mixed materials. He described the preparation of the mixed films using magnetron sputtering in deuterium and argon atmospheres. The films were analysed using secondary ion mass spectrometry (SIMS) combined with residual gas analysis (RGA), electron probe microanalysis (EPMA), and reflected high electron energy diffraction (RHEED). His principle finding was that the concentration of deuterium in the W-C mixed



film is much lower than expected from the assumption that the mixed film consists of individual tungsten and carbon atoms. Results were presented for a range of target/substrate temperatures.

**E. Zhukovskaya** of the Uzbekistan Academy of Sciences Institute of Nuclear Physics presented the third and final talk of the first session. Dr. Zhukovskaya presented results on the investigation of using neutron induced elastic recoil to analyse hydrogen depth profiles in wall materials. She gave a description of the neutron generation and detection processes and the overall experimental arrangement. A new computer simulation code, named Distribution Recoil Induced Neutron (DRIN) was written for analysis of energy and angular recoil distributions, based on Monte Carlo methods. This code development makes possible the calculation of depth profiles for hydrogen isotopes, as well as boron.

The second session was held in the afternoon of the first day and was chaired by V. Alimov. C. Garcia-Rosales, E. Vietzke, K. Watanabe, and A. Haasz made presentations during this session.

The first presentation of the second session was by **C. Garcia-Rosales** of the Centro de Estudios e Investigaciones Tecnicas de Guipuzcoa. Dr. Garcia-Rosales gave a presentation on the development of improved doped carbon materials for fusion applications. The purpose of this work was the investigation of methods of using doping of carbon to reduce erosion processes and not degrade, but to enhance, if possible, the thermal properties of the graphite. She described the details of the manufacturing procedures used and the selection of doping materials. She then presented summaries of mechanical and erosion properties of the materials considered.

**E. Vietzke** of the Institut für Plasmaphysik, Forschungszentrum Jülich gave the next presentation on siliconisation and silicon doped carbon in Tokamaks. Dr Vietzke first summarized the characteristics of silicon that make it a useful tool for study of plasma behavior with wall materials. He then gave a review of silicon sublimation and chemical erosion for silicon and SiC. This was followed by a report on a number of experiments using the TEXTOR-94 facility. These included investigations of impurity release and recycling behavior of siliconised walls and behavior of a silicon doped test limiter under high heat load. The main conclusion from these investigations is that silicon doped CFC appears to be a suitable material for the ITER divertor plate.

**K. Watanabe** of the Hydrogen Isotope Research Center, Toyama University, Japan gave the third talk of the second session. Dr. Watanabe presented results from his collaboration with Dr. K. Ashida who had tragically passed away before the meeting took place. Dr. Watanabe presented results for investigations in reactions of hydrogen containing carbon films with metal substrates. He first described the method of preparing samples and the subsequent analysis of those samples. He next described experiments of solid state reactions with these samples using thermal desorption spectroscopy, X-ray photoelectron spectroscopy, and X-ray diffraction. He then gave a detailed kinetic analysis of reactions with substrate metals using the nuclei growth method.

**A.A. Haasz** of the Institute for Aerospace Studies, University of Toronto, gave the fourth and final talk of the second session. Dr. Haasz presented results of a number of recent

experiments for a variety of processes on mixed materials. He presented chemical erosion, radiation enhanced sublimation, deuterium retention, and thermal diffusivity/conductivity measurements for graphite doped with a number of materials. He then gave results of studies of deuterium retention as a function of carbon thickness for carbon implanted in tungsten. Next he summarized studies of chemical erosion and thermo-oxidation for boron on graphite. Finally he presented results of new studies of mass loss due to oxidation for tungsten on graphite.

The third session began on the morning of the second day of the meeting. Dr. C. Garcia-Rosales served as chair for this session. M. Guseva, J. Roth, and R. Doener gave presentations.

The first presentation of this session, by M. Guseva the Scientific Research Institute Kurchatov Institut, Russian Federation, was on the investigation of tungsten-carbon-hydrogen co-deposited layers on tungsten and graphite. Dr. Guseva first described the experimental arrangement. Then she described the formation of layers of C+H on tungsten, giving an analysis of the microstructure of the layers, the thickness and density of the layers and the hydrogen and carbon retention properties. She gave similar descriptions for C+H on graphite and for C+W+H layers on tungsten and graphite while irradiated with  $C_2H_2$  plasma.

**J. Roth** of the Max Planck Institut fur Plasmaphysik, Garching gave the next presentation. Dr. Roth presented recent results on studies of mixed material formation and erosion. He first gave the motivation for these studies in terms of the types of materials and their methods of bonding. He then summarized details of formation processes, including reactivity, carbonisation, carbon surface concentration, and diffusion. He then presented results for erosion of materials under bombardment by different ions.

**R. Doerner** of the University of California, San Diego gave the third and final presentation of this session. Dr Doerner described his investigations into mixed material layers and wall cleaning techniques. He first summarized previous studies of film formation and properties. The conclusion from these studies is that boronization of DIII-D has introduced sufficient boron to globally effect plasma-wall interactions. The next part of the presentation dealt with the question of what can be done to remove a mixed-material surface. The method presented is that of the transferred arc (TA) discharge. This method holds some promise for detritiating surfaces after plasma bombardment. It was found that the TA method is able to effectively remove deuterium and plasma deposited carbon from surfaces.

The final session, chaired by **R.E.H. Clark**, was devoted to a summary of the results of the CRP and a discussion of the final reports and the publication of the results. The conclusions reached in this session are summarised in the next section. Brief summaries from each the CRP participants are attached at the end of this report. In addition, most of the CRP participants have also contributed articles to the *Atomic Processes and Material Interaction Data for Fusion* (APID) journal.

### **3. Meeting Conclusions and Recommendations**

The assessment of the overall success of the CRP in achieving its objectives was done on the basis of brief reports of the meeting participants for the activities within each CRP project for the duration of the CRP. Their reports are attached in Appendix 3. Both these reports and the presentations at the RCM indicate that the CRP has achieved its goal of generating (by using experimental and theoretical methods) new data on the erosion properties (physical sputtering, chemical erosion, and radiation enhanced sublimation) and hydrogen retention characteristics (diffusivity, permeation, and recombination) of mixed (doped) plasma facing reactor materials such as Be/c, Be/W, C/W, B-C, Si-C, Ti-C. The CRP has resulted in the collection and critical assessment of all existing information of this type on these systems, and will produce a recommended database for use in fusion energy research and other applications. The degree to which this CRP was successful is best shown in a table of new available data, presented as table 1. One sees that this CRP has added a large amount of new data and basic knowledge of these processes in materials relevant to fusion reactor design.



**Table 1: Available database**

Base Mater. Addition	Be	C, a:C-H	Si	Mo	W
<b>B</b>	- - -	Chem Ero Ret	- Ero -	- - -	- - -
<b>Be</b>	<b>X</b>	Chem Ero ret	- - -	- - -	Chem Ero Ret
<b>C</b>	Chem Ero Ret	<b>X</b>	- Ero -	Chem - Ret	Chem Ero Ret
<b>O</b>	Chem Ero Ret	Chem Ero -	- Ero -	- - Ret	- Ero -
<b>Si</b>	- - -	Chem Ero -	<b>X</b>	- - -	- - -
<b>Ti</b>	- - -	Chem Ero Ret	- - -	- - -	- - -
<b>W</b>	Chem - Ret	Chem Ero Ret	- - -	- - -	<b>X</b>

**Legend:** Chem = Chemical interaction studies  
 Ero = Erosion studies  
 Ret = Hydrogen retention studies



**2<sup>nd</sup> (Final) IAEA Research Co-ordination Meeting of the Agency's Co-ordinated  
Research Programme on "Plasma-Material Interaction Data for  
Mixed Plasma Facing Materials in Fusion Reactors"**

16-17 October 2000, IAEA Headquarters, Vienna, Austria

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**2<sup>nd</sup> (Final) IAEA Research Co-ordination Meeting on “Plasma-Material Interaction Data for Mixed Plasma Facing Materials in Fusion Reactors”**

16-17 October, IAEA Headquarters, Vienna, Austria  
Scientific Secretary: R.E.H. Clark

**Meeting Agenda**

Monday, 16 October

**Meeting Room: B0545**

09:30 - 10:00    Opening: Adoption of Agenda

Session 1: Latest Results

**Chairman: K. Watanabe**

10:00 - 10:45    R.A. Anderl:    Deuterium Retention in Tungsten and Tungsten Carbide

10:45 - 11:15    *Coffee Break*

11:15 - 12:00    V. Alimov:    Deuterium retention in mixed tungsten-carbon films prepared by magnetron sputtering in D2 atmosphere

12:00 - 12:45    E. Zhukovskaya: The method of H-isotopes and boron depth profiling by use neutron flux as a tool for plasma-facing components investigation

12:45 - 14:00    *Lunch*

Session 2: Latest Results II

**Chairman: V. Alimov**

14:00 - 14:45    C. García-Rosales: Development of improved doped carbon materials for fusion application

14:45 - 15:30    E. Vietzke:    The siliconization of TEXTOR and the use of silicon-doped carbon as testlimiter

15:30 - 16:00    *Coffee Break*

- 16:00 - 16:45    K. Watanabe:    Solid-state reactions of hydrogen-containing carbon films with metal substrates (Be, Mo and W)
- 16:45 - 17:30    A.A. Haasz:    The effect of metal surface impurities on the oxidation of graphite
- 17:30 - 19:00    *Reception*

Tuesday, 17 October

Session 3: Latest Results III

**Chairman: C. García-Rosales**

- 09:00 - 09:45    M. Guseva:    Investigation of W-C codeposited layers in plasma accelerator
- 09:45 - 10:30    J. Roth:    Formation and erosion of multi-component materials under fusion conditions
- 10:30 - 11:00    *Coffee Break*
- 11:00 - 11:45    R. Doerner:    Deposition and removal of mixed-material surface layers
- 11:45-12:15    Preliminary discussion of results
- 12:15 - 14:00    *Lunch*

Session 5: Formulation of Final CRP Outcome and Form of Publication

**Chairman: R.E.H. Clark**

- 14:00 - 15:00    Discussion of form of publication of final reports Method of transmitting final outcomes to A+M Data Unit Meeting Conclusions
- 15:00-15:30    *Coffee Break*
- 15:30-16:30    Formulation of Meeting summary
- 16:30 -        *Adjournment of Meeting*

**Summary Reports from CRP Participants**



## **Mixed Plasma-Facing Materials Research at the INEEL**

R. A. Anderl, G. R. Longhurst, R. J. Pawelko  
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### **Introduction**

Mixed-materials research at the Idaho National Engineering and Environmental Laboratory (INEEL) has focused on the Be-C and W-C systems. The primary purpose of this work was to investigate hydrogen isotope retention in these systems. For this purpose, we simulated plasma-mixed material layers using carbon-coated Be and W specimens that were heat-treated and tungsten carbide specimens prepared by chemical vapor deposition (CVD). Hydrogen isotope retention was investigated by means of thermal desorption spectroscopy (TDS) measurements on deuterium-implanted samples. Results of this work on Be-C were published in references 1-3. References 4-6 document the results of our studies on the W-C system and compare deuterium retention data to results derived from previous permeation experiments for tungsten foil [7].

### **Summary**

#### **Be-C**

Sample preparation entailed fabrication of carbon-coated Be foils, heating of the composite samples at different temperatures and depth-profile Auger electron spectrometry (AES) analyses. Coated specimens were prepared by exposing Brush Wellman ingot metallurgy foils (type IF-1, 99.8% Be, 150 ppm BeO) in a vacuum chamber to carbon vapor generated from an arc discharge between high-purity carbon electrodes. Specimens with a carbon layer thickness of 10, 50, 100 and 130 nm were prepared. AES depth profile analyses for annealed coated specimens indicated that for a temperature of 300°C, no Be/C inter-diffusion was observed during a two-hour annealing. For a temperature of 400°C, Be diffused into the carbon over-layer at a rate of ~20 nm/h, whereas for a temperature of 500°C, Be diffusion in the carbon over-layer was quite rapid, with complete migration through a 130-nm C layer in less than 2 hours. The characterization analyses indicated a mixed layer of Be<sub>2</sub>C and possible graphitic phases throughout the original carbon layer, with a thin BeO layer at the original C-Be interface.

Deuterium retention studies were made by exposing pure Be and C-coated Be specimens to a fluence of  $\sim 2 \times 10^{23}$  D/m<sup>2</sup> at a flux rate of  $6 \times 10^{19}$  D/m<sup>2</sup> for 1-keV D ions. Samples were heated to 400°C during the implantation. Post-implantation TDS measurements (23 to 1000°C, 30°C/min) indicated that for comparable implantation conditions, the quantity of deuterium retained in the C-coated Be sample was about 40% more than that retained in the un-coated Be samples. Thermally-induced release of deuterium occurred predominately at lower temperatures for bare Be (principal D<sub>2</sub> desorption peak at ~250°C) than that for C-coated Be (principal D<sub>2</sub> desorption peak at ~400°C). These results suggest that, for the mixed C-Be layer, deuterium retention is affected by the presence of possible graphitic phases of C. Details of these studies have been published [1-3].

## W-C

Many different sample types were prepared and characterized for our experiments on deuterium retention in W-C systems compared to pure W. All specimens were 16.5-mm in diameter with a thickness of 25  $\mu\text{m}$  for W foil, 0.5-mm for W1%La and 0.75-mm for CVD-W and CVD-W<sub>2</sub>C products. W foil samples were punched from high purity foil (99.95 % by weight), a reduction-rolled, powder-metallurgy product. This material was from the same lot as that used for earlier permeation studies [7]. These foils were implanted “as-received” and after annealing at 1200°C. Samples of 0.5-mm W1%La discs were polished to a mirror finish and in some cases were annealed at 1000°C or coated with a carbon surface layer. Tungsten and tungsten carbide samples were made using proprietary CVD processes. The CVD-W material, machined from a deposit collected in one CVD cycle, had a columnar microstructure, no indications of porosity or second-phase interstitials, a density of 19 g/cm<sup>3</sup> and a micro-hardness of 525 VHN. In contrast, fabrication of the CVD-tungsten carbide material required multiple CVD deposition cycles, resulting in a microstructure that contained distinct columnar layers and interfaces between the layers, a density of 18.3 g/cm<sup>3</sup>, and a micro-hardness of 2040-2450 VHN. Based on XRD analyses, the CVD-tungsten carbide consisted primarily of W<sub>2</sub>C, with some indication of trace C, most likely at the grain boundaries of the structure.

Carbon-coated samples were prepared by exposing polished W1%La discs to carbon vapor generated from an arc between two carbon electrodes in a vacuum system. Nominal coating thickness ranged from 100 to 150 nm. Carbon-coated samples with 100-nm thickness were used in annealing studies to determine inter-diffusion properties of the C-W system. Depth-profile Auger analysis of three different samples heated to 400°C, 600°C, and 800°C for 3 hours each revealed that some intermixing was observed at the C-W interface only for the sample annealed at 800°C. No intermixing was observed in the other samples.

Test samples were implanted with a deuterium ion beam (500 eV/D,  $\sim 3 \times 10^{19}$  D/m<sup>2</sup>-s) to a fluence of  $\sim 3 \times 10^{23}$  D/m<sup>2</sup>. Sample temperatures were set at values between 23°C and 400°C during implantation. Retained quantities of deuterium were measured using TDS (23 to 1000°C, 30°C/min). Principal findings of this work included the following:

- (1) TDS retention data for the 25- $\mu\text{m}$  W foil are consistent with retention values derived from the permeation experiments for material from the same batch.
- (2) Annealing of 25- $\mu\text{m}$  W foil to 1200°C results in a retention reduction by a factor of 4 for deuterium implanted at 200°C, with similar retention reductions observed for other W types that were annealed.
- (3) Retention in CVD-W and W1%La is below that in W foil for implantation temperatures <200°C, indicating that different fabrication processes for disc and foil materials and the resulting intrinsic defect structures influence the retained quantities.
- (4) Above 200°C, TDS results indicate the opposite trend, possibly due to higher bulk retention in thicker CVD-W and W1%La samples, resulting from diffusive transport into the bulk.
- (5) Retention in CVD-W<sub>2</sub>C was somewhat higher than that in CVD-W for temperatures below 300°C, most likely because of trapping at trace C impurities in the bulk, at impurities and porosities in micro-structural defects or at beam-induced damaged sites produced by recoil C.

- (6) Desorption peaks were observed at similar temperatures (130, 300, 400 and 530°C) for these W and W<sub>2</sub>C materials, with the relative magnitudes varying from material to material.
- (7) Implantation into C-coated tungsten specimens results in retention that is orders of magnitude greater than that in a pure tungsten surface. Annealing studies for C-coated W indicate little inter-diffusion of C and W for temperatures less than 800°C.

Results of this work [4-6] demonstrated that retention of deuterium implanted into tungsten and tungsten carbide samples is highly dependent on the material type and structure, anneal condition and the presence of free carbon. Consequently, retention in W-C mixed material layers and carbon-covered tungsten PFC components may be dominated by uptake in the carbon.

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**Report on activities related to CRP on “Plasma-material interaction data for mixed plasma facing materials in fusion reactors”**

V. Alimov

At the 1<sup>st</sup> Research Co-ordination Meeting (October, 1998) Dr. V. Alimov reported on oxygen and deuterium retention in the surface layers of beryllium and beryllium oxide. It has been shown that the irradiation of beryllium with D-ions in O<sub>2</sub> atmosphere at 300 and 700 K leads to oxygen incorporation into the sample. The total amount of incorporated oxygen increases stepwise with D-ion fluence. At the first accumulation stage, the oxygen atoms are incorporated into the surface layers. At the second accumulation stage, the development of interconnected gas porosity in the Be matrix and penetration of oxygen molecules through the system of open pores result in the oxidation of new-formed beryllium surface; in so doing the marked increase of the oxygen content in the ion stopping zone takes place. The maximum content of oxygen incorporated into the surface layers and the bulk increases with the oxygen pressure and decreases when the irradiation temperature increases from 300 to 700 K.

Deuterium implanted into beryllium oxide film at irradiation temperatures of 300 and 700 K was found to reside as both molecules and atoms. Irradiation at these temperatures leads to the formation of tiny D<sub>2</sub> bubbles of 0.6-0.7 nm in radius and of high volume density  $\cong 4.5 \times 10^{24} \text{ m}^{-3}$ . These bubbles together with the intercrystalline gaps are responsible for the accumulation of a molecular fraction of the implanted deuterium. At both irradiation temperatures the D<sub>2</sub> concentration reaches its maximum of 0.01 D<sub>2</sub>-molecules/BeO. The major part of implanted deuterium is present in BeO film in the form of D atoms, probably, chemically bound to O atoms. The maximal D atom concentration is 0.18 D-atoms/BeO for 300 K and 0.05 D-atoms/Bo for 700 K. Thus, the maximum concentration of deuterium accumulated in the both states in BeO at 300 and 700 K reaches value of 0.20 D/BeO and 0.08 D/BeO, respectively.

At the 2<sup>nd</sup> Research Co-ordination Meeting (October, 2000) Dr. V. Alimov presented recent experimental results regarding deuterium retention (i) in graphite, W single crystal, and chemical vapor deposited (CVD) carbon-containing tungsten W<sub>0.75</sub>C<sub>0.25</sub> implanted with D ions and (ii) in carbon and tungsten-carbon mixed films prepared by dc magnetron sputtering of graphite and tungsten cathodes in D<sub>2</sub> atmosphere. It has been shown that the maximum

concentration of deuterium accumulated in the ion stopping zone of graphite irradiated with 3 keV D ions at 300 K reaches value of  $\sim 0.40$  D/W. In the hydrogenated carbon films prepared by dc magnetron sputtering in  $D_2$  atmosphere, the deuterium concentration is varied from  $\sim 0.4$  D/C at deposition temperature of 400 K to  $\sim 0.02$  D/C at 973 K. Irradiation of W single crystal with 6 keV D ions at 300 K leads to the accumulation of D atoms both in the ion stopping zone and at depths up to several  $\mu\text{m}$ . Besides, in the stopping zone deuterium is additionally accumulated in the form of  $D_2$  molecules. After D ion implantation at 650 K,  $D_2$  molecules are absent in the W sample and deuterium is accumulated in the form of D atoms alone at depths up to 500 nm. The maximum concentration of D accumulated in both states in the ion stopping zone reaches value of  $\sim 0.08$  D/W at 300 K. After irradiation at 650 K, this value is found to be  $\sim 0.015$  D/W. The maximum concentration of all deuterium (D atoms and  $D_2$  molecules) trapped in the ion implantation zone of CVD carbon-containing tungsten  $W_{0.75}C_{0.25}$  implanted with 6 keV D ions is  $\sim 0.06$  D/host atom for  $T_{\text{irr}} = 300$  K and  $\sim 0.01$  D/host atom for  $T_{\text{irr}} = 650$  K.

Tungsten-carbon mixed films prepared by dc magnetron sputtering in  $D_2$  atmosphere contain inclusions of tungsten carbides WC and  $W_2C$ . At 400 K, the concentration of the D atoms and  $D_2$  molecules in the hydrogenated W-C mixed film is  $\sim 0.007$  D/host atoms and  $\sim 0.008$   $D_2$ /host atom, respectively. Thus, in spite of the presence of carbon atoms in the W-C mixed films (up to 30%), the concentration of  $D_2$  molecules in these films is much lower than it is expected from the assumption that the hydrogenated W-C mixed film consists of individual tungsten and carbon atoms.

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## FINAL REPORT FOR RESEARCH PROJECT IAEA

“Development of the Neutron-Induced Elastic Recoil Detection (NERD) Method for the Study of Hydrogen Isotope Retention in Plasma-Facing Components of a Thermonuclear Reactor”

IAEA Research Contract No. 10100/RO

*Institute of Nuclear Physics Academy of Science Uzbekistan, Tashkent*  
*Chief Scientific Investigator: Dr. E. Zhukovskaya*

This project is devoted to exploration hydrogen isotope components accumulation processes in tokamak first wall. Such an investigation is used to make decision in a choice of optimal fusion reactor plasma-facing components. The project is considered the following guidelines of studies, framed with the mentioned general task.

Our group had been working on this project in two directions. First, we had been persuading the goal of adapting Neutron induced Elastic Recoil Method to plasma-facing material exploration. Second, we want to prove effectiveness and perspective of such investigations.

The results of our investigations can be summarised in the following way. It has been implemented for the improvement of the experimental method:

- Changing a design of the scattering chamber for the possibility of detectors telescope location at arbitrary (nonzero) angle for a depth resolution improvement.
- Optimisation of geometry of the spectrometer as a whole, for obtaining a highest possible rate of useful counts (for different angles of registration) with required energy resolution.
- Improvement of the energy resolution of spectrometer by the reduction of the effect of neutron rescattering on constructive elements.

As a result of the development of algorithm of DRIN code it takes into account now the following:

- Measurements and analysis of depth profile H, D on tokamak JET and B<sub>4</sub>C coated samples with dT neutrons.
- Measurement and analysis of contents and profile concentration H, D on magnetic and tokamak JET samples with dD neutrons.
- The measurements of the concentration profiles and contents of boron and hydrogen isotopes have been carried out for graphite samples with B<sub>4</sub>C coating as well as tungsten.
- As a result of the development of DRIN code algorithm a possibility of calculation of depth profile concentration is appeared not only for hydrogen isotopes but also for boron. It is important for investigation of hydrogen isotope retention in plasma-facing components (B/C, B/W,...) of a thermonuclear reactor.
- The possibility for determination of hydrogen isotopes concentration profiles in material containing boron without using the background samples, as well as without the additional B(n,a) reaction measurements is motivated, since (n,a) - method reduces greatly a depth of analysis.



# **Plasma-Material Interaction for Mixed Plasma-Facing Materials in Fusion Reactors studied in TEXTOR-94**

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## **Introduction**

The main activities in this project were concentrated to analyse erosion and deposition processes including local particle transport, to study the hydrogen recycling and to investigate the behaviour of several test limiters made from different materials or from different doped carbon during TEXTOR discharges. The materials investigated in comparison to pure carbon were thin boron or silicon films on all walls (so called boronisation and siliconisation), silicon-doped CFC material (NS31), tungsten and tungsten-coated graphite, tantalum and thick B<sub>4</sub>C coated copper as limiter material. NS31 is a reference material for the lower divertor target foreseen for ITER and B<sub>4</sub>C coated copper is foreseen for the first wall of the stellarator W7-X in Greifswald.

## **Modelling of erosion and deposition processes by the ERO-TEXTOR code**

Simulating erosion and redeposition processes in fusion devices lead to a better understanding of the involved processes. The 3-dimensional Monte-Carlo code ERO-TEXTOR [1,2] has been developed to model the plasma-wall-interaction and the transport of eroded particles in the vicinity of test-limiters exposed to the edge plasma of TEXTOR. Important problems concerning the lifetime of various wall materials (high  $Z$  vs. low  $Z$ ) under different plasma conditions and the transport of eroded impurities into the main plasma can be treated with the ERO-TEXTOR code. Recently, the divertor geometry was implemented to carry out simulations for JET, ASDEX and ITER [3].

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J. Nucl. Mat. 290-293 (2001), 238



## Solid-state Reactions of Hydrogen-containing Carbon Films with Metal Substrates

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Hydrogen-containing carbon films were prepared on tungsten, molybdenum and beryllium as model systems to simulate changes in physiochemical properties of carbon depositing on the inner wall of a tokamak, in which carbon tiles are used in combination with two or more plasma facing materials. The properties of the co-existing layers and their solid-state reactions at elevated temperature were studied by means of infrared, Raman, x-ray photoelectron (XPS), x-ray diffraction (XRD) and thermal desorption (TDS) spectroscopies. In this report, however, much attention was focused on observations for the carbon films deposited on tungsten.

[1] Sample preparation: Hydrogen-containing carbon films were prepared on tungsten, silicon and quartz-glass plates by inductive RF discharge (13.56 MHz with 40 W) of methane or ethylene at 40 Pa. The size of each plate was 10 X 10 X 0.5 mm and the thickness of deposited films was about 0.4 micron. Hereafter, the films are denoted as C(H)/W, C(H)/SiO<sub>2</sub>, C(H)/Si, etc. The first one was used for XPS, XRD and TDS measurements, C(H)/SiO<sub>2</sub> was prepared as a reference for TDS measurements. The last one was for FT-IR and Raman spectroscopies.

[2] Characterization of as-prepared carbon films: The compositions of the carbon films were determined from the changes in the total and partial pressures during the discharge. The methane discharge yielded a hydrogen-containing carbon film of [H]/[C]  $\approx$  1.7 in atomic ratio. In the case of ethylene discharge, the hydrogen content in the film was determined to be [H]/[C]  $\approx$  1.4. Both of the films showed very similar FT-IR and Raman spectra in the frequency regions of the C-H and C-C stretch bands, respectively. They indicated that the C-C and C=C bonds make the principal structure of the as-prepared carbon films, which contain hydrogen as -CH<sub>3</sub> and >CH<sub>2</sub>.

[3] Solid-state reactions at elevated temperature:

[3.1] TDS measurements: The C (H)/W and C (H)/SiO<sub>2</sub> samples were heated in vacuum with a temperature ramp of 9 C/min from room temperature to 1000 C. Both of the films showed a desorption peak of H<sub>2</sub> about 450 C, which was accompanied by desorption of other species as C1-, C2- and C3-hydrocarbons. The C (H)/W film gave another hydrogen desorption peak about 920 C, whereas the C (H)/SiO<sub>2</sub> did not form any noticeable desorption peaks above 450 C. These observations indicate that the low temperature peak is due to thermal decomposition of the deposited carbon films, and the substrate dependent high temperature peak is ascribed to a reaction of the carbon film with the substrate.

[3.2] XPS and XRD measurements: The carbon films deposited on tungsten were heated step by step in vacuum at a given temperature for 10 minutes up to 1000 C to observe changes in surface and crystallographic states. Below 400 C, only carbon (about 80%) and oxygen (20%) were detected by XPS, where the C1s peak was at a position of amorphous carbon type (285.5 eV) and the O1s peaks appeared at the positions of -OH (533.0 eV) and -CO (530.8 eV) types. The W4f peaks began to appear at 500 C and grew with temperature in compensation with the reduction in the peak intensities of O1s, which disappeared at 600C. Above this temperature, the peak

intensity of C1s decreased with temperature in compensation with the growth of W4f peaks. Finally the surface atomic fraction attained to  $[W]/[C] \approx 2$ , where both C1s and W4f peaks appeared at the binding energies corresponding to carbide. During this process, the binding energy of C1s continuously decreased from 285.5 to 283.2 eV, whereas the binding energies of O1s were kept at 530.8 and 533.0 eV. The XRD measurements confirmed the formation of tungsten carbide,  $W_2C$ , by vacuum heating at elevated temperatures.

[3.3] Comparison with C(H)/Mo and C(H)/Be films: Very similar features were observed for C(H)/Mo and C(H)/Be by TDS, XPS and XRD spectroscopies. The solid-state reactions of C(H)/Me can be divided into two types. One is the thermal decomposition of C(H)-film itself around 400-450 C and then is substrate independent. The other is the carbide formation at higher temperatures and depends on the substrate. There was found good relations between the reaction temperature and physicochemical properties of the substrates as melting point, surface tension, etc. For example, the halfway temperature at which the extent of carbide formation attained to 50% showed a good linear relation with the melting point of substrate. Namely, the reactivity of the carbon film is in the order of  $Be > Mo > W$ .

[4] Kinetic analysis: Reaction curves observed for C(H)/Be at 600 and 700 C was analyzed by a nucleation and growth model by assuming two dimensional crystal growth. The kinetic equation is described as

$$-\ln(1-x) = \left( \frac{\pi h N_0 f D}{2} \right) \exp(-ft) t^2$$

where  $h$  is the thickness of the deposited film,  $N_0$  the number of nucleation site per unit volume,  $f$  the nucleation frequency per site and  $D$  the diffusion constant of carbon. However, since the diffusion constant and the number of nucleation sites are not known, the above equation was rearranged as

$$-\ln(1-x) = C f \exp(-ft) t^2$$

where  $C$  is a constant, and fitted to the observed reaction curves by non-linear fitting. It was found that this equation reproduced fairly well the reaction curves, but the nuclear frequency,  $f$ , was extremely small. Namely, the equation can be simplified as

$$\ln(1-x) = k / t^2$$

where  $k$  is the apparent rate constant. It was found that this equation can reproduced well the reaction curves for C(H)/Me (Me=Be, Mo, W) observed by XPS and XRD.

[5] Conclusions: The hydrogen-containing carbon film undergoes two distinct reactions at elevated temperatures. One is the thermal decomposition of the film itself and the other the reaction with substrate metals to form carbide of  $Me_2C$ . The reactivity of the film to form carbide was in the order of  $Be > Mo > W$ . It was found that the reactivity is strongly dependent on physicochemical properties of substrate properties as melting point, surface tension, heat of formation and so on. The reaction kinetics can be explained well by a nucleation and growth model.

**IAEA RCM on “PMI Data for Mixed Plasma-Facing Materials,” Vienna, 2000oct16-17**

***Brief statements regarding the benefits and impact of the CRP***

- Excellent forum to bring together international experts in the fusion/PMI community
- Meetings provide excellent opportunity to share latest research results in a setting that is conducive to informal and open discussions.
- The CRP engages researchers in the field to collectively identify critical-path issues for fusion/PMI R&D.
- The CRP also enables the participants to plan their respective research directions and programs in a coordinated fashion addressing the critical issues that have been collectively identified.
- At the conclusion of this particular CRP on ‘Mixed Materials’ the participants have agreed to produce a brief summary report on their respective presentations at the 2000oct16-17 RCM in Vienna [UTIAS report is attached below], and a series of brief articles that will be collected in an ‘IAEA Green Book.’ The articles will be grouped under the following three main subject headings:
  - (1) Materials: Formation and Characterization
  - (2) Erosion Processes
  - (3) Hydrogen Inventory and Removal

**Studies of Mixed Materials**

**at the University of Toronto Institute for Aerospace Studies**

A.A. Haasz, J.W. Davis, C.G. Hamilton,

R. G. Macaulay-Newcombe, M. Poon and P.B. Wright

**Contents**

- (1) ***Doped Graphites***
  - Chemical erosion and radiation-enhanced sublimation (RES)
  - Thermal diffusivity/conductivity
  - D retention
- (2) ***Carbon Implanted in Tungsten***
  - D retention as a function of C thickness

- (3) **Boron in/on Graphite from DIHD**  
Chemical erosion due to  $D^+$   
Thermo-oxidation in  $O_2$
  - (4) **Tungsten on Graphite [new results]**  
Thermo-oxidation of EK98 graphite with W surface coverage
- 

(1) **Doped Graphites**

**Chemical erosion and RES:** Chemical erosion and radiation-enhanced sublimation are considered to be major drawbacks for the use of carbon as a plasma-facing material in fusion reactors. In order to improve graphite's erosion resistance, doping with other elements has received extensive attention. We have studied the effect of doping on both chemical erosion and RES using specially fabricated specimens provided by a Canadian company, Ceramics Kingston (CKC). Dopants used were: B, Si, Ti, Ni, and W. We found that doping with B was most effective in reducing chemical erosion, mainly at high temperatures and high energies [Allen Y.K. Chen, A.A. Haasz, J.W. Davis, J. Nucl. Mater. 227 (1995) 66]. Ti was seen to reduce RES in some cases [P. Franzen, A. A. Haasz, J. W. Davis, J. Nucl. Mater. 226 (1995) 15]. These findings led us to test graphite doped with  $TiB_2$ , with favourable results obtained for chemical erosion [J.W. Davis, A.A. Haasz, J. Nucl. Mater. 255 (1998) 214]; no RES studies have been done on this material.

**Thermal diffusivity/conductivity:** The thermal diffusivity and conductivity of the CKC doped specimens were measured and were found to be anisotropic. Significant reduction in thermal conductivity with increased dopant concentration, especially at low temperatures [ $\sim 300K$ ], was found in the 'high' conductivity direction. In the 'low' direction, the reduction due to dopants was not significant, but the absolute levels themselves were considerably lower than even the doped 'high' conductivity levels. [B.N. Enweani, J.W. Davis, A.A. Haasz, J. Nucl. Mater. 224 (1995) 245-253; A.A. Haasz, J.W. Davis, J. Nucl. Mater. 252 (1998) 150].

**D retention:** With increasing  $D^+$  fluence, doped graphites retain relatively higher levels of D than pure carbon. The increased retention is relatively much larger for Si and W dopants than for B and Ti, the latter two being similar to EK98 graphite [A.A. Haasz, and J.W. Davis, J. Nucl. Mater. 232 (1996) 219-225]. It is not clear whether the increased retention is due to the dopants, or the increased porosity of the materials.

(2) **Carbon Implanted in Tungsten**

**D retention as a function of C thickness:** With Be, C and W in ITER, the effect of 'mixed materials' on plasma-surface interactions becomes very important. For example, how will the presence of carbon on W affect D uptake and transport? Using W specimens [foils of 25  $\mu m$  thick polycrystalline, 99.95 wt% pure W, annealed at  $T > 1470 K$  for 1 hour], we have implanted  $C^+$  in the near surface to build up a controlled C layer. We found that D retention in  $C^+$ -implanted W strongly depends on  $C^+$  fluence and implantation temperature. With low  $C^+$  fluence, W carbides

are formed, leading to decreased D retention at low  $D^+$  fluences – possibly due to enhanced surface recombination. With high  $C^+$  fluence, and low  $D^+$  fluence, the presence of a graphitic surface layer increases D retention above the pure W case. With increasing  $D^+$  fluence, preferential sputtering removes both the graphitic surface layer and the implanted C from the bulk, such that the D retention of the specimen tends to pure W at high  $D^+$  fluences.

**Surface analysis:** XPS surface analysis confirmed the formation of WC for the low  $C^+$  fluence case, and graphitic carbon at high  $C^+$  fluences. RBS analysis for the high  $C^+$  fluence case at 300K implantation temperature showed evidence of C diffusion into the bulk, with a tail of C, ~2 at%, extending 170 nm into the specimen. With high  $C^+$  fluence implantations at 500 K, C diffusion into the bulk is extended to ~ 300 nm, at ~1 at% C.

### (3) *Boron in/on Graphite from DIII-D*

Spectroscopic measurements in DIII-D at the midplane have shown no reduction in the CD band intensity over time. On the other hand, at the lower divertor, the CD band intensity was seen to decrease progressively over time [D. Whyte et al., 14<sup>th</sup> PSI Conf., Rosenheim]. In order to further probe the observed behaviour, specimens from both of these locations were obtained, and controlled laboratory experiments were performed.

#### **Surface analysis:** *[midplane specimen]*

SEM: Appearance of a globular, porous-looking surface, similar to other plasma-deposited films, is evident. Cross-section show photos show the film thickness to be ~ 2  $\mu\text{m}$ .

EDX, XPS and SIMS: We see a thick boron-rich layer [~2  $\mu\text{m}$ , introduced during boronization]. On top of the boron, is a thin C/B:D layer of ~ up to ~ 100 nm, containing a significant amount of C [~ 20 at%]. The surface C layer implies a net deposition of C from the plasma on the midplane tiles.

**Ion-beam studies of chemical erosion due to  $D^+$ :** On the lower divertor tile, after a very low fluence of  $D^+$  exposure, the chemical reaction yields of the products [hydrocarbons] reached steady-state levels similar to those for graphite. This implies that whatever B-containing surface layer existed on the specimen, the  $D^+$  beam must have removed it rather ‘fast,’ exposing the graphite substrate.

For the midplane tiles, on the other hand, at fluences comparable to those received during a DIII-D discharge [ $<10^{22}$ /shot], we see erosion yields comparable to B-doped graphite. As the fluence is increase, the yields are reduced by about a factor of 2, consistent with preferential removal of C, leading to a B-enriched film. The fact that the yield does not go to zero may indicate that the erosion is limited by the removal of B, or by the diffusion of C to the surface. The fact that the steady-state yields are higher at the higher temperatures indicates that C diffusion may be important. If we extrapolate these results to plasma operation in DIII-D, where the fluence is  $<10^{22}$ D/shot, we would not expect the thin carbon layer on the B-rich layer to be removed.

**Thermo-oxidation of DIII-D Specimen in  $O_2$ :** We have performed thermo-oxidation experiments on a variety of specimens obtained from operating tokamaks [e.g., J.W. Davis and A.A. Haasz, J.

Nucl. Mater 266-269 (1999) 478]. The main objective of such experiments is to assess the tritium removal efficiency of oxidation at elevated temperatures [ $\sim 600\text{K}$ ] from codeposited C:H layers. D content in the specimens was measured by laser thermal desorption as function of oxygen exposure time. The initial D concentration of  $\sim 10^{22}\text{D/m}^2$  was reduced by only 25-30%, consistent with the removal of the surface C layer [with D in it], leaving a stable B-rich surface behind. The remaining D may have been trapped during boronization or have been codeposited with C during tokamak discharges, and later buried during B deposition.

#### (4) Tungsten on Graphite [new results]

In Sec. 2 above, we discussed mixed W/C surfaces that were produced via implantation of C in W. Conversely, in the divertor, the graphite or CFC target surfaces could be contaminated with W that gets sputtered from the divertor wall. As a first attempt to study such surfaces, we have evaporated W on one face of fine-grain EK98 graphite [estimated 1, 3 and 10 monolayers – based on W temp ( $\sim 2,600\text{K}$ ) and geometry ( $\sim 2\text{cm}$ )]. Subsequent to W evaporation, thermo-oxidation experiments were performed at  $\sim 600\text{K}$ , to assess the effect of W on the oxidation rate of carbon. From earlier experiment with pure graphites, we have found the oxygen erosion rate of pure EK98 found to have an upper limit of  $4 \times 10^{-10}\text{C/O}_2$  [Davis et al. “O<sub>2</sub> erosion of graphite tile substrates,” J. Nucl. Mater., accepted]. First results for the W-contaminated C show negligible mass changes after prolonged O<sub>2</sub> exposure, however XPS measurements indicated much lower W surface concentrations than expected. Further work is in progress.

### Title of CRP: Plasma-material Interaction Data for Mixed Plasma Materials in Fusion Reactors

#### Publication related to mixed materials R&D

##### During 2000

- [1] J.W. Davis, P.B. Wright, A.A. Haasz, “Chemical erosion of boronized films from DIII-D first wall tiles,” *J. Nucl. Mater.*, accepted.
- [2] A.A. Haasz, M. Poon, R.G. Macaulay-Newcombe, J.W. Davis, “Deuterium retention in single crystal tungsten,” *J. Nucl. Mater.*, accepted.
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## Report on activities related to CRP on “Plasma-material interaction data for mixed plasma facing materials in fusion reactors”

M. Guseva

Dr. M.I. Guseva presented on the recent experiments in the Kurchatov Institute of C-H and W-C-H codeposition on W and graphite. Experiments were done on the stationary plasma accelerator with closed electron drift and elongated acceleration zone named «TE type accelerator» with the 300 eV  $C_2H_2$  - plasma flux at 3 different doses:  $2 \cdot 10^{23}$ ,  $8 \cdot 10^{23}$ ,  $2 \cdot 10^{24}$   $C_2H_2/m^2$  for W target and  $4 \cdot 10^{24}$   $C_2H_2/m^2$  for graphite target. The kinetic energy of C and H ions was equal 11,5 eV for  $H^+$  and 138,5 eV for ions. The two typical microstructures of codeposited layers on tungsten - a uniform smooth films under doses  $2 \cdot 10^{23} m^{-2}$  and the globular structure at higher doses were obtained. A similar film with globular structure was detected on the graphite. With the irradiation dose growth integral carbon concentration and the thickness of the deposited layers rise from 1 to 10  $\mu m$ . The density of deposited films is in the range 0.52-0.79 of that of graphite. The content of hydrogen in the deposited films is determined by their structure. The integral hydrogen concentration in the deposited films is  $7,2 \cdot 10^{21} H/m^2$  for uniform film and  $3,2 \cdot 10^{21} m^{-2}$  for globular structure. If globular structure covers all the surface the hydrogen concentrations in the films on graphite and on W for highest dose irradiation are close. The mixed W+C+H- layers on tungsten and graphite were produced under simultaneous irradiation by 300 eV  $C_2H_2$  plasma and tungsten redeposition. The hydrogen accumulation in such mixed layers is small.



## **Mixed-Material CRP Summary of Research Conducted by UCSD/PISCES**

R. Doerner

The PISCES-B plasma device has examined the formation conditions and properties of mixed-material surface layers, which can form on plasma-facing components. The formation of the layers is a complicated process governed not only by the material's properties of the surfaces (such as temperature, reflection coefficients and sputtering yield), but also on the conditions in the adjacent plasma (redeposition probability and impurity content). Control of these parameters is necessary for understanding. PISCES is ideal for such a purpose and has demonstrated the necessary exposure control to make quantitative assessments on the importance of certain material mixes. The work in PISCES has focussed primarily on the Be/C and W/C systems.

The PISCES-B device has been modified to investigate mixed-materials in several ways. An impurity gas puffing system has been installed in the target interaction region. Experiments have focused on puffing deuterated methane ( $CD_4$ ) into the plasma. In addition, a beryllium gettering ball can be inserted upstream in the plasma column to seed the plasma with beryllium as it flows toward the target. This evaporator is necessary because of the long mean-free-path of sputtered beryllium in the PISCES-B plasma geometry prevents introduction of beryllium into the plasma by ionization of sputtered particles.

The formation of any surface layer is a competition between deposition and removal processes. Deposition from a plasma will be influenced by such factors as the impurity concentration of the plasma, the sticking coefficient of the impurity (and hence the type of impurity - molecular, atomic, ionic) on the surface, the incident energy of the impurity and probably many others. Removal will be influenced by chemical bonding on the surface, the plasma ion energy, the probability of redeposition and again probably many other factors. Influential in both the removal and deposition processes is the temperature of the surface. The temperature will determine the chemical structure of the surface as well as the interdiffusion of impurities and the bulk material. At elevated temperatures, molecules such as carbides are more likely to form and impurities deposited in the near surface region can more easily diffuse away from the surface and become more unlikely to remove. Therefore, it is more likely to form a surface layer at higher temperatures, all other variables being constant. In principle, a study of the formation of mixed-material surface layers should include variations of all of these quantities. In practice, this work focused on variations of the temperature of the surface and the concentration of impurities in the incident plasma [1] to develop a two dimensional map to determine the formation of mixed-material layers [2].

Work performed for this CRP, on the Be/C system, has examined the impact of mixed-material surfaces layers on the erosion rate of the substrate material [3], the formation conditions and growth rates [2], the hydrogen retention properties of the resultant material [4,5] and the chemical properties of the final mixed Be/C surfaces [4]. Similar work on the W/C system also examined growth rates of the layers [6], hydrogen retention [6] and properties of the resultant surfaces [7].

Measurements were also performed concerning the effects of dopants on the plasma-interaction behavior of carbon-based materials in PISCES. The impact on the chemical erosion of SiC doping of graphite, in the NS31 material, was found to be small due to the fact that the dopant was not dispersed uniformly throughout the graphite structure [4]. In-situ doping of carbon due to beryllium atom seeding of the incident plasma was also demonstrated to reduce the chemical erosion of the graphite substrate by more than the change in the surface concentration of carbon [4]. These studies in PISCES naturally led into investigations of the DIII-D tokamak to examine the role of in-situ doping of the graphite tiles in DIII-D due to repeated boronizations.

This line of research has been motivated by two results. Firstly graphite tiles that have been exposed in the DIII-D tokamak since 1993 have been shown to possess lower erosion yield. The tile surfaces have been modified extensively by the DIII-D plasma exposures and boronizations. The cause(s) of the reduced chemical erosion are not yet clear, but are thought to be a combination of boron dopant effects and plasma conditioning. Secondly, preliminary results from the PISCES-B carbon flux dependence experiments showed an apparent reduction in chemical yield after the sample had been exposed for several hours, resulting in a net erosion of > 40 microns. The DIII-D and PISCES-B results together suggest that long-term conditioning of carbon PFC can dramatically alter their erosion properties.

Finally, an effort was made to evaluate the effectiveness of the transfer arc cleaning process [8], developed at LANL, for removing plasma deposited mixed-material surface layers. Transfer arc (TA) cleaning utilizes a DC plasma torch and a secondary power supply attached between the torch (anode) and the part to be cleaned (cathode). The torch produces a plasma that becomes the electrical conduction path for the cleaning arc. The arc attachment to the cathode is influenced by the surface shape, the surface roughness and surface contaminant layers (oxide, organic and/or metal film). The cathode contaminant layers are preferential sites for arc attachment. This is caused by ion charge buildup on the contaminants producing an enhanced electric field that increases in strength until break down of the film occurs resulting in arcing to the cathode. Joule heating of the cathode causes erosion of the contaminant layer and the bulk cathode material beneath. As the contaminant is removed, electron emission ceases and the arc is displaced to another area with remaining contaminant. This process continues until the contaminants are removed.

Transfer arc cleaning proved successful in removing all surface impurity films deposited on test samples exposed to impurity containing plasmas in PISCES-B [9]. In addition to removing the surface layers the TA technique also removed all measurable deuterium that was trapped in the layers. Finally, the concentration of carbon that had migrated into the bulk of the samples during the exposures at high temperature was also significantly reduced by TA cleaning. Although questions still exist as to the practicality of the use of a TA cleaning technique in a fusion reactor, the results obtained so far have been promising.

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