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EURATOM-MHEST

Slovenska Fuzijska Asociacija
Slovenian Fusion Association

Association EURATOM – MHEST

ANNUAL REPORT 2006

Ljubljana, November 2007

Slovenska Fuzijska Asociacija
Slovenian Fusion Association

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The Annual Report 2006 of the Association EURATOM-MHEST
covers the period 1 January to 31 December 2006

Compiled by Milan Čerček and Bojan Žefran

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Association EURATOM-MHEST

Institute Jožef Stefan
Jamova 39
SI-1000 Ljubljana
Tel. +386 1 5885 450
http://www.sfa-fuzija.si

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Slovenian fusion association EURATOM-MHEST

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1. INTRODUCTION

This report presents the research and development results for 2006, the second year of the Slovenian Fusion Association. The SFA was established in 2005 during the signing of the Contract of Association between EURATOM and the Ministry of Higher Education, Science and Technology. In 2006, several new tasks were included in the work programme of the Research Unit.

At the Faculty of Mechanical Engineering of the University of Ljubljana, a group of researchers who have been collaborating in a cost-sharing action on edge-plasma modelling since 2004, decided to continue their work in the field of tokamak modelling, focusing on the development of core-edge coupling procedures. This task is well integrated into the work programme of the EFDA Integrated Tokamak Modelling Task Force. At the Jožef Stefan Institute, experience in the field of vacuum science and technology enabled researchers in the Department for Surface Engineering and Optoelectronics to investigate hydrogen retention in fusion-relevant materials. This task is integrated into the work programme of the EFDA Plasma Wall Interaction Task Force. The volume of work in this important field of the European fusion programme is already quite substantial and the SFA was happy to organize the 5th General EFDA Plasma Wall Interaction Task Force meeting, which was held at the Nuclear Training Centre of the Jožef Stefan Institute on 13–15 November 2006. The meeting was a success and helped us to consolidate our work programme and focus on the objectives of the Task Force programme.

Another important milestone in the development of the SFA is the start of a collaboration with JET. Together with colleagues from the UKAEA and the Romanian Association, who run the task, we will work on upgrading the gamma-ray spectrometry systems. The objective of this particular project is to estimate the influence of the proposed neutron attenuators in the spectrometer on the neutron fields.

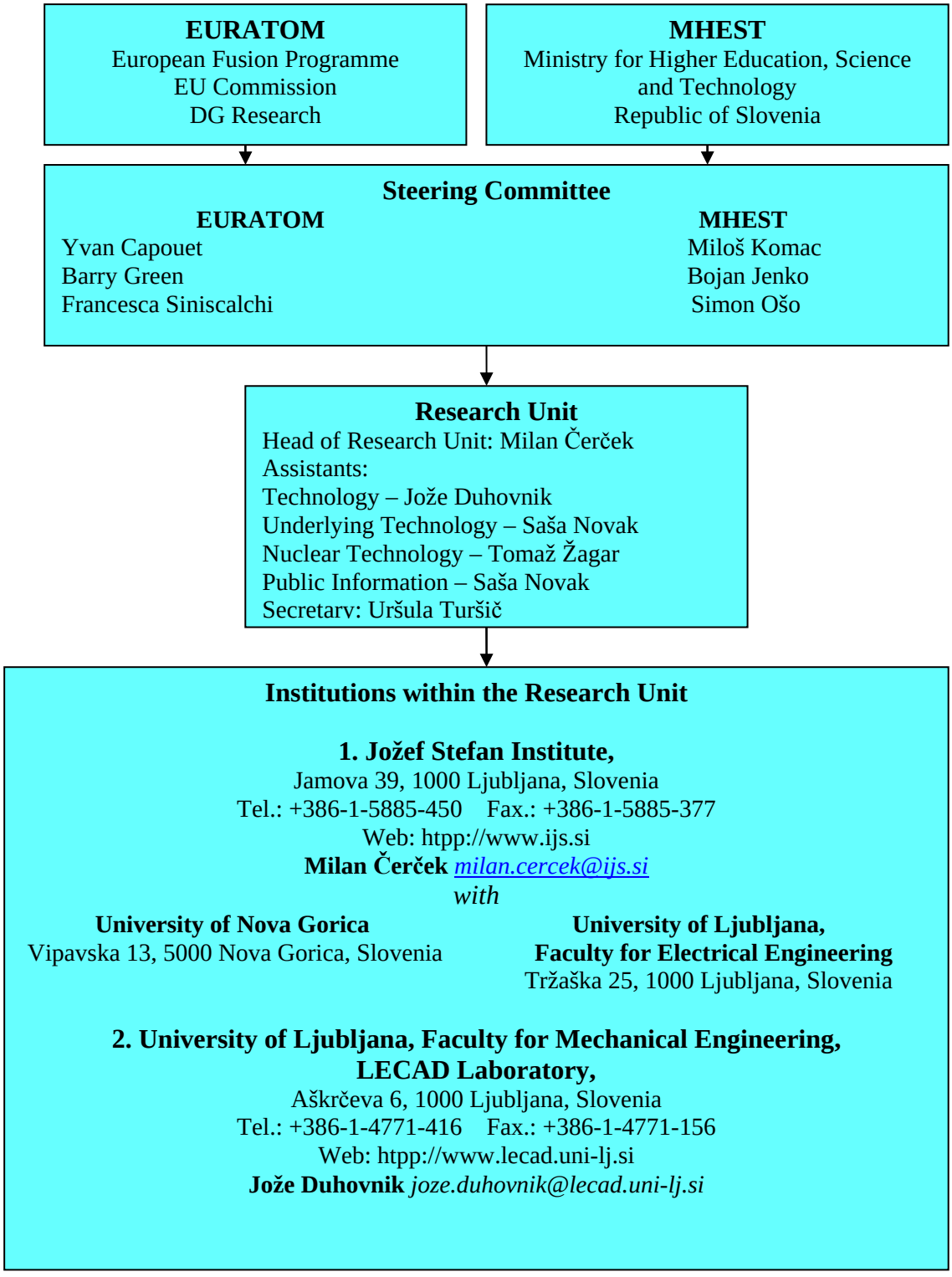
In 2006 the fusion community witnessed a further important step in the realization of our vision to harness fusion as a sustainable energy source for mankind. Representatives of the ITER parties – the European Union, the Russian Federation, Japan, the Republic of Korea, China, India and the United States of America – met in November in Paris to sign the international agreement to build ITER.

The researchers in the SFA are strongly committed to contributing to this endeavour. Up until now their efforts have been generously supported from various sides: by the Ministry of Higher Education, Science and Technology, as the Associate; by the National Research Agency, as the financing body; by the European Commission; and by their home institutions. We strongly believe that this support will continue in future.

Milan Čerček, Head of Research Unit

1.1. General Information

Association EURATOM-MHEST Management structure



Slovenian representatives in the European commmittees relevant to fusion research and development

Consultative Committee for the EURATOM Specific Programme on Nuclear Energy Research – Fusion (CCE-FU)
Miloš Komac Ministry for Higher Education, Science and Technology of Republic of Slovenia

Milan Čerček Head of Research Unit, Jožef Stefan Institute, Ljubljana

EFDA Steering Committee
Jože Duhovnik University of Ljubljana

EFDA Scientific and Technical Advisory Committee (STAC)
Milan Čerček Head of Research Unit, Jožef Stefan Institute, Ljubljana

EFDA Administrative and Financial Advisory Committee (AFAC)
Darko Korbar Jožef Stefan Institute, Ljubljana

Public Information Group
Saša Novak Jožef Stefan Institute, Ljubljana

1.2. Financial Information

Table 1.1 Expenditure for 2006

	Expenditure (Euro)
General Support (20% EU contribution)	**1,253,115**
Physics	760,654
Underlying Technology	430,083
JET	62,378
EFDA Technology	**256,027**
EFDA Art. 5.1a (20% EU contribution)	39,945
EFDA Art. 5.1b (40% EU contribution)	216,081
Mobility (100% EU contribution)	**17,306**
TOTAL	**1,526,448**

1.3. Statistics

Table 1.2 R&D projects in the Association EURATOM-MHEST for 2006

	JSI	ULJ	
Physics	5	1	**6**
Underlying Technology	2	-	**2**
JET	1	-	**1**
Technology	1	1	**2**
TOTAL	9	2	**11**

Table 1.3 Manpower for 2006

	Professional	Non professional	TOTAL
JSI	26	12	38
ULJ	4	0	4
	30	**12**	**42**

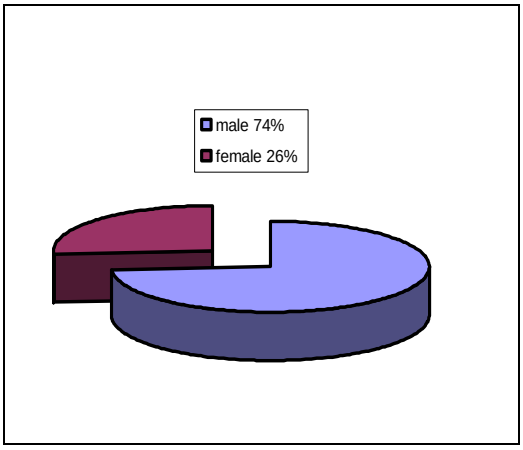


Figure 1. Association EURATOM-MHEST staff in 2006 by gender

INTEGRATED TOKAMAK MODELLING WITH EXTERNALLY COUPLED CORE AND EDGE TRANSPORT CODES

Mladen Stanojević, Tomaž Kolšek, Marjan Korošec, Janez Krek, Ivan Demšar, Nikola Vukašinović and Tanja Mavrič

LECAD Laboratory, Faculty of Mechanical Engineering, University of Ljubljana, Aškerčeva Str. 6, SI-1000 Ljubljana, Slovenia
e-mail: mladen.stanojevic@lecad.uni-lj.si

1 INTRODUCTION

The P1 project started in October 2006. It is closely related to the EFDA's Integrated Tokamak Modelling Task Force (ITM-TF) Integrated Modelling Project 3 (IMP3): Transport Code and Discharge Evolution, in particular to its topic 3E: Interfaces to boundaries. The P1 project is focused on the core-edge coupling and includes the following activities:

1. review of the "state-of-the-art" in the integrated tokamak modeling with the existing fluid transport codes (in 2006 - 2007);
2. analysis of the physical models used in the existing core and SOL transport codes, which are main candidates for the coupling, with the objective to identify and formulate possible necessary changes and improvements to be implemented in these codes in order to achieve more efficient coupling (in 2006 - 2007);
3. analysis of the software architecture and numerical methods used in the codes mentioned above with the objective to identify and formulate possible necessary changes or improvements to be implemented in these codes in order to achieve more efficient coupling (in 2007);
4. implementation and testing of the changes or improvements identified and formulated in the activities 2. and 3. (in 2007);
5. specification and prototyping of the interfaces for the core-edge coupling (in 2007 - 2008);
6. development of either the interface module for at least one example of the core-edge coupling with the existing codes or the standardized interface module for all core and edge transport codes considered in the project according to the recommendations of the ITM-TF (in 2008);
7. testing, verification and validation of the modules produced (in 2008 - 2009);
8. documenting the work and results in the activities 1. - 7. (in 2006 - 2009).

In 2006, the activity 1. was started. The work performed in 2006 is briefly presented in the next section.

2 WORK PERFORMED IN 2006

In 2006, the structure of the COCONUT code package was analyzed. COCONUT stands for COmbined COdes Numerical Utility for Tokamaks [1, 2]. Its main components are self-consistently coupled 1.5D core transport code JETTO [3, 4] and the 2D edge (or scrape-off-layer, SOL) transport code EDGE2D/NIMBUS [5, 6]. In a COCONUT run, JETTO calculates the plasma density, electron and ion temperatures, heat fluxes and transport coeffcients in the core and passes the values of the particle and heat fluxes at the separatrix as boundary conditions for EDGE2D, whereas EDGE2D calculates the corresponding quantities in the SOL and passes the density and electron and ion temperatures at the separatrix as boundary conditions for JETTO. The boundary between the 1D JETTO computational mesh and 2D EDGE2D computational mesh is at the separatrix and both codes are called at each time step. The interaction of the plasma with the wall and the distribution of particle and energy sources due to neutrals is optionally calculated by the Monte-Carlo code NIMBUS. The transport of the inpurities in the core can be computed by transport code SANCO, which uses prescribed radial profiles of diffusion coefficients and convective velocities. Thus if all codes within COCONUT are used in the simulation, each one will be coupled with three other codes. However, NIMBUS exchanges data in both directions only with EDGE2D, whereas in the couplings with JETTO and SANCO it only prescribes the neutral particle fluxes at the separatrix, which are used in the boundary conditions of these two codes.

The coupling of these codes is based on a splitting scheme adapted from the method of fractional steps. One code uses a Dirichlet boundary condition (e.g., JETTO), whereas the other is run with a flux boundary condition (e.g., EDGE2D). The boundary values for the variables and their fluxes are supplied by the respective other code at each time step, thereby imposing the continuity of densities and temperatures and of the corresponding particle and heat fluxes. The boundary for JETTO is the separatrix itself, whereas the EDGE2D boundary is generally placed a few millimiters inside the separatrix. If any poloidal variations a little further inside are to be taken into account, the location of the boundary should be modified and care should be taken to avoid double counting of radiation losses.

In the couplings, the following numerical scheme is used:

$$n_{i,C}^{\mathrm{n}+1/2} = n_{i,E}^{\mathrm{n}}, \quad T_{e,C}^{\mathrm{n}+1/2} = T_{e,E}^{\mathrm{n}}, \quad T_{i,C}^{\mathrm{n}+1/2} = T_{i,E}^{\mathrm{n}}, \qquad \text{for JETTO,}$$

$$\Gamma_{z,C}^{\mathrm{n}+1/2} = \Gamma_{z,E}^{\mathrm{n}}, \qquad \text{for SANCO,}$$

$$\Gamma_{i,E}^{\mathrm{n}+1/2} = \Gamma_{i,C}^{\mathrm{n}}, \quad P_{e,E}^{\mathrm{n}+1} = P_{e,C}^{\mathrm{n}+1/2}, \quad P_{i,E}^{\mathrm{n}+1} = P_{i,C}^{\mathrm{n}+1/2}, \quad n_{z,E}^{\mathrm{n}+1} = n_{z,C}^{\mathrm{n}+1/2}, \qquad \text{for EDGE2D,}$$

where n_i and n_z are the main positive ion component and impurity density respectivly, T_e and T_i are the electron and ion temperature respectively, Γ_i and Γ_z are the main positive ion component and impurity particle flux respectively, P_e and P_i are electron and ion energy or heat flux respectively, n is the time step, and *C* and *E* refer to the core and edge codes respectively. Poloidal averages over the innermost layer of computational cells are used for the quantities that are passed from EDGE2D to the core codes. The fluxes received by EDGE2D are distributed uniformely in the poloidal direction. While the natural flow direction from the core to the edge is used for the main ion component particle fluxes and heat fluxes, for the impurity fluxes it was found

advantageous to pass the flux from the edge to the core. The neutral fluxes to the core and the energy of these neutrals are provided by NIMBUS.

The impurity code SANCO is called after the completion of every JETTO time-step and receives electron and main ion component densities, electron and ion temperatures computed by JETTO. If fusion reactions are taken into account, JETTO can also pass the alpha particle source to SANCO. The transport of the impurities is evolved for the same time interval but allowing smaller time steps. On completion, SANCO returns the impurity densities of all charge states and the radiated power due to impurity line radiation. The coupling of SANCO to all codes supports taking into account neutrals and single- or multi-positive ions of several impurity species.

An important element is the flexibility of the COCONUT code package. Depending on the physics situation under investigation, the most suitable subset of codes can be selected. For example, impurities can be modelled in the SOL but not in the core or the impurities can be modelled everywhere but with a frozen background plasma. The freezing can be limited to the core or include the SOL.

The possibility to adapt COCONUT to the relevant time-scales is essential to avoid big overheads in computing time. The CPU time required is dominated by the computations of the SOL code. In the fully coupled mode when core and SOL are updated in each time-step, the simulation is therefore limited to processes on the SOL time-scale. In order to model slow core transport processes, COCONUT can be run in a partially coupled mode, in which the core is run stand-alone for extended decoupled phases with relatively short fully coupled phases to update the edge. Thus using the partially coupled mode it is possible to perform integrated tokamak simulations. However, when running COCONUT it has been proven essential to use the individual codes first to analyze the core and SOL as well as main ions and impurities separately. This serves to identify appropriate transport coefficients and to prepare initial states for each code before they run in a coupled mode. Therefore the accuracy and speed of the integrated tokamak simulations with COCONUT are very sensitive to these parameters.

According to the private communication with R. Zagórski from the Institute of Plasma Physics and Laser Microfusion, Warsaw, Poland, who is working on a similar project, COCONUT-like approach is also used in the coupled codes RITM-TECXY and COREDIV [7], the latter including 1D core transport code describing the radial transport and 2D multifluid code EPIT describing the plasma transport in the SOL.

3 CONCLUSIONS AND OUTLOOK FOR 2007

The analysis of the COCONUT code package has given a valuable information for developing new and more advanced core-SOL coupling schemes and interfaces. However, due to limited portability and accessibilty of the COCONUT code package, which is located at JET computer system, we have decided to continue our work on this project with the core transport code ASTRA [8] and SOL transport code SOLPS-B2.5 [9]. In 2007, the latest standard or master versions of these codes will be installed at LECAD Laboratory computer system and modified according to the needs and requirements of the project. Regarding the coupling of the core and SOL codes at a specific location, which is usually the separatrix, we have identified the following physical and computational problems to be investigated and solved in the continuation of the project:

- since the exact location of the separatrix may be unknown or may change during the tokamak operation, coupling core and SOL transport codes at a prescribed location of the separatrix may give unrealistic results for the plasma parameters and related fluxes near the separatrix;
- it is well-known from the experiments and theory that the plasma transport in the core edge or pedestal region has a crucial role in tokamak plasma confinement (especially in the H-mode), so that the coupling at the separatrix or at a location within the core, which includes poloidal averaging of the plasma parameters and related fluxes or simplification of the transport models in that region, may give very unrealistic results;
- although the computational mesh of the SOL transport codes can be extended deeply into the core, they do not include appropriate transport coefficients and various source terms (e.g., due to plasma heating) appropriate for the core region, therefore modifications and improvements of the SOL codes in that sense will be necessary in order to obtain more realistic results of the coupled core and SOL plasma simulations, even if only few layers of the SOL code computational cells within the core are used in the simulation.

In addition, we intend to supplement the latest version of the SOLPS-B2.5 code with the boundary conditions (or boundary source terms) developed in our previous research project on the boundary conditions for fusion plasmas (Cost-Sharing Action: Contract No. FU06-CT-2003-00321).

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INTERACTION OF VIBRATIONALLY EXCITED HYDROGEN WITH FUSION RELEVANT MATERIALS

Iztok Čadež, Milan Čerček, Zvone Grabnar, Sabina Markelj, Primož Pelicon, Dušan Rudman, Zdravko Rupnik
"Jožef Stefan" Institute, Ljubljana
e-mail: iztok.cadez@ijs.si

Tomaž Gyergyek
Faculty for Electrical Engineering, University of Ljubljana

Vida Žigman
University of Nova Gorica

1 INTRODUCTION

The main goal of this project is to providing quantitative data for processes involving vibrationally excited hydrogen molecules that are needed for modelling fusion edge plasma and plasma-wall interaction. We also search for specific phenomena with such molecules that might have high cross sections. Studies are performed with H_2 and D_2 in order to understand isotope effect and especially as deuterium is the main constituent of tokamak plasma. In the following we will use abbreviation VEHM for any isotopologue of vibrationally excited hydrogen molecule, e.g. $H_2(v)$, HD(v), $D_2(v)$.

In particular we are interested in the following processes at surfaces of fusion relevant materials:

- Vibrational distribution of molecules released from surfaces by thermal desorption and recombinative desorption for different surface temperatures and composition.
- Ratio of atomic to molecular species released from a surface and its variation with surface parameters.
- Interaction of vibrationally excited molecules with plasma-facing materials:
  - Change of vibrational distribution caused by interaction with the surfaces.
  - Transfer of vibrational energy to the wall and its effects on erosion yields.
  - Wall sticking probability for the excited molecules.

Due to importance of volume reactions in divertor plasma we are also interested in:

- Binary collisions of vibrationally excited hydrogen molecules with other atomic particles.

For studying processes with VEHM we are using experimental set-up (DTVE-B) for vibrational spectroscopy of hydrogen molecules that was developed in our

laboratory during recent years. It incorporates an electron gun and an extraction system for light ions (H^- and D^-) created by dissociative electron attachment. Weak homogeneous magnetic field is used for guiding low-energy incident e-beam and also, together with specially designed penetrating electrostatic field, to separating and guiding extracted ions to ion detector (channeltron).

Measuring method for vibrational spectroscopy of hydrogen molecules is based on the properties of dissociative electron attachment (DEA) in AB:

$$AB(X\ ^1\Sigma_g^+, v) + e \rightarrow AB_2^-(X\ ^2\Sigma_u^+) \rightarrow A + B^-,$$

where A and B stands for any of hydrogen isotopes – in our case H or D. This, lowest energy DEA in hydrogen occurs at 4 eV incident electron energy for molecules in the ground vibrational state. Main properties of this process that make it suitable for vibrational spectroscopy are:

- Very strong rise of cross section (CS) with vibrational and rotational excitation of target molecule (reaching 10^{-15} cm^2 range for high v!),
- CS is peaked at the threshold exhibiting vertical onset,
- DEA threshold for excited molecule is displaced towards lower energy by internal excitation energy,
- Pronounced isotope effect for low v.

This method for hydrogen vibrational spectroscopy based on DEA was first developed at Université Pierre et Marie Curie, Paris in late 80ties.

Vibrational distribution of target molecules is obtained from experimental spectra (H^- or D^- ion yield vs. electron energy) by deconvolution procedure taking into account apparatus function and theoretical CSs for DEA. Present method does not allow determination of individual low rotational state populations due to the limited energy resolution of electron beam. Only a mean rotational distribution characterised by rotational temperature can be estimated as it determines the width of individual vibrational peak.

We are studying vibrational distribution of molecules created by surface recombination of atoms, thermal dissociation and importance of VEHM for the processes in plasma.

During recent years we also developed and tested procedures for quantitative depth profiling of H and D in materials. For this we use ion beam analytical (IBA) method ERDA (Elastic Recoil Detection Analysis) that is set at 2MV tandem accelerator in our laboratory. Depth distribution of H and D is determined by analysis of energy spectra of ejected particles after studied sample is irradiated by high energy $^7Li^{2+}$ ion beam. By using ERDA we are performing in situ and in real time studies of interaction of neutral hydrogen atoms and molecules (H, D, H_2 and D_2) with fusion relevant materials. For these studies we are employing specially designed hydrogen exposure cell (HEC).

2 WORK PERFORMED IN 2006

Having main experimental procedures developed during previous years we concentrated our efforts in 2006 on some particular studies.

2.1 Hydrogen interaction with surfaces

2.1.1 *Study of production of VEHM by surface recombination*

DTVE-B has been used for the studies of vibrational excitation of molecules produced by surface processes [1], [2], [3], [4].

Study of vibrational excitation of H_2 and D_2 molecules from the test source used previously were performed for different sample materials. A test source (TS) is shown in figure 1 as mounted on the top of detection block of DTVE-B. It is made of copper and has cylindrical shape. Water or air flow through the top part of TS cools the source. Hydrogen is introduced into the TS where it is partially dissociated by the resistively heated tungsten filament. A sample disk made of studied material was mounted on the cold finger that is facing the exit orifice directed towards the interaction region of DTVE-B. The following processes are occurring in the source: molecule dissociation at the hot filament, atom recombination at different inner wall surfaces of the cell, elastic and inelastic collisions of molecules with the wall, volume collisions among the present neutral particles. Main process of interest for us is the production of VEHMs by atom recombination on the sample surface that is mounted in front of the exit orifice. Molecules formed by the atom recombination on the sample disc together with unrecombined atoms and other molecules leave the source through the exit orifice to the interaction region of DTVE-B where they are intercepted by the probing e-beam and later pumped out from the vacuum chamber by a turbo pump.

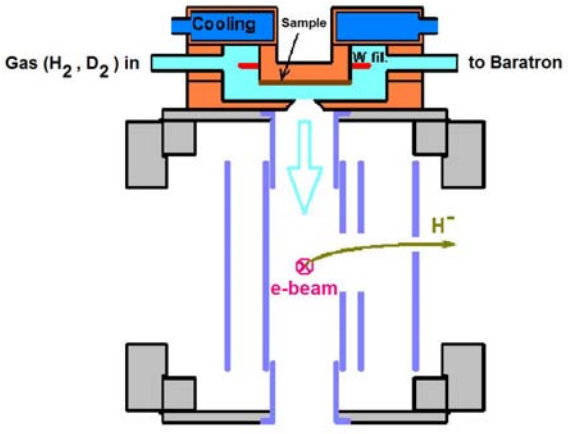

Figure 1. Test source for production of vibrationally excited molecules used in the present study.

Different processes mentioned above contribute to the observed experimental vibrational distributions. In order to evaluate importance of particular one and also to extract quantitative data on this one needs self consistent modelling

Measurements were performed with Ta, Cu, W, and C (graphite surface). Measurement was performed also with a teflon disk where no recombination should occur in order to get information on characteristics of the TS and contribution of internal surfaces to the observed signal.

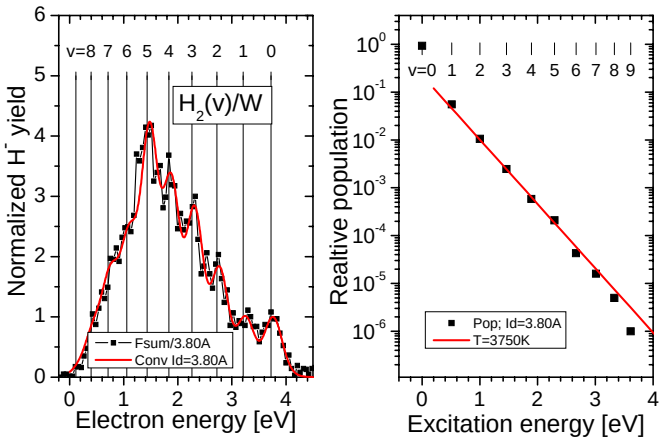

Figure 2. Spectrum of $H^-/H_2(v)$ yield for dissociation filament temperature about 1550K (left) and corresponding relative vibrational population distribution (right) for W sample plate in TS. Positions of individual v-state on energy scale are indicated on both figures.

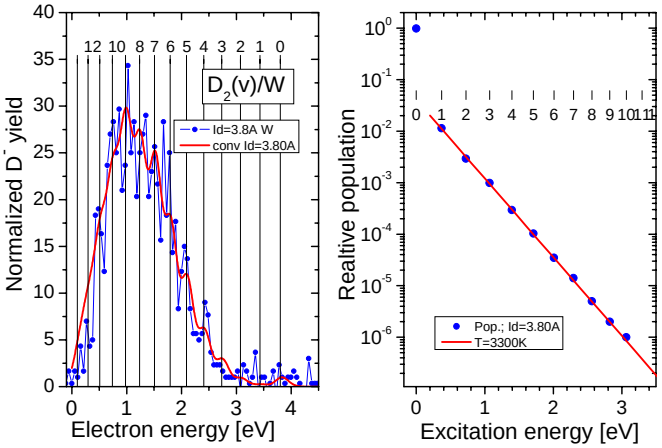

Figure 3. Same as in figure 2 but for the case of D^-/D_2 (v).

Result of measurements with W sample plate mounted in TS is shown in figures 2 and 3 for H_2 and D_2, respectively as an example of measurements.

For the deconvolution $H^-/H_2(v)$ and $D^-/D_2(v)$ spectra the most recent theoretical cross sections of Horáček et al. were used. Dr. P. Kolorenč and Prof. J. Horáček from Charles University in Prague, Czech Republic kindly performed additional CS calculations for more ro-vibrational states which we needed for deconvolution of spectra in deuterium.

2.1.2 *Study of hydrogen recombination after permeation through the membrane*

We have continued study of vibrational state distribution of molecules created by recombination of atoms after permeation through a membrane. Spectra of H^-/H_2 did not indicate production of vibrationally excited states by recombination on the vacuum side of a Pd membrane. Study with Ta membrane, that is our main goal was not successful and only sporadic H_2 signal was observed at highest (about 300°C) temperatures even with 2 bar overpressure. Permeation through both these membranes was studied by ERDA as well (see bellow) and it was shown that contamination of the high-pressure side of Ta membrane was the reason for absence of permeation. New set-up with Ta membrane that was produced with special precaution in order to avoid contamination also did not work as expected so that further efforts are needed.

2.1.3 *In-situ studies of hydrogen interaction with tungsten by ERDA*

Extensive measurements were performed on experiment with sample material exposure to the controlled neutral hydrogen atmosphere [5], [6]. For these studies we are employing special hydrogen exposure cell (HEC) shown schematically in figure 4. Processes on the surface and below the surface are followed by H and D depth profiling using ion beam analytical technique ERDA. HEC was modified with respect to the previous version in that sense that sample was not on the line of sight of the dissociation filament as it was the case previously.

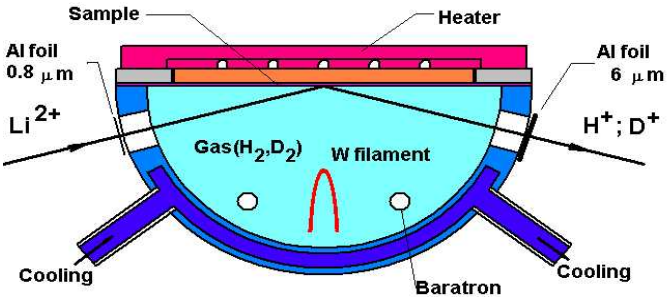

Figure 4: Hydrogen exposure cell used for real time studies of hydrogen interaction with material. A 4.2 MeV $^7Li^{2+}$ probing beam is used for hydrogen depth profiling.

Measurements were performed with tungsten sample. Due to low hydrogen solubility in W most of hydrogen remains adsorbed on the surface so that surface reactions could be clearly followed. Control of the exposure conditions in the HEC are performed by variation of gas pressure or composition (H_2 or D_2 in present case) variation of tungsten dissociation filament temperature and sample and wall temperatures. Tungsten filament is used similarly as in the case of the TS for VEHM, to dissociate molecules that are present in the cell. By such arrangement processes as chemisorption, isotope exchange and thermal desorption can be studied in real time.

Three ERDA spectra are shown in the figure. The black one is obtained before sample was exposed to deuterium and only pre-existing hydrogen is observed mainly at the surface (peak at around channel 85). The blue spectrum is obtained after sample was

exposed to D_2 and low intensity peak appears at around channel 170 that is due to the chemisorbed deuterium at the surface. Finally, when the tungsten dissociation filament is turned on and D atoms are present in the cell strong adsorption of D occurs.

An example of time variation of H and D surface concentration when exposure conditions are varied is shown in figure 6. Integral surface D and H intensity are obtained as individual peak integrals from consecutive spectra as those shown in figure 5. Integral signal is shown at the top part of the figure while gas pressure, sample temperature and dissociation filament current are shown at middle and lower part.

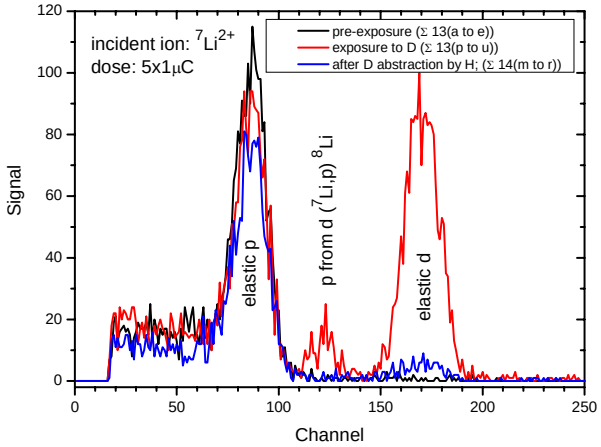

Figure 5. ERDA spectra before (black), and after W-sample exposure to D_2 (blue) and D (red).

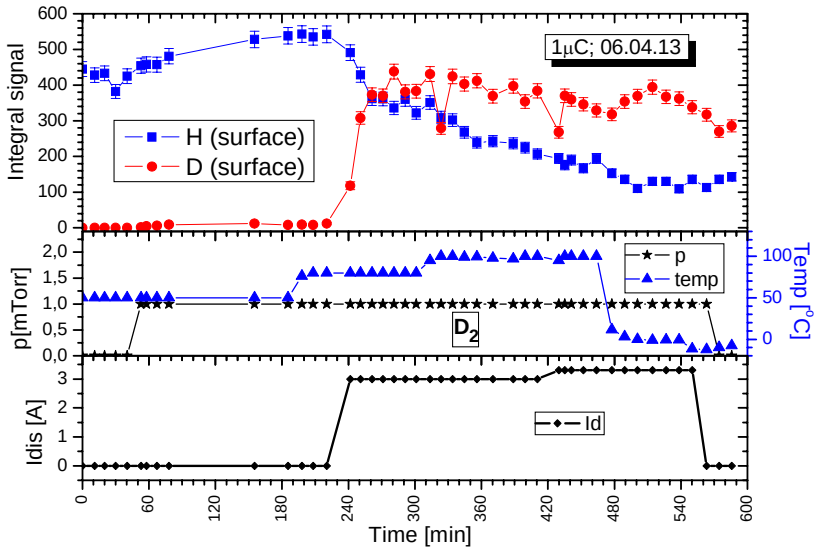

Figure 6. Temporal evolution of D and H surface concentration obtained by plotting integral signal of corresponding peaks for a series of successive spectra.

Only small adsorption of D can be observed when the sample is exposed to D_2 but as soon as dissociation filament is turned on one can observe strong rise of D and simultaneous decrease of H – isotope exchange occurs. Change of surface concentrations is also observed later, when stationary exposure conditions were present but sample temperature was decreased. Present experimental facility will allow detailed

studies of such processes to be performed. Our long term goal is to elucidate the role of VEHM that are present in HEC on the studied surface processes.

2.2 Test atmospheres with vibrationally excited hydrogen molecules

2.2.1 *A pilot study of molecular presence in H-atom source based on hot tungsten capillary*

Preliminary measurements of vibrational excitation of hydrogen molecules issuing from H-atom source have been performed. These measurements were done in collaboration with Dr. Thomas Schwarz-Selinger from IPP, Garching. H-atom source developed and currently in use at IPP, Garching was brought to JSI and attached to the vibrational spectrometer DTVE-B at Microanalitical center, JSI. H-atom source uses hot tungsten capillary where hydrogen is dissociated while flowing through it. It was observed that vibrationally excited molecules are present when the source operates. More work is needed to elucidate the influence of background signal to these measurements what is due to the geometry and pumping conditions of DTVE-B.

2.2.2 *Modelling the elementary processes in the test-cell hydrogen atmosphere with $H_2(v)$*

We have further developed the modelling of the hydrogen-hydrogen and hydrogen-wall interaction occurring in the hydrogen cylindrical test-cell source.

By Monte Carlo simulation the number of molecule-molecule collisions in the bulk and molecule-wall collisions, i.e. the total number of collisions before cell exit have been estimated. To gain statistical significance, the procedure was iterated in 1000 runs at each pressure value considered, covering the range from 2 to 40 mTorr.

According to the plan for the year 2006, we have extended the preliminary model relying on elastic collisions only, along the following issues:

1. Molecule-molecule interaction: diffusive particle-wall scattering and variable free-path for bulk collisions have been introduced.
2. Implementation of the hot filament/surface in the cell-model in order to allow for the thermal dissociation and vibrational excitation of H_2 molecules, as well as adsorbtion and desorption processes.
3. Adjusting the model to other cell geometries, like the ‘half-cylinder’ hydrogen **e**xposure **c**ell (HEC) that have been used or are foreseen in future experiments.

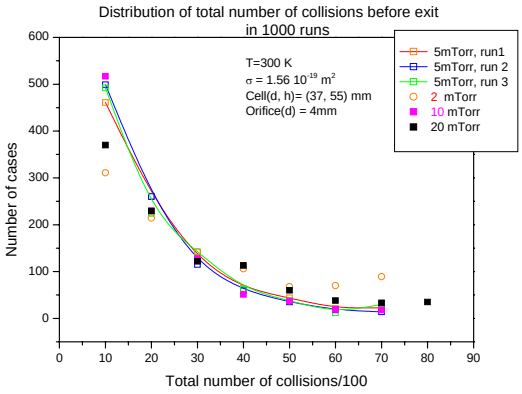

Figure 7. Total number of collisions before cell exit at different pressures. Elastic H_2 –H_2 scattering and elastic H_2-wall collisions have been assumed. Monte Carlo simulation of the particle life-history from entrance to exit in 1000 runs. The curves indicate the results of three repeated independent cycles, at 5mTorr.

Preliminary results were presented at a conference [7].

2.3 Hydrogen plasma

In the year 2006 we have measured plasma parameters of the discharge hydrogen plasma in linear magnetized plasma device (LMPM) at JSI. Measurements were performed mostly by Langmuir probes. We have learned from these measurements that hydrogen plasma can be created in somewhat narrower range of discharge parameters (discharge voltage, discharge current and pressure) when compared to argon that was mostly used before. Some tests were performed on LMPM also in order to get information on its performance as a high intensity electron beam source that will allow specific spectroscopic experiments. Electron distribution profile was measured by Langmuir probe and integral current vs. acceleration voltage (i.e. electron energy) was determined with ionizing filaments on and gas feed off.

Preliminary emission spectroscopy measurements of hydrogen plasma in LMPM were also performed. The main objective of planned experiments is to study the influence of vibrationally excited molecules created by wall recombination on the emission spectrum from plasma. These experiments are performed in collaboration with Dr. S. Brezinsek from FZ Jülich.

In figure 8 the Langmuir probe characteristic signal (left) is shown together with an emission spectrum (right) obtained during these measurements. Observed spectrum is characteristic of rather low ro-vibrational excitation of molecules in plasma. Due to the low acceptance angle of optical system that was in turn defined by physical dimensions of the viewing port available at LMPM, the signal was rather weak. That is planned to be improved in the following experiments.

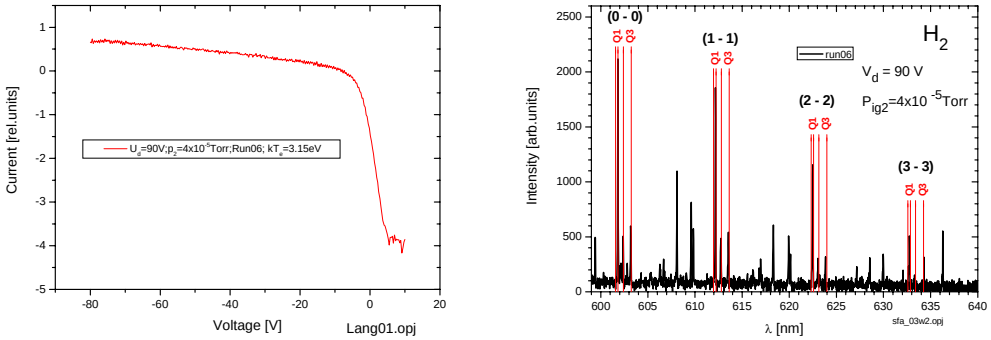

Figure 8. Langmuir probe characteristics (left and emission spectrum in the range of Fulcher band (right) observed for plasma discharge voltage of 90 V. Only ΔJ=0 (Q) lines of vibrationally diagonal transitions between d and a triplet electronic states are indicated.

We have continued with computer simulations and theoretical modelling of potential formation in front of a negative electrode immersed in hydrogen plasma. This subject is mostly related to plasma diagnostic using Langmuir and emissive probes. In this area we have developed a one-dimensional fluid model of the sheath formation in front of a negatively biased electron emitting electrode that is immersed in plasma containing a hot maxwellian electron population. The emitted electrons are modelled as monoenergetic, but the initial velocity is allowed to be larger than zero. The singly charged positive ions are considered as monoenergetic and at rest at large distance from the electrode. If the temperature of the hot electrons is high enough the electron emission goes from temperature limited regime into space charge limited regime and back as the electrode bias is varied. Because of that the current-voltage characteristics may exhibit even 3 different floating potentials [8], [9]. Very similar results with the fluid model were obtained – namely that in some cases the electron emitting electrode may have up to 3 floating potentials.

Using a computer PIC simulation we have analyzed potential formation in front of a floating electrode containing negative ions and two electron temperature velocity distribution [10]. Multiple double layers predicted by the model were observed also in PIC simulation. With basically the same method plasma containing two species of positive ions [11] was also analyzed. It was found that the pre-sheath potential drop is independent on the density ratio of both ion species suggesting that each ion population satisfies it own Bohm criterion at the sheath edge. The dependence of the floating potential on the density ratio of both ion species could be used for the diagnostic purposes. No special potential structures (like double layers) were found. We have also analyzed current-voltage characteristics of an electron emitting electrode immersed in a two-electron temperature plasma using a fully kinetic one dimensional model of a bounded plasma system [12], [13]. Very similar results with the fluid model were obtained – namely that in some cases the electron emitting electrode may have up to 3 floating potentials.

We are also starting with the analysis of the sheath formation in the presence of an oblique magnetic field. Here we are trying to reach the present state of the art.

2.4 Hydrogen depth profiling

An effort was done to put D depth profiling by ERDA on absolute scale by measurements of standard samples. For this purpose samples of deuterated amorphous carbon (a-C:D) produced by plasma vapour deposition at the Institut für Plasmaphysik Forshungszentrum, Jülich (Dr. H. G. Esser) were studied. If absolute deuterium content in the sample is determined by TDS then elastic cross sections needed for our Li ERDA measurements can be determined. Until now we analysed ERDA spectra using SIMNRA software and assuming Rutherfords cross sections for elastic recoil of D by Li ions in order to obtain an initial estimate for absolute concentration. Another possible method of calibration could be by using cross section for nuclear reaction d(^{7}Li,p)^{8}Li that we observe in measurements on W (peak at channel 120 in figure 5).

In order to complement the study of vibrational excitation of molecules after permeation we have performed H and D depth profiling in Pd [14], [15] and Ta membranes by consecutive ERDA spectra recording. This study gave us a clear time evolution of permeation dynamics in Pd (e.g. figure 9) and also helped us identifying the problem with Ta membrane.

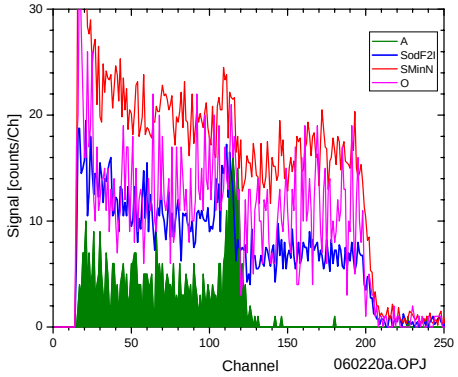

Figure 9. Few initial consecutive ERDA spectra from the moment when membrane was exposed to deuterium. Build-up of deuterium is visible but once the temperature becomes high enough and fast permeation starts the concentration of both D and H drops.

Although Pd is not a fusion relevant material, this study helped us in preparing future experiment with Ta and other materials.

2.5 Vibrational spectroscopy

Efforts were devoted to improving DTVE-B operation. Ion extraction was further studied and computer control programme was upgraded to include control of VEHM source. Vacuum chamber was enlarged in order to house the new VEHM source. Closed water cooling system was put in operation also for VEHM source. Second gas line is added that will enable mixing of gasses and also study of methane effect to the VEHM production.

3 CONCLUSIONS AND OUTLOOK FOR 2007

Last year we started to use experimental set-ups and procedures that were developed in previous years. Different processes where the role of vibrationally excited hydrogen molecules could be important were started to be studied. This work allowed us to get better insight into our possibilities and also to help us defining priorities for the time ahead.

Integration into EU efforts in the field of fusion research was continued by intensifying collaborations with other fusion associations in particular with FZJ [16] and IPP Garching [17]. Our association has organized 5th annual general meeting of EU Task Force for Plasma Wall Interaction in Ljubljana, from 13 to 15 November 2005 and co-workers from this project took main activity in this action [18], [19], [20].

For 2007 we plan to continue experiments initiated in previous time in particular studies of production of vibrational hydrogen molecules by recombination in a special cell, study of VEHM production by recombination after permeation and by thermal interaction with hot tungsten capillary, spectroscopic studies of Fulcher band emission from plasma into which VEHM is injected from outside and hydrogen interaction studies by HEC at new ERDA line that is under reconstruction. New experiments will be initiated in order to study vibrational relaxation of molecules by wall collisions. Special emphasise will be also on a study of possible influence of VEHMs on chemical erosion of hydrogenated carbon layers. Further work on development of experimental procedures will also be continued.

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HETEROGENEOUS SURFACE RECOMBINATION OF NEUTRAL HYDROGEN ATOMS ON FUSION RELEVANT MATERIALS

Miran Mozetič, Alenka Vesel, Aleksander Drenik, Tatjana Filipič, Janez Trtnik

Department for Surface Engineering and Optoelectronics, Plasma Laboratory,
Jozef Stefan Institute, Jamova 39, 1000 Ljubljana, Slovenia
email: miran.mozetic@guest.arnes.si

1 INTRODUCTION

Neutral hydrogen, deuterium and tritium atoms (thereafter: H atoms) are present in the cold edge and divertor plasmas in tokamaks as well as in vacuum components between the divertor and the pumps. The atoms in the plasma edge and divertor regions take part in a variety of collision processes. Hydrogen atoms form molecules with elements of plasma - facing components, presently predominating hydrocarbons arising from carbon surfaces, as well as with impurities on other surfaces. Unlike molecules that would not react with carbon even at elevated temperature, hydrogen atoms are reactive already at room temperature. It applies not only for hot protons coming from plasma but also for cold atoms that are found in the plasma edge layer. Slow atoms were detected in the plasma edge layer of a tokamak with several different diagnostic methods. Measurements in TEXTOR-94 showed that the atomic hydrogen flux from the carbon, deduced from Balmer line Hα measurements, is comparable to molecular flux at low carbon temperature. By heating of the test limiter surface the molecular intensity dropped and the intensity of atomic species increased. At high temperatures (above carbon temperature of 1500K) the atomic release dominates and molecules are practically negligible. Such behaviour was also found in ion beam experiments.

Determining hydrogen atom densities in remote parts of a tokamak presents a particular challenge. Atoms can reach surfaces that are otherwise shaded from the plasma and where otherwise used spectroscopic techniques are unavailable. An alternative method for measuring neutral atom density is the catalytic probe. A catalytic probe takes advantage of the exothermal nature of a recombination reaction, i.e. the event in which two hydrogen atoms recombine into a hydrogen molecule on the surface of the probe. The dissociation energy is absorbed by the probe. Rate of recombination is proportional to neutral atom density in the vicinity of the probe and thus it can be determined by observing the temperature of the probe. The catalytic probe seems a much cruder method than spectroscopy techniques, however it is a very non-demanding measuring technique, requiring neither a lot of space nor an environment free of vibration, etc (which is hard to procure during tokamak operation). Furthermore, it provides a reasonable spatial resolution of atom density.

So far we have developed several types of catalytic probes of which the Fibre Optic Catalytic Probe (FOCP) has proved the most successful design so far. Other, older types usually feature a small piece of metal foil – the catalyst surface – attached to thermocouple wires. The thermocouple wires can be highly susceptible to electromagnetic interference which hinders observing probe's temperature during plasma operation and can in that case be accurately measured only after plasma operation has ended. Moreover, to overcome heat losses through the thermocouple wires, the metal foil itself has to be of rather large proportions so that the heat absorbed from recombination becomes decisively greater than heat lost through the wires. The large surface of the probe means that the probe creates a significant disturbance in the atom density and the large mass of the probe lessens the probe's temporal density.

In a FOCP, the thermocouple is abandoned in favour of optical detection of probe's temperature. The catalyst in this case is a much smaller and lighter piece of metal foil that is wrapped around a small glass sphere (diameter typically 0.3 mm), which in turn is attached to an optical fibre. The fibre transfers thermal radiation emitted by the metal foil to a detector. Thus the signal is immune to electromagnetic interference and the probe is small enough not to significantly disturb atom density in its vicinity.

A key issue in both determining atom densities and predicting atom density profiles in a reactor is knowledge of recombination coefficient values of involved materials. The recombination coefficient – defined as the probability that an impinging atom will form a molecule with an adsorbed atom – determines the fraction of impinging atoms that recombine on the probe surface. Also, with the knowledge of recombination coefficient values it is possible to predict penetration depths of atoms in confined areas such as reactor pump ducts, etc.

2 WORK PERFORMED IN 2006

Our work in 2006 was divided into work perfomed at TEXTOR-94 or directly pertaining TEXTOR-94 and research of recombination coefficients for neutral hydrogen atoms on fusion-relevant materials.

2.1 Catalytic probe for TEXTOR

We constructed a catalytic probe for use in TEXTOR-94 in order to determine densities of neutral hydrogen atoms in remote parts of the reactor.

Discussions pertaining the design of the probe were finally complete in March 2006. Initially we intended using a Fibre Optic Catalytic Probe for that purpose, however the current configuration of the TEXTOR limiter-lock system does not allow an optic fibre feedthrough. Consequently we had to revert to the thermocouple design. Moreover, the strong magnetic field in TEXTOR (2.2 T) prohibits use of ferromagnetic materials which further influenced the design of the probe.

The probe for measuring the H atom density in TEXTOR is shown schematically in Figure 1. A photo of the probe is shown in Figure 2. The probe is made from a W-Re thermocouple wires mounted on the prescribed sample holder. The thermocouple wires are insulated with alumina tubes. On one end, the wires are connected to a standard W-Re plug. The plug fits the existing feedthrough system at TEXTOR. The other end of thermocouple is covered with catalytic material. We use 99.9+% pure gold foil with the thickness of 0.1mm. The foil covers the entire thermocouple joint as shown in Figure 1. The mass of the gold catalyst is 3.0g and thus exceeds the mass of the thermocouple joint for a factor of 2. The thermal connection between the gold catalyst and the thermocouple joint is satisfactory so that it is assumed the thermocouple shows the actual catalyst temperature.

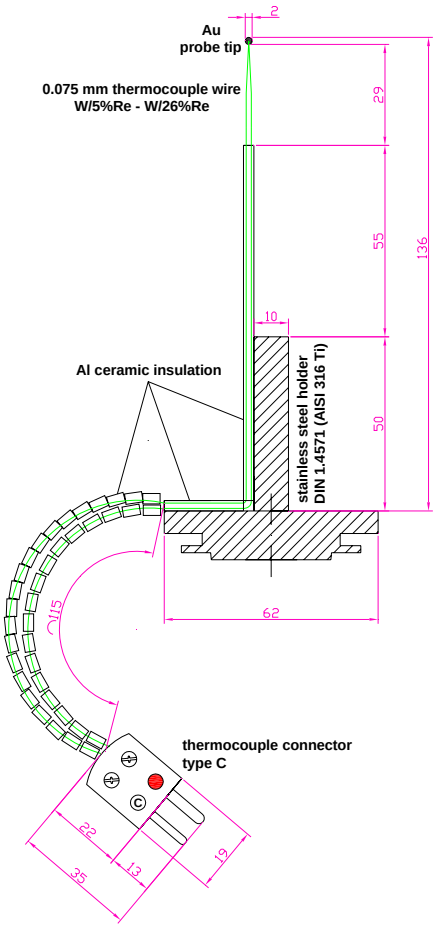

Figure 1: Schematics of a catalytic probe specially designed for measurements of H-atom density in TEXTOR.

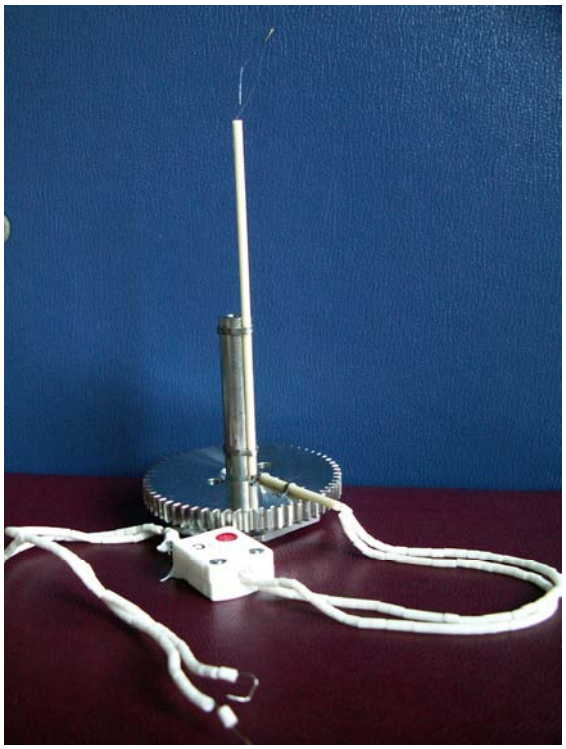

Figure 2: Photograph of the probe

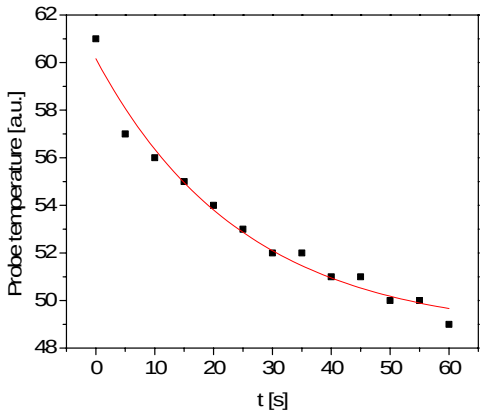

Figure 3: Temperature curve of the probe immediately after the glow discharge in TEXTOR. H density is determined by observing cooling of the probe.

The probe was tested in afterglow chambers of both a 5 kW and 300 W plasma reactors. The plasma in the glow chamber of the 5 kW reactor is definitely too aggressive and the probe would melt immediately if mounted into this part of the system. However, exposed only to dissociated hydrogen molecules, the probe withstood the test. The degree of dissociation determined with the probe was between 25% and 50%, which is a reasonable result, in accordance to our previous measurements with FOCP.

In TEXTOR the probe was mounted in the limiter lock system and tested in a glow discharge in hydrogen. At that time a connector for the C type thermocouple was unavailable so the probe was plugged in a different connector, which reduced the thermal resolution of the signal. That is most evident in the measurement performed during glow discharge, shown in Figure 3. Regardless, we managed to verify whether the probe was working or not. Also, we were able to determine some basic thermodynamic characteristics of the probe. After the successful test in the glow discharge, a few measurements were performed during regular tokamak discharges.

2.2 Recombination of neutral hydrogen atoms on fusion-relevant materials

Experiments on recombination of neutral hydrogen atoms on fusion-relevant materials have been performed at our radio frequency plasma reactors in Ljubljana, Slovenia, and microwave plasma reactor in Font Romeu, France. Samples of stainless steel and carbon were mounted in plasma reactors and exposed to hydrogen radicals created in plasmas. The stainless steel samples have been pre-treated by polishing and different cleaning procedures. The recombination coefficient was

found to be heavily dependent on the pre-treatment procedures. While chemically cleaned samples shower a rather stable recombination coefficient of the order of 0.1, other cleaning procedures showed completely different behaviour. The samples cleaned by oxygen plasma treatment were found particularly peculiar in terms of hydrogen atom recombination. While the samples cleaned at room temperature showed the recombination coefficient close to 0.1, the recombination coefficient for the samples cleaned at elevated temperatures show different values that were also time-dependent. The results showed that the recombination coefficient depends on particular phase formed on the surface during cleaning. The SEM analyses showed increased surface roughness of plasma – cleaned samples, while XPS, AES and XRD analyses showed formation of different phases during cleaning. While these experiments have not yet brought decisive results and more work will have to be performed, it is clear that the recombination coefficient can be modified by different pre-treatment of the material.
Preliminary results with carbon samples showed that the recombination coefficient for carbon depends heavily on the deposition conditions. The coefficient can vary for more than and order of magnitude. As in the case of stainless steel, more experiments will be performed in the next future in order to understand this phenomenon.

3 CONCLUSIONS AND OUTLOOK FOR 2007

A thermocouple gold-tipped catalytic probe for H density measurement in TEXTOR was designed. The probe was tested in the laboratories of Jožef Stefan Institute, then installed in TEXTOR and tested both during glow discharges and regular tokamak discharges. In 2007 further measurements are to be performed with the catalytic probe. Experiments will be performed at probe position few cm below the plasma edge.

Extensive experimental work on behaviour of fusion relevant materials in hydrogen plasma is foreseen in 2007. The results of 2006 showed interesting variations of the recombination coefficient during evolution of surface peculiarities. The goal of 2007 campaign is to perform systematic measurements that should give an insight of the hydrogen atom interaction with samples prepared by different means. The measurements on recombination of H atoms on carbon surface will be performed for samples prepared by different deposition techniques. Recombination coefficients will be measured during exposure to aggressive plasma when chemical erosion takes place. The resulting evolution of the surface roughness that causes variation of recombination probabilities will be monitored by suitable techniques so it will be possible to explain the γ coefficient versus the surface morphology.
More experiments with stainless steel will be performed at extreme conditions (i.e. high sample temperature and high flux of hydrogen radicals in order to explain variations in recombination coefficient.

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APPLICATION OF ION BEAM ANALYTICAL METHODS TO THE STUDIES OF PLASMA WALL INTERACTION IN TOKAMAKS

Primož Pelicon, Iztok Čadež, Zvone Grabnar, Darko Hanžel, Matjaž Kavčič, Zdravko Rupnik, Jure Simčič

Department for Low and Medium Energy Physics, Jožef Stefan Institute, Jamova 39, P.O.B. 3000, SI-1000 Ljubljana
e-mail: Primoz.Pelicon@ijs.si

1 INTRODUCTION

The interaction of the tokamak plasma with plasma-facing components results in erosion, transport, re-deposition of wall materials as well as fuel retention in the vessel wall. Fuel retention in deuterium-fueled fusion experiments is currently intensively studied to allow predictions for the tritium retention in ITER, which may lead to a forced cleaning intervention due to excess safety limits in the vessel. The project is dedicated to detection of the retained fuel and re-deposited material in inner walls of tokamaks by Ion Beam Analytical methods, based on the interaction of accelerated ion beams with materials. The isotope sensitivity and ability of obtaining depth profiles without object destruction is main reason for application of IBA techniques in the field of fusion research. Focused ion beam facility at tandem accelerator laboratory of Jožef Stefan Institute additionally provides lateral resolution in micrometer range.

2 WORK PERFORMED IN 2006

Attention has been focused to the measurements of a fuel adsorbed in the surfaces exposed to tokamak plasma. Among several available measurement methods for detection of hydrogen isotopes with fast ion beams, Elastic Recoil Detection Analysis (ERDA) [1] and Nuclear Reaction Analysis (NRA) with $D(^3He,p)^4He$ reaction [2] were selected as candidates for lateral mapping of adsorbed fuel.

The ERDA method enables detection of ^1H, ^2D and ^3T simultaneously. The NRA method based on $D(^3He,p)^4He$ is used to detect deuterium. Focusing of the accelerating ion beams offers an option of lateral-resolved methods. Scanning of the focused beam over the sample surface reveals lateral distribution of hydrogen isotopes.

2.1 Isotope-resolved micromapping of retented fuel in tokamak plasma-exposed surfaces with Elastic Recoil Detection Analysis

Elastic Recoil Detection Analysis (ERDA) with ^{7}Li beam is well developed method for detection of hydrogen isotopes in thin films and surfaces at Jožef Stefan Institute [3,4]. In order to enable lateral-resolved equivalent method, the 3 MeV ^{7}Li beam has been injected in the microbeam-forming system [5]. The required parameters for the lenses excitation were obtained via numerical simulation in the first-order ion-optical calculation, developed at JSI (fig.1).

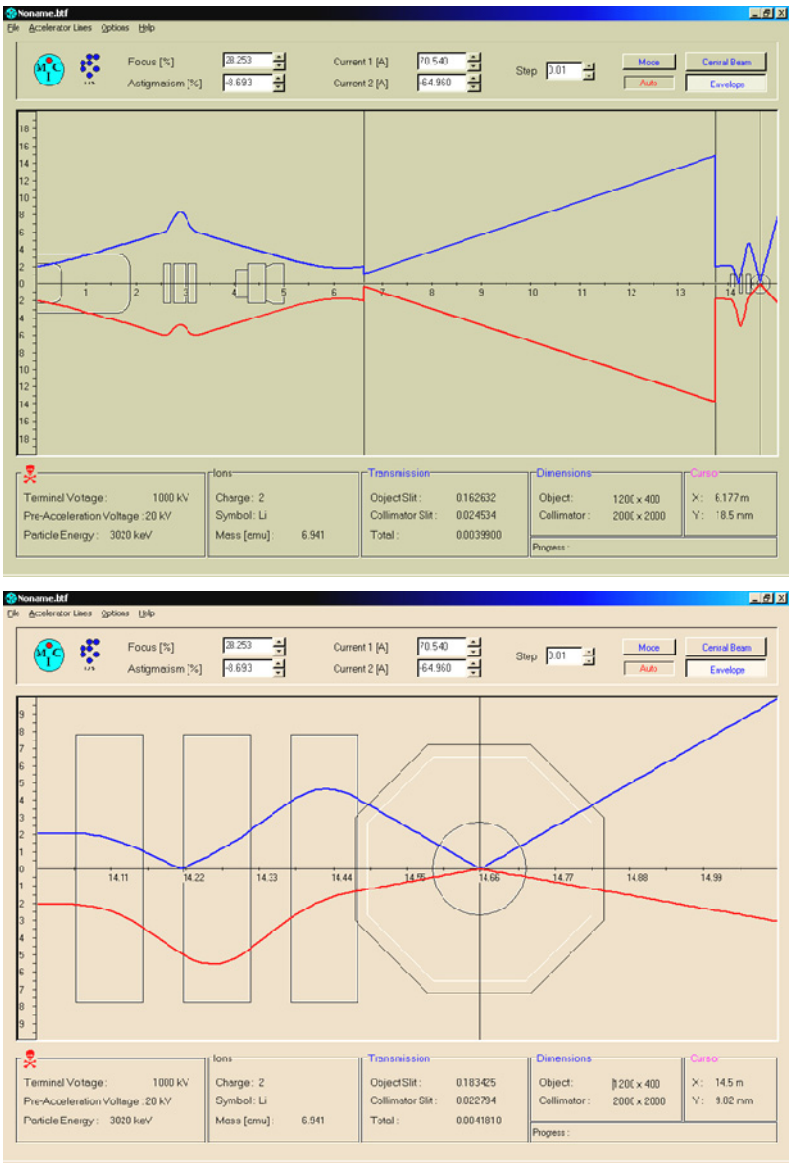

Fig. 1: The 3 MeV ^{7}Li microbeam forming setup. Up: entire focusing system action -accelerator, electrostatic lens, switching magnet and quadrupole lens. Bottom: zoom on the action of the magnet quadrupole triplet lens [5].

2.2 Isotope-resolved micromapping of retented fuel in tokamak plasma-exposed surfaces with Elastic Recoil Detection Analysis

The method in the first measurements revealed hydrogen distribution at the surface of castellated limiter (Fig. 1) exposed on a load-lock system of TEXTOR tokamak, Forschungzentrum Juelich. Its low roughness enabled good mapping of the ^{1}H with focused beam ERDA. The erosion-dominated plasma conditions during the tokamak experiment [6] resulted in ^{2}D concentration levels below 10^{16} D at/cm^2. The measurement revealed ^{1}H distribution anti-correlated with re-deposited molybdenum [7] (fig. 2). Result is expected, taking into account low hydrogen sticking affinity at molybdenum surface in respect to graphite.

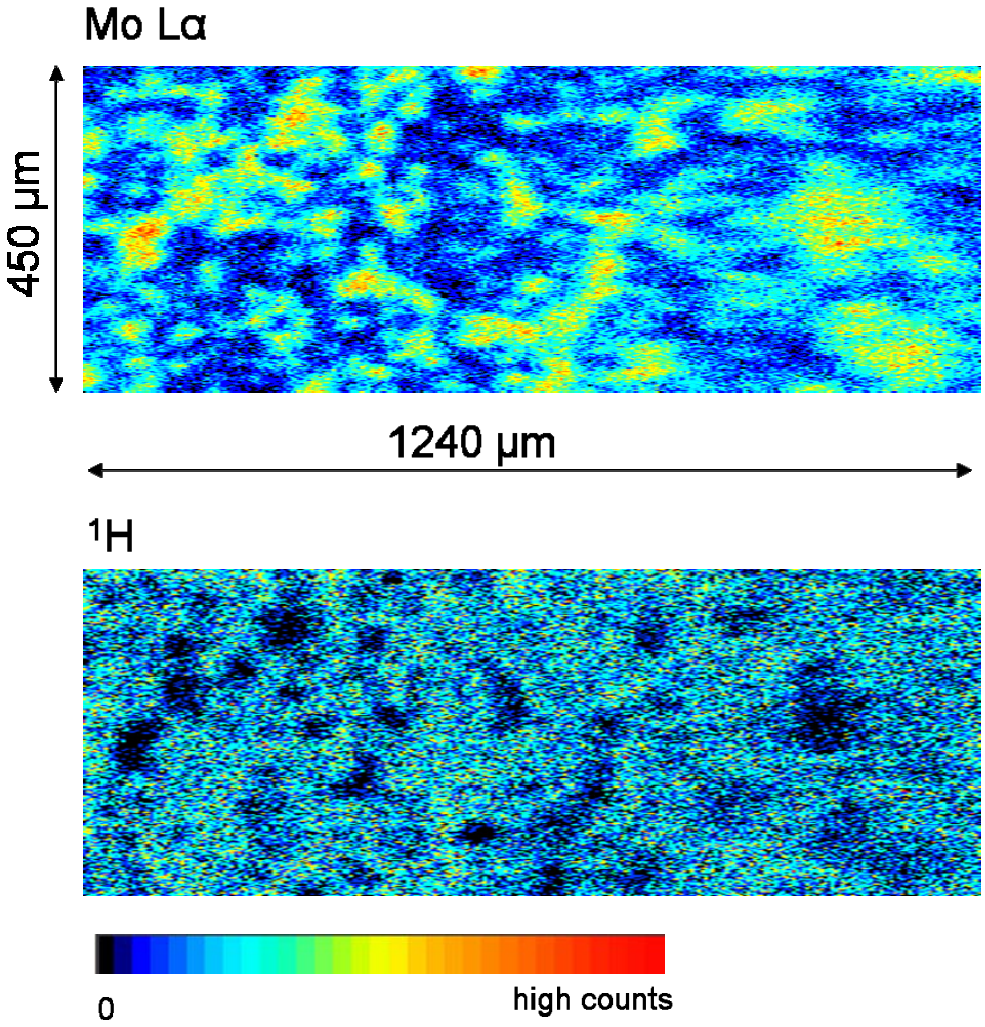

Fig. 2: Lateral distribution of molybdenum and hydrogen measured simultaneously in the same region of the surface of castellated limiter. The limiter consisted of Mo and graphite sections[6] to simulate the proposed divertor structure of ITER. The exposure in the tokamak plasma of TEXTOR tokamak, FZJ, has been performed under erosion-dominated conditions. The experiment resulted in island-like re-deposited Mo on graphite section The hydrogen distribution shows anti-correlated feature: in the Mo island locations, adsorbed hydrogen areal density is much lower. The molybdenum distribution has been measured by detection of Mo Lα X-rays and ^{1}H distribution by detection of ^{1}H recoils with ERDA method.

2.3 Deuterium retention in carbon-based tokamak wall materials

Experiment consisted of controlled deuterium-rich plasma exposure in the TEXTOR tokamak and post-mortem analysis of exposed surface layers [8]. Three carbon-based materials were exposed: a former ITER reference Carbon Fiber Composite (CFC) NB31 produced by Snecma, JET CFC DMS780 produced by Dunlop and fine-grain graphite EK98 produced by Ringsdorff. The limiter (fig. 3a) was exposed to 32 reproducible ohmic discharges with a total duration of 177 s. The total deuterium fluence was $2.9 \cdot 10^{25}$ D/m^2. The sectioning scheme of exposed materials is shown on fig. 3b. The section selected for focused ion beam measurements is labeled with MB.

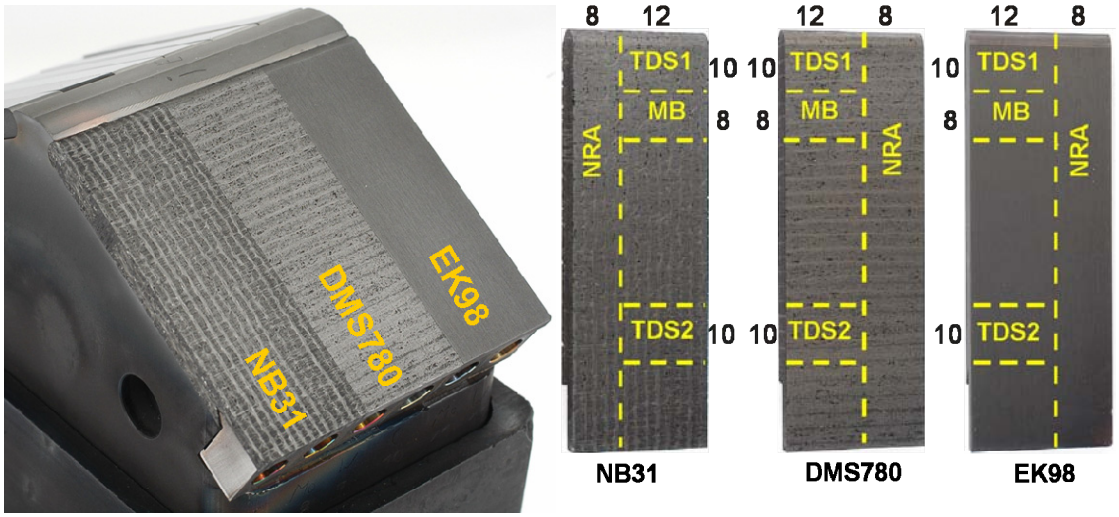

Fig. 3: Left: A photograph of limiter constructed for deuterium plasma exposure experiment in TEXTOR tokamak, Forschungszentrum Juelich. Right: Distribution of specimen for post-mortem analysis, where TDS means Thermo-desorption (TDS) analysis at FZJ (Juelich), NRA Nuclear Reaction Analysis by VR (Stockholm, Uppsala), MB Micro-beam analysis by JSI (Ljubljana).

Deuterium lateral distribution map has been measured using ERDA method with focused ^{7}Li beam. Result of mapping is shown on fig. 4. In the case of Carbon Fiber Composites (CFCs), large inherent surface roughness introduces artifacts in ERDA distribution image. Surface image obtained by secondary electrons provides qualitative image of surface topography, which is reflected in the maps of ^{1}H and ^{2}D lateral distribution. Large influence of surface topography on measured isotope lateral maps is caused by large beam impact angle used in ERDA [9]. Taking into account the topography effect, the distribution of retained fuel is well homogeneous including regions, where fibers in CFCs protrude from bulk to the surface.

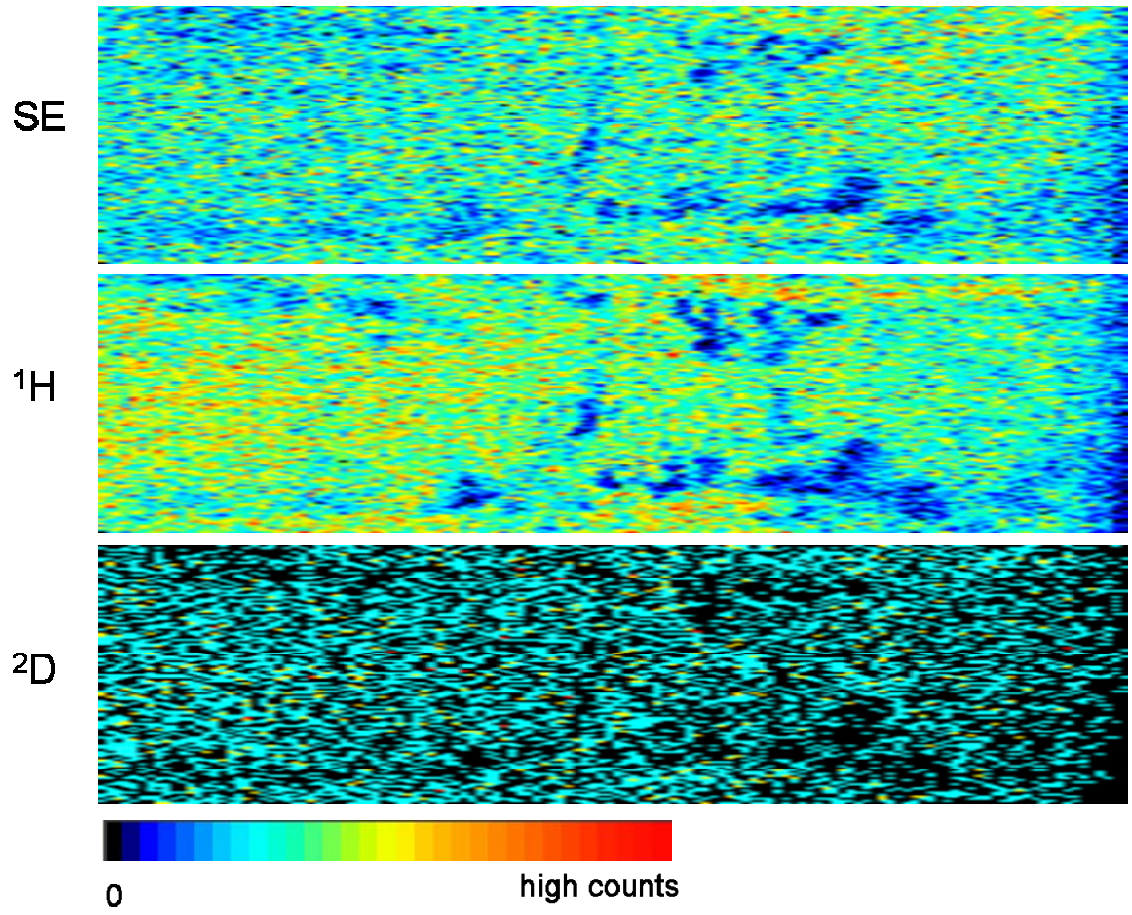

Fig. 4: Lateral maps obtained with ERDA method on Carbon Fiber Composite material Carbon Fiber Composite (CFC) NB31 after exposure in TEXTOR. Maps present secondary electron image (quantitative surface topography), hydrogen distribution and deuterium distribution, respectively. Map size is 1500 x 6000 μm^2. Hydrogen map shows strong inhomogenity due to sensitivity of ERDA method on surface topography (roughness).Taking into account the topography effect, the distribution of retented fuel is well homogeneous including regions, where fibers in CFCs protrude from bulk to the surface [9].

In order to enable measuring of deuterium lateral distribution, Nuclear Reaction Analysis (NRA) with D(^{3}He,p)^{4}He reaction has been developed as alternative method since November 2006. In this case the ^{3}He ion focused beam is directed perpendicularly on analyzed surface. The D(^{3}He,p)^{4}He reaction produces high energy protons with energies of over 10 MeV [2]. In such configuration, the topography (surface roughness) influence on lateral maps is significantly lower. The method is, however, expensive due to demanding beam formation on tandem accelerators as well as due to considerable ^{3}He gas consumption.

3 CONCLUSIONS AND OUTLOOK FOR 2007

The evaluation of retention processes in wall materials rely mostly on deuterium quantification in the tokamak plasma-exposed wall materials, since hydrogen (^{1}H) may

be additionally introduced in the form of moisture during sample atmospheric exposure and tritium handling imposes severe safety measures. Several available analytical techniques for deuterium detection provide distribution information with a lateral resolution of typically 1 mm^2. Deuterium detection techniques with high-energy focused ion beam include Elastic Recoil Detection Analysis (ERDA) and Nuclear Reaction Analysis (NRA) with ^3He beam employing $D(^3He,p)^4He$ reaction. With larger depth range and perpendicular geometry applied, the NRA employing $D(^3He,p)^4He$ reaction is less sensitive on the roughness in comparison with ERDA. Mean surface roughness exceeding 10 micrometers is inherent to several samples of interest, including cleaved bulk wall samples of candidate materials for the ITER walls and even the surface of Carbon Fiber Composites (CFCs) selected for construction of ITER divertor. Strong influence of surface topography on the ERDA method favors the NRA technique for the analysis of the real materials used in fusion devices.

Deuterium distribution maps of wall materials exposed in the deuterium plasma of the tokamak TEXTOR will be measured by NRA method in 2007. Participation of the project group with facilities and methods described here in Deuterium Inventory in Tore Supra (DITS) project is initiated.

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DEUTERIUM RETENTION AND RELEASE FROM METAL SURFACES – A COMPLEMENTARY METHOD TO NUCLEAR TRITIUM METHODS

Vincenc Nemanič, Bojan Zajec, Marko Žumer

Department of Surface Engineering and Optoelectronics (F4), Jožef Stefan Institute, Jamova 39, 1000 Ljubljana, Slovenia
e-mail: vincenc.nemanic@ijs.si

1 INTRODUCTION

The results obtained in 2006 were within the frame that was made in the working plan. Two main activities could be reported separately since they cover two problems: a) the sensitivity limit of the applied UHV system to deuterium adsorption at low pressure and room temperature, which was in fact "the blank run" needed for the work in 2007 b) the residual hydrogen contained in the sample made of stainless steel AISI 316. This amount is also important when the isotope effect and exchange with deuterium is expected in high temperature exposures planned for 2007.

2 WORK PERFORMED IN 2006

2.1 The first part of the year

In the first period of project duration, (i.e. the first half of 2006), we investigated the UHV system response to well defined deuterium exposures. This phase was in fact needed to prove the proposed principle of our alternative method as well as its accuracy. This is particularly needed when an arbitrary selected conductive sample (metal or graphite) will be tested for deuterium absorption and release. It is worth to mention that the same data are crucial for prediction of tritium inventory in material exposed to gaseous tritium to ensure safe handling and decommissioning of future fusion reactors, as reported a few years ago by UKAEA group [1].

It is quite a natural question what is a response of an UHV system after a well defined exposure to gaseous hydrogen or its isotopes at low pressures. Intensive search in UHV related literature for such a report gave no score, so we had to obtain our own data.

Measurements were performed in an all metal UHV system. The components were made of AISI 304 or AISI 316L, mounted in the system for a few years. The outgassing behaviour of regularly thermally treated system was well known since it was

a part of each study done on the system. The analysis of inner surfaces was not made since native oxides of ~1 nm thickness are known to be formed on stainless steels and represents the protective layer. For the initial pump-down as well as for gas removal after deuterium exposures, a turbo-molecular pump was applied. Leak testing and offset point setting of passive gauges was done by a quadrupole mass spectrometer (QMS-1), mounted just above the turbo-molecular pump. The pressure in the system in long term observations was registered by two calibrated capacitance manometer heads (1 mbar and 0.05 mbar full-scale, MKS). The volume V = 0.56 L and estimated surface area, used for calculations, A ≅ 500 cm^2. Another external QMS-2 (Prisma 100, Pfeiffer), pumped by its own ion-getter pump, was attached to the system and separated by the valve for the most of the experimental time. Experiments could be realized by hydrogen but deuterium was selected instead to distinguish it from the hydrogen background. HD molecules were undoubtedly formed at the surface via exchange reaction. To obtain accurate results, a careful calibration of the QMS-2 with pure D_2 and H_2 and their mixtures in different proportions had been made to determine the HD formation rate within the QMS-2 itself.

For all results in the study, it was thus of crucial importance to evaluate the residual hydrogen outgassing rate determined after an overnight heating at 120 °C. The temperature was uniform during the heating by circulating air except for the externally mounted CM heads hold at 45 °C and short connecting bellows. The unheated surfaces represent a tiny fraction of the whole system surfaces. After cooling to room temperature and closing the valve to the pump, the pressure increasing as a result of background outgassing was recorded by the 0.05 mbar CM head for several ten hours. The accumulated gases were analyzed by the QMS-2. Hydrogen usually represented more than 98 % of the evolved gas, while the rest was mass number 28 which could be released from the QMS-2 by the hydrogen impact, but practically no water signal was detected.

The results of this work were published in journal *Vacuum,* [2]. The main findings may be presented simply:

Deuterium exposures in a well outgassed UHV chamber made of stainless steel AISI 316 (V= 0.56L, A = 500 cm^2) lasted 24 hours. The registered pressure change $\Delta p \approx 1\times10^{-2}$ mbar during the exposure of 1 mbar D_2, (303 K, 84 h) corresponds to absorption of 5.4×10^{14} D atoms /cm^2. The slope $dp/dt \sim 3.4\text{x}10^{-8}$ mbar/s corresponds to sticking probability ~ $1\text{x}10^{-12}$. So far, such low values could only be registered by applying tritium [3,4].

Beside the kinetics of deuterium absorption we could also follow the changed kinetics of deuterium desorption which was observed after it had been pumped out of the system. The measuring cycle lasted at least 72 hours. As expected, deuterium influenced outgassing rate substantially, for three orders of magnitude. Initially, deuterium in the form of D_2 prevailed. The gas that prevailed after 24 hours and manifested the isotope exchange effect was HD. Its abundance in QMS spectra was present also after 72 hours which shows that some deuterium could migrate deeper into the bulk. This was confirmed also by a quantitative comparison of deuterium that has been absorbed and D_2 and HD that have been released in subsequent periods.

2.2 **The second part of the year**

The role of strongly bound hydrogen on the observed kinetics of hydrogen outgassing from austenitic stainless steel was recognized to be important as it was shown in publications based on our previous studies. Before we could determine the amount of deuterium that could be absorbed in stainless steel at elevated temperature we had to determine the amount of nascent hydrogen in the sample. Precise measurements were performed in several heating cycles at 1040 K during some months. They reveal that the initial hydrogen abundance may be substantially higher than generally expressed by the solubility relation. A re-population of regular hydrogen sites in the bulk proceeded spontaneously by keeping the sample at room temperature for some days in high vacuum. To eliminate hydrogen absorption from residual atmosphere, some heating cycles were continued after the sample was stored in deuterium at 1 mbar. The same phenomenon was observed. Moreover, when the same sample was charged with deuterium up to 1 bar at high temperature, its fast and complete release followed within some minutes at 1040 K. Subsequent hydrogen evolution was not affected by this exposure. These new findings may improve the traditional picture of hydrogen interaction within the bulk of stainless steel. The old picture based mainly on the results of permeation measurements, where strongly bound states do not play a noticeable role. Contrary, they must be respected when high energy D or T ions from plasma hit the vacuum chamber. Tritium removal by subsequent thermal treatment is not as simple as predicted by the generally accepted picture.

The main result of this part of our experimental work is that a high amount of nascent hydrogen was found to be stored in the bulk having a long-term persistence in the sample. The unexpected behaviour of this residual hydrogen is most easily shown by time dependence of its kinetics. The outgassing did not approached to zero after the first peak ceased, but started to rise again after approximately 3 minutes. The hydrogen in the first peak may be reasonable well described as “diffusive peak”. It was confirmed by measuring of the outgassing rate by the QMS (throughput method) instead of measuring the pressure rise in the closed volume that hydrogen peak is not an artefact or any other side effect.

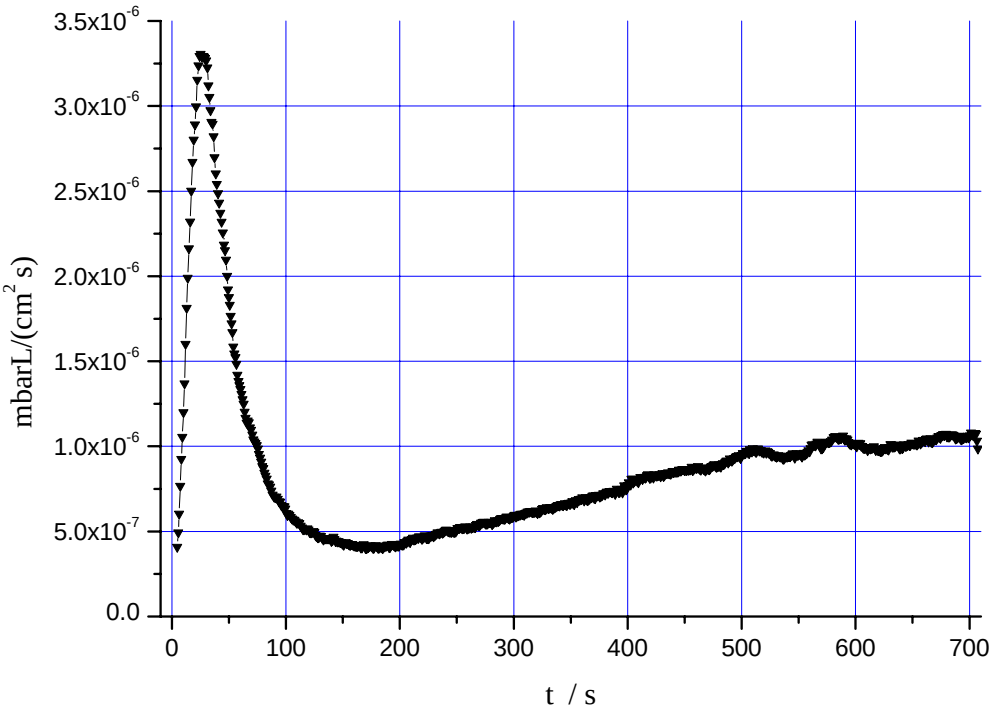

Fig. 1: Hydrogen evolution kinetics during the first heating cycle at 1040 K. After the first peak which is quite a regular phenomenon, a slow but persistent increase in outgassing followed which does not fit in any of proposed models.

To ensure that broad anomalous slow but persistent increase in outgassing, which appeared after some storage time, does not represent hydrogen that was accumulated from the surrounding hydrogen, which always represents the main constituent of residual atmosphere, the sample was also kept for some days in deuterium at 1 mbar. It was pumped before the next heating cycle at 1040 K. Surprisingly, there was no deuterium found in spectra but mainly hydrogen. The surprising result is presented in fig. 2. The shape of the curves, all recorded at the same temperature, can not be explained by any of the existing models. The intensity should have no delay since the maximum should appear immediately after the temperature reached the set value. It was well confirmed that it is the storage time at room temperature that influences the kinetics of the next cycle.

The same shape of the maximum, but being several times more intense than the narrow peak in Fig. 1, was found to appear after the sample had been charged with deuterium at 1 bar. Our stainless steel sample thus exhibited regular or expected properties described by Sievert's law and diffusivity. Most of researches presumably built their models on permeation rate measurements where strongly bound residual hydrogen indeed participate a negligible fraction to the total flux.

Unfortunately, the anomaly described in this report represents a permanent source of hydrogen delivered into well outgassed UHV chamber made of stainless steel. Every thermal treatment and cool down thus promotes some residual hydrogen to occupy regular sites. A good confirmation of these phenomena was published some years ago [5]. This hidden hydrogen presumably hinders achieving very low outgassing rates needed in UHV technique. Anyhow, it gives a different outlook on very scattered data on diffusivity and solubility of many poly-crystal pure metals. In that paper it was shown that diffusive hydrogen may even be the minor constituent compared to strongly bound species. The relevance of residual strongly bound hydrogen in fusion related materials and their use has not been published.

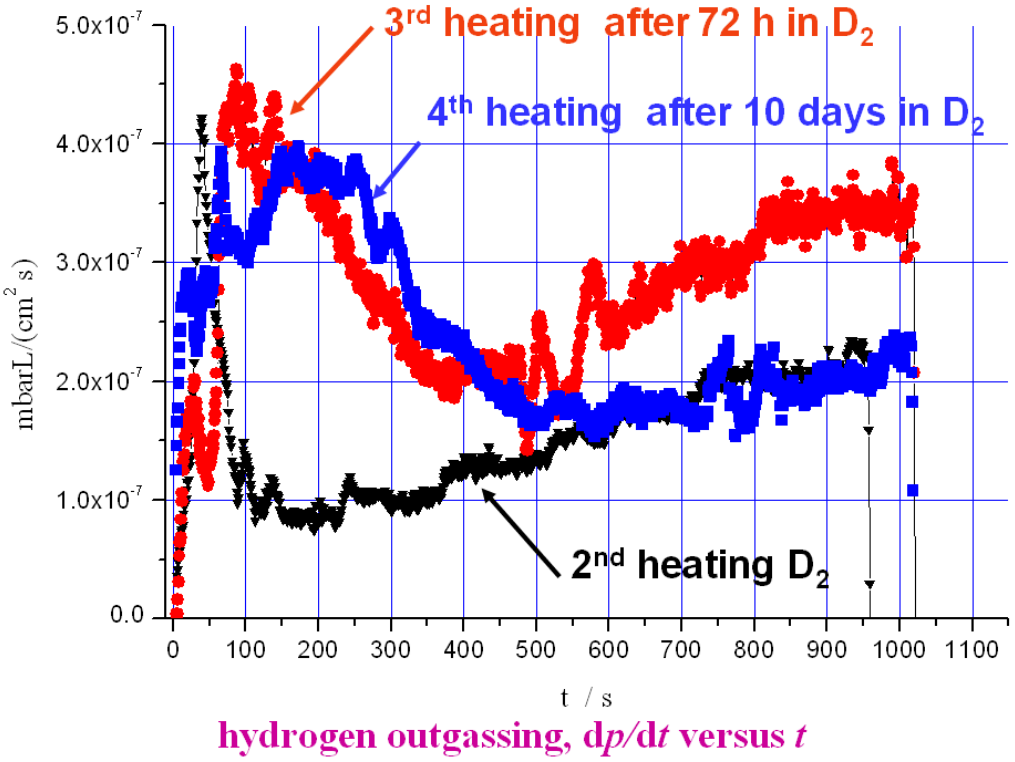

Fig. 2: Hydrogen evolution kinetics during some heating cycle at 1040 K. The shape of the maxima after some days of storing in deuterium is presented. The storage time in D_2 before the 2nd heating was 24 hours.

3 CONCLUSIONS AND OUTLOOK FOR 2007

Two main characteristics were determined in the first year of studies needed for refined experimental work planned for 2007. These characteristics are a) the sensitivity limit of the applied UHV system to deuterium adsorption at low pressure and room temperature, b) the amount of hydrogen contained in the sample made of stainless steel AISI 316A.

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COLLABORATION IN DEMO WORKING GROUP

Matjaž Ravnik, Andrej Trkov

Jožef Stefan Institute, Reactor Physics Department
e-mail: Matjaz.Ravnik@ijs.si

1 INTRODUCTION

The purpose of the project is to contribute and to make available to the DEMO Working Group the knowledge and the expertise on the conventional aspects of the DEMO power station as a nuclear installation.

The main work objective of the DEM Working group in 2006 was the assessment of the fusion development strategy and of the program objectives of the DEMO project development. Our interest from was mainly in the technology of the secondary side (balance of plant) and in the neutronics. However, the main objectives and open problems of the DEMO project are still in the basic fusion technology. Practically, our work in the DEMO Working Group consisted of studying and assessing of the materials prepared by EFDA-CSU and related scientific and technical documentation. The materials were discussed and co-ordinated by exchange of information and at the meetings of the working group. Our contributions are included in the materials (2) that are the final result of the team work of the Working Group.

2 WORK PERFORMED IN 2006

In 2005, the DEMO Working Group accomplished work on the European Power Plant Conceptual Study (1). The document treated general and conceptual aspects of the future fusion power plant. On the basis of the conceptual study, a strategic document entitled 'Fusion Development Strategy – Physics, Engineering and Technological Challenges' was prepared by EFDA-CSU and assessed by the DEMO Working group (2) in 2006. On its basis, the detailed programme of the project will be elaborated in 2007 (3).

The document on the fusion development strategy (2) is a conceptual scenario of the development from the present stage technology to the final goal - a commercial fusion power plant. It is based on the "Fast Track" programme with a single step between ITER and a FPP. It involves three main components of the fusion programme – ITER, IFMIF and DEMO (DEMO being the single step between ITER and a FPP). The scenario, prepared by the EFDA-CSU Garching, has been reviewed by the DEMO Working Group with the main purpose to highlight the unresolved physics, engineering and technological challenges. It also includes the safety, environmental, societal and economic features of fusion power. One of the main questions addressed is also the

time-frame of the DEMO project, in particular, when will the fusion power become commercially available.

It is assumed that the design of DEMO can start after the completion of ITER construction, and develop as data from ITER and IFMIF operations becomes available. The licensing and start of construction of DEMO will depend on the availability of data on materials testing from IFMIF and on physics confirmation and component testing from ITER. The granting of a license for DEMO operation represents the first key milestone of the DEMO programme. As the DEMO programme is highly dependant on data from the IFMIF and ITER programmes, delay in the availability of this data will have a direct impact. Delays to the programme could also result from delays in making the two key decisions: (a) to start the design, and (b) to start the construction.

Main scope of the work was related to the critical physics issues, critical technological and engineering issues, duration of stages and in defining the design activities. As our main interest was in the technological and engineering issues related to the conventional technology, here we resume this part of the studies while the rest (physics in particular) may be found in the main paper (2).

Main gaps between the existing and required technology are related to

- the level of operational availability of DEMO (assumed from 33%-50% during the first phase of operation)
- the neutron wall loading ($2MW/m^2$)
- the qualification of the most exposed components to resist the design neutron fluence (up to 50dpa)
- the architecture and the qualification of the remote handling procedures
- integral experimental qualification of the blanket and divertor structural material.

The availability is identified as one of the main problems. If the overall availability of DEMO (at least 33%) is to be achieved, the availability of the individual systems must be much better (up to 90%). On the other hand, the testing of some components (and their availability in future power plants) depends on the availability of the DEMO plant as they can not be tested anywhere else as only DEMO will achieve the wall neutron loading comparable to the commercial fusion power station reactor. However, main tests and qualifications will have to be result of ITER. The fusion development scenario assumes that the required physics for DEMO is demonstrated during ITER Stage 1 operations. The scenario also identifies a number of technological objectives which need to be achieved during the second stage of ITER operations to provide input which is critical to the DEMO programme.

The following objectives for ITER stage 2 operation are derived (2):

(a) Validation of the breeder blanket technology in terms of thermo-hydraulic, thermo-mechanical and TBR properties, such that DEMO would be self sufficient in tritium production
(b) Validation of divertor functional performance in terms of thermo-hydraulic and thermo-mechanical properties.
(c) Qualification of the required tritium technology.

(d) Qualification of the required technology for H&CD systems.
(e) Qualification of relevant PFCs: the only suitable first wall material for DEMO and a FPP identified to date is tungsten, and the reference scenario implicitly assumes that DEMO has a 100% tungsten first wall from Day 1 of operation.

The DEMO physics scenario can only be considered fully validated in ITER by operation with a tungsten first wall. This can only be accommodated in ITER by a change in phase 2 of the exploitation. To minimise risk it would also be important to study aspects of operation with a tungsten first wall (for example, plasma performance and thermal fatigue behaviour) under high power loads within the accompanying programme, using devices such as JET.

Although main technology gaps will be solved with the results from ITER, there are several aspects that will require also other parallel activities. Among them, the conventional side of the plant deserves special attention not to be overlooked in the entire scheme. It is usually assumed that the components of the balance of plant (the cooling and power conversion systems in particular) do not require any development. This would be true for water cooled DEMO with water at PWR conditions (325ºC, 155bar) as there are hundreds of PWR's in operation. For a helium-cooled DEMO, key equipments for the primary heat transfer system and for the power conversion system do not exist, they must be developed (heat exchangers, blowers, turbo-generators, etc.). Either these developments are undertaken by another community, e.g. Gen. IV fission reactors, or the fusion community should assess whether it could support these developments on its own. The resources required would be considerable and the availability of these systems could be a major concern. It is worth recalling that, in the 70's, the major causes of PWR's unavailability were BoP components and systems.

The draft fusion energy development scenario was developed. It is summarized as follows (2):

- DEMO design and licensing is assumed to last 10 years, starting immediately after completion of the construction of ITER.
- DEMO construction is assumed to last 8 years, followed by 2 years for DEMO commissioning. DEMO Phase 1 would last 8 years (average availability 33%) and the DEMO shutdown 1 year. If an average availability of 50% is assumed for DEMO Phase 1 then the overall programme is accelerated by 2 years.
- The design of FPP is estimated to last 8 years and it is assumed that FPP design starts before the completion of DEMO Phase 1. However, the design of FPP internal components (5 years) would only start following the successful completion of DEMO Phase 1 and of the shutdown. This assumption provides an acceleration of the programme by 3 years.
- With an estimated FPP construction period of 7 years, FPP operation would start at the beginning of 2057.

The electricity would be delivered to the grid early during the first phase of operation of DEMO.

3 CONCLUSIONS AND OUTLOOK FOR 2007

In 2007 the work continues according to the STAC guidelines and DWG program. The goal of this project (P-7) will not change. Main work is planned on the DEMO project development programme as the conceptual stage has been practically accomplished.

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GAS IMPERMEABLE COATING FOR SiC_f/SiC

Saša Novak[1], Goran Dražić[1], Nataša Drnovšek[1], Katja Mejak[1], Tea Toplišek[1], Tomaž Žagar[2], Bruno Riccardi[3]

"Jožef Stefan" Institute, [1]Department for Nanostructured Materials, [2]Reactor Physics Department, [3]ENEA Frascatti, Italy
e-mail: sasa.novak@ijs.si

1 INTRODUCTION

The task is aimed to overcome the drawback of the CVI-processed SiC/SiC composites, i.e. residual open porosity, by application of a dense layer of the SiC-based material having properties adequate for use in the fusion reactor. The main problem to be solved to achieve this goal is linked to high temperatures and pressure needed for the densification of SiC and therefore a part of the work has been focused in development of a suitable coating material, taking into account the specific requirements for the fusion-related materials, in particular low neutron activation. Another part has been oriented in development of a suitable technique for the coating. Electrophoretic deposition (EPD) was introduced as a perspective method, mainly due to suitable electrical conductivity of SiC/SiC(CVI) composites and due to ability of production dense compacts. The investigations in the year 2006 comprised mostly evaluation of the EPD process parameters as a function of the ceramic slurry composition and the conditions during deposition, wetting characteristics of the secondary phase and investigation of microstructure and physical characteristics of prepared coating materials.

2 WORK PERFORMED IN 2006

2.1 Electrophoretic deposition of low-activation SiC-based coating on SiC/SiC (CVI) substrate

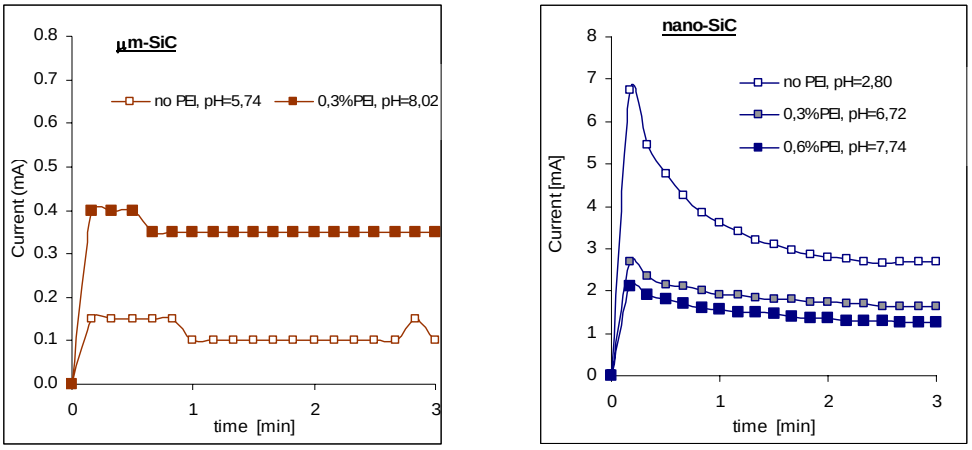
Electrophoretic deposition (EPD) is a colloidal process wherein ceramic bodies or coatings are shaped directly from a stable colloid suspension by a DC electric field which causes the charged particles to move toward and deposit on the oppositely charged electrode. Our preliminary tests implied that the SiC/SiC (CVI) composites exhibit sufficiently high electric conductivity to be employed as a deposition electrode that enables deposition of the SiC particles from suspensions to form the coating on it. In order to enable the formation of dense sintered coating at SiC/SiC, the SiC particles and sintering additives containing suspension for EPD must be suitably conductive and the particles must have high surface charge (zeta-potential, ZP).

Therefore, suspensions of micron-sized and nano-sized SiC powders in ethanol were first analysed for the zeta-potential as a function of operational pH of suspensions, various surface active additives and additives for sintering. Due to a low reproducibility of the results, different techniques were tested and finally Electrokinetic Sonic Amplitude (ESA) was selected as the most accurate. The device ZetaProbe (Colloidal Dynamics, USA) was purchased and delivered in October 2006. The slurries for the deposition were prepared by the homogenization of micron-sized and nano-sized SiC powders in ethanol and deposited at various applied DC voltage (5 - 60 V/cm) and various solids content (7 to 70 wt.%). The results showed significantly different behaviour of both powders due to different composition in surface layer at the particles (ref.1). Further, polyethylene imine (PEI) was found to increase the ZP of micron-sized SiC particles from ~10 mV to ~35 mV and at the same time increases conductivity of suspension. In contrast, in the nano-sized powder suspension it caused a decrease (Figs. 1a and 1b). Figures 1a and 1b also illustrate the current drop during the deposition of the suspensions containing micron- or nano-sized SiC powder, sintering additive and PEI, while Figures 1c and 1d show dry resulting deposits formed at SiC/SiC placed as cathode after 3 minutes at 60V. Apparently, to produce a few hundreds of microns thick coating, less than a minute is needed.

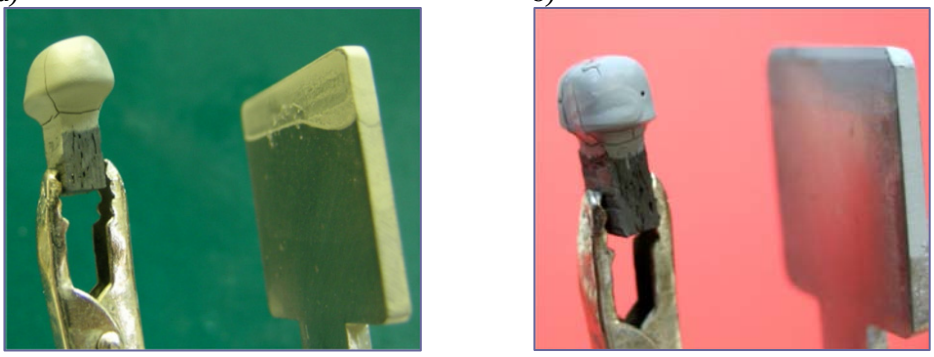
The effect of the PEI in SiC-ethanol suspensions for EPD was thoroughly analysed in diploma work of Mrs. K. Rade (ref.2). The results were also presented at the 11th International Ceramics Congress in Italy (ref.3) and were published in Journal of Materials Science (ref.4) and Key Engineering Materials (ref.5).

The EPD technique as a tool for coating of SiC/SiC was also a research topic of Dr. Sasa Novak during her visit at Imperial College London in the period from October to December 2006, under the staff mobility scheme. The visit was in part supported by Royal Society, UK, and partly by EC under the Staff mobility agreement. The work at the Imperial College was focused in development of electrophoretic deposition of SiC from aqueous suspensions containing sintering additive either in solution or deposited previously on the SiC particles. Based on the experience at the hosting laboratory, a suspension containing CTAB (cetyl-trimethilammonium bromide) as a surfactant was developed that enabled the deposition of both uncoated and AlOOH-coated SiC on steel electrode as well as on SiC-fibres. Further, the CTAB-dispersed suspension allowed addition of Mg-ions proposed as a substitution for aluminium. EPD experiments were first performed at voltage 30 V/cm using steel electrodes, while in another set of experiments we used 2-dimensional SiC-fibre woven Tyrano SA as an electrode. Finally, 3-D SiC-fiber preform was infiltrated. Both types of the SiC-fiber woven were provided by dr. Alberto Ortona, FN, Italy. The experiments of deposition of SiC on SiC substrates were slower than that on the steel, due to the lower conductivity of the SiC. It is proposed that the deposition could probably be enhanced by applying much higher voltage, e.g. 100 V/cm. The advantageous effect of higher voltage was also found in our previous work, where the density of the deposits increased with increasing voltage. In further work, electric field in the deposition cell will have to be adapted in order to prevent electrolysis in suspensions, resulting in bubbles formation in the deposit. The results of the work performed at Imperial College are summarised in common manuscript of the paper to be submitted to J. Mat. Research (ref.7) and will be presented at the Conf. of the Eur. Cer. Soc. 2007 in Berlin.

The work performed at Imperial College in collaboration with Jožef Stefan Institute hence provided us with the knowledge on the processing of the SiC-suspensions for EPD. The EPD experiments confirmed that the EPD could be an effective tool for the deposition of SiC with sintering additives in SiCSiC performs.



a) *b)*



c) *d)*

Figure 1: (a), (b) Current drop during the deposition of the ceramic slurries of micron-sized and nano-sized powder, respectively; (c) and (d) the corresponding deposits on the SiC/SiC(CVI)

2.2 Low-temperature densification of low-activation SiC-materials for the coating

The proposed material for the coating is a composite of SiC particles bonded with a small amount of secondary phase based on the system SiO_2-XO-P_2O_5, where XO stands for $Al2O_3$ or MgO. As explained in previous reports, selection of this system is based on its' ability to produce transient liquid phase at temperatures below 1500°C, defined as a limited temperature range in order to prevent the SiC-fibers (Nicalon type) from detrimental structural changes. Moreover, this system was also selected due to a significantly lower neutron activation of the elements involved in comparison to the composition used in the NITE, suggested by Japanese group, in particular yttria.

Although the above mentioned system was shown to enable production of dense SiC-based material, the main issue in this system is, however, insufficient reproducibility of the process, frequently resulting in residual porosity. To establish the reason for that, within a diploma work of Mrs. N. Drnovsek thorough sintering studies were performed. Main stress was given to analysis of the oxygen content throughout the processing steps from the powder to the sintered ceramics and to wetting the SiC material with the secondary phases at elevated temperatures. Using IR-spectroscopy (Eltra ON 900-Oxygen Nitrogen Determinator) the oxygen content in the samples was closely inspected throughout the bulk material preparation process. It was found that the processes such as mixing, grinding, suspension preparation, etc. must be performed in glove-box, filled with Ar. Also the firing should be done in the atmosphere with very pure argon (99.9999%). For the illustration, in Fig. 2 the comparison of the chemical composition of the surface layer of SiC micron and nano-particles, determined with XPS is shown. It is obvious that nano-powder contains more oxygen, most probably due to much higher surface than in the case of the micro-powder. It was also found the strong influence of the purity of firing atmosphere (Ar) on the total content of oxygen in the samples. Firing a sample in 99.99 % Ar at 1300 °C resulted in around 3 wt.% of oxygen content in the sample, while after firing in 99.9999% Ar just 1.3 wt.% of oxygen was present in the samples.

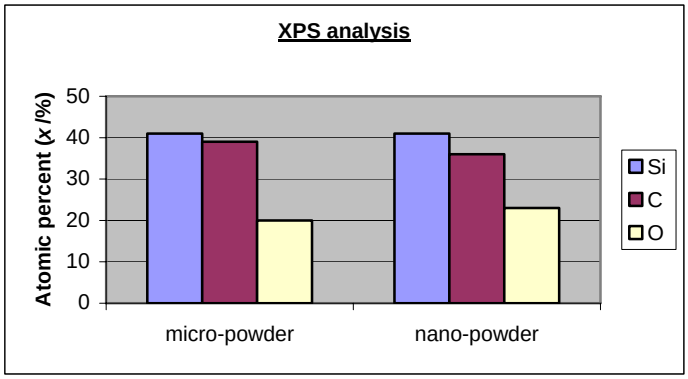


Fig. 2 XPS analysis of the surface layer of micro and nano-powders

For sintering studies various compositions were prepared and wetting angles between liquid phase and SiC substrate were measured using optical dilatometer. It was found that the thickness of oxide layer on the SiC particles plays an important role in

wetting. Figures 3 and 4 illustrate the wetting of the SiC disc (lower, flat sample) with the secondary eutectic phase precursors SiO_2-Al_2O_3-P_2O_5 (Al-p) or SiO_2-MgO-P_2O_5 based (Mg-p), (upper sample) during heating in optical dilatometer. The interface was studied in details by EDX analysis and was reported in diploma work (ref. 8)

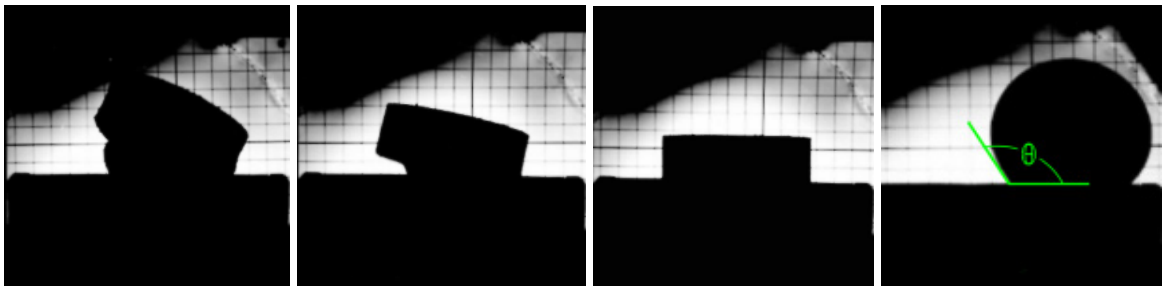


Figure 3: Melting of the secondary phase precursor (SiO_2-Al_2O_3-P_2O_5) and wetting of the SiC-sample in Ar atmosphere (first image at the left is taken at 25°C, before heating experiment, last image at 1155 °C, which is above eutectic temperature for this system).

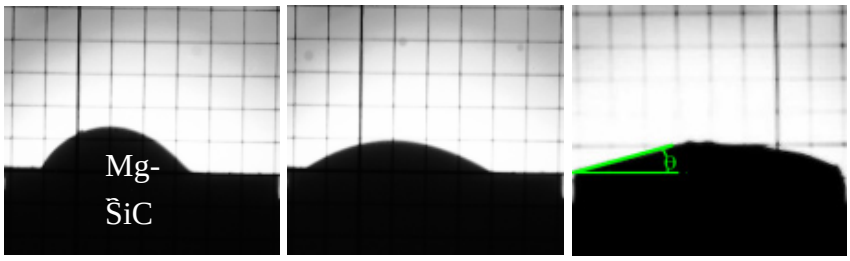


Figure 4: Melting of the secondary phase precursor (SiO_2-MgO-P_2O_5) and wetting of the SiC-sample in Ar atmosphere (from the left: pictures taken at 1312 °C, 1341 °C and 1358 °C).

In order to establish effects of the secondary phase content (amount of sintering additive), sintering temperature and time, the samples with different composition and sintering procedure were prepared. Porosity of the sintered samples was estimated from microstructures of polished samples by SEM and for the selected samples the microhardness was measured. The porosity decreases with increasing amount of the secondary phase, but on the other hand this causes the decrease in hardness as presented in the section “Physical characterisation” (Figure 7). The microhardness also decreases with sintering temperature and with sintering time. In summary, the samples composed of submicron SiC powder and 20 wt.% of the Al-phosphate as sintering additive exhibited the highest hardness by sintering at 1300 °C for 2 hours. The amount of secondary phase could presumably be lowered by application of the sintering additive precursor (Al-p or Mg-p) directly on the SiC powder surface, which will enable better homogeneity.

Comparison of the Figures 3 and 4 suggests that the SiO_2-MgO-P_2O_5 system has much better wetting characteristics than SiO_2-Al_2O_3-P_2O_5 based system. Based on these findings we already performed a few experiments where Al_2O_3 in matrix material was replaced with MgO. At present, the MgO containing samples are quite porous, which appears to be the consequence of evaporation that we are trying to limit by using powder bad during sintering.

In a parallel study we developed a technique for the coating of SiC powders with a thin Al_2O_3 or MgO layer at the surface that will presumably allow us in further work to prepare SiC-ceramics with lower amount of secondary phase, as compared to the present processing technique, where the additives are admixed with the SiC in the form of powders. As mentioned above, the EPD experiments with such coated powders have been performed, while sintering studies are in progress.

2.3 SEM and TEM characterization of coatings

As a part of the collaboration with the ENEA Frascatti, Italy, we coated green preform, composed of Tyrano-SA SiC fiber woven infiltrated with coarse SiC particles, with $SiC\text{-}SiO_2\text{-}Al_2O_3\text{-}P_2O_5$ based material. For the coating we used the micron-sized SiC powder. Prior to coating, the preform was treated with a tenside (SDOSS) in order to improve the wettability and adhesion of the preform with the slurry. The coating was applied by simple dipping the preform into the SiC-based slurry that resulted in formation of rather homogeneous layer. After firing at 1400 °C for 3h in pure Ar (99.9999 %), SEM-EDXS analysis was performed. In Figs. 5a and 5b, SEM micrographs of the cross-section of the coated sample are shown (ref. 10).

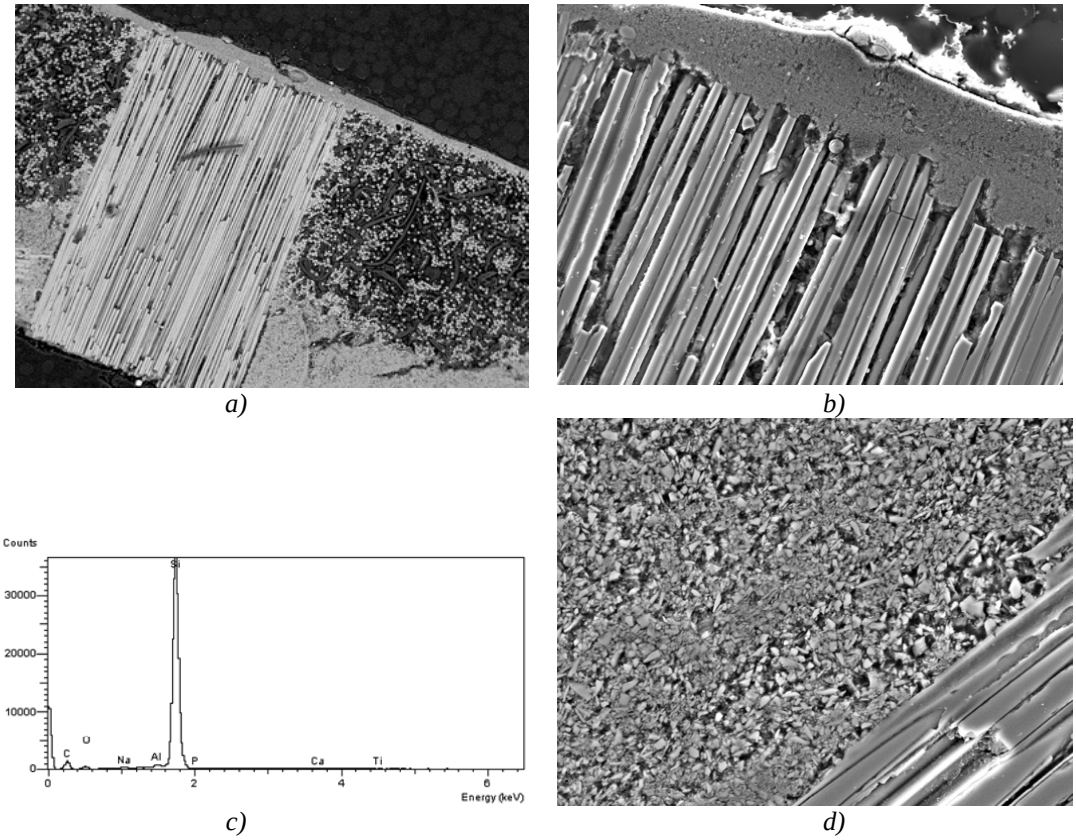


Fig. 5: (a) and (b) SEM micrograph of the cross-section of the Tyrano-SA perform (ENEA) coated with $SiC\text{-}SiO_2\text{-}Al_2O_3\text{-}P_2O_5$ based coating and (c)EDXS spectrum of the coating; (d) the matrix material in the composite after sintering.

The coating is approximately 50 m thick and according to EDXS analysis (Figure 5c), beside Si, C, and some oxygen it contains also very low concentration of Al. As evident, the amount of phosphorus is negligible. For illustration, Figure 5d shows the microstructure of a central part of the sample, suggesting, that the SiC particles introduced into the fiber mat in ENEA (FN), also partly densified by aid of the sintering additive introduced during dipping the sample into the slurry for the coating.

It is obvious from Figure 5b, however, that the SiC suspension did not fill the voids between the fibres close to the surface. For this reason in further step the EPD technique will be used in order to force the SiC particles from the suspension to better fill the voids. We also anticipate better filling the voids by using nano-sized SiC powder or with powder mixture with bimodal size distribution. First experiment with nano-sized powder or composition with 90% submicron and 10% of nano-sized SiC support these presumptions, which is also supported by the fact that the Japanese reference material NITE, developed by Kohyama's group is prepared by nano-SiC powder.

The sintered coating materials were also analysed by transmission electron microscopy (TEM). A characteristic TEM micrograph of a sample with nano-SiC powder is presented in Fig. 6. It is evident that after sintering at 1400 °C the SiC particles size remain nearly unchanged, suggesting that only rearrangement took place during firing. Small mullite particles were also observed in the microstructure. The secondary phase (G) between the SiC particles was found to contain a certain amount of oxygen, presumably resulting from the oxygen containing surface layer at the particles. As it was mentioned above, by using nano-SiC powder coated with AlOOH or MgO, as mentioned before, in further work the amount and the thickness of the secondary phase should be decreased.

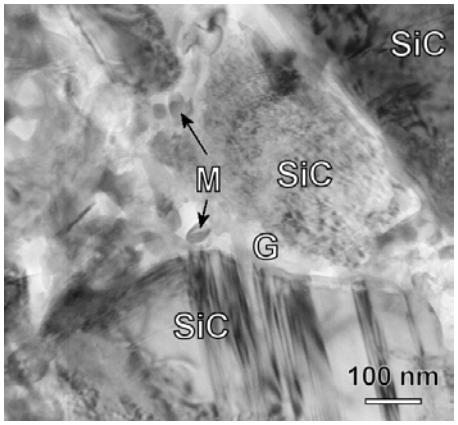


Figure 6: TEM micrograph of the SiC-based coating material, showing SiC particles, the Al_2O_3-P_2O_5-SiO_2 secondary phase (G) and small mullite particles (M).

2.4 Physical characterization of coating material and coated $SiC_{(f)}$/SiC

Since the properties of the coated sample are mostly determined by the properties of the substrate, it was more reasonable to characterise the matrix material itself than the coating on the perform. Hence, the coating material was shaped in small discs, sintered and analysed for the electrical properties at room and elevated temperatures, thermal conductivity, microhardness and elastic modulus. Figure 7 shows

the microhardness of the samples of coating material as a function of composition, sintering temperature and sintering time. The properties of the characteristic samples are collected in Table 1. As presented, the electrical conductivity of the SiC-based coating material is obviously very low at room temperature as well as at 880 °C. Accordingly, thermal conductivity is also quite low.

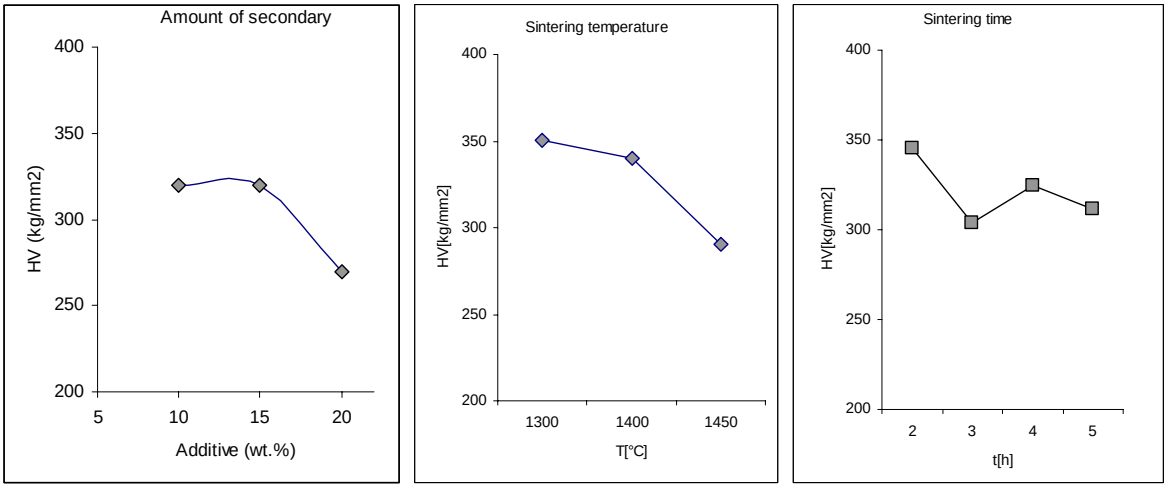


Figure 7: Microhardness of the samples of coating material as a function of starting suspension composition, sintering temperature and sintering time.

	Sample 197*	Sample 174**
Thickness [mm]	6.45	0.9
Resistivity (21 °C) [Ω]	274 x10^{3}	155 x10^{3}
Resistivity (880 °C) [Ω]	1,140	8
Spec. conduct. (21 °C) [S/m]	1.0 x10^{-3}	0.12
Spec. conduct. (880 °C) [S/m]	0.25	2.0
Thermal conductivity [W/mK]	0.83	1.1
Elastic modulus (GPa)	65	48
Microhardness (kg/mm^{2})	350	290

* SiC (submicron powder) + 20% Al-p, 1300 °C
** SiC (nano-powder) + 20% Al-p, 1300 °C

Table 1. Properties of the characteristic samples of the coating material

2.5 Irradiation of the coated samples

In this stage of the investigation, the starting materials, i.e. SiC powders, the transient sintering additives (TSA) and sintered material for the coating were routinely activated and their activation residuals measured at the Reactor Infrastructure Centre of the JSI. Samples were irradiated in central channel of TRIGA Mark II research reactor at fast neutron flux (E > 0.1 MeV) of 8×10^{12} 1/cm^2 s. The total neutron flux in this position was 2×10^{13} 1/cm^2 s and the total neutron fluence was 3×10^{21} 1/m^2. Various coated samples are in the process of irradiation and will be analysed after different periods of time.

3 CONCLUSIONS AND OUTLOOK FOR 2007

- Electrophoretic deposition (EPD) was studied as an alternative way for the production of a coating on a SiC_f/SiC (CVI) composite material. Procedures, compositions and operating parameters were varied with the aim to determine for suitable properties of the deposit. The coating at the SiC-based substrate may be prepared by the deposition from aqueous or ethanol suspension with addition of CTAB or PEI, respectively.
- The formation of eutectic melt in the system SiO_2-Al_2O_3-P_2O_5 and SiO_2-MgO-P_2O_5 and wetting of the SiC with these melts was followed as a function of temperature and composition. It was found that the MgO based secondary phase better wets SiC than the Al_2O_3 based, however, sintering of SiC with this phase results in residual porosity due to evaporation of a volatile phase.
- It was found that in the order to keep the oxygen amount as low as needed the suspension processing must be performed in glove-box with inert atmosphere (very pure Ar). Also the argon atmosphere during firing must be of high purity (99.9999 %).
- As a part of the collaboration with the ENEA Frascatti, Italy, we coated green preform, composed of Tyrrano SiC fiber woven filled with coarse SiC powder. For the coating we used SiC-SiO_2-Al_2O_3-P_2O_5 based coating material. The approximately 50 μm thick coating composed of fine SiC grains embedded in an amorphous secondary phase containing also small mullite particles.
- The specific electrical conductivity of the coating material was measured to be in the range of 10^{-3} to 10^{-1} S/m and increased one or two orders of magnitude at 880 °C. The composition electrical, thermal and mechanical properties of the materials is affected by particle size of the starting SiC powder.
- In continuation, the following steps will be done: SiC powder with bimodal particle size will be used in order to increase particle packing density and hence to minimise the shrinkage of the coating at the SiC/SiC substrate. Experiments with AlOOH-coated and MgO-coated powders should further improve the homogeneity of the sintered material and in particular decrease the amount of secondary phase. This will improve the mechanical, thermal and electrical properties of the coating materials as well as further improve neutron activation characteristics. More stress will be given to MgO-based secondary phase. Larger samples with a thin continuous coating will also be prepared by using both proposed techniques: EPD and dip-coating in order to compare their effectiveness and applicability for the proposed application. Possible application for joining will also be verified.

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NOVEL PROCESSING OF SiC/SiC BY SLIP-INFILTRATION OF SiC FIBRE PRE-FORMS WITH SiC UNDER VACUUM

Goran Dražić[1], Saša Novak[1], Nataša Drnovšek[1], Katja Mejak[1], Tea Toplišek[1], Tomaž Žagar[2], Bruno Riccardi[3] , Aldo Boccaccini[4]

"Jožef Stefan" Institute, [1]Department for Nanostructured Materials, [2]Reactor Physics Department, [3]ENEA Frascatti, Italy, [4]Imperial College London, UK
e-mail: goran.drazic@ijs.si

1. INTRODUCTION

The use of Chemical Vapour Infiltration (CVI) method for fabrication of SiC_f/SiC composite material require very long processing times and moreover, it results in material with high residual porosity. The basic aim of the work in this task was hence substitution of the CVI technique with an alternative method, where the SiC-fiber preform is vacuum-infiltrated with a specially designed ceramic suspension and densified in a way to obtain a dense and impermeable matrix with good adherence to the SiC fibres. Vacuum slip infiltration (VSI) was proposed as a method that offers more efficient way for the preparation of dense and impermeable SiC_f/SiC composites. In our earlier investigations we proved feasibility of the idea to produce a dense SiC/SiC composite by vacuum infiltration of the SiC textile with ceramic slurry of fine SiC powder and low-activation additives, needed for the densification in the temperature range below 1500 °C. The main objective of the work was to reach zero open porosity, good mechanical properties at elevated temperatures, suitable thermal conductivity and the lowest possible neutron activation of the material. The research work in 2006 was focused in improvement of the matrix material by optimisation of its chemical composition and of the efficiency of the infiltration technique by improvement of wetting and infiltration characteristics of the suspension.

2. WORK PERFORMED IN 2006

2.1. Optimisation of the chemical composition of the low-activation matrix material for infiltration

A suspension for infiltration of SiC-fiber woven for production of low activation SiC/SiC composite must meet several quite demanding requirements. First, its composition must allow densification of the material at moderate temperatures, i.e.

below 1500 °C, in order to avoid structural changes (crystallisation) and consequent shrinkage of partly amorphous SiC fibers. Second, the additives for sintering must contain only low-activation elements to bring an advantage over the Japanese NITE material. The amount of the secondary phase in sintered SiC-based ceramics must be as low as possible to achieve best possible high-temperature behaviour. Further, the suspension for infiltration must wet the fibers to efficiently fill the voids between them, for which the viscosity of the suspension must be suitably low, although containing a high amount of the SiC powder, needed to attain high density and low shrinkage of the matrix material between the fibers.

The vacuum slip infiltration (VSI) process is in a way similar to the NITE process for manufacturing of SiC/SiC, since both techniques starts from the powder. In the NITE, however, nano-SiC powder is densified by additives as yttria and alumina, which does not classify the material as low-activation (see Fig.11 below). For this reason, in our work we primarily aim at substitution of yttria with low activation elements for sintering additive. Based on the early studies of phase diagrams, supplemented by studies of activation behaviour of candidate elements for the sintering additives, we showed in previous work that we can approach the above mentioned requirements by using Al-hydrogen phosphate or Mg-phosphate. The phosphorus namely decrease temperature of transient liquid phase during sintering in protective atmosphere and after playing this role it evaporates, resulting in progressively increasing melting temperature of the secondary phase.

Among the main issues in development of the new SiC-based matrix material is strict control of the composition, in particular when nano-sized powder is used. Based on precise analysis of the submicron and nano-sized SiC powders, we found that the amount of SiO_2 at the surface of the starting powder play a major role in densification process. In order to avoid this effect, we put large efforts in modification of the powder's surface with thin layers of AlOOH and/or MgO. The proposed advantage of the coating of SiC powder is twofold: it minimises the amount of SiO_2 and helps to homogeneously distribute a low amount of sintering additives in the material. It was reported recently (Tatli) that the MgO-coating enables sintering of SiC, which is not possible if the MgO is added to SiC in a form of powder. Since magnesium exhibits much lower activation than the aluminium, one of our main activities was development of the coating process and sintering studies of SiC with MgO-containing secondary phase in addition to Al_2O_3-containing system.

2.2. Surface modification of SiC particles and SiC-fibres

Particles' surface modification

For the coating of the SiC powder with AlOOH or MgO, several procedures were verified. Among them, the most effective was the procedure, where Al-nitrate was used as a precursor and by pH adjustment of aqueous SiC suspension, a thin layer was formed at the SiC particles. The coating was followed by thermal treatment and leaching the redundant precursor. Finally, to verify the result, the coated powders were analysed by using two complementary methods: XPS and high-resolution transmission

electron microscopy (HRETM). Figures 1a-c show the submicron SiC particle coated by a few nanometers thick AlOOH and the EDXS spectra for the surface layer, while the composition of the layer at submicron (BF12) and nano-sized (nano) SiC powder, obtained with XPS is presented in Figure 2.

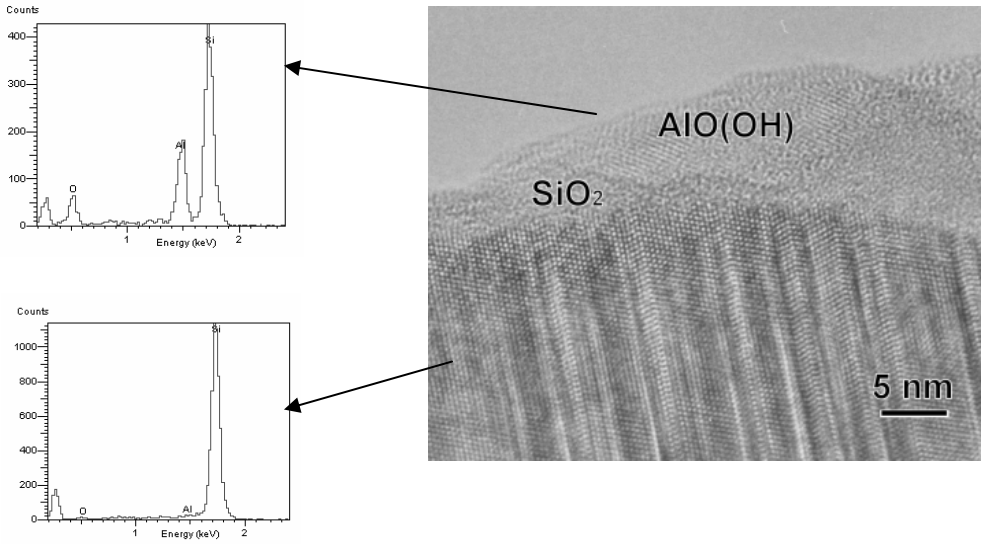


Figure 1: HRTEM image of the AlOOH-coating at the SiC particle and the corresponding EDXS spectra

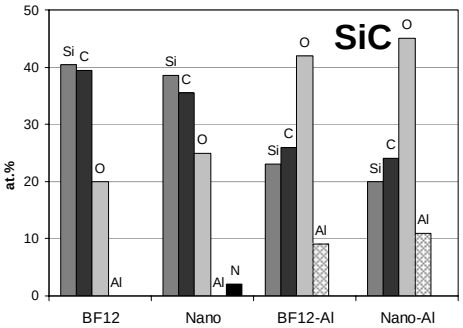
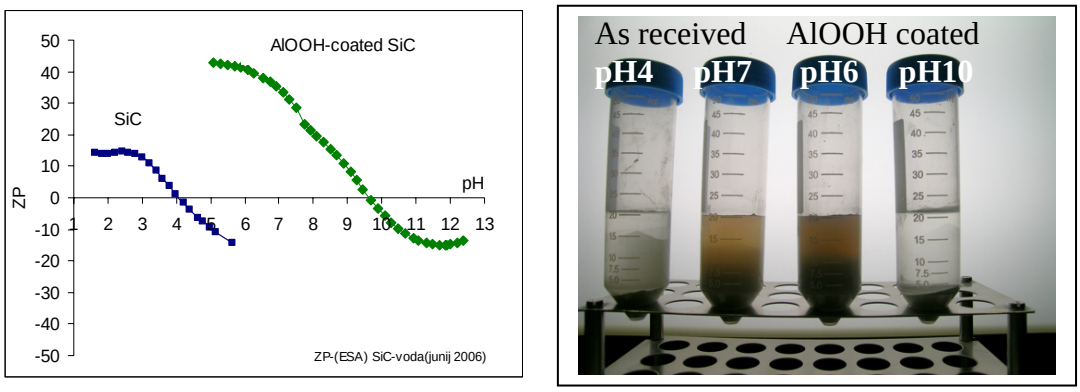


Figure 2: Concentration of elements at the surface region of few nm on as-received SiC powder and on the AlOOH-coated SiC powder showing the presence of Si, C, O and Al (on the coated powder).

From Figure 3, where the zeta potential for non-coated and AlOOH coated SiC powder is show, it is obvious that the thin layer completely changes the behaviour of the SiC powder. The powder that contains only a very small amount of Al, behaves in water as aluminium oxide, which have a beneficial effect on colloidal processing of SiC-based materials. Namely, wet ceramic processing (as slurry infiltration or electrophoretic deposition) requires high zeta potential (ZP) of the suspension in order to get high packing density in green and sintered part. As presented in Fig. 4a, the AlOOH coated powder develop much higher surface charge (ZP>40 mV) than the as-received powder that also reflect in an increased stability of suspension in a neutral pH range (see Figure 3b).

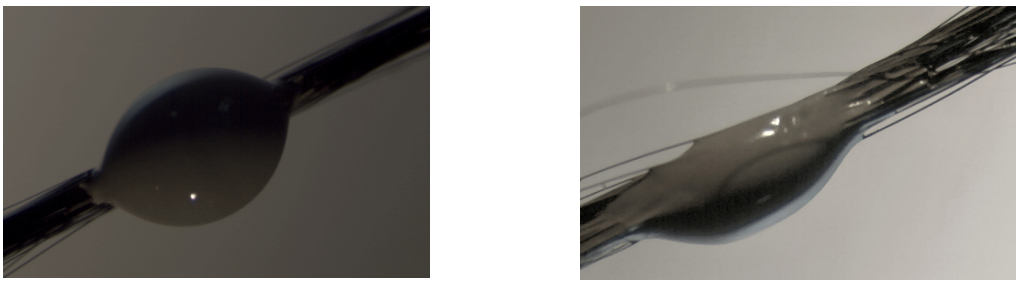


a. *b.*

Figure 3: a) Surface charge (presented as zeta-potential) for the uncoated and AlOOH coated submicron-SiC powder; b) the resulting change in suspensions stability after surface modification of the SiC-powder

Fibers' surface modification

Another important topic in development of slip-infiltration technique to produce dense SiC/SiC is a study of the SiC fibers. In order to improve infiltration of SiC-fibre perform, we analysed the wetting characteristics of the fibres with aqueous SiC-suspension and the effect of different surface active agents. As illustrated in Figs. 6a and 6b, surface modification of the originally hydrophobic SiC fibers with sodium dioctyl sulphosuccinate (SDOSS) considerably improved the wetting with the suspension for infiltration. In contrast to non-treated fibers, a bundle of pre-treated fibers was well infiltrated with the SiC-suspension (Fig. 4b).

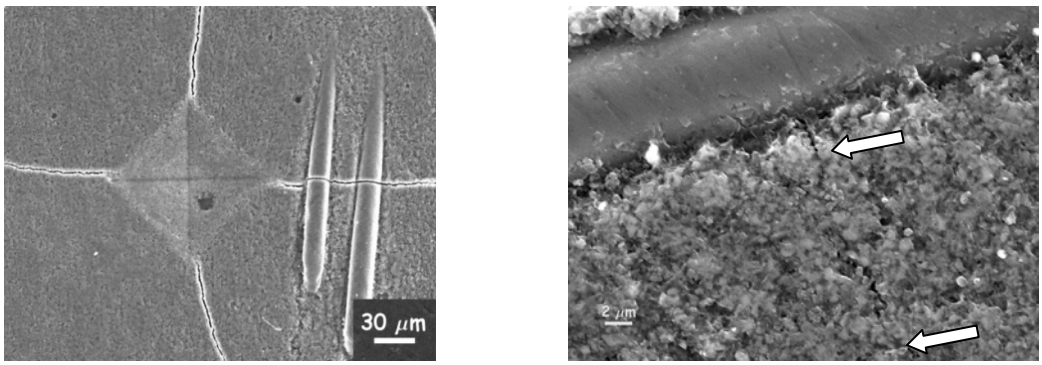


a) *b)*

Figure 4: SiC-suspension on the as-received SiC-fibers (a) and surface modified fibers (b)

As a part of a fractography study of SiC/SiC composites within a PhD thesis of T. Toplisek, the analysis of the surface properties of the SiC fibers were also studied and different options for crack-deflecting interface layers has been tested. Using magnetron sputtering the Nicalon fibres were coated with a thin layer of one or several layers with relevant chemical composition (with regard to the activation, see Figure 11).

First, a few tens of nanometer thick CrN layer was applied and crack propagation studied by observing the microcraks introduced by Vickers indentation. As shown in Fig. 5a, the crack did not stop at the matrix-fibre interface in the sample without interface layer at the SiC fibres, while a 50 nm thick layer of CrN deflected the crack. The CrN, however, appeared to be less suitable due to reaction with matrix material, so that in continuation carbide layers (Cr- and Ti-) and multilayer coatings will be applied and tested.



a) *b)*

Fig. 5: Crack propagation created by Vickers indentation; a) the crack traversing the fibers without interface layer; b) the crack deflecting at the CrN inerphase layer at the SiC fiber

Detailed TEM analysis of the composite without an interphase layer revealed that the SiC fibers are originally coated with a thin SiO_2 protective layer (Figure 6). Although this layer presents a weak interface that could, according to some literature data resulting in debonding of the fiber-matrix interface, the brittle silica layer does not meet the expectations, as illustrated in Fig. 5a.

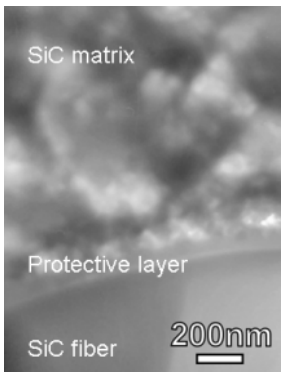


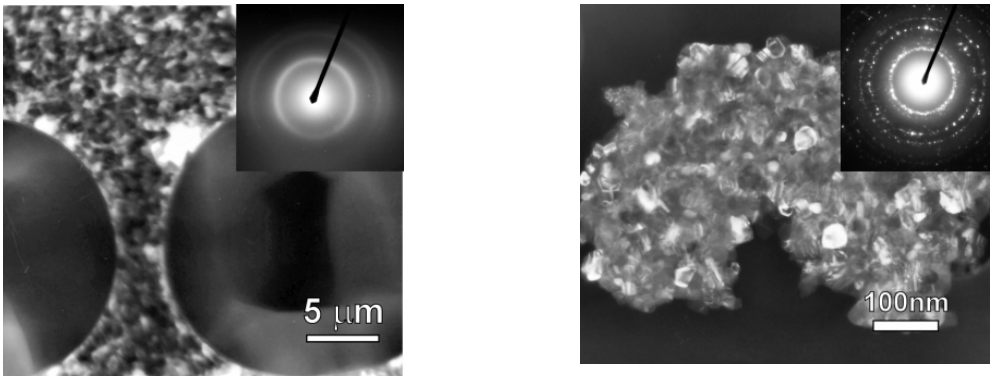
Fig. 6: TEM micrograph of SiC-matrix at the contact with the SiC-fiber, showing the brittle silica layer at the fiber.

2.3. Low-temperature densification of the infiltrated SiC-textile

In order to verify the temperature limit for the sintering of the SiC-matrix material, we analysed the behaviour of the available SiC-fibers (Nicalon S) after heating at high temperatures, in particular their structure. It was confirmed that heating the fibers at temperature higher than 1600 °C caused crystallisation. Accordingly, figures 6a and 6b present TEM micrographs of SiC-fiber preform infiltrated with SiC-P_2O_5-Al_2O_3 composition and heated at 1350 or 1600 °C in Ar for 2 hours. From Figure 7a it is

evident, that after sintering at 1350 °C the fibers surrounded with SiC-matrix remained amorphous (see the inset SAED pattern typical for amorphous materials), while heating at 1600 °C caused crystallisation (see the inset pattern in Figure 7b indicating the crystalline SiC structure). The crystallisation led to brittleness of the sample that also caused breaking of the sample during the preparation for TEM analysis. This confirms that a use of low-temperature regime in sintering is necessary when Nicalon fibers are used.

The Tyrano S fibers were provided only recently by FN, Italy (A. Ortona) and the analysis is still in progress. We also plan to analyse thermal stability of the Tyrano fibers used in NITE material, that should be provided in accordance to the recent agreement with dr. A. Kohyma. It should be pointed out here, that although good thermal stability is an advantage of the Tyrano fibers, their chemical composition represents a drawback due to the presence of yttria.



a) *b)*

*Figure 7: (a) TEM micrograph (bright field) and SAED pattern of fibres fired at **1350 °C***
*(a) TEM micrograph (dark field) and SAED pattern of the fragment of fibre fired at **1600 °C***

We also measured the microhardness of the SiC-composites sintered at 1350 °C in argon. The microhardness for the matrix was 270-385 kg/mm^2 and for the SiC-fibers (polished cross-section) 1300-2600 kg/mm^2. The variation in the matrix material can be ascribed to non-homogeneous amount of secondary phase, while for the SiC fibers the explanation could be the non-homogeneous structure across the diameter.

After infiltration of Tyrano S fiber woven with the same matrix material and sintering at 1350 °C, thick interphase layer on the fibers are well visible, Figure 8. The infiltrated matrix material is well adhered at the fibers as well as it fills even some small voids between the fibers. The as-received perform was already infiltrated with coarse SiC powder (by FN and ENEA, Italy) and further infiltration with a suspension of fine SiC powder with addition of Al-phosphate led to formation a dense ceramics. This result encourage us in presumption that highly dense composite could be obtained by a combination of infiltration with a coarse powder technique, as introduced by FN and ENEA, and further infiltration with the sintering additive containing suspension of fine, nano-sized or bimodal SiC powder.

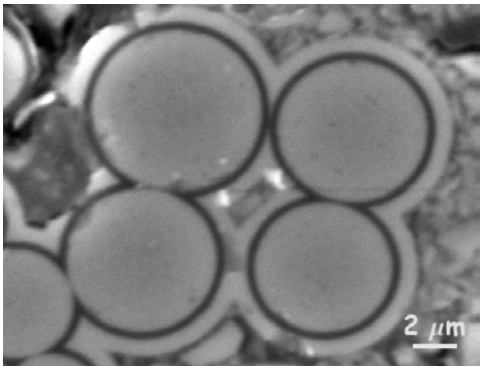
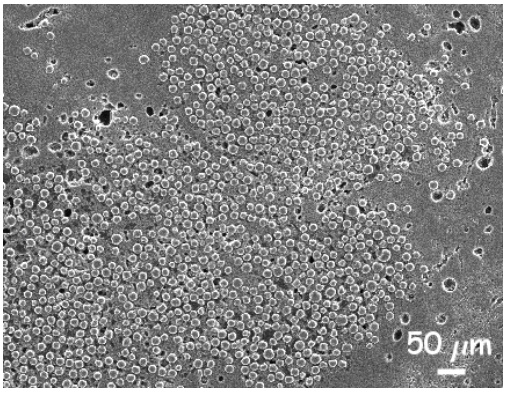


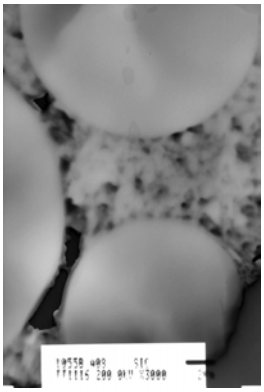
Figure 8: SEM micrograph of slip infiltrated Tyrano S fibre perform. The fibers coated with thick interface layers are surrounded with the matrix materials prepared by infiltration with suspension of submicron SiC and Al_2O_3-based secondary phase

2.4. SEM and AEM characterisation of the $SiC_{(f)}$/SiC samples

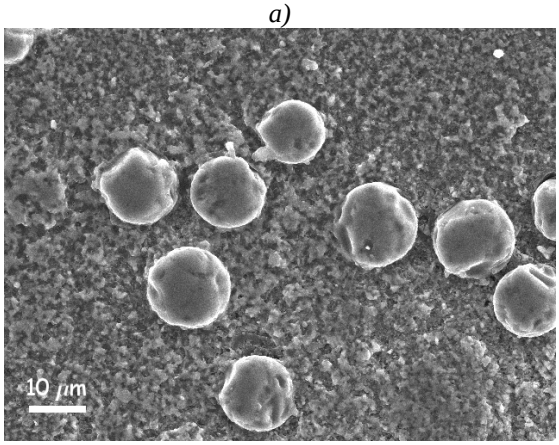
The prepared SiC/SiC samples with different chemical composition and amount of additives were routinely characterized with scanning electron microscopy. Main stress was given to observation of residual porosity, homogeneity and chemical composition. The composite samples were formed by slip-infiltration of Nicalon S fibre preform with SiC-suspension containing 20% Al-dihydrogen phosphate. As a novel technique - electrophoretic deposition was also tested for the infiltration of 2-D and 3-D SiC-woven. The study of the EPD technique was performed at Imperial College in the frame of "mobility" (dr. S.Novak).



a)



c)



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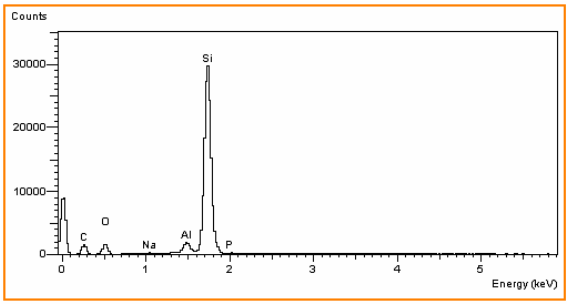
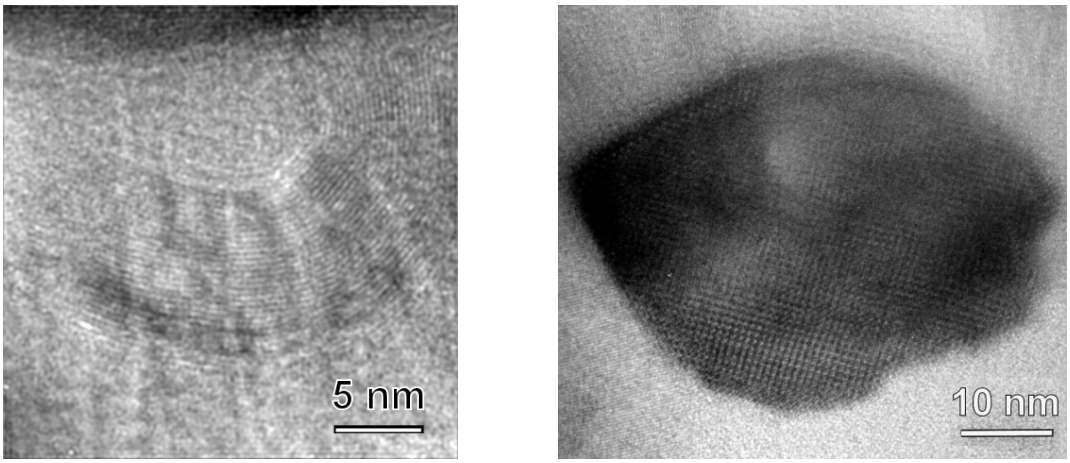


d)

Fig. 9: SEM (a,b) and TEM (c) image of a SiC/SiC sample fired at 1400 °C in argon; EDXS spectrum of matrix phase (d)

Figs. 9a-c show polished surface of a characteristic sample where bunch of SiC fibres were first pre-treated with SDOSS and impregnated with matrix suspension prepared from submicron SiC powder with addition of Al-phosphate. It is evident (Fig. 9a), that the remaining porosity is very low and that only closed pores are present - at some places the matrix material did not completely fill the voids between the fibres. SEM and TEM micrographs confirm a very good contact between fibres and matrix. The composition of the matrix material analysed by EDXS (Fig. 9d) revealed the presence of a small amount of aluminium and negligible amount of phosphorus. Detailed analysis of the secondary phase was also performed by using XPS and HRTEM. The analyses revealed, that the SiC particles are embedded in secondary phase composed of Si, O and a small amount of Al and P. Beside the silica-rich phase with a layered structure (Fig. 10a), mullite particles were also detected in the secondary phase (Fig. 10b). The structure of this phase is a topic of further study focused in structural engineering.



a. *b.*

Fig. 10: HRTEM micrographs of nanoparticles in the secondary phase of the SiC-matrix material (a. – layered silicate, b. – mullite)

2.5. Irradiation and activation analysis of the SiC-based samples

In order to establish a list of the elements that may be used in sintering additive and the range of acceptable amounts, we performed a calculation of neutron activation expected in a fusion flux. The results are presented in Fig. 11 as maximum allowable concentration in wt.% of the element.

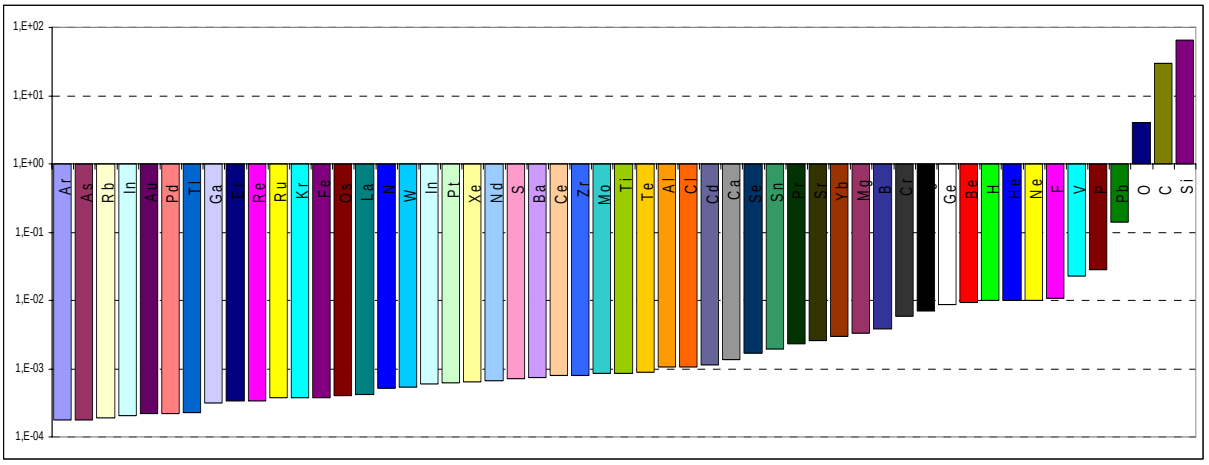


Fig. 11 Maximum "allowable" concentration of individual element (in wt. %)

Irradiation analysis of the infiltrated SiC/SiC samples in comparison with Nicalon and Tyrano SiC fiber performs, SiC/SiC (CVI) composite and also with Eurofer that was recently provided from FRG Petten, will be finished with the first months of 2007, while the NITE material will be irradiated and analysed later, since the samples has not been delivered yet.

3. CONCLUSIONS AND OUTLOOK FOR 2007

- The matrix material for infiltration has been developed and used for infiltration the Nicalon and Tyrano SiC-fibre perform. The properties of the matrix material were found to highly depend on the oxygen content at the powder's surface as well as on the purity of the argon atmosphere during densification processes.
- In order to decrease the amount of secondary phase in the matrix and to improve the homogeneity of the phase distribution, the procedure for the application of the sintering additive precursor directly on the SiC powders was developed. The advantageous effect of AlOOH-coating was confirmed, while sintering of the MgO-containing compositions requires further improvement in order to prevent the evaporation and the resulting residual porosity. Further work will be more focused in the MgO-based secondary phase as it may further improve the neutron activation behaviour.
- Pre-treating the fibers with surfactant resulted in improved wettability with the slurry for infiltration. The Nicalon and Tyrano fiber-preforms were infiltrated and sintered at 1300-1400 °C. The analysis confirmed good adhesion of the matrix material with SiC fibers. Some remaining closed porosity, observed mostly in the central parts of thick performs, will have to be eliminated by adapting the vacuum infiltration technique.
- As an alternative technique for infiltration, the electrophoretic deposition was analysed. The part of the study was performed in collaboration with Imperial College

London within the staff mobility programme, at the end of the year 2006. The suspension for electrophoretic infiltration was developed and infiltration experiments performed, while the major part of the microstructural analysis will be done at JSI.

- Coating the fibres with a thin layer of one or several materials with fusion-relevant chemical composition and structure and the fractographic analaysis of their effect is in progress. The CrN, although appropriate in respect to the neutron activation, was found to react with the SiC-based matrix, while better results are expected with the carbide-based mono- or multilayer coatings.
- The progress of the SiC-fibers development within the last implies that the fibers are more and more thermally stable which suggests that possibly the initially set limitation on the sintering temperature at 1500 °C is not justified anymore. Moreover, the proposition for introduction of Tyrano fibers or NITE material as a reference, also put in doubt the basic requirement for using low-activation materials.

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MCNP (NEUTRON PHOTON) CALCULATIONS FOR GAMMA-RAY CAMERAS - NEUTRON ATTENUATORS PROJECT

Igor Lengar, Andrej Trkov, Luka Snoj, Bojan Žefran

"Jožef Stefan" Institute, Reactor Physics Division
e-mail: igor.lengar@ijs.si

1 INTRODUCTION

A coherent set of upgrades of the gamma-ray spectrometry systems are planned at JET in order to better support the experimental program. In particular, by using adequately chosen neutron-attenuating materials to reduce both the neutron background, and the neutron-induced gamma-ray background, the measurements of the fast ions could be performed in every JET discharges, even those with the highest neutral beam power. With the existing diagnostics capabilities the gamma-ray signal is completely wiped out by the neutrons even in JET discharges using a few MW's of neutral beams.

The aim of the larger project is the installation of attenuators for neutrons, which should shield the KN3 camera (neutron-gamma vertical camera at JET). The attenuator material is water with a thickness of 24 cm along the line of sight. The task for the particular project was to estimate the influence of the proposed KN3 neutron attenuators on the neutron field. The calculations were performed on the basis of Monte Carlo method using the MCNP code. The three major problems addressed were:

- attenuation factors for neutrons and gammas in the neutron attenuator
- neutron induced gammas in the neutron attenuator
- possible inscattering of neutrons into the detector region from the attenuator in the parking position.

The most important of the problems was the last one, addressing the question, whether neutron measurements are disturbed, when the planned attenuator is not in working position but moved from the line of sight. The attenuator is namely used only for gamma measurements.

2 WORK PERFORMED IN 2006

The original model used for the calculations was a well tested MCNP model, developed on JET. The model was then modified in accordance with the needs of the calculation, i.e. a neutron attenuator was inserted. The sector of interest of the MCNP model with the inserted neutron attenuator is presented in Figure 1

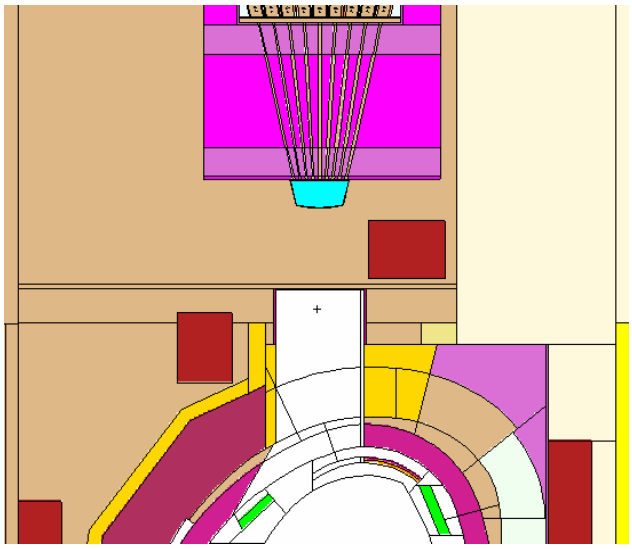


Figure 1: Cross-section of the region of interest of the JET tokamak (MCNP model). White color presents in the figure the vacuum part of the tokamak with the vertical vacuum port. The gamma detectors are in the central uppermost part of the figure. The neutron attenuator is presented in blue color.

After initial calculations, it has been established, that an exact calculation of any of the three tasks through a straight-forward Monte Carlo method is not possible due to the severe degradation of the neutron flux from the plasma to the detector region. The attenuation the flux is of the order of 10^{-9} what is schematically presented in Figure 2. The flux below the vacuum port is still far from our detector region, which is shielded by another 140 cm of borated concrete, accounting for further attenuation despite the flight tubes, which are seen in Figure 1.

The most effort was thus spend in finding appropriate approximations for the calculations of the three desired results.

2.1 Neutron attenuation factors and induced gammas

Calculations were performed for a monodirectional flux below the attenuator, oriented parallel with the flight tube, with a spectral distribution as established at the entrance surface of the neutron attenuator. Due to this simplification the statistical accuracy of the results was greatly improved. The reason is that in a dispersed neutron beam only few neutrons pass through the narrow flight tube from the attenuator to the detector region. Through the simplification of transporting a beam parallel with the flight tube, only the neutrons, that would reach the detectors, are actually dealt with. The results showed an attenuation factor of approximately 100 for 2.45 MeV neutrons and induced gamma ratio of 10-5 per incident neutron. The main induced gamma line corresponds to thermal neutron capture on hydrogen in water. The permeability of plasma gammas ranges from 20% for 1 MeV gammas to 60% for 10 MeV gammas and would be somewhat better in the case of the usage of an attenuator, filled with heavy water.

2.2 Estimation of the in-scattering

Estimation and an upper limit for the effect of scattering neutrons into the detector region from the attenuator, placed in parking position was set.

The purpose was to estimate, how many neutrons possibly scatter from the neutron attenuator in the parking position into the detector as a comparison to the case, when no neutron attenuator is present. Due to the fact, that with a straight forward method, the task could not be accomplished, it was addressed in an indirect way.

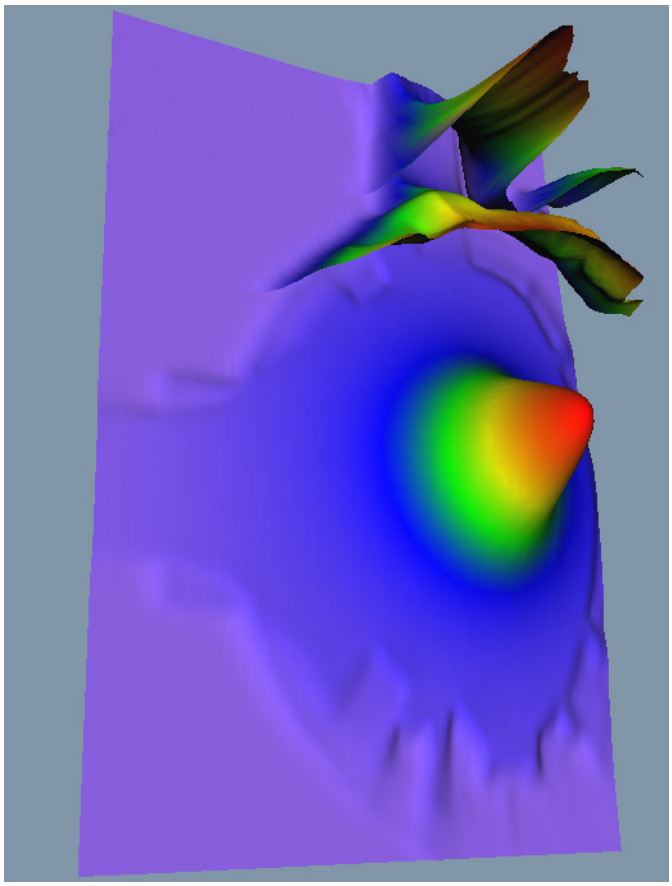


Figure 2: Schematic presentation of the attenuation of the neutron flux in the torus and the vertical port. In the figure three different scales are used to present the flux starting from the highest flux in the plasma (lowest in the figure) to the flux below the vacuum port (the scale is 10^4 times larger). The contour of the torus is clearly seen as the shape of the neutron intensity.

The attenuator is not in the line of sight of any of the detectors and any part of the attenuator is from the view of the detectors protected with a layer of at least 50 cm of borated concrete. Direct inscattering is thus not possible and a neutron has first to pass through a thick layer of concrete before it reaches the detector, or alternatively it has to be a second time scattered from the shield material.

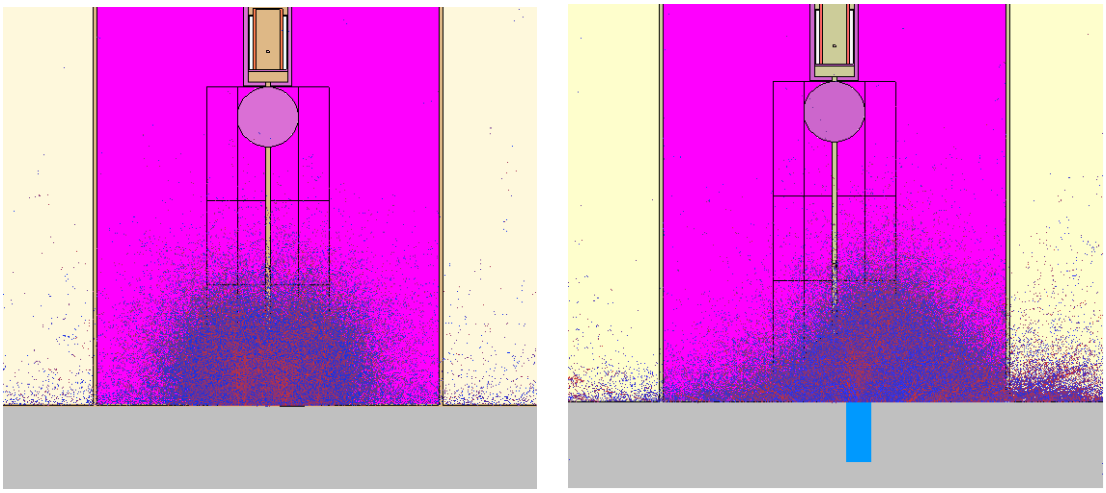
The possible positions of the neutron scattering events which can lead to a hit in the detector have thus been studied for the general case of the plasma neutron source. From the results a qualitative understanding of the scattering situation could be acquired and the conclusion could be made, that most of the scattering events in the concrete shield, that lead to the neutrons scattering into the detector, appear near the corresponding flight tube. The possible scattering positions for neutrons, exiting the neutron attenuator, will be similar, and neutrons will be a second time scattered into the detectors from the region around the corresponding flight tube.

The total neutron flux and the flux of neutrons, scattered from the attenuator, is presented in Figure 3.

The calculations have been preformed for two cases:

1. neutron flux originating directly from the plasma
2. the flux scattered from the attenuator in the parking position

The ratio between the fluxes in both cases is thus assumed to be representative for the amount of *additional* inscattering from the attenuator. It has been found, that this ratio in any region of the shield does not exceeds the value of 3%. The highest values are obtained in cells with low value of the flux where the lower statistical accuracy contributes to the overall ratio. The value of 3% was thus set as the upper limit for the additional inscattering, which can be attributed to the positioning of the neutron attenuator in the parking position.



(a) (b)

Figure 3: Neutron flux of plasma neutrons in the KN3 neutron shield without the presence of a neutron attenuator (a) and the flux, scattered from the neutron attenuator (b). The flux is presented graphically in the form of neutron scattering points. The scale for the number of points in Figure (b) is 100 times the scale in Figure (a) (the real flux is smaller in Figure (b)).

3 CONCLUSIONS AND OUTLOOK FOR 2007

Three main problems were addressed in connection with the disturbance of the neutron flux in the KN3 detector system through positioning of neutron attenuators – attenuation factors for neutrons and gammas, induced gammas in the neutron attenuator and inscattering into the detector from the attenuator in the parking position.

Monte Carlo method was employed using the MCNP code. Exact calculations were not possible due to the severe degradation of the neutron flux from the plasma to the detector region and the corresponding low Monte Carlo statistical accuracy. All results were thus obtained through indirect estimations.

The main task of the project was the estimation of the neutron scattering from the attenuator, placed in parking position, into the detector region. It has been found, that the value of additional innscattering from the attenuator in the parking position amounts to less than 3% of the overall inscattering into the detectors from the shield.

The attenuation factors of the attenuator for DD neutrons were found to be roughly 100 and the induced gammas for more than another factor of magnitude lower. The transitivity of the “plasma” gamma rays varies with gamma energy and amounts to approximately 20% at 2 MeV to some 60% at 10 MeV.

It should be noted, that the estimations for the amount of the induced gamma rays were performed in simplified geometry. In 2007 the project should continue with the emphasis on the calculations of the induced gammas also for other approximations of the incident neutron beam and verifying the present results.

SUSD3D DETERMINISTIC CROSS SECTION SENSITIVITY/UNCERTAINTY PRE-ANALYSIS OF TBM – HCLL NEUTRONICS EXPERIMENT

I. Kodeli, A. Trkov, B. Žefran, I. Lengar, S. Slavič

Jožef Stefan Institute, Rector Physics Division, Jamova 39, 1000 Ljubljana, Slovenia
e-mail: Andrej.Trkov@ijs.si

1 INTRODUCTION

A new benchmark experiment is under preparation at the Frascati Neutron Generator (FNG) 14 MeV facility [1, 2, 3] in cooperation between the ENEA Frascati, FzK, TUD and IJS. The NEA Data Bank also participates in this study to perform computational studies as well as to assure the final compilation and dissemination of the data in order to make the results of this valuable work (financed partly by EU funds) easily available to international community.

The objective of this new experiment is to study the Helium Cooled Lithium Lead (HCLL) Tritium Breeding Blanket Module (TBM) concept, which together with the HCPB concept (already analysed in 2005 FNG experiment) represents a candidate for the blanket module design for the DEMO plant. Test blanket modules of both designs will be tested in ITER. In the HCLL design helium is used for heat removal, and lithium lead plays the role of breeder and multiplier. The FNG experiment will contribute to the validation of the computational tools and nuclear data needed for the neutronics analysis. In this study the neutron fluxes, tritium production rate (TPR) and detector activation rates were calculated, together with their sensitivity to the underlying cross sections and the corresponding uncertainties using the deterministic approach. The study contributes to the understanding of the neutronics properties of the HCLL mock-up and permits us to assess the uncertainty on TPR due to the uncertainty in the basic nuclear data. This information will be used to anticipate the relative merits obtained from the experiment and thus guide and optimise its design, and to assess the nuclear data needs.

2 HCLL BREEDING BLANKET MOCK-UP MODELLING

The TBM mock-up geometry is shown in Figure 1 and consists of 12 stainless steel layers, each 0.65 cm thick, with 3.6 cm thick layers of LiPb bricks in between. Seven detector positions are planned between 3.7 cm and 27.7 cm deep in the central brick layer to measure the Tritium production rate (TPR) using Li_2CO_3 pellets.

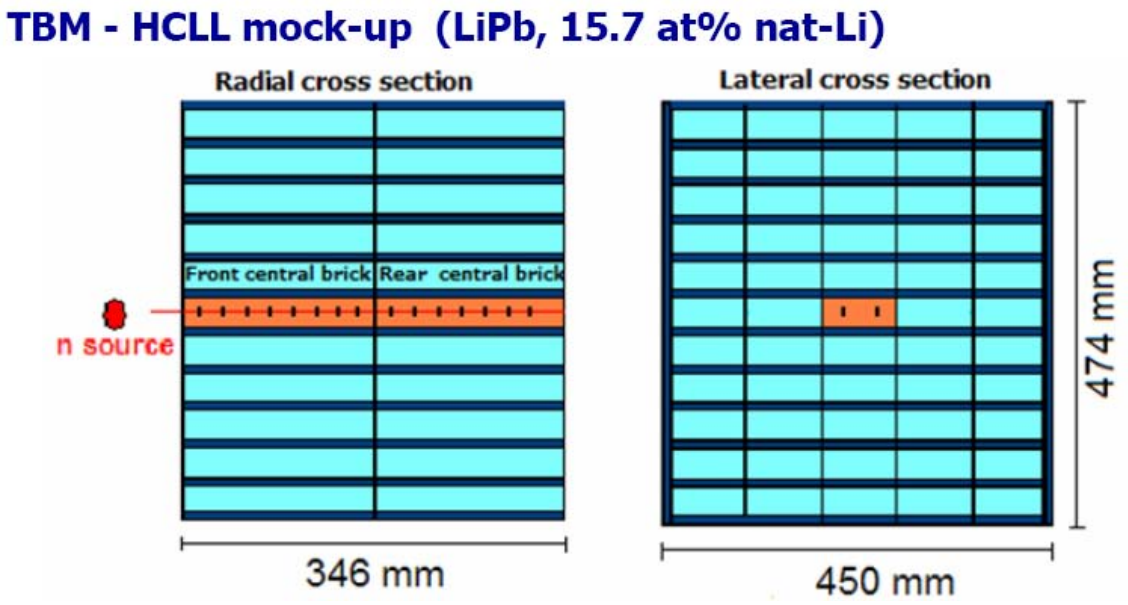


Figure 1: Schematic diagram of the TBM - HCLL mock-up showing sttel (dark blue) and LiPb (light blue) layes. The LiPb test zone is marked orange.

The modelling of the TBM HCLL mock-up geometry requires full 3D description to achieve the required accuracy of calculations. However, a 2D approximate model was developed and validated for certain sensitivity studies because the full 3D TORT calculation requires several days of CPU on a Linux computer, compared to a few minutes for 2D DORT analysis. Like in the previously analysed HCPB Tritium Breeder Module Mock-up Benchmark experiment, the 3D calculational procedure was based on the following codes:

- TORT [4] discrete ordinates transport code for 3D geometries,
- GRTUNCL-3D [5] code to prepare uncollided and 1st collision neutron source for TORT,
- SUSD3D [6] code for the cross section sensitivity and uncertainty analysis.

The SUSD3D code performs sensitivity/uncertainty analysis and assessment of uncertainties in calculations using discrete ordinate approach based on the first-order perturbation theory where sensitivity coefficients are derived from the direct and adjoint flux moments calculated by one of the discrete ordinates codes.

The preparation of cross section for deterministic codes is case-dependent, particularly due to the self-shielding phenomena, which are different than in the previous experiments. Among the available multigroup cross-section libraries for deterministic codes the FENDL-2 and -2.1 libraries [8] dedicated to fusion applications were found the most suitable; the latest FENDL-2.1, was used in these analyses.

The response functions from IRDF-90 and the recently released IRDF-2002 [9] data were processed.

The covariance matrices of lead were needed for the uncertainty analyses in addition to those already processed for the previous experiments. The most recent data were found to be available in the ENDF/B-VI.8 evaluation and were processed using the NJOY-99 code [10].

3 RESULTS OF TRANSPORT CALCULATION

The 3D transport calculations using the TORT code required up to several days of CPU. Both direct and adjoint calculations were performed, adjoint ones being even more time consuming (~2 days runs). The calculated TPR from the ^{6}Li(n,t) and ^{7}Li(n,nt) reactions at the 7 planned detector positions are shown in Figure 2. The results are consistent with those reported in [1], obtained using the MCNP Monte Carlo code. Likewise, the recommendation given in [1] to use enriched Li instead of the natural one is confirmed, leading to smaller gradients between the front and back detector positions and in this way assuring that the TPR is above the detector limit for the deep detector positions.

The fast flux contribution to the TPR using natural Li is important in the first detector positions due to the TPR from the high energy ^{7}Li(n,nt) reaction, as evident from Table 1, which gives the ratio between the TPR from the reactions on ^{6}Li and ^{7}Li. In the case of enriched Li most of tritium is produced by the ^{6}Li(n,t) reactions sensitive to thermal neutrons.

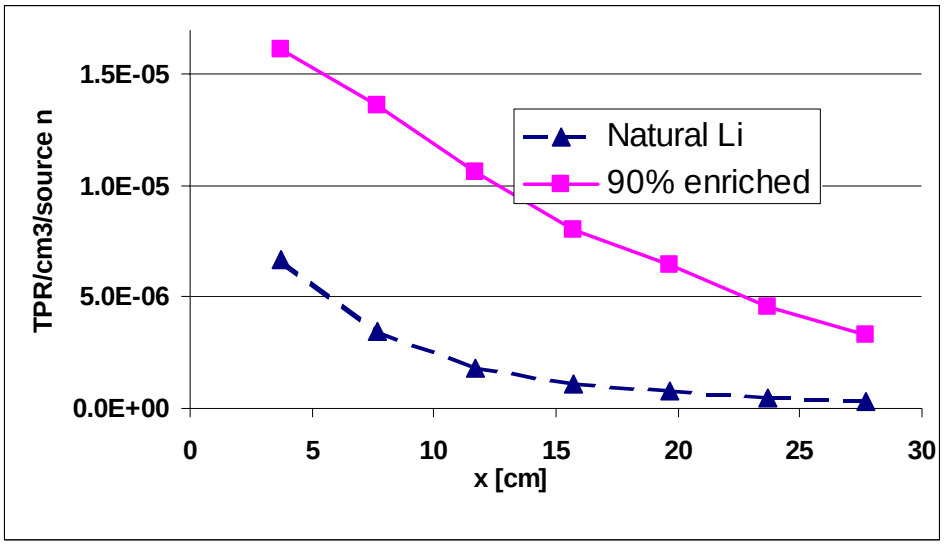


Figure 2: Tritium production rate for the natural and enriched Li pellets.

Table 1: Contribution of ^{6}Li(n,t) and ^{7}Li(n,nt).

^{6}Li(n,t)/ $^{6+7}$Li(n,t)		
Y(cm)	Natural	90% enriched
3.7	19 %	96 %
7.7	32 %	98 %
11.7	48 %	99 %
15.7	62 %	99 %
19.7	69 %	100 %
23.7	77 %	100 %
27.7	80 %	100 %

4 RESULTS OF CROSS SECTION SENSITIVITY-UNCERTAINTY ANALYSIS

The cross section sensitivity and uncertainty calculations were performed using the SUSD3D code, which requires as input the direct and adjoint angular moment fluxes calculated by the Discrete ordinates codes (DORT or TORT), as well as the transport cross sections and covariance matrices for the relevant materials and reactions. Both 3D and 2D sensitivity and uncertainty analysis were performed, using respectively fluxes calculated by the TORT code and from DORT simplified geometry calculations. Good consistency between the 2D and 3D calculations was observed, demonstrating again that the sensitivity analysis does not require extremely precise models; this can be explained by the fact that the sensitivities are derivatives and as such relative quantities.

The sensitivities and uncertainties in the calculated TPR for two detector positions using 2D and 3D computational model are presented in Tables 2 and 3. The sensitivities at the D2 position correspond to the use of natural and 90 % enriched Li; those at the D7 position are relevant for the case of natural or enriched Li.

We can also observe that the sensitivities to the Pb cross sections are relatively low (below 1% of change in TPR per 1% of change in cross section). The most sensitive reaction is the elastic scattering on Pb. However, this is not the reaction causing the largest uncertainty, since the corresponding cross section uncertainty is relatively low. On the other hand, the precision in the (n,2n) and (n,3n) reactions on Pb isotopes is poor; consequently most of the overall uncertainty in the calculated TPR is caused by the uncertainty in these reaction cross sections.

Comparison of the sensitivities at the front detector positions (D2) for the natural and 90% enriched Li in Table 3 indicates rather large differences in the sensitivities. In particular, in the case of using natural Li in the front positions the sensitivity to the (n,2n) reaction on Pb is negative, and positive in the case of using enriched Li and for deep positions (both for natural or enriched Li). This has an important and very beneficial consequence for the cross section validation studies. Use of both enriched and natural Li provides different (and complementary) information on the cross sections and would contribute to the validation of the data at different energy ranges and for different nuclear reactions.

Table 2: Sensitivity of TPR to cross-sections for two detector positions (SUSD3D results based on the TORT 3D neutron fluxes).

^{6}Li(n,t) D7 (~27 cm)	Sensitivity (% / %)				
Reaction	^{206}Pb	^{207}Pb	^{208}Pb	^{6}Li	^{7}Li
Total	0.27	0.25	0.42	0.96	0.07
El.	0.16	0.16	0.37	0.004	0.06
Inel.	0.08	0.06	0.002	$-5\cdot10^{-4}$	
(n,2n)	0.03	0.02	0.06		
(n,3n)	$7\cdot10^{-5}$	$4\cdot10^{-5}$	$6\cdot10^{-4}$		
(n,t)				0.96	0.01
(n,γ)	-0.004	-0.003	-0.001		
Uncertainty	2.1 %	2.0%	6.4%	0.2%	0.1%

Table 3: Sensitivity of TPR to cross-sections for two detector positions. Both natural and 90% enriched Li were considered (SUSD3D results based on the DORT 2D neutron fluxes).

$^{6+7}$Li(n,t) Li NAT. D2 (~7 cm)	Sensitivity (% / %)				
Reaction	^{206}Pb	^{207}Pb	^{208}Pb	^{6}Li	^{7}Li
Total	0.02	0.02	0.01	0.31	0.69
El.	0.04	0.04	0.09	1E-3	0.01
Inel.	0.01	0.01	-0.003	-3E-4	-1E-3
(n,2n)	-0.03	-0.03	-0.08		
(n,3n)	$2\cdot10^{-5}$	$1\cdot10^{-5}$	$2\cdot10^{-4}$		
(n,t)				0.31	0.68
Uncertainty	0.9 %	0.8%	3.0%	0.9%	0.1%
$^{6+7}$Li(n,t) Li-90% enr. D2 (~7 cm)	Sensitivity (% / %)				
Reaction	^{206}Pb	^{207}Pb	^{208}Pb	^{6}Li	^{7}Li
Total	0.29	0.25	0.50	0.97	0.06
El.	0.13	0.13	0.28	$3\cdot10^{-3}$	0.04
Inel.	0.07	0.05	0.03	$7\cdot10^{-5}$	0.01
(n,2n)	0.10	0.08	0.19		
(n,3n)	$1\cdot10^{-4}$	$6\cdot10^{-5}$	$1\cdot10^{-3}$		
(n,t)				0.97	0.02
Uncertainty	2.0 %	1.8%	9.7%	0.3%	0.1%
^{6}Li(n,t) D7 (~27 cm)	Sensitivity (% / %)				
Reaction	^{206}Pb	^{207}Pb	^{208}Pb	^{6}Li	^{7}Li
Total	0.31	0.27	0.48	0.96	0.07
El.	0.17	0.17	0.37	0.004	0.06
Inel.	0.09	0.07	0.01	$-3\cdot10^{-4}$	
(n,2n)	0.05	0.04	0.10		
(n,3n)	$7\cdot10^{-5}$	$4\cdot10^{-5}$	$6\cdot10^{-4}$		
(n,t)				0.96	0.01
(n,γ)	-0.004	-0.003	-0.001		
Uncertainty	1.9 %	1.8%	6.5%	0.2%	0.1%

5 CONCLUSIONS

DORT and TORT/GRTUNCL-3D/SUSD3D computational scheme was used in the pre-analysis of TBM-HCLL benchmark mock-up on the FNG facility. The following main conclusions can be drawn from this study:

- Large and potentially advantageous differences in the sensitivities of TPR to the Pb cross sections between natural and enriched Li pellets in the front detector positions were observed. Combined use of natural and enriched Li pallets is

therefore strongly recommended for the TPR measurements at the front detector positions, if the neutron flux levels are sufficient for the measurements.

- Using the ENDF/B-VI.8 covariance matrices, the uncertainties in TPR due to cross section uncertainties were estimated to be between 5 – 10 %. The TPR values are most sensitive to the Pb elastic scattering cross sections, but these are known with a fairly high accuracy. The reactions (n,2n) and (n,3n) on Pb cause the highest uncertainty, although the corresponding sensitivities are not relatively low. Inelastic cross sections were also found to be important for some Pb isotopes.

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PUBLIC INFORMATION

Saša Novak, David Jezeršek

Public Information, Association EURATOM-MHEST
e-mail: sasa.novak@ijs.si

1 Internet

- Web page of Slovenian fusion association EURATOM-MHEST www.sfa fuzija.si editing
- Web page “Unofficial ITER fan club” *(January 2006)*
- New domain of "Unofficial ITER fan club" - iterfan.org *(November 2006)*

2 Public information material:

- Translation of the EFDA JET web page (J. Mlynar)
- Translation of the brochure “Cleaner Energy for the Future” (A. Dauermaier)
- Translation of the subtitles to the film (H. Desmedt)

(January - December 2006)

3 Expositions, posters, lectures

Figure: Permanent Fusion Expo at Nuclear Technology Information Centre (ICJT) is a much visited destination for secondary school students.

- Permanent Fusion Expo at Nuclear Technology Information Centre (ICJT)
- Lectures to the secondary schools (L. Snoj et al): In the year 2006 there were 14 visiting schools, 629 visitors altogether and in the year 2007 till April 23th there were 7 visiting schools, with 257 visitors.
- Lecture »ITER, opportunity for Slovenian Industry« during the Meeting »Opportunity for Industry«, »Stefans' Days«, J. Stefan Instutute, (given by M. Čerček); present: Minister for Science, high education and technology Dr. J. Zupan, (ex-) Minister for development (dr. J. P. Damjan), National TV *(March 23th 2007)*
- Invited lecture “Fusion, energy for the future” at the national conference “Slovenski kemijski dnevi” (Slovenian Chemical Days) *(September 2006)*
- Lecture about fusion by Luka Snoj at "Študentska arena" *(October 2006)*
- Lecture by Milan Čerček at Materials and Technologies conference "Raziskave fuzije v Sloveniji – stanje in načrti" (Fusion research in Slovenia, state of the art and future plans) *(October 16-18th 2006)*

- Poster by David Jezeršek at Materials and Technologies conference "Unofficial ITER fan club – An opportunity to be a part of international fusion community" *(October 16-18th 2006)*

4 Meetings, events

- Participation at the Public information group meeting, Vienna *(April 4th, 5th 2006)*
- Press-releases and reports on the Launch event *(May 24th 2006)*
- Organisation of the Information day ”ITER- Opportunities for industry” Web page – For industry *(June 2006)*

5 Popular articles

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- NOVAK, Saša. "Reaktor ITER je priložnost za slovenska podjeta" in Finance, 2006, št. 13, p. 24.

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1 Journal articles

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[2] T. Gyergyek, M. Čerček, A fluid model of the current-voltage characteristics of an electron emitting electrode immersed in a two electron temperature plasma. To be published in The European Physical Journal D.

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