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INDC INTERNATIONAL NUCLEAR DATA COMMITTEE

1st IAEA Research Co-ordination Meeting on "Plasma-Material Interaction Data for Mixed Plasma Facing Materials in Fusion Reactors"

October 19-20, 1998, Vienna, Austria

SUMMARY REPORT

Prepared by:
R.K. Janev and G. Longhurst

December, 1998

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

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Abstract

The proceedings and conclusions of the 1st IAEA Research Co-ordination Meeting on "Plasma-Material Interaction Data for Mixed Plasma Facing Materials in Fusion Reactors", held on December 19 and 20, 1998 at the IAEA Headquarters in Vienna, are briefly described. This report includes a summary of the presentations made by meeting participants, a review of the data availability and data needs in the areas from the scope of the Co-ordinated Research Project (CRP) on the subject of the meeting, and recommendations regarding the future work within this CRP.

Reproduced by the IAEA in Austria
December, 1998

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1. INTRODUCTION

The IAEA 1st Research Co-ordination Meeting (RCM) on "Plasma-Material Interaction Data for Mixed Plasma Facing Materials in Fusion Reactors" was held on October 19 and 20, 1998 at the IAEA Headquarters in Vienna, Austria, and was organized as part of the activity within the IAEA Co-ordinated Research Project (CRP) on the same subject. The objectives of the Meeting were:

a) to review the work carried out by the CRP participants within their CRP related projects since the inception of the CRP;
b) to review the overall status of the data availability and needs for erosion and hydrogen retention properties (and underlying processes) of fusion relevant mixed materials;
c) to formulate and adopt conclusions and a plan of action regarding focussing the future CRP activities with an aim to achieve the CRP objectives and to establish a close work co-ordination among the CRP participants.

The meeting was attended by ten CRP project chief scientific investigators, two observers and the staff of the IAEA Atomic and Molecular Data Unit (see Appendix 1: List of Participants).

After the opening of the Meeting by the Scientific Secretary, Dr. R.K. Janev, the work of the meeting proceeded in four sessions (see Appendix 2: Meeting Agenda). The first three sessions were devoted to presentation of the results obtained within the individual CRP projects, while during the last meeting session a review of the available data and the data needs was carried out, including a discussion on re-focussing the CRP objectives and the future work co-ordination within the CRP.

2. BRIEF MEETING PROCEEDINGS

Dr. J. Roth was the first technical speaker, addressing the experimental investigation of tungsten sputtering by carbon and methyl ions at different temperatures. He described a process by which these ions may erode the surface or deposit films on the surface or both, depending on ion energy and surface temperature. Experimental weight-change data showed that at ambient temperature, incident carbon first removed material by sputtering, then as a protective carbon film grew on the surface, the tungsten began to be shielded, and there was a net carbon deposition to arbitrary thickness. As the temperature of the tungsten was increased in successive tests, the rate of film growth was slower and slower until at 800°C, there was no observed growth, only a slowing of the erosion rate with increasing ion fluence. The same kinds of effects were seen at constant target temperature when carbon and methyl ions were applied to pure tungsten and to carbon-covered tungsten targets. Carbon ions seemed to be more effective in stopping the erosion and in generating film growth compared with methyl ions. A two-parameter theoretical model was developed that seemed to predict this behavior

quite well. Similar experiments are planned to investigate carbon segregation at the carbon-tungsten interface under carbon ion bombardment, the formation of carbides in tungsten, titanium, and silicon, and the results from implanting carbon monoxide into beryllium.

Dr. W. Eckstein was the next speaker. He discussed modeling of sputter yields using TRYDIN, the dynamical TRIM.SP code. The code calculates the dependence on ion fluence to the surface of such features as surface recession, surface and subsurface composition profiles, sputter yields, and reflection coefficients. Results are sensitive to angle of incidence and energy of the ions. Analyses were presented for carbon, methane, and deuterium incident on beryllium and tungsten. Results were in general agreement with experimental observations, though some concern was expressed because of the sensitivity of the results on computational step size.

Dr. M. Guseva reported on the investigation of tungsten erosion using a new field ion microscopy technique that has been developed for the determination of the sputtering threshold energy and sputtering yield for ions up to 500 eV. It offers the advantages of *in situ* cleaning of the surface by evaporation in the field, polishing of the surface by atomic desorption, and extremely high resolution to observe single vacancies. She described the preparation of tungsten oxide and mixed tungsten-carbon layers and sputtering of those layers at near threshold energies. Sputtering is influenced by the chemical affinity of the substrate for oxygen. An oxide film reduces sputtering by a factor of about ten. Sputter yield energy dependencies were found that agreed well with those of other researchers. The production of vacancies in the energy range under study was only observed in the first surface layer.

Dr. E. Vietzke addressed thermal stability and some erosion phenomena in silicon carbide. Experiments measured silicon sublimation rates from SiC30 and a-C/Si:H films and found them to be temperature dependent and in a range between the rates of pure silicon and silicon carbide. After the materials were heated to 2000 K, the sublimation rates were essentially those of silicon carbide. Physical sputter yields for silicon are proportional to the fractional coverage of the surfaces by silicon as compared with impurity films. The chemical sputtering yield of silane from silicon surfaces was small for 200-eV D ions and 500°C target temperature, but it may be higher at low temperatures and low ion energies. Oxygen was also observed to induce chemical sputtering from silicon surfaces. Experiments demonstrated that carbon films build up non-uniformly on boron films. Hence, boron erosion by hydrogen can take place simultaneously with carbon deposition. Dr. Roth commented that there is no silane erosion observed when silicon carbide is present on the surface.

Dr. Yu. Martynenko was the next speaker. He described investigations of carbon composite and mixed graphite erosion. Sputter yields of carbon fiber composite material were found to be in the range of $5 \pm 3 \times 10^{-3}$ and to show little sensitivity to impacting deuterium ion energy. That suggests chemical sputtering is the dominant erosion mechanism. For titanium doped RGT graphite exposed to stationary plasma in LENTA and to a pulsed plasma beam in

MKT, the surfaces became very textured as the titanium carbide clusters generated sputter-cone-like structures. These structures were stable to thermal shocks. Removable grains of carbon were seen on surfaces and in filters. Surface structure is a main factor in determining the amount of erosion taking place, for example, during a disruption.

Dr. G. Longhurst presented recent experimental results of hydrogen interactions with carbon-coated beryllium and tungsten. Experiments to characterize films of carbon on beryllium and tungsten showed that for beryllium there was strong intermixing between the beryllium and the carbon at temperatures above 400°C, while with tungsten, the mixing only began above 800°C. Composition ratios evident in sputter Auger analyses suggested carbide formation. Retention of implanted deuterium in carbon-coated samples was much greater than in non-coated samples. Indeed, without the carbide formation, retention in the carbon films was essentially that of pure carbon. Even in tests where erosion had removed much of the carbon, deuterium retention was still dominated by the remaining carbon. Issues related to ITER safety were discussed.

Dr. A. Haasz next discussed hydrogen retention in pure tungsten, in tungsten exposed to the PISCES A plasma, in lanthanum oxide doped tungsten, and in carbon-implanted tungsten. The objectives of his work included measuring the retention of deuterium as a function of ion fluence, ion energy, and material temperature. He found that in polycrystalline tungsten foils at 300 K, the amount retained approached saturation values of about 6×10^{20} D/m^2 for high (> 10^{23} D/m^2) fluences. At higher temperatures, to 600 K, higher retention levels were observed, but they depended in rather predictable ways on ion energy, ion fluence, and implantation history. Pure single-crystal tungsten showed no such saturation effects, but the amount retained seemed to be diffusion limited. Retention in the lanthanum oxide doped material was similar to the single crystal results except at high fluences at 500 K where saturation appeared to occur. Results for the PISCES-exposed tungsten were similar to the other pure tungsten specimens at 300 K and at 500 K for high fluences. For 500 K and low fluences, retention is at least an order of magnitude lower than other pure tungsten specimens. Retention in carbon-implanted tungsten varies with the extent of carbon implantation and tends to the level seen in pure tungsten. Micrographs showing blister formation with overlayers much thicker than the implantation depth generated considerable discussion.

Dr. V. Alimov reported on oxygen and deuterium retention in the surface layers of beryllium and beryllium oxide. SIMS depth profiling was used to investigate oxygen incorporation following irradiation with 3-keV D ions in an ^{18}O enriched oxygen atmosphere in the 10^{-5} to 10^{-3} Pa range. Sample temperatures were 300 and 700 K. Two stages of oxygen accumulation were observed. At fluences less than 5×10^{17} D/cm^2, the oxygen atoms are incorporated into the oxide initially present on the metal surface and in the metal layers adjacent to the oxide. Above 10^{18} D/cm^2, interconnected porosity develops, and oxygen is found to occupy sites on the newly opened surface area. Deuterium implantated into beryllium oxide films at fluences of 2.2×10^{15} to 6.7×10^{18} D/cm^2 was found to reside as both molecules

and atoms. Concentrations were up to 0.2 D/BeO for atoms and 0.08 D/BeO as molecules. Small bubbles 0.6 to 0.7 nm in radius were seen to form at 2 x 10^{17} D/cm^2 ion fluence and are responsible for the retention of molecular deuterium. Most of the deuterium exists as atoms, probably bound to oxygen atoms in the BeO film to form $Be(OD)_2$. That inventory diminishes in time after plasma exposure has stopped.

Dr. K. Ashida was the next speaker, discussing carbide formation in C(H)Be and C(H)-Mo binary systems and associated hydrogen release. He observed that a hydrogenated carbon layer deposited on beryllium, when heated in vacuum to 400°C, released hydrogen and carbon monoxide by the reaction of the carbon with adsorbed water molecules. Above 500°C, the carbon was transformed to beryllium carbide by the movement of beryllium through the carbon. That also resulted in the hydrogen in the film being liberated to come off as molecular gas or to diffuse into the bulk of the sample. When similar experiments were conducted with hydrogenated carbon films on molybdenum, the reaction with adsorbed water was also seen at about 400°C, while the carbon reacted with the molybdenum to form carbide at about 600°C. In addition, there was a reaction at that temperature between the carbon film and molybdenum oxide, liberating hydrogen, carbon monoxide, and forming molybdenum carbide.

Dr. V. Ulanov spoke next about using the nuclear induced elastic recoil detection (NERD) method for hydrogen depth profiling. Using this method, a target sample up to tens of micrometers thick containing hydrogen isotopes is bombarded by energetic neutrons. Recoil hydrogen isotope atoms coming from the test sample are then detected using two or three Si-detectors. By analyzing the signals from the various detection stages using the well-known dE-E method, making use of techniques reminiscent of those used in nuclear reaction analysis, it is possible to obtain the energy spectra for the hydrogen isotope. Treatment of the experimental spectra by the DRIN code (Distribution of the Recoil nuclei Induced by Neutrons) allows to extract the concentration profiles for hydrogen isotopes in the test samples. Iterative convergence using various profiles leads to the one giving the best agreement with the measured signals from the detectors. DT neutrons (14.1 MeV) give for hydrogen about 60-μm resolution in a 2-mm thick carbon sample with 10^{-4} atom fraction sensitivity. DD neutrons (2.5 MeV) give 8-μm resolution in 100-μm thick targets with 10^{-5} atom fraction sensitivity.

Dr. C. Garcia-Rosales next addressed the manufacturing and characterization of mixed carbon materials. Centro de Estudios e Investigaciones Técnicas de Guipúscao (CEIT) in San Sebastian, Spain has been investigating effects of doped graphite microstructure on chemical erosion and hydrogen retention. Dopants such as boron, silicon, titanium, and tungsten are being considered to reduce both chemical erosion and hydrogen isotope retention in plasma-facing materials. Chemical erosion includes thermally activated processes, which may be enhanced by radiation effects, and surface processes. Doping with boron enhances the recombinative release of hydrogen, shifting it to lower temperatures, and reduces chemical sputtering. Silicon, titanium, and tungsten dopants produce a small shift in temperatures for

hydrogen desorption, but they result in a much smaller reduction of the sputter yield than does boron. At high fluences of low-energy hydrogen, carbon is eroded preferentially, resulting in some concentration of the dopants on the surface. Eventually, carbidic grains protect the underlying carbon from erosion. A fine grain structure of carbidic precipitates (sub micron size) or atomic dispersion is desirable for reducing erosion. Hydrogen retention can be by implantation, migration to bulk trapping sites, and the buildup of codeposited layers with carbon. Doped graphites tend to retain more hydrogen than pyrolitic graphite, possibly because of their greater porosity.

Dr. R. Doerner was the concluding speaker. He spoke on material interaction studies in the PISCES program. These investigations include the beryllium/carbon, tungsten/ carbon, and silicon-doped carbon fiber composite systems. Experiments showed that a mixed material layer (carbon) on beryllium reduces the net sputter yield. They have studied the flux-rate dependence of chemical erosion of graphite and have not seen a definite effect. They have experimentally and theoretically studied the importance of the plasma impurity species concentration to film formation on plasma-facing surfaces. Spectroscopic measurements of carbon impurity in the plasma agree quite well with residual gas analysis determinations. Temperature and plasma carbon concentration conditions for experimentally observed film growth support the theoretical concepts arrived at, and film growth rates observed using successive profilometry measurements agree quite well with simple calculations using their analytical model. XPS measurements confirm that some beryllium carbide was formed athermally during low-temperature carbon deposition.

3. REVIEW OF DATA STATUS AND NEEDS

At the last session of the meeting (chaired by Dr. Roth) analysis was undertaken of the data availability for erosion and hydrogen retention properties of mixed materials of interest to fusion energy research. The analysis included also the chemical interaction properties of the relevant mixed materials. Most of the analyzed information was based on the recent and past results obtained by the laboratories participating in the present CRP, but due attention was paid also on information generated outside the CRP group.

The result of this analysis is presented in Table 1. As seen from this table, there are many systems (all of which have relevance to fusion) for which data on the above mentioned properties are not available at all. For some systems, however, there exist a significant body of data, and the required information can be completed in a reasonably short period of time. These systems are indicated in Table 1 by an asterix and were selected as candidates on which the CRP could focus its future activities to achieve completion of the corresponding databases. This, however, does not mean that the CRP activities will be limited to these systems.

Table 1: Work done or in progress within present CRP

Base Mater. / Addition	Be	C	Si	Mo	W
D	- - Ret	- Ero -	- Ero -	- - -	- Ero Ret
Be	X	Chem * Ero -	- - -	- - -	- - -
C	Chem * Ero Ret	X	- Ero -	Chem - Ret	- * Ero Ret
O	- - Ret	- - -	- Ero -	- - Ret	- Ero -
Si	- - -	- * Ero -	X	- - -	- - -
Ti	- - -	- * Ero -	- - -	- - -	- - -
W	- - -	Chem * Ero -	- - -	- - -	X

Legend:

Chem	=	Chemical interaction studies
Ero	=	Erosion studies
Ret	=	Hydrogen retention studies
*	=	System selected for focused CRP studies

4. CONCLUSIONS AND RECOMMENDATIONS

On the basis of the analysis of data status and needs, the meeting participants concluded that the database for erosion and hydrogen retention properties of mixed materials of interest to fusion research, as well as for the chemical interaction properties of these materials, is, with a few exceptions (Be-C, C-W, and Si, Ti-doped graphites), essentially in its initial stage of development. Completion of the entire database on these properties for all fusion relevant mixed materials would require considerable resources and a large-scale effort for several years. Given the available resources and limited size and duration of the present CRP, the meeting participants took the view that concentration of the CRP efforts in the remaining period of its duration to a limited number of mixed materials would be a reasonable approach, if the CRP is to produce a specific final, deliverable result of its three year activity. The selected systems (see Table 1) for such a focused effort are those for which already a considerable body of information exists, but the corresponding databases are still far from being complete. For achieving this objective, the work co-ordination within the CRP should be significantly enhanced, including also direct collaboration between certain laboratories that participate in the CRP. The focused and co-ordinated CRP activities for the remaining period of its duration (i.e. until end of 2000), which have been agreed upon by the meeting participants, are given in Table 2. The participanting CRP institutions in these activities are also shown in the table.

While generation of accurate data for the erosion and hydrogen retention properties of selected mixed materials, as well as for the corresponding chemical interactions, remain the prime objectives of the future CRP activities, the meeting participants emphasized the need for understanding the basic mechanisms underlying the experimental observations. Therefore, the experimental efforts should be also oriented towards providing the necessary information for constructing analytical models for the considered processes (or observed behaviour). The constructed models would also need further validation by other, independent experiments. Construction of general, self-consistent and physically well founded models for erosion and hydrogen retention properties of mixed materials, which would have also a good level of predictive power, would be another important objective of the future CRP efforts.

The meeting participants observe that the study and data gneration for the other systems shown in Table 1 (those not covered in Table 2) have also significant importance for the fusion energy research (e.g. W-Be, O-W, Si-C, etc). Extension of CRP efforts to these systems is, therefore, highly recommendable. This extension would, however, require an extension of the CRP beyond its present three year duration. It is hereby recommended that the IAEA, in its programme planning for the 2001-2002 period, includes also a two year extension of the present CRP.

Table 2: Co-ordinated CRP activities for the remaining period of CRP duration

Mixed Material	Research Activity	Participating Institution (#)
W/C	Film fabrication	RAS, SRC, UCSD
	Chem. interaction and C diffusion	Toyama, IPPG, UCSD
	Hydrogen retention parameters (W, W-C depos. layers, implantation)	RAS, SRC, UzAS, UITAS, INEEL, FZJ, UCSD
	Hydrogen retention modeling	INEEL
	Erosion (physical, chemical)	UCSD, IPPG, SRC, UTIAS, FZJ
	Erosion modeling	IPPG
	High heat flux, high fluence, struct. effects	SRC
Be/C	Film fabrication	UCSD, SRC
	Chem. interaction (including oxygen)	Toyama, IPPG, UCSD
	Hydrogen retention and permeation	UTIAS, UzAS, IPPG, UCSD
	Surface structure at high fluxes, porosity	SRC, INEEL
	Erosion (physical, chemical)	UCSD, SRC, IPPG
Doped graphites (Ti, Si, ...)	Production	CEIT
	Fabrication (bulk, films)	SRC
	Microstructure analysis	CEIT
	Thermal conductivity	UTIAS
	Erosion (chemical, RES)	UTIAS, IPPG, FZJ
	Hydrogen retention	UTIAS, UzAS
	Saturation concentration	IPPG, FZJ

(#): Institution code (key person)

CEIT:	Centro de Estudios e Investigat. Techn.	(C. Garcia-Rosales)
FZJ:	Forschungszentrum Jülich	(E. Vietzke)
INEEL:	Idaho Natl. Eng. and Environment. Lab.	(G. Longhurst)
IPPG:	Max-Planck-Inst. Plasma Phys., Garching	(J. Roth)
RAS:	Russian Academy of Sciences	(V. Alimov)
SRC	Scientific Res. Centre "Kurchatov"	(M. Guseva, Yu. Martynenko)
Toyama:	Toyama University	(K. Ashida)
UzAS:	Uzbekistan Academy of Sciences	(V. Ulanov)
UCSD:	Univ. of California, San Diego	(R. Doerner)
UTIAS:	Univ. of Toronto Inst. Aerospace Studies	(A. Haasz)

Appendix 1

First 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"

19-20 October 1998, IAEA Headquarters, Vienna, Austria

List of Participants

CRP members

Dr. A. A. Haasz
University of Toronto
Institute for Aerospace Studies
4925 Dufferin Street
Toronto, Ontario, CANADA
Fax: 1-416-667-7925
aahasz@utias.utoronto.ca

Dr. J. Roth
Max-Planck-Institut für Plasmaphysik
Surface Science Division
Plasma Wall Interaction
Boltzmannstrasse - 2
Garching bei München, GERMANY
Fax: 49-89-3299-1149
jor@ipp-garching.mpg.de

Dr. E. Vietzke
Forschungszentrum Jülich GmbH
Institut für Plasmaphysik
Postfach 1913
D-52425 Jülich, GERMANY
Fax: 49-2461-612660
E.Vietzke@fz-juelich.de

D. Kan Ashida
Hydrogen Isotope Research Center
Toyama University
Gofuku 3190, Toyama 930, JAPAN
Fax: 81-764-456931
ashida@hrc.toyama-u.ac.jp

Dr. V. Kh. Alimov
Institute of Physical Chemistry
Russian Academy of Sciences
Leninsky Prospect 31
117915 Moscow, RUSSIA
Fax: 7-095-334-8531
alimov@ipcras.msk.ru

Dr. Yu.V. Martynenko
Scientifc Research Centre
"Kurchatov Institute"
Ploshchad I.V. Kurchatova
Moscow D-182, 123182, RUSSIA
Fax: 7-095-943-0073
martyn@qq.nfi.kiae.su

Dr. C. Garcia-Rosales
Centro de Estudios e Investigaciones
Tecnicas de Guipuzco
Materials Science and Technology
Po Manuel Lardizabal 15
P.O. Box 1555
E-20009 San Sebastián, SPAIN
Fax: 34-43-213076
cgrosales@ceit.es

Dr. Russ Doerner
University of California, San Diego
UCSD Fusion Program
Experimental Research Division
9500 Gilman Dr.
La Jolla, California, USA
Fax: 1-619-534-7716
rdoerner@ucsd.edu

Appendix 1 Contd.

Dr. Glen Longhurst
Lockheed Martin Idaho Technologies
2525 Freemont Avenue
P.O. Box 1625
Idaho Falls, Idaho, USA
Fax: 1-208-526-2930
gxl@inel.gov

Dr. V. Ulanov
Uzbekistan Academy of Sciences
Institute of Nuclear Physics
Laboratory of Material Research by
Nuclear Reaction Methods
702132 Ulugbek, Taskent,
UZBEKISTAN
Fax: 371-264-2590
ulanov@pc202.suninp.tashkent.su

Observers

Dr. W. Eckstein
Max-Planck-Institut für Plasmaphysik
Boltzmannstrasse - 2
Garching bei München, GERMANY
Fax: 49-89-3299-2591
wge@rzg.mpg.de

Dr. Maria Guseva
Scientifc Research Centre
"Kurchatov Institute"
Ploshchad I.V. Kurchatova
Moscow D-182, 123182, RUSSIA
Fax: 7-095-943-0073
martyn@qq.nfi.kiae.su

IAEA AMDU staff

Dr. R. K. Janev
Atomic and Molecular Data Unit
International Atomic Energy Agency
Wagramerstrasse - 5, P.O. Box 100
A-1400 Vienna, AUSTRIA
Fax: 43-1-26007
janev@ripcrs01.iaea.or.at

Dr. J. Stephens
Atomic and Molecular Data Unit
International Atomic Energy Agency
Wagramerstrasse - 5, P.O. Box 100
A-1400 Vienna, AUSTRIA
Fax: 43-1-26007
j.stephens@iaea.org

Appendix 2

First 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"

19-20 October 1998, IAEA Headquarters, Vienna, Austria

MEETING AGENDA

Monday, October 19 **Meeting Room: A-27-15**

09:30 - 10:00	- Opening - Adoption of Agenda	
Session 1:	*Erosion and Related Properties of Elemental and Mixed Materials*	
Chairman: A. Haasz		
10:00 - 10:30	J. Roth:	Experimental investigation of W irradiation with C and CH_3 at different temperatures
10:30 - 11:00	W. Eckstein:	Computer simulation of C implantation into W at different temperatures
11:00 - 11:30	*Coffee break*	
11:30 - 12:00	M. Guseva:	Investigation of tungsten erosion
12:00 - 12:30	E. Vietzke:	Si/C material: thermal stability and some erosion phenomena
12:30 - 13:00	Yu. Martynenko:	Investigation of C-C composite and mixed graphite erosion
13:00 - 14:30	*Lunch*	
Session 2:	*Particle Retention and Release, and Related Processes*	
Chairman: E. Vietzke		
14:30 - 15:00	G. Longhurst:	Hydrogen interaction with carbon coated Be and W
15:00 - 15:30	A. Haasz:	Hydrogen retention in C-doped tungsten

15:30 - 16:00 *Coffee break*

16:00 - 16:30 V. Alimov: Deuterium and oxygen retention in the surface layers of Be and BeO, irradiated with D-ions

16:30 - 17:00 K. Ashida: Hydrogen release from and carbide formation in C(H)-Be and C(H)-Mo binary systems

Tuesday, October 20 **Meeting Room: A-27-15**

Session 3: *Other Processes and Research Methods/Techniques*

Chairman: Yu. Martynenko

09:30 - 10:00 V. Ulanov: Simulation and experimental investigation of depth profile of hydrogen isotopes by the NERD method in simple and mixed materials

10:00 - 10:30 C. Garcia-Rosales: Manufacturing and characterization of mixed carbon materials

10:30 - 11:00 *Coffee break*

11:00 - 11:30 R. Doerner: Results and plans for material interactions studies in the PISCES program

11:30 - 12:00 Discussion of the first year accomplished results by the CRP

12:00 - 14:00 *Lunch*

Session 4: *Specific CRP objectives and methods of work co-ordination*

Chairman: J. Roth

14:00 - 17:00
- Setting more specific, achievable CRP objectives with a view on fusion needs and delivering a final product in 3 years time;
- Work co-ordination and task distribution

17:00 - *Adjournment of the Meeting*