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# **BOOK OF ABSTRACTS**

**5<sup>th</sup> IEA INTERNATIONAL WORKSHOP ON BERYLLIUM  
TECHNOLOGY FOR FUSION**

Moscow, Russia, October 10-12, 2001



**A.A. BOCHVAR RESEARCH INSTITUTE  
OF INORGANIC MATERIALS**



# 5<sup>th</sup> IEA INTERNATIONAL WORKSHOP ON BERYLLIUM TECHNOLOGY FOR FUSION

MOSCOW, RUSSIA  
OCTOBER 10 – 12, 2001

**Wednesday, October 10, 2001**

9:30 – 10:00	Welcome and introduction	A. Khomutov Yu. Sokolov A. Shikov
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## **Session A: Production, Characterization & Forming and Joining (Chairpersons: A. Khomutov, W. Haws)**

10:00 – 10:20	Status of Beryllium Technology for Fusion in Russia	D. Davydov
10:20 – 10:40	Beryllium Production at "Ulba Metallurgical Plant" PJSC	V. Savchuk
10:40 – 11:00	Experience of Commercial Production of Spherical Powders from Beryllium and its Alloys at Ulba Metallurgical Plant	V. Savchuk
11:00 – 11:20	Manufacturing and Testing of Be Armoured First Wall Mock-up for ITER	A. Gervash
	BREAK	
11:40 – 12:00	Manufacturing and Testing of a Divertor Baffle Mock-up with Beryllium Armour	M. Merola
12:00 – 12:20	Development of Be/DSCu HIP Bonding	M. Uchida
12:20 – 12:40	HIP Bonding of Beryllium and PH-Copper (CuCrZr)	M. Uda
12:40 – 13:00	Development of the Process of Diffusion Welding of Metals "Stainless Steel – Copper – Beryllium" into a Single Composite	G. Goriayev
13:00 – 13:20	Thermal Fatigue Properties and Results of In-Pile Integrated Test of Be/CuCrZr and Be/GlidCop Joints Produced by Fast Brazing	A. Gervash
	LUNCH	

**Session B: High Heat Flux Performance, Thermal and Mechanical Properties, Plasma/Tritium Interactions & Basic Properties (Chairpersons: M. Roedig, I. Mazul)**

14:30 – 14:50	High Heat Load Property of HIPed Be/DSCu Joint	M. Uchida
14:50 – 15:10	Simulation of Vertical Displacement Events (VDE) and Thermal Fatigue on Actively-Cooled Beryllium-Copper Mock-Ups	M. Roedig
15:10 – 15:30	Measurements of Heat Transfer Through Deformed Single Size Beryllium Pebble Beds	G. Piazza
15:30 – 15:50	Retention of Deuterium Atoms and Molecules in Beryllium Pre-Irradiated with Helium Ions	V. Alimov
	BREAK	
16:10 – 16:30	Some features of Beryllium-Laser Beam Interaction	D. Davydov
16:30 – 16:50	Features of Iron-Contained Phases Precipitating in Commercial Beryllium at 600°C	V. Petrov
16:50 – 17:10	Modelling of Joint Operation of Different Plasma Facing Materials	A. Zimin
17:10 – 17:30	Modelling of Arcing at the Plasma Facing Components from Beryllium	V. Ivanov

**Thursday, October 11, 2001**

**Session C: Neutron Irradiation Effects, Chemical Compatibility and Corrosion, Health and Safety Issues & Disposal/Reclamation and Recycling (Chairpersons: F. Scaffidi-Argentina & B.N. Kolbasov)**

8:30 – 8:50	Damage of Beryllium under High Dose Neutron Irradiation	V. Chakin
8:50 – 9:10	The Effect of Irradiation Dose on Tritium and Helium Release from Neutron Irradiation Beryllium	I. Kupriyanov
9:10 – 9:30	The Effect of Structural Defects on Helium Swelling of Beryllium with Large Volumetric Increase During Out-Of-Pile Annealing	Yu. Shumov
9:30 – 9:50	Helium and Tritium Release and Sub-Microscopic Restructuring in Irradiated Beryllium Pebbles	E. Rabaglino
9:50 – 10:10	Preliminary Evaluation of High He Generation Effect of Beryllium by He Ion and Electron Implantation Test	M. Uchida

	BREAK	
10:30 – 10:50	Mechanical Properties Variation and Swelling of Beryllium under High Temperature Neutron Irradiation. A Role of Doping with Niobium and Nickel.	G. Serniayev
10:50 – 11:10	Change of Optical Properties of Beryllium Mirrors under Deuterium Ion Bombardment	D. Orlinskij
11:10 – 11:30	Behavior of Implanted Deuterium in Be and Be <sub>12</sub> Ti	N. Yoshida
11:30 – 11:50	Analytical Study of Steam Diffusion and Chemical Interaction with Beryllium Powder on Hot Surface Inside the Grooves	V. Filatov
11:50 – 12:10	Experimental Study of Steam Chemical Reactivity with Beryllium Powder on Hot Surface Inside the Grooves	V. Kuznetsov
12:10 – 12:30	Electrical Behaviour of a Beryllium Pebble Bed at High Temperature in a Reducing Atmosphere	E. Alves
	LUNCH	
14:00	Tour of Moscow	
19:00	Social Event	

**Friday, October 12, 2001 (Continuation of Session C)**

9:30 – 9:50	Interaction of HCPB Breeding Blanket Beryllium Pebbles with Air and Steam	F. Druyts
9:50 – 10:10	Medical and Sanitary Security of Beryllium Facility Employees of "Ulba Metallurgical Plant" PJSC and Analysis of Occupational Disease Incidence	A. Kovyazin
10:10 – 10:30	Conditioning Methods for Irradiated Beryllium Waste	J. Fays
10:30 – 10:50	Practical Beryllium Resource Recovery Based on Dry Method Utilizing Gaseous Reaction	K. Tatenuma
10:50 – 11:10	Beryllium-Steam Interaction Experiments and Self-Sustained Reaction Studies (Integral Validation Testing)	P. Mikheyev
	BREAK	

**Session D: Beryllides & Other Fields (Chairpersons: H. Kawamura, V.Chernov)**

11:30 – 11:50	Status of Beryllium Study in Japan	H. Kawamura
11:50 – 12:10	Status of Beryllium Technology Activity in NGK	N. Franz
12:10 – 12:30	Design of Heat Resistant Alloys Strengthened by Beryllides	Y. Mishima
12:30 – 12:50	Thermal Property of Neutron Irradiated Be <sub>12</sub> Ti	M. Uchida
	LUNCH	
14:00 – 14:20	Compatibility of Be <sub>12</sub> Ti and SS316LN	H. Kawamura
14:20 – 14:40	Preliminary Neutronic Estimation for Demo Blanket With Beryllium Intermetallic Compound	H. Yamada
15:00 – 15:20	Microstructure Analysis of Be <sub>12</sub> Ti and its Irradiation Response by Dual Beam Irradiation	H. Takahashi
15:20 – 15:40	Tritium Release from Neutron Irradiated Be <sub>12</sub> Ti	E. Ishitsuka
	CLOSING REMARKS	

**SESSION A**

**Production, Characterization & Forming and Joining**

**Chairpersons:**

**A. Khomutov & W. Haws**



## STATUS OF BERYLLIUM TECHNOLOGY FOR FUSION IN RUSSIA

D.Davydov, V.Chernov, V.Gorokhov

*SSC RF-A.A.Bochvar Research Institute of Inorganic Materials  
(SSC RF-VNIINM), 123060 Moscow, P.O.Box 369, Russia*

Many conceptual designs for a thermonuclear reactor consider the use of beryllium as a plasma-facing armour materials and as a neutron multiplier in the breeding blanket. SSC-RF VNIINM – head institute of Minatom of RF on beryllium problems- provides a lot of studies and developments from metallurgy of beryllium to safety and environmental. The paper is a review of some studies performed in Russia at last time. Chemical interaction of dense, porous beryllium and powders with steam (in temperature range from 400 to 700°C) and air (at 500-1000°C) was investigated. Mathematical models describing it were developed. Chemical interaction of beryllium powder with carbon was studied too. Chemical reactivity of beryllium dust located in gaps under interaction with overheated steam was investigated. Plasma disruption loads on different beryllium grades were simulated using GOS-1001 high power solid state laser. Aerosols formation and its particle distribution in size during heating up to 1000° and cooling of dense and porous beryllium were studied. A modeling of irradiated beryllium zone melting from view point of irradiated material recycling is considered. A study the structure, swelling, tritium and helium release from irradiated beryllium of different varieties during its stepwise and linear post irradiation anneals has been carried out. A scrap-free low-temperature (250-300°C) pressing technique for beryllium articles with inherently open porosity has been designed. Two methods have been developed to manufacture granulated beryllium of different particle size. Two models of a DEMO breeding blanket zone, composed of tritium breeding material and beryllium neutron multiplier have been designed and manufactured for testing in IVV-2M fission reactor RDIPE&VNIINM). New method of isothermal working of commercial beryllium has been developed and used for manufacturing precision forging and port-limiter armour tiles(VNIINM). Two port-limiter small skale mock-ups were manufactured and tested (RDIPE&Efremov Inst.).



## BERYLLIUM PRODUCTION AT “ULBA METALLURGICAL PLANT” PJSC

V.V. Savchuk, Yu.V. Shakhvorostov, V.G. Khadeyev, B.A. Kuznetsov

*“Ulba Metallurgical Plant” PJSC  
102, Abay Ave, Ust-Kamenogorsk, Kazakhstan  
Tel: (3232) 475043  
Fax: (3232) 240683  
Email: [umz@sigma.kz](mailto:umz@sigma.kz)*

### 1. Background

Beryllium production was established in Ust-Kamenogorsk in early 50-s for the needs of defense complex of the USSR. This is the only enterprise on the territory of CIS operating in a complete process cycle: from ore to finished products. The facility is provided with the complete required infrastructure, including research, analytical and environment protection laboratories, medical surveillance, etc.

The main products are as follows:

- vacuum cast beryllium
- beryllium alloys and master alloys
- beryllium products
- beryllium oxide products.

### 2. Shut-down and laying-up of the facility

In early 90-s the number of orders for beryllium products was drastically reduced. The basic facilities, such as, hydrometallurgical facility, production of beryllium ingots, powders and Be structural grades, beryllium oxide were gradually shut-down.

In late 90-s the plants of distilled Be production and production of Be oxide and products were laid up.

### 3. Return of the facility back to service

The favorable change of the situation in the beryllium market resulted in making decision to return the facility back to service. In 2000 the plant of beryllium ingots production was successfully put into operation. In 2001 the hydrometallurgical plant and beryllium oxide plant resumed their operation.

### 4. Development prospects

Establishment of large production of master alloys by carbothermic method. Establishment of beryllium coppers production. Construction of a new plant for beryllium hydroxide production with higher output.

### 5. Technical capabilities of the facility

“Ulba Metallurgical Plant” PJSC possesses technologies of production of reactor grade beryllium, spherical powders of beryllium and beryllium based alloys.

Unique presses, including hydrostatic and gasostatic presses make possible to produce large size products with high physical and mechanical properties. Under certain conditions the facility can recondition the “Sphere” installation and to start production of spherical powders.





## EXPERIENCE OF COMMERCIAL PRODUCTION OF SPHERICAL POWDERS FROM BERYLLIUM AND ITS ALLOYS AT ULBA METALLURGICAL PLANT

V.V. Savchuk<sup>2</sup> D.Ph., A.V. Babun<sup>1</sup> D.Ph., Yu.V. Shakhvorostov<sup>2</sup>, Ph.S. Tuganbayev<sup>2</sup>

*<sup>1</sup>The National Science Center "Kharkov Institute of Physics and Technology"*

*61108, Academicheskaya str.1, Kharkov, Ukraine*

*Tel: (0572) 35-6027*

*Fax: (0572) 35-1662*

*<sup>2</sup>"Ulba Metallurgical Plant" PJSC, 492026,102, Abay Ave, Ust-Kamenogorsk, Kazakhstan*

*Tel: (3232) 47-5043*

*Fax: (3232) 24-0683*

*E-mail: umz@sigma.kz*

The report to be presented illustrates results of investigations obtained in the commercial production of spherical powders from beryllium and BeAl alloys at Ulba Metallurgical Plant. The report contains also the following information:

- The diagram of gas atomization plant;
- The basic process parameters of powders production;
- The characteristics of spherical powders, including the data on a particle-size distribution, specific surface, bulk density, shape and structure of powder particles and impurities content;
- The results of investigations of physical and mechanical properties of compact billets obtained from spherical powders.

As a whole, the practice of commercial production of spherical powders and compact billets from them and the results of investigations show the following:

- Gas atomization of alloy makes possible to obtain spherical powders of the size less than -315 microns from beryllium and BeAl alloys on industrial scale;
- Spherical powders obtained by gas atomization possess high bulk density (more than 68% of theoretical density of beryllium) and low oxygen content, and this is their advantage in comparison with the powders obtained by traditional mechanical grinding;
- The compact billets from spherical powders of beryllium possess practically isotropic physical and mechanical properties;
- The gas atomization is used for production of spherical powders from Be-Al alloys that in its turn makes possible to remove restrictions on the size of billets from these alloys used in the traditional metallurgical technology and specified by thermodynamic properties of the alloys.



## MANUFACTURING AND TESTING OF Be ARMoured FIRST WALL MOCK-UP FOR ITER

A. Gervash, I Mazul, N. Yablokov

*Efremov Research Institute.*

*Russia, 196641 St. Peterburg, Metallostroy, Sovetsky pr., 1*

*Tel: +(812) 462 78 72*

*E-mail: gervash@sintez.niiefa.spb.su*

One of the reference design options of ITER first wall (FW) -option B- is an option of separable first wall. Copper alloy such as CuCrZr is used as structural heat sink material for this component. The reference method used until now for bonding Be armour, copper alloy heat sink and stainless steel (SS) support structure is hot isostatic pressure (HIP). In order to avoid a number of problems inherent for HIP method (precise machining, canning, annealing of CuCrZr), authors propose alternative technology of the first wall manufacturing, which uses casting to join CuCrZr heat sink with SS and brazing to join Be to CuCrZr

Basing on results of previous investigations an actively cooled FW mockup with SS cooling tubes inserted into CuCrZr heat sink was manufacturing by casting. The mockup dimensions are 500x115x50 mm. The details of manufacturing (device for casting, casting parameters) as well as heat treatment of CuCrZr after casting are described. In particular, ultimate tensile strength of heat treated CuCrZr was about 320 MPa. To check a quality of the mockup after casting, non-destructive X-ray introscopy, which allows detecting pores and cracks having size more than 2 mm, was done. No defects were found in the mockup. After non-destructive inspection one half of the FW mockup surface was armoured with different (55x55x10mm and 55x85x10mm) Be tiles using Russian fast brazing method.

Armoured part of the mockup then successfully withstood 1000 thermal cycles at 1 MW/m<sup>2</sup>. Paper presented infra-red (IR) images of Be tiles during the testing. Aiming to check a thermal response of cast CuCrZr with inserted SS cooling tubes, another part of the mockup (without Be armour) was loaded by 1000 thermal cycles at about 4 MW/m<sup>2</sup>. Testing revealed no losing a thermal contact between CuCrZr and SS cooling tube.

The details of the first results of the FW mockup testing as well as further testing plans are presented and discussed.



## MANUFACTURING AND TESTING OF A DIVERTOR BAFFLE MOCK-UP WITH BERYLLIUM ARMOUR

M. Merola<sup>1</sup>, A. Erskine<sup>2</sup>, K. Cheyne<sup>2</sup>, P. Sherlock<sup>2</sup>, M. Rödiger<sup>3</sup>, W. Kühnlein<sup>3</sup>

<sup>1</sup>EFDA Close Support Unit, Boltzmannstr. 2, D-85748 Garching, Germany

<sup>2</sup>NNC Ltd, Warrington Rd, Risley, Cheshire, WA3 6BZ, UK

<sup>3</sup>Forschungszentrum Jülich, D-52425 Jülich

This paper describes the manufacturing, the high heat flux testing and the destructive examination of a beryllium armoured mock-up manufactured by NNC Ltd under EFDA contract and tested at JUDITH facility in FZJ. The mock-up had a straight geometry, and an overall length and width of 146 and 24 mm, respectively.

The cooling tube (10/12 mm ID/OD) was made of dispersion strengthened copper (DS-Cu), type GlidCop, and had a 0.2-mm thick stainless steel liner joined by Hot Isostatic Pressing (HIP) at 940 °C, 2 hrs, 140 MPa.

The heat sink consisted of two half-plates, made of GlidCop, with two grooves obtained by machining where the cooling tube was inserted. During this HIP cycle, at 940 °C, 2 hrs, 140 MPa, a stainless steel back plate was also joined on the rear side of the heat sink.

The final step consisted in the HIP of the armour onto the heat sink at 580 °C, 2 hrs, 100 MPa. The armour was made of beryllium tiles with a width, axial length and thickness of 24, 20 and 4 mm, respectively. Each tile had a 2-mm deep castellation of approximately 6 x 6 mm obtained by EDM.

Ultrasonic examinations were performed after each joining process as well as pressure and leak test on the final component.

During the high heat flux testing, the mock-up endured 100 cycles at 1 MW/m<sup>2</sup>, 100 cycles at 2 MW/m<sup>2</sup>, 1000 cycles at 3 MW/m<sup>2</sup> and 1000 cycles at 5 MW/m<sup>2</sup> before overheating was observed after 90 cycles at 7 MW/m<sup>2</sup>. It is worth mentioning that the divertor baffle has a design heat flux of 3 MW/m<sup>2</sup>, which is well below the achieved experimental values.

Final destructive examinations revealed that the failure occurred not in the armour to heat sink joint but in the joint of the two half plates forming the heat sink.



## DEVELOPMENT OF BE/DSCU HIP BONDING

T. Hatano<sup>1</sup>, M. Enoda<sup>1</sup>, T. Kuroda<sup>1</sup>, M. Uchida<sup>2</sup>, V. Barabash<sup>3</sup> and M. Akiba<sup>1</sup>

<sup>1</sup>*Naka Fusion Research Establishment, Japan Atomic Energy Research Institute, Naka-machi, Naka-gun, Ibaraki-ken, 311-0193, Japan*

<sup>2</sup>*Oarai Research Establishment, Japan Atomic Energy Research Institute, Oarai-machi, Higashi-Ibaraki-gun, Ibaraki-ken, 311-1394 Japan*

<sup>3</sup>*ITER Garching Joint Work Site, Max-Planck-Institute fuer Plasmaphysik, Boltzmannstrasse 2, D-85748 Garching bei Muenchen, Germany*

*Tel: +81-29-270-7570*

*Fax: +81-29-270-7539*

*E-mail: hatano@naka.jaeri.go.jp*

Beryllium (Be) has been selected as an armor material to be bonded metallurgically on copper alloy heat sink of the first wall in ITER. The joining technology for this component to withstand severe loading conditions has been required.

The objective of this study is to optimize the joining condition of Be/alumina dispersion strengthened copper (DSCu) using hot isostatic pressing (HIP) technology for the first wall. Especially, optimization of the interlayer between Be and DSCu is important to prevent from forming brittle compounds but providing a compliant layer at the interface. HIP conditions and interlayer materials were investigated with trial fabrication, four-point-bending tests and metallurgical observation of the joints. Following to the screening tests, two HIP conditions were determined as shown in Table 1. The No.2 specimen showed the highest bending strength of 258 MPa at room temperature among the trial joints. Though the No.1 specimen had the second highest bending strength, high thermo-mechanical performance is expected due to its thicker Al working as a compliant layer.

To evaluate thermo-mechanical performance of the HIP-bonding joints, FEM analyses were performed. In the analyses, lower of thermal stress at the Be/DSCu interface was observed in the No.1 specimen.

Table 1. Be/DSCu HIP conditions determined

No	Pre-HIP coating				HIP conditions			
	On Be	On DSCu			Interlayer	Temp. (°C)	Holding (hr)	Pressure (MPa)
1	Al	Al	Ti	Cu	Al/Si/Mg	555	2	150
2	—	Cu			—	620	2	150



## HIP BONDING OF BERYLLIUM AND PH-COPPER (CUCRZR)

M. Uda<sup>1</sup>, T. Hatano<sup>2</sup>, T. Iwadachi<sup>1</sup> and Y. Ito<sup>1</sup>

<sup>1</sup> *New Metals Division, NGK Insulators, LTD.,*

*1 Maegata-cho, Handa, Aichi 475-0825, Japan*

<sup>2</sup> *Naka Research Establishment, Japan Atomic Energy Research Institute,*

*Naka-machi, Naka-gun, Ibaraki 311-0193, Japan*

*TEL : +81-569-23-5807, FAX : +81-569-23-5856, E-mail : uda@ngk.co.jp*

Beryllium has been selected as an armor material to be bonded on copper alloy heat sink of the first wall in ITER. The joining technology for this component to withstand severe loading conditions has been required. Hot Isostatic Pressing (HIP) bonding using interlayer for Be/dispersion strengthened copper (DSCu) has been studied before. The objective of this study is to survey the joining condition of Be/precipitation hardened copper (PH-copper) that is another candidate copper alloy for heat sink.

Interlayer material and HIP conditions were investigated by four-point-bending tests and metallurgical observations of the joints. An interlayer is the combination of Cu, Cr, Ti and Al. The interlayer was PVD-coated on Be first and HIPed with CuCrZr. HIP temperature were 550 °C, 580 °C and 610 °C. HIP pressure and holding time was 150 MPa and 1 hour, respectively. Four-point-bending tests were performed at R.T., 200 °C and 400 °C. Metallurgical observations were performed by microscopy and EPMA.

In these tests, the joints that had PVD-coated Cr layer and PVD-coated Cu layer on Be showed higher fracture strength at any HIP temperatures. The joints were not fractured at the HIP-bonded interface and bent in CuCrZr region up to 400°C. The other specimens were fractured at the HIP-bonded interface. From the results of the microscopy and EPMA, it appears that PVD-coated Cr layer was clearly observed in the interface without forming intermetallic Be compounds. Other interlayer formed intermetallic Be compounds. It was also observed that Cr element and Zr element in CuCrZr diffused into the PVD-coated Cu layer. It was discussed that PVD-coated Cr layer prevented from forming intermetallic Be compounds and PVD-coated Cu layer promote the diffusion bonding between CuCrZr and Cu.

In this study, the joining condition of Be/CuCrZr was surveyed and new HIP procedure utilizing the Cr layer to prevent from forming intermetallic Be compounds and the Cu layer to promote the diffusion bonding was found. The optimization of the parameters is necessary for ITER first wall application. This new technology is expected to expand the possibility to apply Beryllium for any applications that require ductile joining with other material.



## DEVELOPMENT OF THE PROCESS OF DIFFUSION WELDING OF METALS “STAINLESS STEEL – COPPER – BERYLLIUM” INTO A SINGLE COMPOSITE

G. Goriayev, O. Maslennikov, N. Vesselkov, A. Vechkutov, B. Zorin, S. Bogacheva

*“Ulba Metallurgical Plant” PJSC  
102, Abay Ave, Ust-Kamenogorsk, Kazakhstan  
Tel: (3232) 475043  
Fax: (3232) 240683  
Email: [umz@sigma.kz](mailto:umz@sigma.kz)*

The report shows results of investigations on the development of the process of obtaining of triplicate composite “beryllium/copper/stainless steel”. Diffusion welding and soldering of beryllium with copper and soldering of copper with stainless steel was used in the investigation. It is expected to use this composite in the thermonuclear reactor ITER in its first wall in which beryllium is turned to plasma. Diffusion welding of beryllium and copper billets, copper and stainless steel was carried out by methods of hot vacuum pressing, gasostatic pressing and just by vacuum heating. Soldering was carried out with small crystal solders.

The best results were obtained in hot isostatic pressing of stainless steel with copper at 900°C, and copper with beryllium at 750 °C at the pressure of about 1000 kg/cm<sup>2</sup>. The strength of diffusion seam between the stainless steel and copper is 23.5 kg/mm<sup>2</sup>, and that between beryllium and copper 9 kg/mm<sup>2</sup>.

The strength of soldered seam between stainless steel and copper is 10.3...11.2 kg/mm<sup>2</sup>, and that between copper and beryllium 2.7 kg/mm<sup>2</sup>.

Results of investigations determined the process of production of triplicate tube models manufactured by method of welding of stainless steel with copper and beryllium. The models are intended for testing of beryllium interaction with steam.

The models are coaxial cylinders of beryllium, copper and stainless steel pressed into each other and welded together by diffusion welding. The models were welded in the gasostatic apparatus. The gasostatic pressing of stainless steel and copper was carried out at a temperature of 850 °C and at the pressure of 1000 atm. during 2 hours. The gasostatic pressing of copper and beryllium was carried out at the temperature of 750 °C at the pressure of 1000 atm. during 1.5 hours.

The models were successfully tested in SNC RK.



# **THERMAL FATIGUE PROPERTIES AND RESULTS OF IN-PILE INTEGRATED TEST OF Be/CuCrZr AND Be/GlidCop JOINTS PRODUCED BY FAST BRAZING**

**A. Gervash<sup>1</sup>, I. Mazul<sup>1</sup>, N. Litunovsky<sup>1</sup>, A. Pokrovsky<sup>2</sup>**

*<sup>1</sup>Efremov Research Institute. Metallostroy  
196641 St. Petersburg, Russia, Sovetsky pr. 1  
Tel: ++7-812-462-7872  
Fax: ++7-812-464-4623  
E-mail: gervash@sintez.niiefa.spb.su*

*<sup>2</sup>Research Institute of Atomic Reactor (SSC RF RIAR)  
435510 Dimitrovgrad-10, Russia*

Proposing beryllium as plasma facing armour and fast brazing as Be/Cu-alloy joining technique, this paper presents the recent experimental results. In particular, it was shown that fast brazing of Be onto both PH-copper like CuCrZr and DS-copper like GlidCop provides reliable joint that does not failure under heat flux up to melting of Be surface.

Investigating the influence of neutron irradiation on Be/Cu-alloy joint behaviour, in-pile integrated test in a core of nuclear reactor SM-2, Dimitrovgrad, Russia was carried out. In this test actively cooled Be/CuCrZr and Be/GlidCop joints were simultaneously subjected to high heat flux (1000 cycles,  $\sim 7.5 \text{ MW/m}^2$ ) and neutron irradiation (total fluence  $2.8 \times 10^{20} \text{ n/cm}^2$ ,  $\sim 0.13 \text{ dpa}$ ). Both type of tested joints successfully survived applied heat and neutron loads. Optic and TEM metallography after irradiation as well as analysis of chemical composition in the brazing zone with X-ray analyser were done to investigate brazing zone stability. Flash method thermal conductivity measurements of Be itself and Be/Cu-alloy joint before and after in-pile test were done to study its degradation.

The results of metallography, microhardness and thermal conductivity measurements are discussed, main conclusions and future plans are also presented.

**SESSION B**

**High Heat Flux Performance, Thermal and Mechanical  
Properties, Plasma/Tritium Interactions & Basic  
Properties**

**Chairpersons:**

**M. Roedig, I. Mazul**





## HIGH HEAT LOAD PROPERTY OF HIPed Be/DSCu JOINT

E. Ishitsuka<sup>1</sup>, M. Uchida<sup>1</sup>, T. Hatano<sup>2</sup>, V. Barabash<sup>3</sup>, A. Kikukawa<sup>4</sup> and H. Kawamura<sup>1</sup>

<sup>1</sup>*Ourai Research Establishment, Japan Atomic Energy Research Institute, Ourai, Higashi-Ibaraki, Ibaraki 311-1394 Japan*

<sup>2</sup>*Naka Fusion Research Establishment, Japan Atomic Energy Research Institute, Naka, Ibaraki, 311-0193, Japan*

<sup>3</sup>*ITER Garching Joint Work Site, Max-Planck-Institute für Plasmaphysik, Boltzmannstrasse 2, D-85748 Garching bei München, Germany*

<sup>4</sup>*SANGYO KAGAKU CO., LTD.,*

*441-3 Muramatsu, Tokai, Naka, Ibaraki, 319-1112, Japan*

*Tel: +81-29-264-8368*

*Fax: +81-29-264-8480*

*E-mail: uchida@ourai.jaeri.go.jp*

The high heat load test of first wall mockups (Be/DSCu) fabricated by HIP were carried out to evaluate the heat removal performance and the durability of these joints. The mock-up consists of the beryllium armor (S65C) and the DSCu heat sink (Al<sub>2</sub>O<sub>3</sub>: 0.5 wt%). The dimension of armor and heat sink is <sup>1</sup>20 x <sup>w</sup>15 x <sup>h</sup>7 and <sup>1</sup>40 x <sup>w</sup>15 x <sup>h</sup>19 mm with  $\phi$ 8 mm cooling water channel, respectively. Two types of mock-up were fabricated with the interlayer of Al/Ti/Cu (1 mm: Type 1) and pure Cu (10 mm: Type 2), and tested by the OHBIS (Ourai Hot-cell electron Beam Irradiation System).

The heat removal performance tests were performed with the surface heat flux of 1, 2, 3, 4 and 5 MW/m<sup>2</sup>. The heat removal performance was measured by the thermocouple installed into the Be armor and DSCu heat sink, and from the surface temperature that was measured by infrared camera. The thermal cycle tests were performed with heat flux of 5 MW/m<sup>2</sup> up to 1000 cycle. The temperature of the joint interface under electron beam heating between Be and DSCu is 200°C, the same as during ITER operation.

The result of the heat removal tests showed good linear relationship between the heat flux and the temperature at a part of DSCu for both types of joints. Also the result calculated by ABAQUS code agreed with the measured value. These meant that both mock-ups had durable joint interface without thermal resistance. As the result of the thermal cycle tests, "Type 1" maintained good heat removal performance up to 1000 cycles, but "Type 2" showed the delay of the surface temperature response at the corner of mock-up in cooling stage. The detachment at the corner of "Type 2" was observed by microscopy. It was considered that the detachment caused by the thermal fatigue because the joining strength of "Type 2" was smaller than that of "Type 1".



## **SIMULATION OF VERTICAL DISPLACEMENT EVENTS (VDE) AND THERMAL FATIGUE ON ACTIVELY-COOLED BERYLLIUM-COPPER MOCK-UPS**

**M. Rödig<sup>1</sup>, W. Kühnlein<sup>1</sup>, P. Lorenzetto<sup>2</sup>, M. Merola<sup>2</sup>, J. Linke<sup>1</sup>**

<sup>1</sup>*Forschungszentrum Jülich, EURATOM Association, D-52425 Jülich, Germany.*

<sup>2</sup>*EFDA Close Support Unit, Boltzmannstr. 2, D-85748 Garching Germany*

*Tel: +49-2461-616383*

*Fax: +49-2461-616435*

*E-mail: m.roedig@fz-juelich.de*

Beryllium is one of the main candidate materials for the first wall and the baffle of future tokamaks. Components will consist of castellated or non-castellated beryllium tiles joint to heat sinks made from copper alloys. Joining techniques are silver-free brazing and hot isostatic pressing (HIP).

During normal operation of the tokamak, the components are stressed by cyclic thermal gradients which may have a fatigue effect on the joint. In addition off-normal events like disruptions or vertical displacement events (VDE) may occur. Both effects may lead to premature failure of the Be-Cu joint, and in the latter case to heavy erosion of the beryllium armour. VDEs and thermal fatigue have been simulated by means of the electron beam facility JUDITH.

Several mock-ups produced by different joining techniques and by different producers have been tested under thermal fatigue conditions at power densities between 1 and 7 MW/m<sup>2</sup>. A wide range of failure limits was found depending on the joining process and on the sample design (e.g. for First Wall or Divertor) and geometries (e.g. castellations, Be thickness).

Three Be-CuCrZr mock-ups with 3, 5 and 8 mm beryllium armour, produced by silver free brazing, were loaded by VDE shots of 100 ms and 300 ms, respectively. The power density of each shot was 60 MJ/m<sup>2</sup>. In spite of heavy melting of the beryllium, no indications for a detachment of the armour were observed. Afterwards, the mock-up with 5 mm armour thickness underwent a thermal fatigue experiment of 1000 cycles at 5 MW/m<sup>2</sup>. Again no failure was observed during this loading condition. Thermal fatigue testing of the other two mock-ups and metallographic inspections are in progress.



## MEASUREMENTS OF HEAT TRANSFER THROUGH DEFORMED SINGLE SIZE BERYLLIUM PEBBLE BEDS

G. Piazza<sup>1</sup>, G. Hofmann<sup>1</sup>, J. Reimann<sup>1</sup>, S. Malang<sup>1</sup>, A. Goraieb<sup>2</sup>, H. Harsch<sup>2</sup>

<sup>1</sup>*Forschungszentrum Karlsruhe, IKET, P. O. Box 3640, D-76021, Karlsruhe, Germany*

<sup>2</sup>*Goraieb Versuchstechnik, In der Tasch 4a, D-76227 Karlsruhe, Germany*

In the helium cooled pebble bed blankets for fusion power plants, beryllium in form of pebble bed is foreseen as neutron multiplier. Beryllium pebbles with a diameter of about 1 mm are arranged between flat cooling plates. Because of different expansions of the pebble beds and the containing structure, large compressive stresses might cause considerable plastic pebble deformations. For the proper thermal mechanical design, therefore, the thermal conductivity of these beds as a function of deformation must be known.

An experimental activity to measure the heat transfer parameters of strongly deformed pebble beds, namely the thermal conductivity and the heat transfer coefficient to the containing wall, has been started with the construction of the test section HECOP at the Research Centre of Karlsruhe. In this facility the heat transfer through uniaxially compressed beryllium pebble beds is being studied.

The design of the HECOP facility was guided by minimisation of uncontrolled heat losses, a reliable measurement of the temperature gradients in the bed and the possibility to adjust independently temperature and pebble bed strain. The test section consists of the following main parts:

- electro-hydraulically controlled mechanical press ( $p_{\max} \approx 6$  MPa);
- electrical heat supply and control system ( $T_{\max} \approx 650$  °C);
- cooling system including heat exchanger;
- test section: heating plate, cooling plate, guard heaters;
- instrumentation: pressure and displacement transmitters, thermocouples, electric power measurement device, flow meters, hardware and software for experiment control and data acquisition.

In the present paper the experimental device is described and first preliminary results are presented.



## RETENTION OF DEUTERIUM ATOMS AND MOLECULES IN BERYLLIUM PRE-IRRADIATED WITH HELIUM IONS

V. Alimov

*Institute of Physical Chemistry, Russian Academy of Sciences,  
Leninsky prospect 31, 117915 Moscow, Russia  
E-mail: alimov@ipc.rssi.ru*

Present concepts for ITER consider beryllium as a candidate material for the plasma-facing component of the first-wall system. Reliable data on the hydrogen behaviour in Be under irradiation are of great importance for the application of Be tiles in fusion devices. Due to neutron irradiation, helium is accumulated in the bulk of Be matrix. The presence of helium is thought to affect the trapping and release characteristics of hydrogen isotopes.

In-depth concentration profiles of deuterium atoms and molecules in Be pre-implanted with 24 keV  $^3\text{He}$  ions to fluences in the range from  $1 \times 10^{20}$  to  $2 \times 10^{21}$   $\text{He}/\text{m}^2$  at 300 K and then irradiated with 2 keV D ions up to a fluence of  $2 \times 10^{23}$   $\text{D}/\text{m}^2$  at 300 and 700 K have been investigated by SIMS and RGA (residual gas analysis) methods in the course of sputtering of the surface with Ar ions.

It has been established that defects created by He ion irradiation (helium-vacancy complexes and helium bubbles) play a role of trapping centres where deuterium is retained both in the form of D atoms and  $\text{D}_2$  molecules. The maximum amount of deuterium trapped at these defects increases with the concentration of He atoms pre-implanted and is estimated to be about 0.7 and 0.2 D per He atom (for He concentration below 5000 appm) at irradiation temperatures of 300 and 700 K, respectively.



## SOME FEATURES OF BERYLLIUM-LASER BEAM INTERACTION

A. Laushkin, D. Davydov, Yu. Kostuk, A. Tselishev.

*SSC RF-A.A.Bochvar Research Institute of Inorganic Materials  
(SSC RF-VNIINM), 123060 Moscow, P.O.Box 369, Russia*

Plasma disruption loads on different beryllium grades were simulated using GOS-1001 high power solid state laser. The impinging energy density was  $0.5 \text{ MJ/m}^2$  with pulse length 3 microsecond. Thermal shock cracking, aerosols formation and propagation response as well as erosion were studied. Aerosols formation and its particle distribution in size during beryllium-laser beam interaction were studied too. The dependence of damage initiation and evolution on the laser beam parameters such as pulse energy, number and length was studied for five beryllium grades. An average ablation depth was calculated using the size of the laser beam impact area and mass loss measurements of the samples. The measurements of the sample mass loss were performed with "Sartorius" microbalance ensuring weighting accuracy of 5 microgram. The surface morphology was studied with a scanning electron microscope S-800. The studies have revealed that more intensive cracking takes place in coarse-grained structures. Fine-grained beryllium is relatively resistant to the thermal shock loads. A net of bulges is being formed on sample surface as result of melted beryllium crystallization. Structure of these bulges is porous. Probable mechanisms of beryllium interaction with laser beam are discussed.



## FEATURES OF IRON-CONTAINED PHASES PRECIPITATING IN COMMERCIAL BERYLLIUM AT 600°C

Petrov V.I., Gladkov V. P., Dubinskaya Yu. I., Rumyantsev I. M.

*Moscow State Engineering Physics Institute  
(Technical University)*

*31 Kashirskoe shosse Moscow Russia 115409*

*Tel: (095) 324-8441*

*Fax: (095) 324-2111*

*E-mail: petrov@physecon.mephi.ru*

The working resource of a metal product at high temperatures depends on developing changes of phase condition of impurities in metal. It is well known, for instance, that the mechanical properties of some beryllium grades being candidate for thermo-nuclear project depend on developing changes of the phase condition of impurities in metal. Modern experimental technique and mathematical methods of results processing allow to study and to predict similar processes.

We have carried out diffusion research and have studied phase condition of iron impurity in beryllium during long anneal up to 1000 hours at 600 °C. by means of Messbauer spectroscopy. It was determined that the concentration dependence of Messbauer effect allows to supervise impoverishment and enrichment of solid solution within the real limits appropriate to technical beryllium grades. The mixed spectrum of solid solution and precipitated phase is resolved satisfactorily. The low equilibrium solubility of iron in a number of beryllium grades results in rapid precipitating of significant part of iron impurity from initial supersaturated solid solution. The speed of disintegration of solid solution in hot-pressed beryllium is highest, and it is much higher, than in a cast material of the same chemical structure. The time of precipitating of the same quantity of iron-contained phase essentially differs. For example, hot pressed beryllium is required to be sustained at 600°C during 15 hours for removing 40 % of iron from the solid solution, and cast - during 90 hours.

Diffusion mobility of iron in beryllium of commercial purity smelted in the induction furnace, coincides with the literary data received for a pure monocrystal. However for hot-pressed beryllium prepared of above-mentioned smelted material it is 2 times higher. The raised mobility, apparently, arises because of high amount of structural defects facilitating volumetric diffusion.

Connected to this fact diffusion way necessary for formation of precipitations is also rather small.

The data obtained are offered to be taken into account at a choice of beryllium grades for use in thermo-nuclear project. At a complex thermal-temporary mode, characteristic for the mentioned project, it is useful to know the mechanism of transformations, to simulate them in real temporary dependence on changes of working temperature and to study in elements of constructions.



## MODELING OF JOINT OPERATION OF DIFFERENT PLASMA FACING MATERIALS

Yu.A. Axyonov<sup>1</sup>, T.A. Belykh<sup>2</sup>, L.S. Danelyan<sup>3</sup>, N.G. Elistratov<sup>4</sup>, V.M. Gureev<sup>3</sup>,  
M.I. Guseva<sup>3</sup>, B.N. Kolbasov<sup>3</sup>, V.S. Kulikauskas<sup>5</sup>, N.N. Vasiliev<sup>3</sup>, E.V. Vygovskij<sup>1</sup>,  
V.V. Zatekin<sup>5</sup>, A.M. Zimin<sup>4</sup>

<sup>1</sup>*Institute of Beryllium, Association «Kompozit», Korolyov, Moscow Region, RF.*

<sup>2</sup>*Uralsky State Technical University, Ekaterinburg, RF.*

<sup>3</sup>*RRC «Kurchatov Institute», Nuclear Fusion Institute, Moscow, RF.*

<sup>4</sup>*N.E. Bauman Moscow State Technical University, RF.*

*e-mail: [zimin@power.bmstu.ru](mailto:zimin@power.bmstu.ru), tel.: - (095)263 60 67*

<sup>5</sup>*Institute of Nuclear Physics, M.V. Lomonosov Moscow State University, RF.*

Beryllium and tungsten are candidate materials for armor of plasma facing components of International Thermonuclear Experimental Reactor (ITER). At interaction of armor surface with plasma these materials will be sputtered, mutually re-deposited and co-deposited with hydrogen isotopes.

A magnetron sputtering system with compound cathodes-targets (50% W + 50% Be and 75% W + 25% Be) was used for material sputtering and re-deposition modeling. Energy of bombarding D-ions was 230 and 320 eV respectively. Average dose was about  $4 \cdot 10^{25} \text{ m}^{-2}$ .

After interaction with plasma the targets had distinct annular structure with zones of predominant sputtering and re-deposition. Deuterium and protium concentration was measured in the depth up to 250-300 nm from specimen surface by elastic recoil detection technique using beams of nitrogen ions with energy of 10.5 MeV (cyclotron) and helium ions with energy of 1.9 MeV (Van-de-Graaf accelerator). Chemical composition in the near-surface layers was determined by Rutherford backscattering of helium ions using Van-de-Graaf accelerator. Spectra for chemical composition determination were calibrated on Au and Al standards. VD and VH were used to measure D and H concentrations respectively.

Microstructure and target phase composition were analyzed. Erosion profiles resulted from ion bombardment were measured. The studies have shown that:

- Since sputtering yield of Be is by 2 orders of magnitude greater than that of W, mixed Be-W layer in the re-deposition zone is practically not formed.
- Thickness of co-deposited layers on Be and W is approximately the same and is equal to 200-300 nm.
- Re-deposited layers of Be-sectors contain 50% Be and 40% O. W is available in the whole volume of the layers, but its concentration is <1 at. %. Integral concentration of deuterium in the layers is  $(1.5-2) \times 10^{21} \text{ m}^{-2}$ .
- D-concentration in re-deposited layers is determined by Be-deposit and practically does not depend on original material of the target. Atomic ratio D/Be is  $\sim 0.1-0.12$ .
- Oxygen concentration in the sputtering zones of Be-sectors is  $\sim 2$  at.%. Atomic ratio D/Be there is by an order of magnitude less than in re-deposition zones. W is available only on the surface, its concentration is  $\sim 2 \cdot 10^{18} \text{ m}^{-2}$ .



## MODELING OF ARCING AT THE PLASMA FACING COMPONENTS FROM BERYLLIUM

B. Jüttner<sup>2</sup>, V.A. Ivanov<sup>1</sup>, A.M. Zimin<sup>3</sup>

<sup>1</sup>*Institute of General Physics of Russian Academy of Sciences, Moscow, RF,*

<sup>2</sup>*Institute of Physics of Humboldt University, Berlin, Germany,*

<sup>3</sup>*Bauman Moscow State Technical University, Moscow, RF*

*E-mail: ivanov@fpl.gpi.ru*

The arcing took place on different plasma facing components practically of all fusion devices. Beryllium is main candidate material for armour of plasma facing components of International Thermonuclear Experimental Reactor (ITER), but the experimental data on the arcing at the beryllium surface are absent in the scientific literature.

The modelling experiments of arcing processes were carried out in the experimental facility of Institute of Physics of Humboldt University in Berlin. The arc discharges were excited by a high electric voltage 12 kV submitted between the cathode (Be) and copper (Cu) anode in high vacuum chamber at residual pressure  $10^{-7}$  Pa. After electrical breakdown of a vacuum gap (100 microns) between the cathode and anode the arc discharge was developed with amplitude of electrical current 60 A. The character of a current time evolution during the arc discharge was a decreasing exponent with the time constant about 400 microseconds. The voltage drop between the cathode and anode under the arc discharge was about 30 V.

Dynamics of cathode spots on the beryllium was studied by a method of a super-high speed photo registration with the help of the CCD-camera: shooting 7 staff with duration of the staff 100 ns, time interval between the staffs - 500 ns and space distribution 3,3 micrometers.

It is established, that the number of cathode spots during the arc discharge can reach 10, and average electric current on one spot was about 5-7 A. Two types of cathode spots on beryllium were revealed, the speeds of which moving on a surface strongly differ. The first type of spots was excited on beryllium cathode with a beryllium-oxide film, which formed under low vacuum condition ( $10^{-1}$  Pa). The speed of these spots, which moved across the surface, can reach 450 m/s. The second type of cathode spots moved with speed on two order smaller than the first type spots. These second type spots were raised on beryllium cathode after cleaning processes on its surface by the arc discharges.

For the first time a phenomena of arcing on the beryllium was investigated in high vacuum. It was shown that two types of cathode spots were excited on real surface of beryllium in vacuum arc discharges. The arcing process on a beryllium leads to the dissipation of the beryllium-oxide films on its surface.



**SESSION C**

**Neutron Irradiation Effects, Chemical Compatibility  
and Corrosion, Health and Safety Issues &  
Disposal/Reclamation and Recycling**

**Chairpersons:**

**F. Scaffidi-Argentina & B.N. Kolbasov**



## DAMAGE OF BERYLLIUM UNDER HIGH DOSE NEUTRON IRRADIATION

V.P.Chakin<sup>1</sup>, V.A.Kazakov<sup>1</sup>, R.R.Melder<sup>1</sup>, G.A.Shimansky<sup>1</sup>, S.V.Belozеров<sup>1</sup>, D.N.Suslov<sup>1</sup>,  
R.N.Latypov<sup>1</sup>, Z.E.Ostrovsky<sup>1</sup>, Yu.D.Goncharenko<sup>1</sup>, I.B.Kupriyanov<sup>2</sup>

<sup>1</sup>SC RF RIAR, 433510, Dimitrovgrad, Ulyanovsk region, Russia

Tel: (84235) 3-20-21

Fax: (84235) 3-56-48

E-mail: fae@niiar.ru

<sup>2</sup>SC RF VNIINM, Box 369, 123060, Moscow, Russia

Tel: (095) 190-80-15

Fax: (095) 925-59-72 / 925-28-96.

E-mail: vniinm.400@g23.relkom.ru

Using of beryllium as a neutron multiplier in a solid breeder blanket implies the knowledge about properties of this material under irradiation at high neutron doses. In the paper there are presented the results of investigation of some Russian beryllium grades manufactured by hot extrusion and hot isostatic pressing and irradiated in the SM and BOR-60 reactors at temperatures of 70-400°C up to fluences of  $(0.5-16) \cdot 10^{22} \text{ cm}^{-2}$  ( $E > 0.1 \text{ MeV}$ ).

Neutron irradiation leads to degradation of physical and mechanical properties and swelling of beryllium for all investigated grades. Tensile and compression tests of irradiated specimens result the presence of radiation embrittlement and decrease in strength. At that there are the dependence for an extent of degradation of mechanical properties from irradiation and testing parameters (temperature and neutron fluence). Irradiation leads also to decrease for heat conductivity of beryllium. The effect depends from irradiation parameters significantly. In particular irradiation in the SM reactor at 70°C up to  $2 \cdot 10^{22} \text{ cm}^{-2}$  ( $E > 0.1 \text{ MeV}$ ) leads to decrease of heat conductivity of beryllium with small grain size to 50 W/m·K, that is in four times on comparison with initial state. Irradiation of beryllium with great grain size in the BOR-60 reactor at 400°C up to  $1 \cdot 10^{23} \text{ cm}^{-2}$  ( $E > 0.1 \text{ MeV}$ ) decreases the heat conductivity to 120-145 W/m·K, that is to 30% only.

Under low temperature (70-200°C) irradiation there is formed practically one type of radiation defects – dislocation loops. There were analyzed the nature of loops and found out that they were of interstitial type. There were made the short annealing of irradiated specimens at temperatures of 300-1200°C, investigated the influence of the annealing on microstructure evolution. Helium bubbles shown by TEM are appeared at 500°C only. Further on the increase of temperature leads to increase of bubble size and decrease of volume density. With increase of the annealing temperature there is the change for dislocation structure too. Irradiation at 400°C leads to formation of qualitatively other type of radiation defects. These are the flat pores with hexagonal form, which are placed presumably in a base plane of a crystal lattice. The pores are in a grain body and on a grain boundary. Last case they have some bigger sizes than first. There are observed deplete zones along grain boundaries.

In the work there are also presented the results for helium and tritium accumulation in irradiated beryllium.



## THE EFFECT OF IRRADIATION DOSE ON TRITIUM AND HELIUM RELEASE FROM NEUTRON IRRADIATION BERYLLIUM

I.B. Kupriyanov<sup>1</sup>, V.A. Gorokhov<sup>1</sup>, V.V. Vlasov<sup>2</sup>, A.M. Kovalev<sup>2</sup>, V.P. Chakin<sup>3</sup>

<sup>1</sup>*A.A. Bochvar Research Institute of Inorganic Materials (VNIIM), 123060,  
Moscow, Box 369, Russia*

<sup>2</sup>*RRC Kurchatov Institute, 123181, Moscow, Russia*

<sup>3</sup>*SSC RIAR, 435510, Dimitrovgrad, Ulyanovsk region, Russia*

The effect of irradiation dose on helium and tritium release from beryllium is described. Beryllium samples were irradiated in SM reactor with neutron fluence ( $E > 0.1$  MeV)  $(0.5-5.0) \times 10^{22} \text{ cm}^{-2}$  at 70–100°C. Mass-spectrometry technique was used in out of pile tritium and helium release experiments during stepped-temperature anneal within 200–1300°C temperature range. The total amount of helium accumulated in irradiated beryllium samples varied from 520 appm to 6000 appm.

The first signs of tritium release detected at temperature of 312–472°C and helium – at 540–700 °C. It was shown that the increase of irradiation dose results in reduction of temperature level (from 950 to 750 °C), where helium release rate becomes in factor 10 faster, while helium generation level significantly affect on finish temperature of tritium release (from 1000 to 740 °C). For the samples irradiated with fluence  $2.0 \times 10^{22} \text{ cm}^{-2}$  maximum tritium release rate concurs with a strong acceleration of helium release rate. On the basis of data obtained, the diffusion coefficients of tritium and helium in beryllium were calculated.



THE EFFECT OF STRUCTURAL DEFECTS ON HELIUM SWELLING  
OF BERYLLIUM WITH LARGE VOLUMETRIC INCREASE  
DURING OUT-OF-PILE ANNEALING

Yu.V. Shumov, B.F. Gromov, A.P. Trifonov, Yu.G. Pashkin

*SSC' RF - The Institute for Physics and Power Engineering,  
249020, Bondarenko sq., 1, Obninsk, Russia*

*Tel: (08439) 9-8211*

*Fax: (095) 230-2326*

*E-mail: [konobeev@nuclmd.obninsk.ru](mailto:konobeev@nuclmd.obninsk.ru)*

Neutron-irradiated beryllium was investigated with respect of volumetric, structural and dimensional changes during the prolonged, at 700 – 850°C, and short time, at 900 – 950°C, out-of-pile annealing.

Samples of warm- and hot-extruded and hot-pressed beryllium produced from powders with 400 and 600  $\mu\text{m}$  particle size were irradiated in the BR-5 reactor at 450 – 475°C to helium accumulation of 1 – 8  $\text{ncm}^3\text{He}/\text{cm}^3\text{Be}$ .

Considerable swelling of beryllium during annealing has been revealed to be increased with rise of temperature, time, and helium concentration, and dependent also on the production technology. Volumetric changes are accompanied by a non-isotropic dimensional change and large structural changes.

The curves of volumetric change as a function of annealing time consist of the two parts: that of fast initial swelling and that of subsequent slow swelling with saturation.

Structural changes are characterized by filling of large grains with helium pores and by appearance of pore-depleted zones on the boundaries of large grains. The depleted zones appear in the structure at a 3 – 7% swelling and stabilize in width at the slow swelling stage.

All the revealed phenomena may be explained by the volumetric increase of closed technological pores during annealing. These pores are filled with helium mainly in the course of irradiation.

The growth of these pores volumes causes the swelling of large grain accumulations at the expense of tensile stresses triggering grain boundary sources of vacancies within those accumulations.



## HELIUM AND TRITIUM RELEASE AND SUB-MICROSCOPIC RESTRUCTURING IN IRRADIATED BERYLLIUM PEBBLES

E. Rabaglino<sup>1,2</sup>, C. Ronchi<sup>2</sup>, F. Scaffidi-Argentina<sup>1,3</sup>, T. Wiss<sup>2</sup>

<sup>1</sup>*Forschungszentrum Karlsruhe, Institut für Kern- und Energietechnik, PO Box 3640, D - 76021 Karlsruhe, Germany Tel.: + 49 7247 82 3476, Fax.: + 49 7247 82 4837, E-mail: elisa.rabaglino@iket.fzk.de*

<sup>2</sup>*European Commission, Joint Research Centre, Institute for Transuranium Elements, PO Box 2340, D - 76125 Karlsruhe, Germany*

<sup>3</sup>*EFDA Close Support Unit Culham, Culham Science Centre, Abingdon Oxon OX14 3DB, United Kingdom*

A classical method to study kinetics of gas-in-solid produced in irradiated materials consists in measuring gas release during laboratory thermal annealing. In order to model the evolution of release as a function of temperature and time, a thorough understanding of gas diffusion phenomena is needed, accounting for microscopic effects due to bubble precipitation, coalescence and venting. Therefore, the measurement of gas release must be coupled with a systematic analysis of the material microstructure, at different temperatures corresponding to distinct stages.

Measurements of helium and tritium release rates from irradiated beryllium pebbles were carried out by means of a Knudsen-cell technique: the samples were annealed in high vacuum up to the melting point, and the escaping gas was measured by a mass spectrometer located in the vicinity of the crucible. This technique makes it possible to obtain very sensitive measurements in a broad temperature range. Furthermore, the evolution of the diffusion phenomena can be detected up to complete release of the initial gas inventory.

The investigated material consists of 2 mm diameter beryllium pebbles irradiated at 770 K in the High Flux Reactor (Petten) ("BERYLLIUM" experiment) up to a neutron fluence of  $1.24 \cdot 10^{21} \text{ cm}^{-2}$ , which produced 480 appm helium and 11 appm tritium. The material microstructure was analysed before and after irradiation. Its evolution during the laboratory annealing experiments was observed at different steps by optical and transmission electron microscopy, with the aim to explain the dependence of release on atomic gas diffusion, radiation damage and bubble formation.



## PRELIMINARY EVALUATION OF HIGH HE GENERATION EFFECT OF BERYLLIUM BY He ION AND ELECTRON IMPLANTATION TEST

M. Uchida<sup>1</sup>, H. Kawamura<sup>1</sup>, T. Shibayama<sup>2</sup> and H. Takahashi<sup>2</sup>

<sup>1</sup>*Oarai Research Establishment, Japan Atomic Energy Research Institute,  
Oarai, Higashi-Ibaraki, Ibaraki 311-1394 Japan*

<sup>2</sup>*Center for Advanced Research of Energy Technology, Hokkaido University Sapporo  
060-8628, Japan*

*Tel: +81- 29-264-8368*

*Fax: +81-829-264-8481*

*E-mail: uchida@oarai.jaeri.go.jp*

Beryllium is a candidate material as the neutron multiplier in a solid breeder blanket for the fusion reactor. By 14 MeV neutron irradiation, it would be degraded by irradiation damage and <sup>4</sup>He would be generated by transmutation of <sup>9</sup>Be. It has been estimated to be over 20,000 appm <sup>4</sup>He generation in DEMO reactor. However no neutron irradiation test has been performed over 4,000 appm <sup>4</sup>He before and it is expected to have neutron irradiation test up to 20,000 appm for determination of multiplier specification. The objective of this study is to evaluate the He irradiation effects up to 20,000 appm He by He and electron implantation test by ion accelerator as preliminarily test before actual irradiation test by testing reactor. The effect of Fe impurities is also discussed.

Microstructure response in two kinds of beryllium which has a different iron impurity content (0.069wt%Fe, 0.180wt%Fe) as a function of irradiation temperature, dose and spontaneous He irradiation effects were evaluated by in-situ experiment using Multi Beam High Voltage Electron Microscope. Nano indentation tests were also performed to investigate mechanical property changes in beryllium before and after He irradiation up to 20,000 appm.

Tiny bubbles and black dots were observed in all specimens irradiated at room temperature. Spontaneous He irradiation is enhanced to the nucleation of bubbles and black dots. The density of black dots in the specimen that contains higher Fe impurity is bigger than another one. It is considered that impurities would create nucleation core of defects and would grow He gas bubble at high temperature.

At the beginning of irradiation as 1,000 appm He, hardness is increased, and to saturate as increase of hardness, and then to decrease with further irradiation. The specimen that contains higher Fe impurities showed higher hardness than another one up to 20,000 appm He.



# MECHANICAL PROPERTIES VARIATION AND SWELLING OF BERYLLIUM UNDER HIGH TEMPERATURE NEUTRON IRRADIATION. A ROLE OF DOPING WITH NIOBIUM AND NICKEL

A. Sernyaev<sup>1</sup>, Yu. S. Strebkov<sup>2</sup>

<sup>1</sup>SUSE "SF NIKIET", P.O.Box 29, Zarechny, Sverdlovsk region, 624250, Russia.

Tel: +7 (34377) 36-264

Fax: +7 (34377) 33-396

E-mail: orbita@zar.ru

<sup>2</sup>SUE "NIKIET" P.O.Box 788, Moscow, 101000, Russia.

Tel: +7 (095) 263-7308

Fax: +7 (095) 263-7492

A high temperature damageability of beryllium is known to be sensitive to its structural peculiarities including the presence of the dispersed phase BeO. There are some indications to anticipate its substantial dependence on the degree of its alloying with some metallic dopes.

This paper continues the developments of the publication G.A. Sernyaev et al. "Strengthening, loss of strength and embrittlement of Be under high temperature neutron irradiation" in Journal of Nuclear Materials, 271&272 (1999), 123-127. The paper investigates the effect of high temperature neutron irradiation ( $T_{irr} = 620^\circ\text{C}$ ,  $\Phi_f = 4.67 \times 10^{21} \text{ cm}^{-2}$  and  $T_{irr} = 680^\circ\text{C}$ ,  $\Phi_f = 5.69 \times 10^{21} \text{ cm}^{-2}$ ) on short-term mechanical properties of warm- and hot- pressed Be grades with the oxygen  $C_O = (0.7 \div 4.9) \text{ wt.}\%$  in content, the grain size  $d_g = (8 \div 50) \mu\text{m}$ , and the initial porosity  $\Pi_0 = (0.81 \div 2.26) \%$ . A weak influence of doping these grades with 0.37 wt.% Nb and 2.7 wt.% Ni is noted.

A graphic representation of the dependence  $(\sigma_{us}^{el})_{T_{test} = 650^\circ\text{C}} = f(\dots, T_{irr}, \Phi_f)$  is shown.

Critical (i.e. fully softening the material) fast neutron fluences are determined (Table 1) and six Be grades are considered in the order of their increasing life-time.

Table 1. Critical Fast Neutron Fluences and Ranking of Increasing Life-Time of Be Grades

Test temperature, $T_{test}$ ( $^\circ\text{C}$ )	650											
Irradiation temperature, $T_{irr}$ ( $^\circ\text{C}$ )	620						680					
Fast neutron ( $E \geq 0.85 \text{ MeV}$ ) fluence, $\Phi_f, 10^{21} \text{ cm}^{-2}$	4.67						5.69					
Grade number	1	2	3	4	5	6	1	2	3	4	5	6
Ultimate strength before irradiation, $(\sigma_{us}^{el})_0, \text{MPa}$	250	190	175	270	100	300	250	190	175	270	100	300
Critical fast neutron ( $E \geq 0.85 \text{ MeV}$ ) fluence, $(\Phi_f)_{cr}, 10^{21} \text{ cm}^{-2}$	9.0	9.85	10.1	8.65	11.2	8.2	7.0	7.85	8.1	6.65	9.2	6.2
Increasing $(\Phi_f)_{cr}$ and thereby life-time for Be grades	6 $\rightarrow$ 4 $\rightarrow$ 1 $\rightarrow$ 2 $\rightarrow$ 3 $\rightarrow$ 5						6 $\rightarrow$ 4 $\rightarrow$ 1 $\rightarrow$ 2 $\rightarrow$ 3 $\rightarrow$ 5					

It is concluded that the sequences of the increasing life-time and the decreasing ultimate strength for these six beryllium grades coincide fully.



## CHANGE OF OPTICAL PROPERTIES OF BERYLLIUM MIRRORS UNDER DEUTERIUM ION BOMBARDMENT

V.G.Konovalov, A.V.Babun, A.F.Bardamid<sup>1</sup>, A.I.Belyayeva<sup>2</sup>, V.N.Bondarenko, A.A.Galuza<sup>2</sup>,  
L.Jacobson<sup>3</sup>, D.V.Orlinskij<sup>4</sup>, I.I.Papirov, I.V.Ryzhkov, A.N.Shapoval, A.F.Shtan',  
S.I.Solodovchenko, A.A.Vasil'ev, V.S.Voitsenya, K.I.Yakimov<sup>1</sup>

<sup>1</sup>NSC "Kharkov Institute of Physics and Technology", 61108 Kharkov, Ukraine; Taras  
Shevchenko National University, 03022 Kiev, Ukraine; <sup>2</sup>Kharkov Technical University, 61002  
Kharkov, Ukraine; <sup>3</sup>Los Alamos National Laboratory, Los Alamos, NM 87545 USA; <sup>4</sup>Kurchatov  
Institute, 123182 Moscow, Russia

It is known from literature [1] that as a mirror material, beryllium has some advantage in comparison to many metals, namely, its reflectance in the nearest UV (in the wavelength range 0.1-0.25  $\mu\text{m}$ ) can reach ~60%, i.e., is higher than in the visible range, and practically does not depend on the wavelength in visible. In connection with the plan to use this material for protection of the fusion reactor first wall [2], interest developed in utilizing beryllium for fabrication of the so called first mirrors (FM) as a part of the in-vessel components of plasma diagnostic system for providing optical and laser measurements. Since FMs are the plasma facing components of plasma diagnostics, they will be subjected to different kinds of radiation emanating from a thermonuclear plasma: neutrons, gammas, x-rays, UV, and charge exchange atoms (CXA). The CXA flux will consists mainly of deuterium and tritium atoms with a wide energy distribution. The long-term effect of simultaneous impact of all those factors on the mirror properties cannot be predicted and special simulation investigations on this subject must be conducted. In addition, the redeposition of materials transported from components subjected to the strongest plasma impact (e.g., the beryllium limiters and first wall protectors) on the FM surface can also be important.

In the present paper we describe and analyze results obtained when mirrors fabricated from different kinds of beryllium are long-term bombarded by ions of deuterium plasma of keV energy range. After a quite short time of exposure (2-5 minutes) to ions of deuterium plasma with ~1keV energy, when the sputtering erosion can be neglected (according to the mass loss measurement), the beryllium mirror reflectance drops 5-8 %, depending on the wavelength of reflecting light ( $\lambda$  = 250-650 nm, normal incidence). We suppose that this effect is explained by the transformation of the BeO film into a Be(OD)<sub>2</sub> film due to bombardment of the mirror by deuterium ions as described in [3]. It was found by use of ellipsometry at  $\lambda$  = 632.8nm that the extinction index of beryllium hydroxide film is not negligible in comparison to a BeO film investigated in [4].

The annealing of the mirror after deuterium ion bombardment at 300-350C during a period of about 1-3 hours resulted in the full or partial decomposition of Be(OD)<sub>2</sub> on the surface of beryllium mirrors and restoration of BeO film with practically full restoration of the initial mirror reflectance in the visible range. The details of process of the transformation of oxide film into hydroxide film and back will be discussed in terms of the results obtained.

With much longer increase of exposure time of Be mirrors to ions of deuterium plasma (more than one order of magnitude longer, i.e., of the order 1 hour), modification of the mirror surface morphology was observed, indicating that the sputtering of the metal itself was in process. As a result of ion etching, the microrelief on the mirror surface increased, being accompanied by the corresponding deterioration of reflectance with much lower rate than observed for initial drop of reflectance. The dynamics of reflectance degradation and development of microrelief will be discussed in the paper.

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BEHAVIOR OF IMPLANTED DEUTERIUM IN Be AND Be<sub>12</sub>TiN. Yoshida<sup>1</sup>, H. Iwakiri<sup>1</sup>, H. Kawamura<sup>2</sup> and Y. Ito<sup>3</sup>

<sup>1</sup>*Research Institute for Applied Mechanics, Kyushu University  
6-1 Kasugakoen, Kasuga, Fukuoka 816-8580, Japan*

<sup>2</sup>*Oarai Research Establishment, Japan Atomic Energy Research Institute,  
Oarai, Higashi-Ibaraki, Ibaraki 311-1394, Japan*

<sup>3</sup>*New Metals Division, NGK Insulators, LTD.,  
1 Maegata-cho, Handa, Aichi 475-0825, Japan*

*Tel: +81-92-583-7716, Fax: +81-92-583-7690, e-mail: yoshida@riam.kyushu-u.ac.jp*

Thermal desorption of deuterium from 8-keV D<sub>2</sub><sup>+</sup> ions irradiated beryllium above room temperature was correlated with microstructural changes during irradiation and annealing to understand the underlying mechanism of retention. Deuterium bubbles are formed at all examined temperatures between 300 K and 873 K. Large roundish bubbles above 200 nm are especially formed above 573 K. They remain even after annealing up to 973 K. Strong retention of deuterium by the bubbles, 90-60% of implanted deuterium for dose of 1×10<sup>21</sup> ions/m<sup>2</sup>, occurs for the irradiation up to 673 K.

In the case of Be<sub>12</sub>Ti intermetallic compound, deuterium retention is much smaller than beryllium. For implantation of 1×10<sup>21</sup> ions/m<sup>2</sup> at 300K, 20% of implanted deuterium is retained and most of them are desorbed around 400K. For implantation at 673K and 873K, retention becomes smaller, about 3-4% for 1×10<sup>21</sup> ions/m<sup>2</sup>, and the majority is desorbed up to 800K and 1000K, respectively. A small portion remains even after heating up to 1500K.



# ANALYTICAL STUDY OF STEAM DIFFUSION AND CHEMICAL INTERACTION WITH BERYLLIUM POWDER ON HOT SURFACE INSIDE THE GROOVES

*V. Filatov*

*D.V. Efremov Institute of Electrophysical apparatus  
Scientific Technical Centre "Sintez"*

*Sovetsky prospect 1, Metallostroy, St. Petersburg, 196641, Russia*

*Phone: +7 (812) 462-7872, fax: +7 (812) 464-4623, e-mail: [filatovv@niiefa.spb.su](mailto:filatovv@niiefa.spb.su)*

Beryllium is the most promising material for plasma-faced armour of vacuum vessel in future tokamak reactor like ITER. Reactor operations are accompanied by beryllium dust (powder) formation inside the vacuum vessel as a result of plasma-faced surface erosion. This dust will accumulate mainly within the grooves on hot surfaces in divertor region. There is a significant safety problem associated with explosion hazard because of hydrogen chemical production from overheated steam on beryllium dust at emergency conditions of steam and air in-leakage to vacuum vessel. However it is expected that this chemical reaction will become extinct due to limited diffusion of steam into narrow grooves. This problem is studied analytically in presented paper.

A comparative analysis of various data on the Be/steam chemical interaction rates at temperature above 600°C has been carried out. The features of this reaction in porous medium (powder bulk) have been considered. The factors of steam/hydrogen mutual diffusion have been determined. A calculation model of reaction simulation is briefly described. The main results of parametrical calculations are presented for various values of temperature and pressure. The results obtained for grooves are compared with these ones for thin flat layers. A rate of hydrogen production in grooves is considerably less than on flat surface. The dust plugs may be also formed at groove necks due to dust swelling during oxidation or sintering at high temperature and pressure (above 800°C and 1 at). These plugs block steam access to the dust located deep into the grooves and this reduces a possible hydrogen production.



## EXPERIMENTAL STUDY OF STEAM CHEMICAL REACTIVITY WITH BERYLLIUM POWDER ON HOT SURFACE INSIDE THE GROOVES

V. E. Kuznetsov, I.B.Ovchinnikov, V.A.Titov

*D.V. Efremov Institute of Electrophysical apparatus  
Scientific Technical Centre "Sintez"  
Sovetsky prospect 1, Metallostroy, St. Petersburg, 196641, Russia  
Phone: +7 (812) 462-7834, fax.: +7 (812) 464-4623,*

### 1. Introduction

An analysis of different emergency scenario for the ITER vacuum vessel (VV) with severe water leakage into the VV shows the following secondary hazard. The beryllium dust stored as a result of FW erosion within the VV (in particular, in the slots on the plasma-faced surface) can quickly produce by reaction with steam a considerable volume of hydrogen that, in turn, can result in an explosion with oxygen.

### 2. Purpose of experiment

A purpose of proposed experiment is to compare a reactivity of beryllium dust oxidation with steam in a dependence on geometry of dust allocation.

### 3. Experimental technique

A measurement of steam disappearance has appeared to be acceptable in view of rate and availability of necessary diagnostic techniques (if measurement of steam concentration will be replaced by measuring the concentration of oxygen in gas phase by a method of an optical spectroscopy). This method allows carrying out experiment as simulation of a severe leakage in ITER during its operation with the subsequent monitoring. Temperature range of beryllium dust is 500-900 °C.

### 4. Conclusions

Be-dust with steam reactivity at temperatures 700 °C and low have no significant dependence on dust allocation geometry.

At 800 °C reactivity vary in few times depend on allocation geometry, pressed slot geometry demonstrate significant reactivity decrease at early stage.

At 900 °C time scales of hydrogen yield in pressed slot and open cases differ one from another ~ 30 times and demonstrate strong dependence on allocation geometry.

Above mentioned results forced to suppose, that among 2 diffusion factors: dust allocation geometry from one hand and BeO dense layer on the Be grain surface from another the first is main at temperatures 800 °C and higher, and the second is main at temperatures 700 °C and lower.



## ELECTRICAL BEHAVIOR OF A BERYLLIUM PEBBLE BED AT HIGH TEMPERATURE IN A REDUCING ATMOSPHERE

E. Alves<sup>1,2</sup>, M. R. da Silva<sup>2</sup>, L.C. Alves<sup>1,2</sup>, F. Scaflidi-Argentina<sup>3</sup>, J.C. Soares<sup>1,2</sup>

*Instituto Tecnológico e Nuclear, EN. 10, P-2686-953 Sacavém, Portugal*

*Centro de Física Nuclear da Univ. de Lisboa, Avenida Prof. Gama Pinto 2, P-1699 Lisboa Codex, Portugal*

*EFDA Close Support Unit, Culham Science Centre, Abingdon OX14 3DB, United Kingdom*

The electrical behavior of a beryllium pebble bed is of relevant interest considering its possible use as a neutron multiplier in a ceramic breeder blanket for a fusion power reactor.

The electrical resistivity of single size and binary beryllium pebbles beds (2mm and 0.1-0.2mm) have been studied in the past at ambient atmosphere as a function of the temperature and of an externally applied mechanical load. In this paper the temperature dependence of the electrical resistivity of a 1mm single size pebble bed in a He+0.1vol% flowing atmosphere is presented and critically discussed. In agreement with our previous studies, the resistivity displays two distinct regimes as a function of the applied load. The first one is characterised by a strong reduction in the resistivity and is likely correlated with the mechanical re-arrangement of the pebbles during the first compression phase. The load range of this regime is strongly reduced by the increase of the temperature. On the other hand the second regime is characterised (in a semi-logarithmic plot) by a linear decrease of the resistivity with the applied load. The ultimate value reached by the resistivity was  $2.9 \times 10^{-4} \Omega \cdot m$  for a pressure of 53 Mpa at all temperatures from 20°C to 550°C. The slope of the resistivity curve decreases with the increase of the temperature, which can be likely correlated with an increase in the plasticity of the pebbles.

Corresponding author:

Eduardo Alves

e-mail: ealves@itn.pt



## INTERACTION OF HCPB BREEDING BLANKET BERYLLIUM PEBBLES WITH AIR AND STEAM

F. Druyts, J. Fays

SCK/CEN, Belgian Nuclear Research Centre

*Boeretang 200, 2400 Mol, Belgium*

*tel.: (32) 14-33.32.38*

*fax: (32) 14-32.35.53*

*e-mail: fdruyts@sckcen.be*

The improved design concept of the European Helium Cooled Pebble Bed (I-HCPB) foresees the use of beryllium as a neutron multiplier in the form of a pebble bed. The current reference pebble material for the HCPB breeding blanket consists of the 0.84-1.19 mm sieving fraction of beryllium pebbles produced by the Rotating Electrode Method. Accidental scenarios, such as a loss of coolant (LOCA), loss of vacuum (LOVA), or loss of fuel accident (LOFA) involve the reaction between beryllium and air or steam. Therefore, kinetic data are needed on the beryllium/steam and beryllium/air reactions for the safety assessment of the HCBP breeding blanket. We performed chemical reactivity tests with coupled thermogravimetry - mass spectrometry (TG-MS) in the temperature range 300-1000°C. The reaction mechanism and reaction rate depend strongly on temperature. At temperatures of 700°C and below, kinetics is approximately parabolic and is associated with the growth of a protective oxide layer. Above 700°C, kinetics is accelerating/linear and oxidation is non-protective. Curve fitting of the TG-MS data allowed us to accurately describe the reaction kinetics by means of the parabolic rate constant in the lower temperature range (up to 700°C) and the linear rate constant in the higher temperature range (above 700°C). An Arrhenius relationship describes the rate constants as a function of temperature. Additional tests were performed on alternative pebble materials to investigate the influence of the manufacturing method on the reactivity in air. In this paper we discuss the results from fysisorption tests (determination of the specific surface area), thermogravimetry and mass spectrometry.



**MEDICAL AND SANITARY SECURITY OF BERYLLIUM FACILITY EMPLOYEES  
OF "ULBA METALLURGICAL PLANT" PJSC  
AND ANALYSIS OF OCCUPATIONAL DISEASE INCIDENCE**

A.A. Urikh, A.G. Kovyasin, L.A. Kovyasina

*Medical and Sanitary Department No.2 Co. (MSD-2)  
Serikbayeva str.1, Ust-Kamenogorsk, Kazakhstan 492022  
Tel: 27-26-21, 29-83-29, Fax: 27-16-07*

The report is based on the results of the long surveillance of MSD-2 (former MSD-22 No.GU at the Ministry of Public Health of the USSR). The report traces the revealability of acute and chronic cases of beryllium lesions of lungs, skin and eyes from the moment of the plant foundation (1951) till the present time and shows the occupational disease incidence depending on sanitary and hygienic conditions at the facility in the period of production foundation, its reconstruction and changes in the engineering process of metal production.

The direct dependence of the level of disease incidence on the duration of exposure to beryllium and its compounds, and labor conditions are revealed.

Speaking about pure beryllium lesions the authors do not exclude the exposure of the body to beryllium and its compounds together with the other industrial emissions of "UMP" PJSC and the Lead and Zinc Complex Works, such as fluorine, sulphur and sulphurous anhydrides, acids, alkalis, etc.

The report shows the comparison of disease incidence and the ability to work among different facilities of "UMP" PJSC (uranium and tantalum facilities) with the beryllium one.

In addition, the issues of occupational selection, diagnostics and curing, invalidization and lethality among occupational patients are touched.



## CONDITIONING METHODS FOR IRRADIATED BERYLLIUM WASTE

J. Fays, P. Druyts

*SCK CEN, Belgian Nuclear Research Centre*

*Boeretang 200, 2400 Mol, Belgium*

*tel.: (32) 14-33.31.28*

*fax: (32) 14-32.35.53*

*e-mail: jfays@sckcen.be*

Future fusion reactors will generate large quantities of radioactive beryllium waste, for which appropriate solutions need to be developed today, in order to be able to estimate in a near future the total environmental and economic impacts of fusion energy. Presently, radioactive beryllium waste is already produced in several fission research reactors, and is most often temporarily stored in the pools of the reactor, as no standard route exists for its management. Recycling in nuclear applications could offer a very interesting solution, but probably not for all the Be waste, and conditioned by the possibility to re-use it for nuclear applications. Therefore, the final disposal of irradiated beryllium is an important option to consider, and an acceptable conditioning method for this new type of waste needs to be found.

In this study, several methods have been identified as potentially interesting for the immobilisation of Be waste: encapsulation of Be-metal in a cement grout, incorporation in a glass matrix, incorporation in phosphate ceramic, and 'no conditioning'. In this latter case, Be is simply put in drums with addition of sand to fill the voids.

A first experimental work has started to gain better insight into beryllium properties in a disposal environment and its compatibility with cement. It includes solubility tests, electrochemical corrosion tests, and leaching of beryllium pebbles conditioned in cement. Results will be compared to geochemical model predictions to determine to what extent existing models and databases manage to properly describe the chemical behaviour of beryllium in complex chemical systems. The experiments will provide first values for the most important parameters that determine the acceptability of this waste in a deep repository: an evaluation of the radionuclide source term and impact of gas generation due to the beryllium metal corrosion.

The presentation will discuss results from the characterisation of existing irradiated beryllium, and the extrapolation to fusion beryllium waste. It will then underline the known advantages and shortcomings of the different available conditioning methods and discuss preliminary experimental results on the 'no conditioning' and 'cement' methods, with their estimated impact on repository safety.



## PRACTICAL BERYLLIUM RESOURCE RECOVERY BASED ON DRY METHOD UTILIZING GASEOUS REACTION

K. Tatenuma<sup>1</sup>, M. Nakamura<sup>1</sup>, T. Iwadachi<sup>2</sup> and H. Kawamura<sup>3</sup>

<sup>1</sup> KAKEN Co., 1044 Hori, Mito, Ibaraki 310-0903 Japan.

<sup>2</sup> NGK Insulators, LTD., 1 Maegata-cho, Handa, Aichi 475-0825 Japan

<sup>3</sup> Japan Atomic Energy Research Institute, Oarai Research Establishment,  
Oarai-Machi, Higashi Ibaraki-Gun, Ibaraki-Ken 311-1394 Japan.

Tel: +81-29-227-4485

Fax: +81-29-227-4082

E-mail: [tatenuma@kakenlabo.co.jp](mailto:tatenuma@kakenlabo.co.jp)

We have proposed the concept of JBR-Process (JAERI Beryllium Recycle Process) for neutron multiplier and plasma facing material in a fusion reactor, and a dry method utilizing chlorination reaction ( $\text{metal-Be or Be-compounds} \xrightarrow{\text{used}} + \text{Cl}_2 \rightarrow \text{BeCl}_2 \text{ <volatile> } \rightarrow \text{Be <recovered> } + \text{Cl}_2$ ) for reprocessing the irradiated beryllium has been developed. By this method, beryllium resource can be recovered and the radioactive nuclides (ex. Co-60, tritium, etc.) contained in beryllium can be separated from recovered beryllium with high efficiencies (Co-60: above 96%, tritium: above 99%).

In this study, in order to develop a practical technology of beryllium resource recovery from used materials, the recovering method of beryllium metal was improved. As the recovering method of beryllium metal, hydrogen gas injection method was applied at a lower temperature (about 450°C) instead of the current pyrolysis method of gaseous BeCl<sub>2</sub> at a high temperature (about 1200-1500°C) using SiC nude heater. By this improvement, it is confirmed that JBR-Process becomes still more practical for recovering beryllium resource.





BERILLIUM-STEAM INTERACTION EXPERIMENTS AND SELF-SUSTAINED  
REACTION STUDIES  
(INTEGRAL VALIDATION TESTING)

P.I. Mikheev<sup>a)</sup>, D.Sh. Kul'zhanov<sup>a)</sup>, E.O. Ishanov<sup>a)</sup>, Ye.A. Kenhzin<sup>a)</sup>, A.N. Kolbaenkov<sup>a)</sup>,  
V.V. Savchuk, O.I. Maslennikov<sup>b)</sup>, B.K. Kuznezov<sup>b)</sup>, I.L. Tazhibaeva<sup>c)</sup>, V.P. Shestakov<sup>d)</sup>,  
B.N. Kolbasov<sup>e)</sup>

<sup>a)</sup>*Institute of Atomic Energy of National Nuclear Centre, Krasnoarmeyskaya 10, Semipalatinsk-21, 490060, Kazakstan, Fax (7-3272)338585, E-mail: [iae@nnc.kz](mailto:iae@nnc.kz)*

<sup>b)</sup>*Ulba Plant, Kazakstan, Ust-Kamenogorsk city, Fax: 7-323-2-64-06-83, E-mail: [umz@sigma-east.com](mailto:umz@sigma-east.com)*

<sup>c)</sup>*National Nuclear Center Republic of Kazakhstan L. Chaykina, 4 Almaty, Kazakhstan Fax: 7-327-2503978, E-mail: [tazhibaeva@ntsc.kz](mailto:tazhibaeva@ntsc.kz)*

<sup>d)</sup>*Science Research Institute of Experimental and Theoretical Physics of Kazakh State University, Tole bi Str., 96a, Almaty, 480012, Kazakstan, Fax (7-3272)503978, E-mail: [istcyova@kazmail.asdc.kz](mailto:istcyova@kazmail.asdc.kz)*

<sup>e)</sup>*Nuclear Fusion Institute Russian Research Center "Kurchatov Institute" Kurchatov sq. 1 123182 Moscow, Russia Fax: (007-095)-196-7909 E-mail: [kolbasov@nfi.kiae.ru](mailto:kolbasov@nfi.kiae.ru)*

In accordance with the Task Agreement G 81 TT 02 FR, Be-steam interaction experiments were performed in order to obtain experimental data for validation of calculation codes analyzing accident situation involving water coolant ingress into the vacuum chamber of International Thermonuclear Experimental Reactor (ITER).

The report describes the experimental facility, specimens used for oxidized beryllium emissivity factor determination and the ITER first wall mock-up used in the experiments on its interaction with steam. Experimental results on Be-emissivity factor after beryllium oxidation versus temperature are given. Four experimental runs of the ITER first wall mock-up interaction with steam were carried out for initial conditions when internal (beryllium) mock-up layer was heated to temperatures of 680, 880 and 1273 K and steam temperature was of 413-423 K. The plots of temperature evolution for beryllium, bronze and stainless steel layers versus time were obtained. Temperature records with 5 s interval are presented. Hydrogen gain in these four experimental runs was measured. The data may be used for computer code validation.

No self-sustained Be-steam chemical reaction at temperatures used in the experiments was observed.

**SESSION D**

**Beryllides & Other Fields**

**Chairpersons:**

**H. Kawamura, V.Chernov**



## STATUS OF BERYLLIUM STUDY IN JAPAN

H. Kawamura

*Oarai Research Establishment,  
Japan Atomic Energy Research Institute,  
Oarai-Machi, Higashi Ibaraki-Gun, Ibaraki-Ken 311-1394 Japan.  
TEL : +81-29-264-8360, FAX : +81-29-264-8480,  
E-mail : kawamura@oarai.jaeri.go.jp*

The beryllium metal(Be) has been studied as a neutron multiplier of fusion blanket for 15 years in Japan. Up to now, the pebble fabrication technology was established and the characterization went ahead.

On the other hand, in order to aim for advanced blanket with higher temperature / high pressure coolant (super critical water) system, main R&D subject concerning neutron multiplier from now to 2005 year is decided on "Development of Beryllium Intermetallic Compounds". First of all, JAERI selected four kinds of beryllium intermetallic compounds, i.e.  $\text{Be}_{12}\text{Ti}$ ,  $\text{Be}_{12}\text{V}$ ,  $\text{Be}_{12}\text{Mo}$  and  $\text{Be}_{12}\text{W}$ , from a point of low radioactivity, high oxidation resistance, high melting point, etc.. Additionally, neutron irradiation test of beryllium metal and beryllium intermetallic compounds up to 20000appmHe is under preparation with SM-3 reactor in Russia.

Concerning the fabrication technology development of beryllium intermetallic compounds by rotating electrode method, the study on ductility improvement of beryllium intermetallic compounds was started on the base of collaboration framework among JAERI, NGK Insulators LTD. and Kitami Institute of Technology (Prof. K.Aoki), in order to endure thermal stress by electric arc.

Concerning the characterization, many kinds of studies, i.e. phase diagram of beryllium intermetallic compounds with University of Tohoku (Prof. K.Ishida), thermal and tritium release behaviors of irradiated material with University of Tokyo (Prof. S.Tanaka), effect of impurity on irradiation damage with University of Hokkaidou (Prof. H.Takahashi), tritium inventory with University of Kyushu (Prof. N.Yoshida), surface composition change by heating with University of Toyama (Prof. K.Watanabe) and mechanical properties at high temperature with Tokyo Institute of Technology (Prof. Y.Mishima) are conducting. The topics and future plan on these studies of beryllium intermetallic compounds will be presented.



## STATUS OF BERYLLIUM TECHNOLOGY ACTIVITY IN NGK

Y. Ito<sup>1</sup>, M. Miyakawa<sup>2</sup> and N. Franz<sup>2</sup>

<sup>1</sup>New Metals Division, NGK Insulators, LTD.,  
1 Maegata-cho, Handa, Aichi 475-0825, Japan

<sup>2</sup>NGK Deutsche Berylco GmbH  
Tabaksmuhlenweg 28, 61440 Oberursel, Germany  
TEL : +81-569-23-5806, FAX : +81-569-23-5856, E-mail : y-ito@ngk.co.jp

NGK is one of the world leading supplier and manufacturer of beryllium and its alloys. The development of the metal business in NGK took place in 1958. It started with the extraction of beryllium oxide from beryl ore, and the commercialization of beryllium copper master alloy. Since then, NGK have become involved in the research and development of the technology to make the most of the excellent qualities of beryllium and its alloys.

One of the biggest item of Beryllium products in NGK is beryllium copper strip. For its excellent spring characteristics as well as its high electrical conductivity, beryllium copper strip is being used in computers, automobiles and recently in mobile phones. Beryllium copper wrought products is used in repeater housings for submarine communication cables, etc. New application of beryllium copper wrought products is chill vent for aluminum die casting.

Alloy 7 is one of the new beryllium copper alloys with high formability and high electrical conductivity. It is mainly being used in mobile phone, and being studied in strength higher with same formability. Ni-Cu-Be alloy is another new developed alloy with high strength, high heat resistance and high corrosion resistance. Corrosion resistance of this alloy can be changed by copper content in nickel depend on the application.

NGK works for the material development of fusion reactor based on our beryllium technology. Rotating electrode method was developed as pebble fabrication process for neutron multiplier. Joint technology between beryllium and copper alloys becomes key technology for ITER first wall. For power generated breeding blanket, NGK is developing intermetallic compounds of beryllium that has unique property for advanced neutron multiplier. Also NGK started to develop the breeder ceramics fabrication process based on ceramics technology that NGK originally has. NGK supports the Fusion Reactor development from the field of material technology.



## DESIGN OF HEAT RESISTANT ALLOYS STRENGTHENED BY BERYLLIDES

Yoshinao Mishima

*Professor, Department of Materials Science and Engineering,  
Tokyo Institute of Technology, 4259, Nagatsuta, Midori-ku, Yokohama 226-8502, Japan  
TEL & FAX: +81-45-924-5612, e-mail: [mishima@material.titech.ac.jp](mailto:mishima@material.titech.ac.jp)*

Up to now, efforts on reducing the density of commercial heat resisting alloys, typically Ni, Co and Fe base superalloys, has been pursued by replacing heavy alloying elements with lighter elements particularly for aerospace applications. However the gain is generally rather small because the replacements are only possible for minor alloying elements.

In the present work, a series of effort is attempted to design a new class of heat resisting structural materials strengthened by beryllium intermetallic compounds in several alloy systems based on Ni, Co and Ti. The efforts include design of two phase alloys consisting of  $\text{Ni}_3\text{Al}(\gamma')$  and NiBe in the Ni-Al-Be ternary system, and Co and Ti alloys strengthened by  $\text{Ti}_2\text{Be}_{17}$  in the Co-Ti-Be and Ti-Al-Be ternary systems. In common, investigations are carried out through alloy preparation by arc melting, critical examination of the ternary phase diagram of interest by differential thermal analysis, X-ray diffraction analysis for the constitution of the alloys, micro structural characterization, and measurement of compressive mechanical properties from room temperature to 1273K.

It is shown that a superior compressive 0.2% flow stress can be obtained at elevated temperatures in each alloy system investigated over that of commercial alloys and that a fairly good room temperature ductility of several to over ten per-cent is exhibited. The alloys developed herein contain several to 20at%Be and hence have a substantial advantage over the conventional alloys in their specific strength.



## THERMAL PROPERTY OF NEUTRON IRRADIATED Be<sub>12</sub>Ti

M. Uchida, E. Ishitsuka and H. Kawamura

*Oarai Research Establishment, Japan Atomic Energy Research Institute*

*Oarai, Higashi-Ibaraki, Ibaraki 311-1394 Japan*

*TEL : +81-29-264-8368, FAX : +81-29-264-8481, E-mail : uchida@oarai.jaeri.go.jp*

Be<sub>12</sub>Ti is expected as the neutron multiplier for fusion DEMO blanket that requires high melting point, good chemical stability and low radioactivity. Thermal conductivity of neutron irradiated Be<sub>12</sub>Ti were measured to evaluate thermal property of the fusion blanket.

Be<sub>12</sub>Ti specimens (φ8 mm × 12 mm) were fabricated by HIP process from beryllium and titanium powder, and were irradiated by JMTR (Japan Material Testing Reactor) with a total fast neutron fluence (E>1MeV) of  $4 \times 10^{20}$  n/cm<sup>2</sup> at 330, 400 and 500 °C. Thermal diffusivity and specific heat of un-irradiated and irradiated one were measured up to 800°C by laser flash method and thermal conductivity was calculated.

Thermal conductivity of un-irradiated Be<sub>12</sub>Ti was constant up to 800°C. It was quarter of that of beryllium at R.T and was half of that of beryllium at 800°C since thermal conductivity of beryllium decreased with temperature increasing. This data showed that Be<sub>12</sub>Ti would not have so big disadvantage on thermal property at higher temperature. Thermal conductivity of irradiated Be<sub>12</sub>Ti was quarter of that of un-irradiated one and increased with temperature increasing. Finally the value became same as un-irradiated one at 800°C. One more heating and measurement was performed for the same specimens and the value maintained constant up to 800 °C. It was considered that irradiation defect decreased thermal conductivity and the recovery of the defect by annealing put the thermal conductivity back as un-irradiated.

Thermal conductivity was measured for un-irradiated and irradiated Be<sub>12</sub>Ti and unique property was obtained. In this workshop, the mechanism of thermal property difference between Be<sub>12</sub>Ti and beryllium will be presented.

COMPATIBILITY TEST BETWEEN  $\text{Be}_{12}\text{Ti}$  AND SS316LNH. Kawamura<sup>1</sup>, V. Shestakov<sup>2</sup> and M. Uchida<sup>1</sup>

<sup>1</sup>*Japan Atomic Energy Research Institute, Oarai Research Establishment,  
Oarai-Machi, Higashi Ibaraki-Gun, Ibaraki-Ken 311-1394 Japan.*

<sup>2</sup>*Science Research Institute of Experimental and Theoretical Physics,  
Kazakh State University, Tole bi Str. 96a, 480012 Almaty, Kazakhstan*

*TEL : +81-29-264-8360, FAX : +81-29-264-8480,*

*E-mail : kawamura@oarai.jaeri.go.jp*

Beryllium metal (Be) is commonest as a neutron multiplier of fusion blanket. However, the melting point of Be is low (1285°C) and the starting temperature of swelling by neutron irradiation is low (~550°C). On the other hand, beryllium intermetallic compounds have good properties like higher melting point (over 1600°C), high oxidation-resistance, etc.. Therefore, in order to widen a design window of fusion blanket, the compatibility test was conducted between  $\text{Be}_{12}\text{Ti}$  which is one of beryllium intermetallic compound and SS316LN which is one of candidate structure materials.

The diffusion couples of  $\text{Be}_{12}\text{Ti}$  and SS316LN were used for this compatibility test. Both surfaces of  $\text{Be}_{12}\text{Ti}$  and SS316LN were polished by buff. Then, the diffusion couples were sealed in an ampule with high purity helium (99.9999%) by TIG welding and were annealed. Annealing temperatures were 600°C, 700°C and 800°C, and annealing periods were 100h, 300h and 1000h, respectively.

From the results of this test, it was obvious that the reaction thickness of SS316LN side between  $\text{Be}_{12}\text{Ti}$  and SS316LN was only 30μm at 800°C for 1000h and was one tenth comparing with the reaction thickness of SS316LN side between Be and SS316LN at same annealing condition. Then, the reaction thickness of SS316LN side between  $\text{Be}_{12}\text{Ti}$  and SS316LN was not observed at same annealing condition, though the reaction thickness of SS316LN side between Be and SS316LN was 30μm at 600°C for 1000h.

In this workshop, the reaction mechanism between  $\text{Be}_{12}\text{Ti}$  and SS316LN will be presented.



## PRELIMINARY NEUTRONIC ESTIMATION FOR DEMO BLANKET WITH BERYLLIUM INTERMETALLIC COMPOUND

K Tsuchiya<sup>1</sup>, Y. Nagao<sup>1</sup>, H. Yamada<sup>2</sup>, M. Nakao<sup>2</sup>, and H. Kawamura<sup>1</sup>

<sup>1</sup> *Oarai Research Establishment, Japan Atomic Energy Research Institute,  
Oarai, Higashi-Ibaraki, Ibaraki 311-1394, Japan*

<sup>2</sup> *Power Plant Division, Kawasaki Heavy Industries, LTD.,*

*2-6-5 Minamisuna, Koto-ku, Tokyo 136-8588, Japan*

*TEL: +81-29-264-8369, FAX: +81-29-264-8481, E-mail: yamada@oarai.jaeri.go.jp*

Some kinds of beryllium intermetallic compounds ( $\text{Be}_{12}\text{Ti}$ ,  $\text{Be}_{12}\text{W}$  etc.) are considered as one of candidate materials as neutron multiplier. In this study, preliminary neutronic estimation was conducted concerning DEMO blanket that beryllium intermetallic compound was packed in the blanket container with multi-layered structure.

The whole inside width of the horizontal cross section of fusion blanket is 390mm, and this horizontal cross section is divided into eight zones by seven plates. Each width of divided eight zones is 10mm, 100mm, 10mm, 50mm, 20mm, 40mm, 60mm and 100mm in turn from plasma side. The neutron multiplier pebbles are packed in both the 2<sup>nd</sup> zone (100mm width) and 4<sup>th</sup> zone (50mm width). In all other six zones, tritium breeder pebbles are packed. The packing fraction of both pebbles is 80%. The structural material and coolant are the ferritic steel (F82H) and pressurized water (15Mpa / 320°C), respectively. And as tritium breeder,  $\text{Li}_2\text{TiO}_3$  was selected, and  $\text{Be}_{12}\text{Ti}$  and Be were selected as neutron multiplier. The neutronic calculations of DEMO blanket were performed using the two-dimensional discrete ordinates code DOT3.5 with multi-group neutron and gamma cross section set (FUSION40) proceeded from JENDL 3.2. The neutron wall loading at the first wall was assumed to be 5MW/m<sup>2</sup> in this study.

From the results of this estimation, it was obvious that Tritium Breeding Ratio (TBR) on blanket with  $\text{Be}_{12}\text{Ti}$  pebbles was 1.1 and that this TBR is 10% smaller than TBR on blanket with Be pebbles. Therefore, there will be the bright prospect concerning the application of beryllium intermetallic compound as neutron multiplier. In this workshop, I will also present the results of thermal estimation, etc..





## MICROSTRUCTURE ANALYSIS OF $\text{Be}_{12}\text{Ti}$ AND ITS IRRADIATION RESPONSE BY DUAL BEAM IRRADIATION

H. Takahashi<sup>1</sup>, T. Shibayama<sup>1</sup>, M. Uchida<sup>2</sup>, H. Kawamura<sup>2</sup> and Y. Ito<sup>3</sup>

<sup>1</sup>*Center for Advanced Research of Energy Technology, Hokkaido University  
Sapporo 060-8628, Japan*

<sup>2</sup>*Oarai Research Establishment, Japan Atomic Energy Research Institute,  
Oarai, Higashi-Ibaraki, Ibaraki 311-1394 Japan*

<sup>3</sup>*New Metals Division, NGK Insulators, LTD.,  
1 Maegata-cho, Handa, Aichi 475-0825, Japan*

TEL : +81-11-706-6767, FAX : +81-11-757-3537, E-mail : takahash@ufml.caret.hokudai.ac.jp

Beryllium is presently a candidate material as the neutron multiplier in a solid breeder blanket system for the international thermonuclear experimental reactor (ITER). On the response of beryllium to 14 MeV neutron irradiation, it would degrade by not only displacement damage and also transmutation products. It has been estimated to be over 20,000 appm  $^4\text{He}$  generation by transmutation of  $^8\text{Be}$  for DEMO reactor. Especially, He gas driven swelling and mechanical property degradation could be severe issue beyond DEMO reactor. It is important to evaluate the effects of He irradiation on microstructure evolution in Be. From high efficiency operation point of view, the requirements to materials performance are increasing.

Recently,  $\text{Be}_{12}\text{Ti}$  has been successfully developed to improve mechanical properties at the elevated temperatures by co-authors.  $\text{Be}_{12}\text{Ti}$  might have an attractive feature for high temperature properties rather than the present.

In this study, microstructure response in  $\text{Be}_{12}\text{Ti}$  as a function of irradiation temperature, dose and spontaneous He irradiation effects were evaluated by in-situ experiment using Multi Beam High Voltage Electron Microscope and compared with the standard Be. Nano indentation tests also performed to investigate mechanical property changes in  $\text{Be}_{12}\text{Ti}$  before and after He irradiation up to 20,000 appm.

TRITIUM RELEASE FROM NEUTRON IRRADIATED Be<sub>12</sub>TiE. Ishitsuka<sup>1</sup>, M. Uchida<sup>1</sup>, H. Kawamura<sup>1</sup> and T. Terai<sup>2</sup>

<sup>1</sup>*Japan Atomic Energy Research Institute, Oarai Research Establishment,  
Oarai, Higashi-Ibaraki, Ibaraki 311-1394 Japan*

<sup>2</sup>*University of Tokyo, 7-3-1 Hongo, Bunkyo-Ku, Tokyo 113 Japan  
TEL : +81-29-264-8368, FAX : +81-29-264-8480, E-mail : ishi@oarai.jaeri.go.jp*

Be<sub>12</sub>Ti is expected as the neutron multiplier of fusion DEMO blanket that requires high melting point, good chemical stability and low activity. Tritium release experiments of Be<sub>12</sub>Ti were carried out to evaluate the tritium inventory in the fusion blanket.

Be<sub>12</sub>Ti specimens (φ8 mm × t2 mm) were fabricated by HIP process from beryllium and titanium powder, and were irradiated by JMTR (Japan Material Testing Reactor) with a total fast neutron fluence (E>1MeV) of  $4 \times 10^{20}$  n/cm<sup>2</sup> at 330, 400 and 500 °C. After irradiation, specimens were heated in a small electric furnace under He+1%H<sub>2</sub> sweep gas condition controlled at 50 cm<sup>3</sup>/min. Heating temperature was 300, 600, 900 and 1100°C, and hold time at each temperature was 30 minutes.

Effective diffusion coefficient of Be<sub>12</sub>Ti shows about two orders larger than that of beryllium at 600~1100°C, and shows about seven orders larger at about 300°C. A small amount of the released tritium from the irradiated specimens at 500 °C was observed because the almost tritium has already released during the neutron irradiation. Generally, titanium is known as typical element to make the metal-hydride, and will act as the traps of tritium. However, this experiments showed the different results from this estimation. Further study is necessary in order to clarify this tritium release mechanism from Be<sub>12</sub>Ti. Effects of the density, grain size and oxide layer should be considered to understand.

Finally, density change of Be<sub>12</sub>Ti after heating was the below 1%, and very small than that of beryllium. These results suggest that the swelling of Be<sub>12</sub>Ti is lower than that of beryllium even in high temperature irradiation.