Steel-based materials surface damage and modification under high power plasma exposures

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Outline

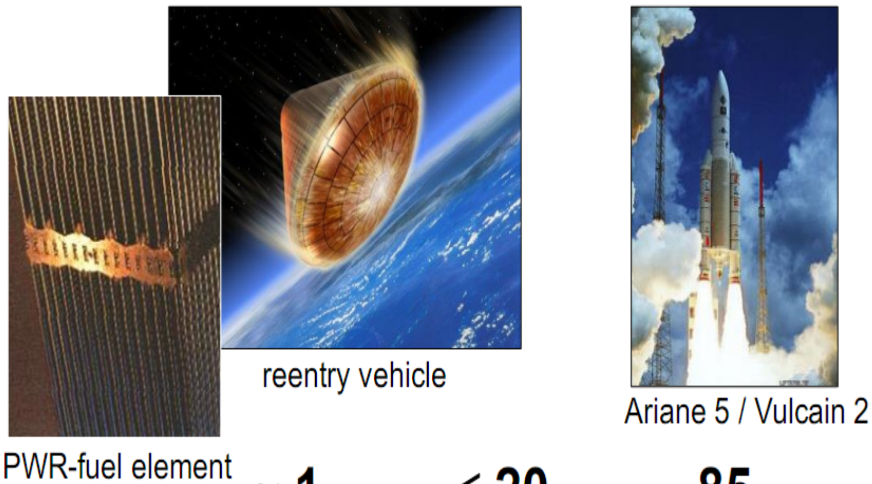
- Introduction

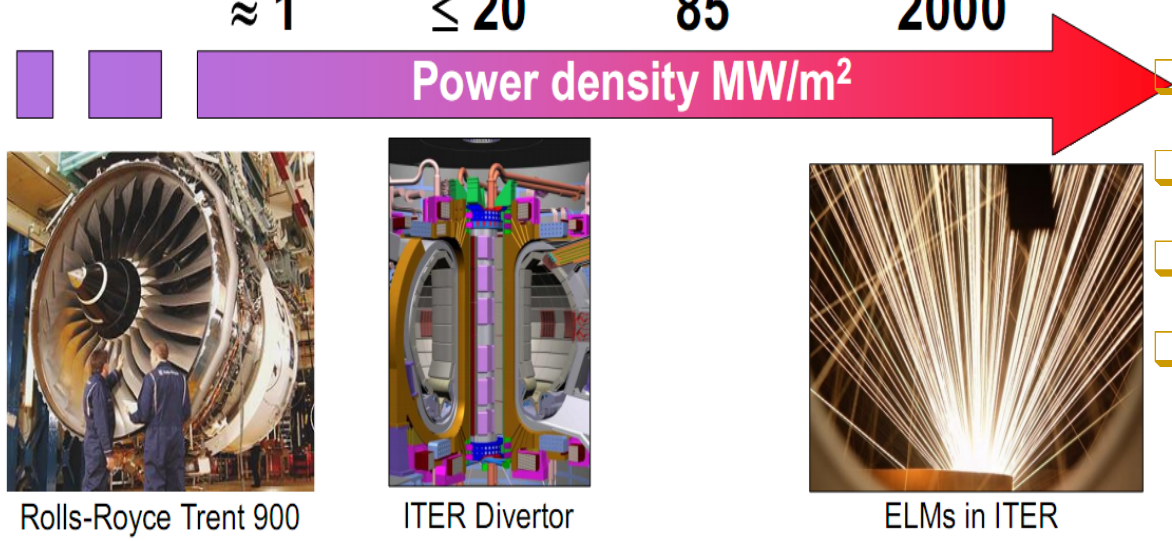
- Experimental facility and samples
  - QSPA Kh-50
  - MPC
  - DSM-2; DSM-1
  - GAS
- Feature of modification and alloying of steels
- First results of hydrogen outgassing of steels modified by pulsed plasma streams

- Conclusions

Introduction

Simultaneous impacts of high energy and particle loads to the material surface are typical for material performance in various extreme conditions:





- turbines
- space apparatus,
- nuclear engineering
- fusion

J. Linke , PFMC-16, Neuss, 15-19, May 2017

Introduction

Overview of Main ITER Components and Materials

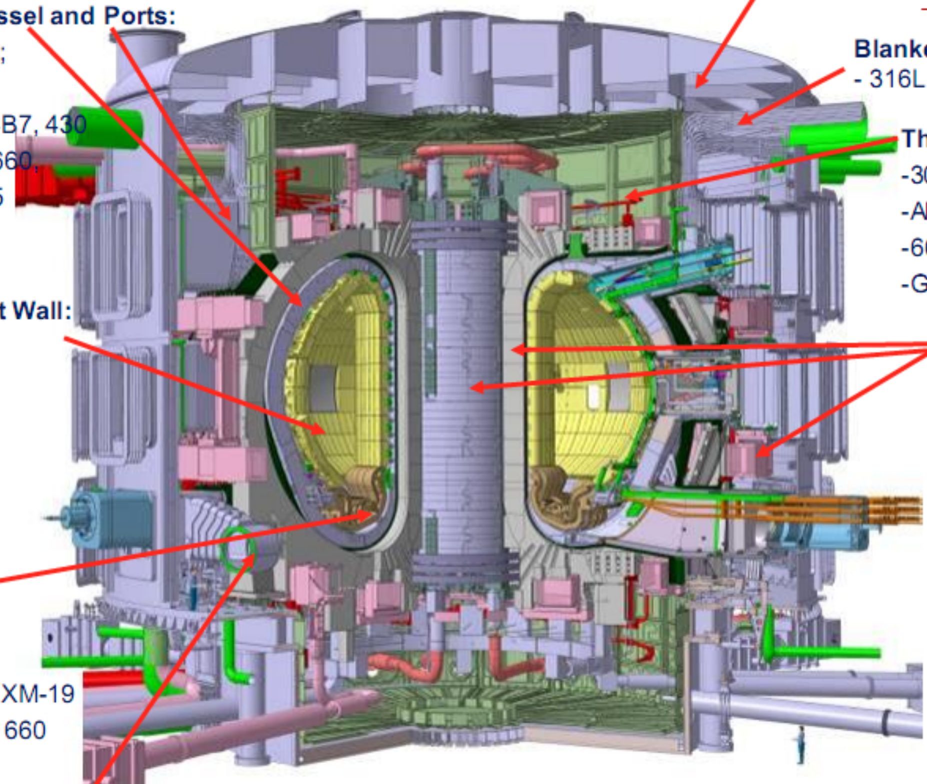
Cryostat:
- 304 /304L
Total weight: ~ 3300 t

Vacuum vessel and Ports:
-316L(N)-IG;
-304L, 304
-304B4, 304B7, 430
-Alloy 718, 660,
-XM-19, 625
Total weight: ~ 5100 t

Blanket manifolds
- 316L
Total weight: ~ 140 t

Thermal Shield:
-304L,304LN
-Alloy 718,
-660
-G10, G11
Total weight: ~ 880 t

Materials issues are key factors for ITER success



Blanket/First Wall:
-Beryllium;
-CuCrZr,
-NiAl bronze
-316L(N)-IG
-Alloy 718,
Total weight: ~ 1530 t

Magnet System:
-Nb_3Sn, NbTi, Cu
-316LN, JJ1, 316L
-Alloy 718,
-Epoxy, glass fiber

Divertor:
-W
-CuCrZr
-316L(N)-IG, XM-19
-NiAl bronze, 660
Total weight: ~ 470 t

Magnets weight:
TF coils – 6117 t
PF coils – 2163 t
CC coils – 191 t
CS coils – 953 t
Feeders ~ 1030 t

Cryopumps:
- 316L
Total weight: ~ 50 t

Total (Cryostat+ VV+ Magnet) ~ 23440 t

iter china eu india japan korea russia usa

ICFRM-17 , Aachen, Germany, 11-16 October 2015

B. Bigot, Key note lecture during ICRFM-17

Introduction

- **Tungsten is chosen as main plasma facing material for ITER and DEMO divertor design.**
- **Tungsten coatings can also be alternative to the monolithic material, especially for large area of the DEMO reactor first wall.**
- **Stainless steels (SS) are one of main material for next step fusion devices. However, the large sputtering rate under high energy partials is main disadvantage of SS as armour material.**
- **One of the potential ways of improving these properties is by alloying theirs surface layer with heavy elements.**
- **Alloying of surface layer by mixing previously deposited thin ($h_{coat} < h_{melt}$) coatings is one of possibility options**

Evaluation of hydrogen/helium retention (outgassing) rate is also important issue for fusion reactor.

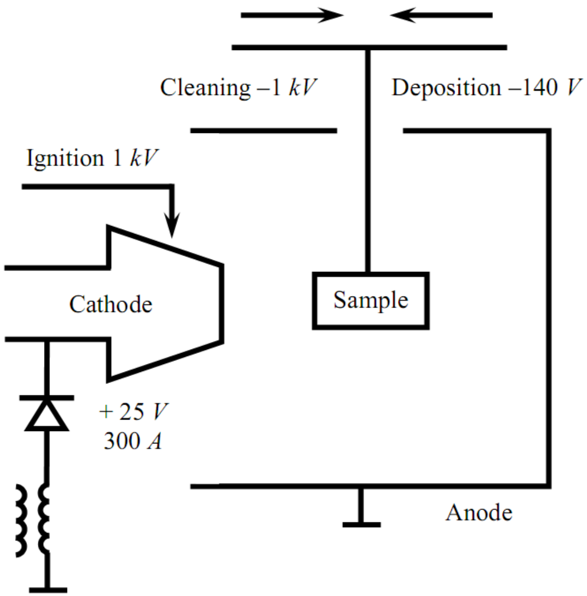
The detailed experimental studies of threshold values for the damaging processes under fusion reactor relevant loading scenarios are required for evaluation of the materials performance under high heat fluxes.

High power plasma streams is unique tool for surface modification

Combination of physical mechanisms:
ion bombardment,
heat load (melting, but no evaporation, thermal quenching),
shock waves,
material alloying with plasma species,
mixing in molten stage…………

The ferritic/martensitic Eurofer and Rusfer steels were modified and alloyed with tungsten by plasma induced mixing.

Stainless steel targets were covered with tungsten by PCD method in facility of Bulat family.



Coatings were deposited by PVD method during 3 min in argon of p = (4…5)·10^{-4} Torr.

Parameters of arc discharge are next: current of arc I_{arc} = 230 A and displacement voltage of U_{bias} = 140 V.

Before depositions of coatings, the ionic clearing (duration 2 min, U_{bias} = 1.5 kV and I_{arc} = 100 A) was applied

The thickness of coatings is about 3 μm

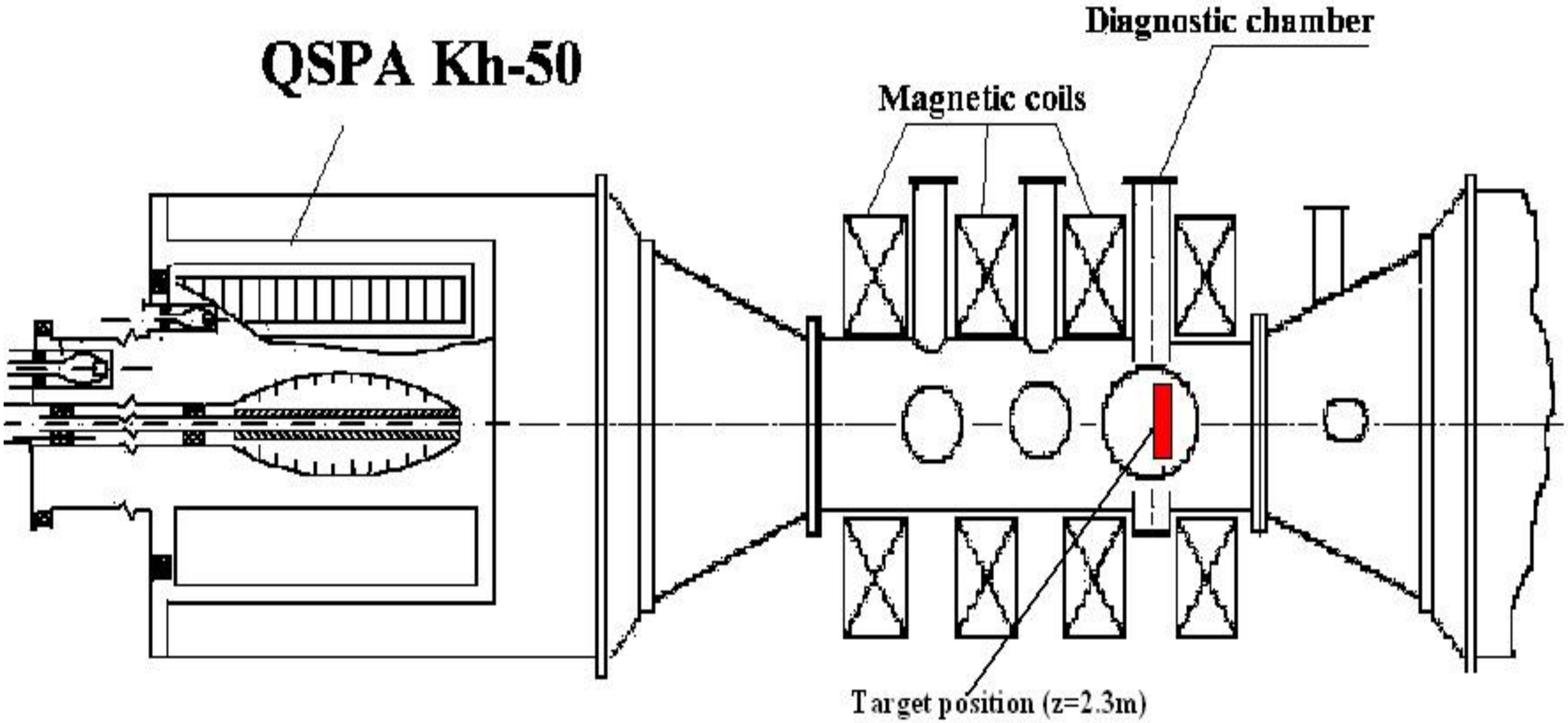
•Ten EUROFER targets of 10mm×10mm×1 mm received from Dr. Dmitry Terentyev, SCK-CEN Belgium Nuclear Research Centre, Mol, BELGIUM
•Two RUSFER steel have been received from Dr. Anna Golubeva, NRC "Kurchatov Institute", Moscow, Russia

- The energy range of ITER disruptions and ELMs will be clearly higher than in the existing tokamaks
- Material response to multiple exposures are studied with ion and electron beams, liner facilities, pulsed plasma guns and QSPA,
- **QSPA is attractive for :**

- Reproduction of heat and particle loads typical for disruptions and ELMs
- Study of plasma/surface interaction (shielding, melting, evaporation, erosion mechanisms) and dynamics of erosion products
- Qualification of PFM&PFCs in extreme conditions
- Comparison of load effects with other simulators. Data for validation of numerical models

QSPA Kh-50 is largest and most powerful device among QSPAs

QSPA Kh-50 Device



Energy density $\rho_w = (0.5\ldots30)$ MJ/m^2,

Plasma pulse duration $\tau \approx 0.25$ ms;

$P_{max} = (3\text{-}18)$ bar, $n = (0.2\text{-}5)\ 10^{16}$ cm^{-3}; $B_0 = 0.54$ T ($\beta \approx 0.3\ldots0.4$);

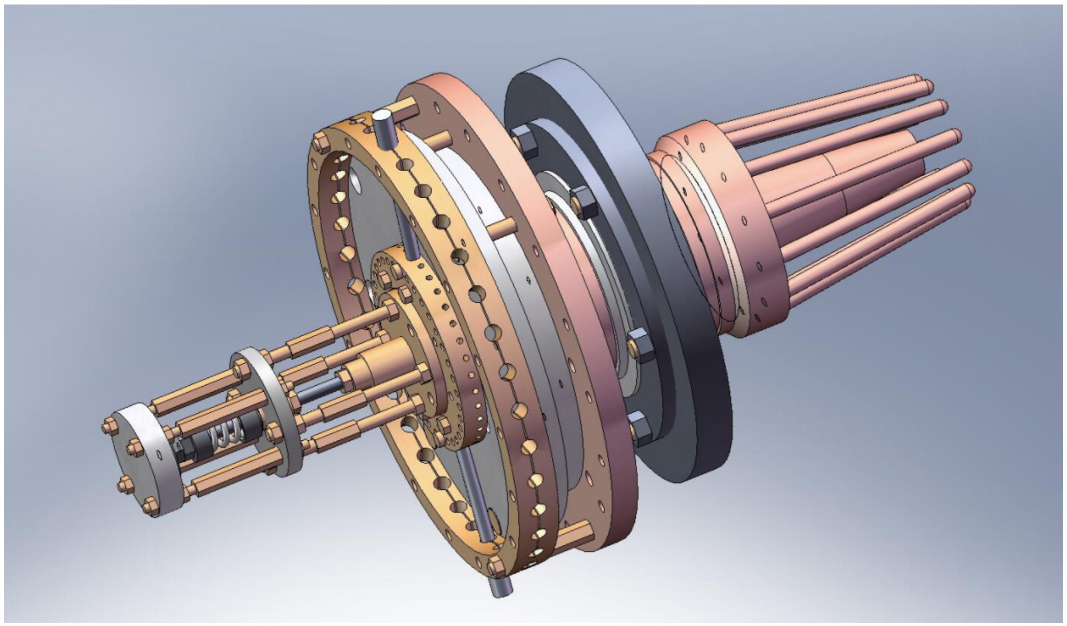
Diameter of plasma stream- 15 cm

Examples of Plasma Parameters in Different Regimes QSPA Kh-50

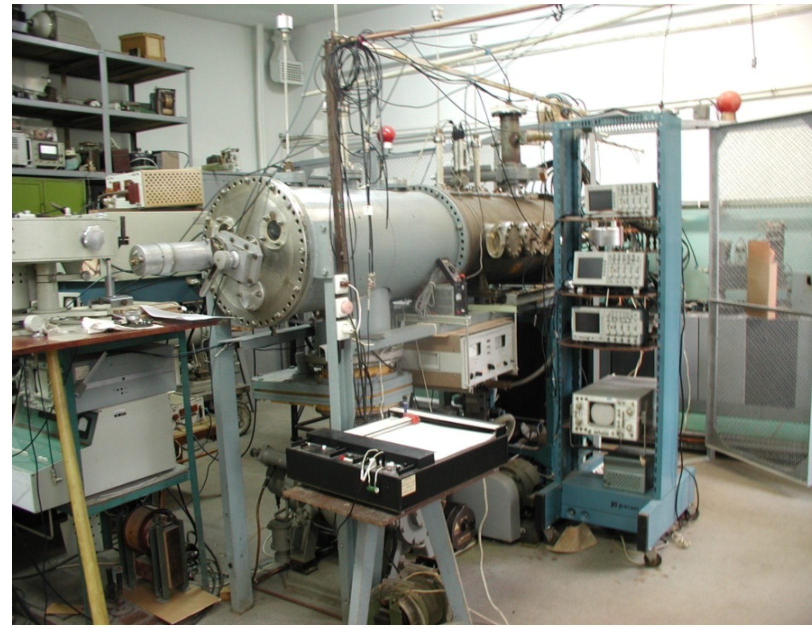
Parameters	ELM 0	ELM 1	ELM 2	ELM 3	Disruption
Plasma stream energy density [MJ/m^2]	0.4	0.9-1.0	1.2-1.5	2.4-2.5	24-30
Target Heat Load [MJ/m^2]	0.22	0.45	0.7-0.75	1-1.1	0.65-0.7 (strong vapor shielding)
Plasma load duration [ms]	0.25	0.25	0.25	0.25	0.2-0.25
Maximal dynamical pressure of plasma stream [MPa]		0.48	0.32	0.45	1,7
Average plasma density [10^{16} cm^{-3}]		1.5-2.5	0.5-0.7	0.2-0.3	4-8
Plasma stream diameter [cm]	12-14	12-14	16	16	14
Surface effects	below crack threshold	no melting	melting	Evaporation start	Strong vapor shield

➢Due to a vapor shield formation the exposed armour target will be protected from the high heat load and erosion by evaporation will be reduced in hundred times

Experimental facility MPC



Electrode system of MPC

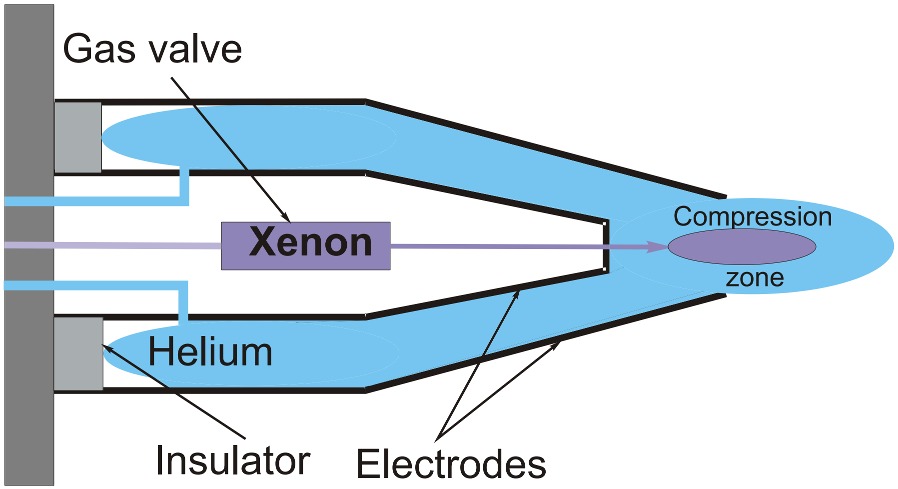


General view of MPC with diagnostics

∅cathode(outer electrode) = 6cm, 3 cm
∅ anode (inner electrode) = 12 cm, 8 cm
Copper rods diameter of 1 cm and of 14.7 cm in length

C_c= 90 µF U_c = 20÷30 kV
C_v = 700 µF U_v = 3 ÷ 5 kV
I_d = 500 kA T_d = 15 ÷ 20 µs
Working gas – helium, Xe+He

Operation modes of MPC

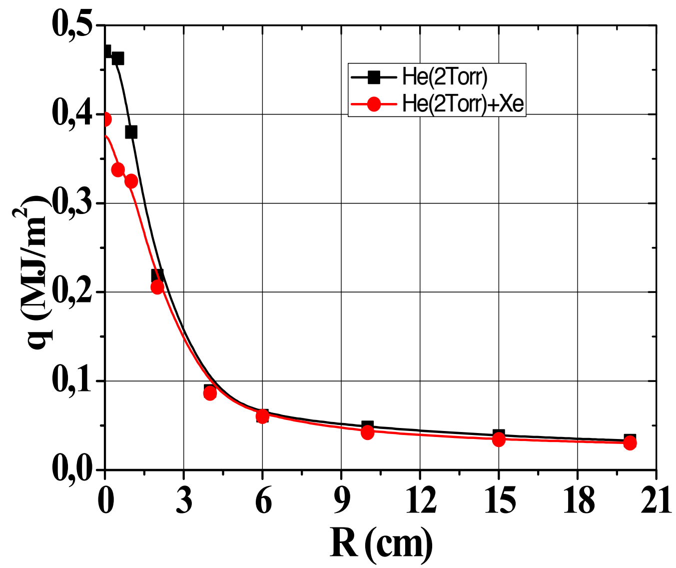


discharges in helium under different residual pressures with additional pulsed injection of Xe directly into the compression zone

Maximal heat load to tungsten surface exposed to helium plasma achieved 0.39 MJ/m².

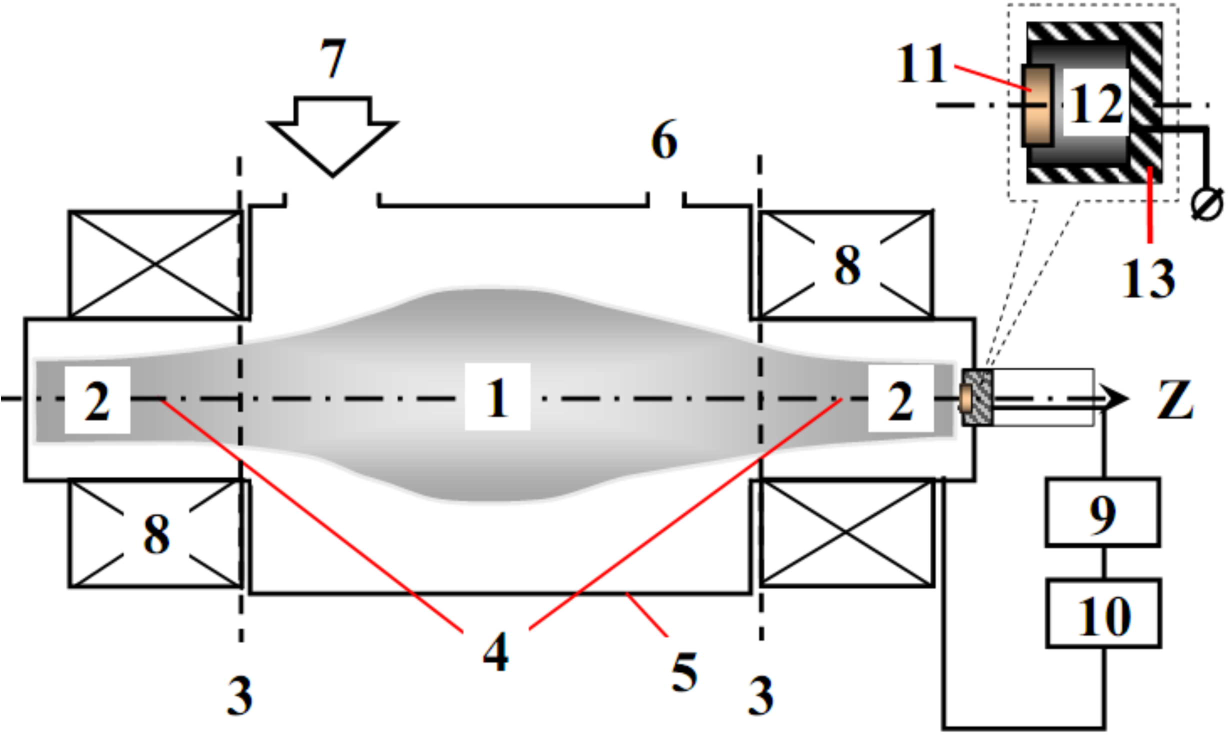
The heat load to tungsten surface exposed under additional injection of xenon is decreased to 0.33 MJ/m².

Radial profile of energy density in MPC plasma stream



Decreasing of total energy measured by colorimeter is caused by losses to ionization of heavy impurities (Xe).

DSM-2 SOURCE FOR MODELING OF LONG-TIME SPUTTERING INFLUENCE ON SURFACE STRUCTURE



1,2 – central and edge plasma volumes with approximate boundaries between them
3, 4 – magnetic mirrors,
5 – vacuum vessel,
6 – gas puffing,
7 – ECR power input,
8 – magnetic coils.
Source of accelerating voltage:
9 – alternating voltage,
10 – direct-current voltage.
11 – mirror sample,
12 – Cu holder of Sample under the test,
13 – teflon insulator.

Ion flux ~10^{16} ion/cm^2,
ion energy range 30 eV – 1500 eV (fixed or time variable),
ion fluence ~$3 \cdot 10^{21}$ ion/cm^2,
sample temperature RT-200 ^{0}C.

Studies of exposed surfaces

Surface analysis carried out with optical microscope equipped with CCD camera,
Roughness of surface was also measured.
XRD fluorescence method was used for measurements of elements content
Changes of phase state on the surface were obtained from XRD spectrum analysis

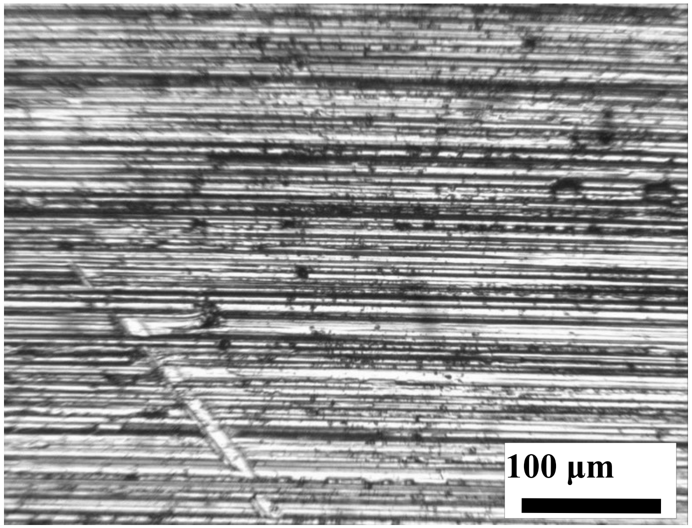
Parameters of structure

$a_0 < a_{ref}$ the surplus of vacancies; $a_0 > a_{ref}$ the surplus of interstitial atoms ($a_{refW} = 0.3165$ nm, $a_{\alpha\text{-}Fe\_ref} = 0.2866$ nm).

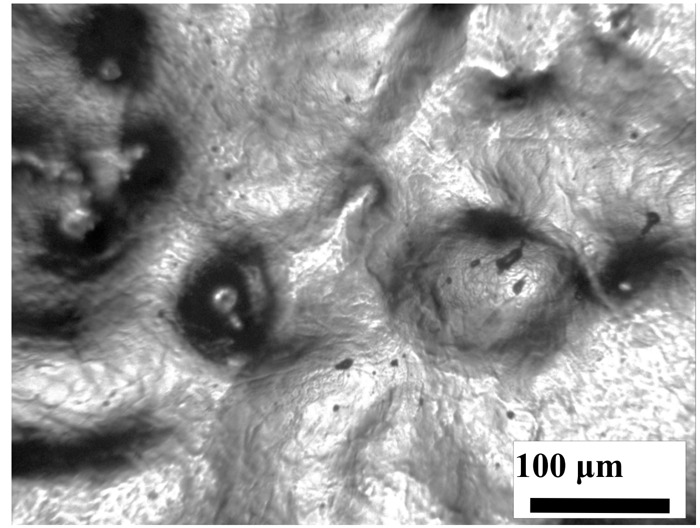
B - the width of the profile is proportional to the number of line defects (dislocations) in the structure.

The asymmetry (δB) is attributed by the presence of complexes of point defects. The sign of δB is caused by the type of defects: vacancies ($\delta B > 0$) or interstitial atoms ($\delta B < 0$).

Alloying and modification with repetitive plasma pulses



Before irradiation



After irradiation

Steel #45 samples covered of PVD coatings.

5 QSPA Kh-50 pulses with energy load of 0.5 MJ/m², pulse duration 0.25 ms

Element content (wt %) of 45Steel Initial

	Cr	Mn	Ni	Fe
Element content	---	0.41	---	99.59

Element content (wt %) of 45Steel+Cr+QSPA+Ni+QSPA

	Cr	Mn	Ni	Fe
Element content	3.346	0.679	2.501	93.474

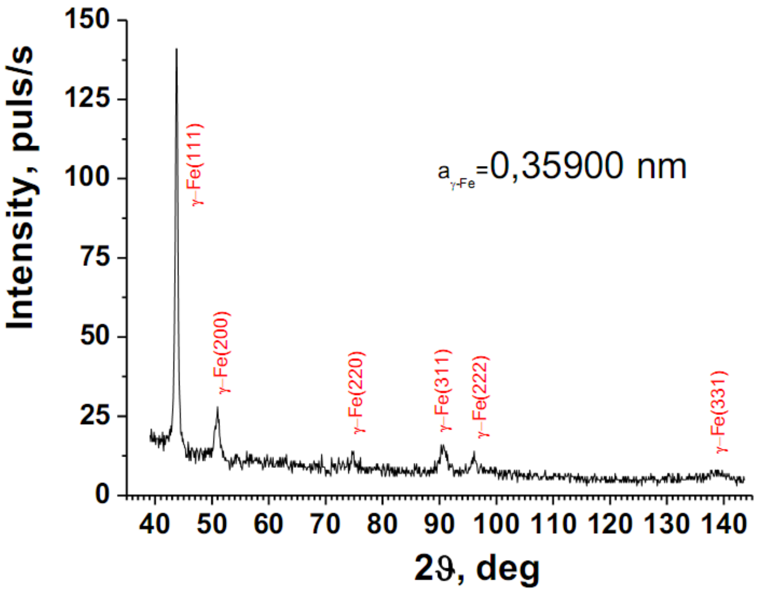
Element content (wt %) of 45Steel+Cr+Ni+QSPA

	Cr	Mn	Ni	Fe
Element content	5.467	0.523	4.227	89.783

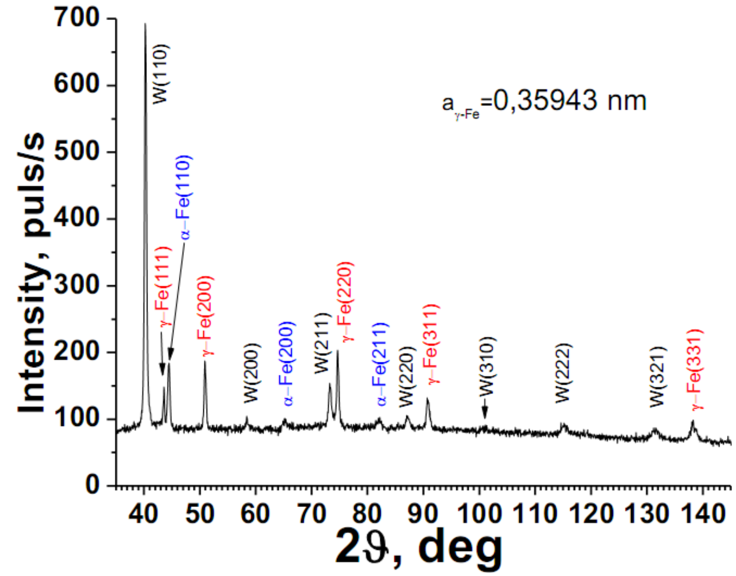
Coatings modification under heat load above melting threshold

Diffraction patterns (Cu-Kα irradiation) of Cr18Ni10Ti stainless steel (analog SS321)

Initial



preliminary coated with tungsten (W) and exposed by powerful plasma streams

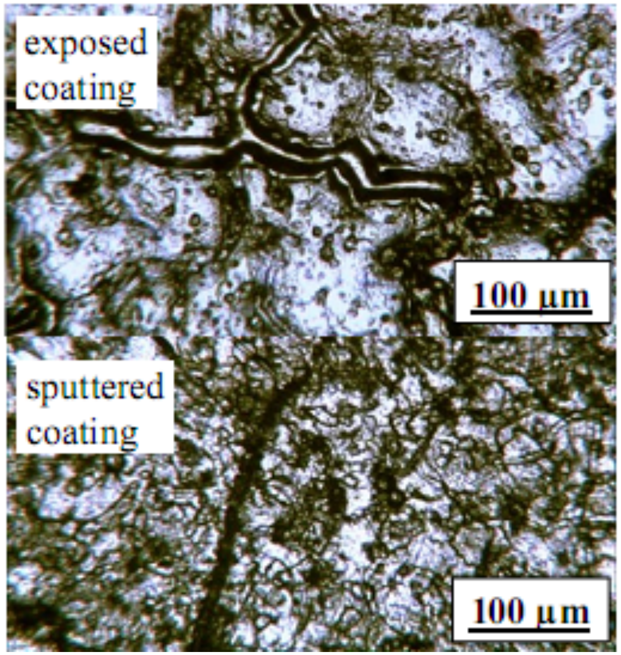


- α-Fe phase is recognized together with lines of γ-Fe phase and W on exposed surfaces.
- Lattice spacing of γ-Fe in stress free state increased from 0.359 nm up to $a_{\gamma\text{-}Fe}$= 0.35943 nm.
- The concentration of tungsten achieved 2 a.p. in modified layer.
- Tungsten penetrated up to 0.38 μm in depth of modified layer.
- Presence of tungsten leads to decrease of sputtering rate of stainless steel surface.

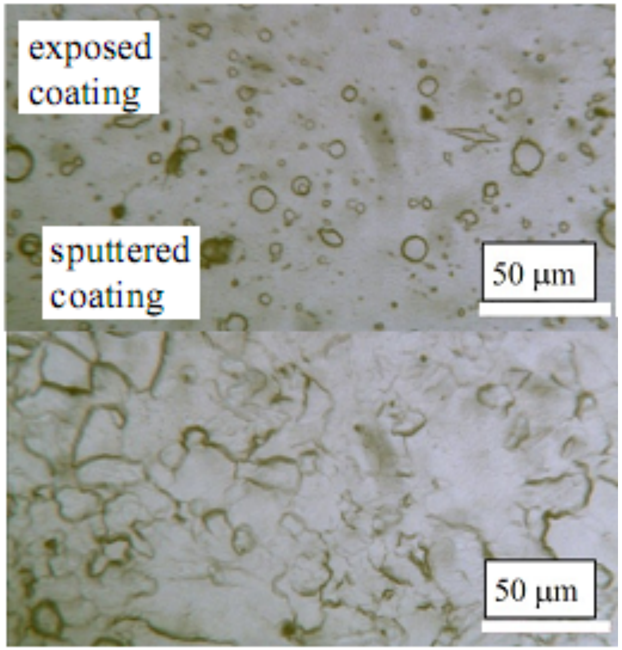
V. A. Makhlaj , et. al. Prob. At,. Sci and Tech 2016, №6(106), p. 129)

Modified tungsten coating

MPC

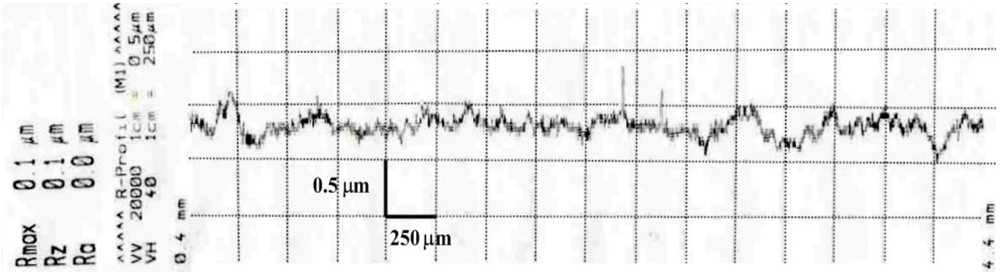


QSPA

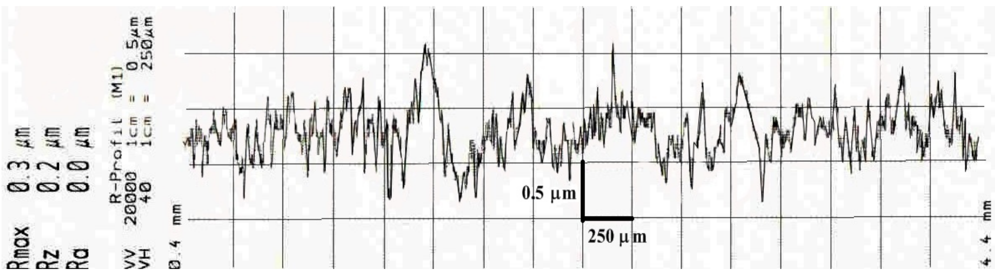


- Melted, re-solidified layer developed on exposed surfaces.
- Modified layer includes of material coating and substrate.
- Macro and micro cracks appear on exposed surfaces

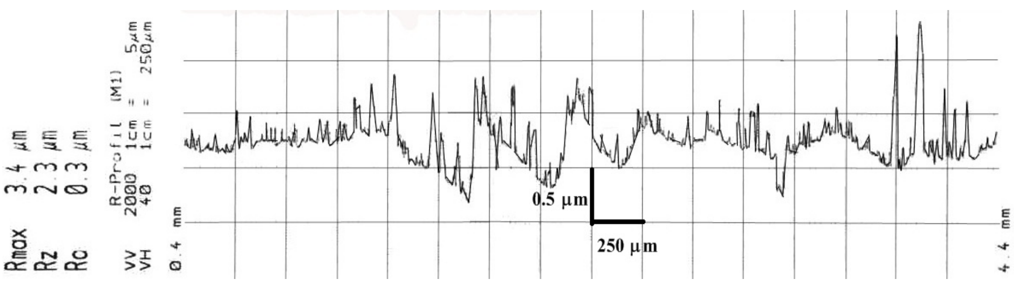
Profiles of Eurofer before and after plasma exposure



Initial



Irradiated by 5 QSPA plasma pulses;

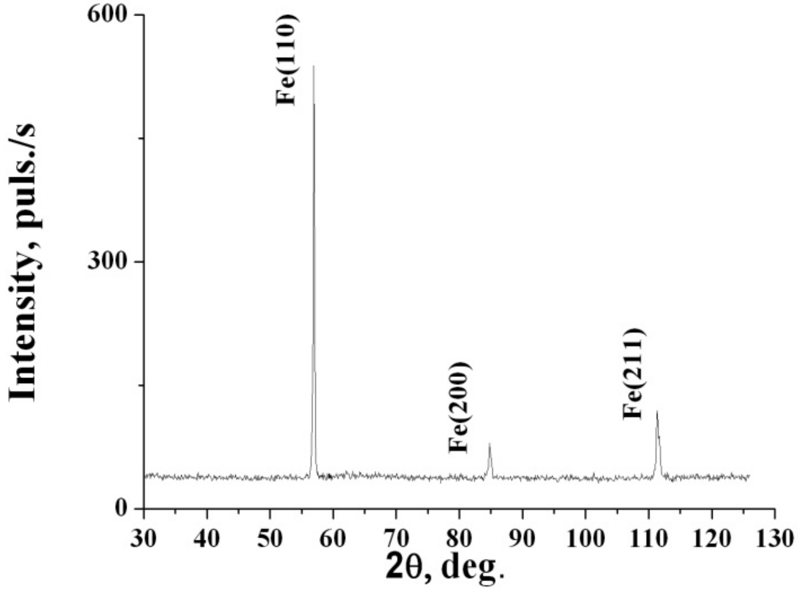


Surface covered by W and exposed to QSPA plasma

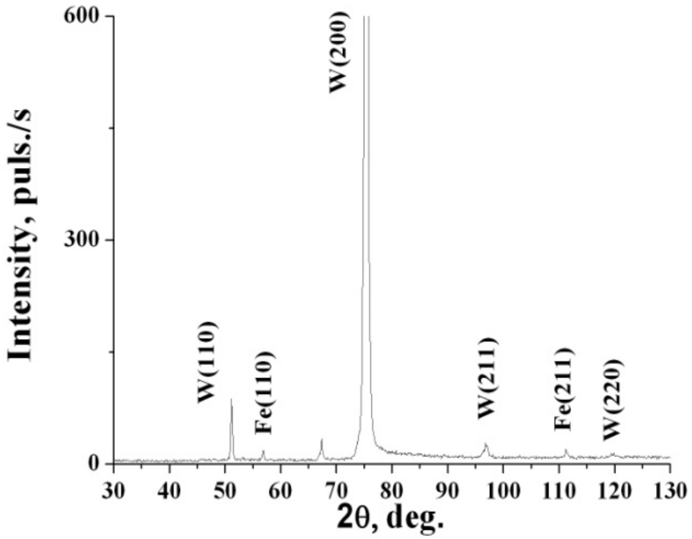
Heat load Q = 0.6 MJ/m^2

The roughness of exposed surfaces increases due to appearing of crack, melting of surface layer as well as etching and sputtering of boundaries of cracks and grains.

Results of XRD spectral analysis



Initial

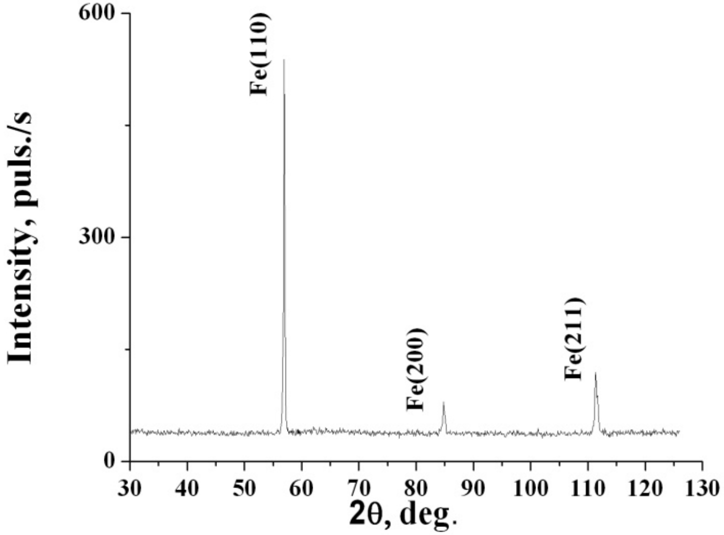


Surface coated by tungsten and exposed to QSPA plasma

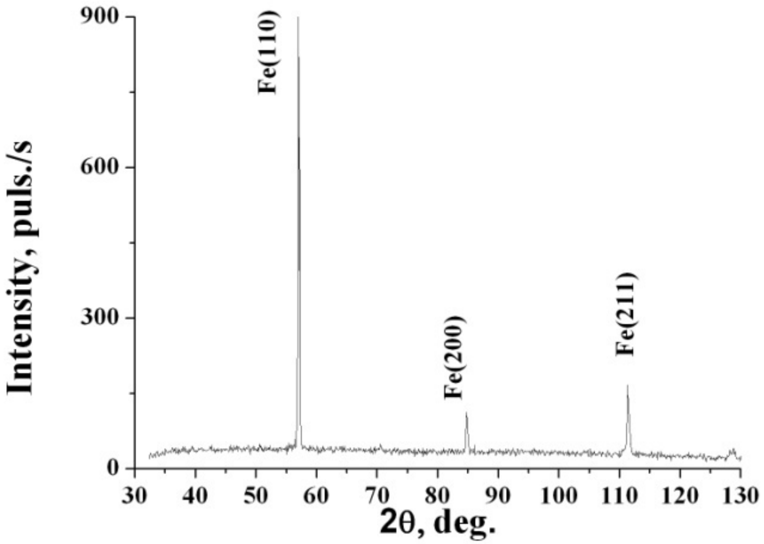
Diffraction patterns (Fe-K α1 irradiation)

- **Only lines of Fe phase are observed on non treatment surfaces.**
- **Surface modification led to penetration of tungsten in affected layer.**
- **W phases are recognized together with lines of Fe phase on treatment surfaces.**
- **Intensity of tungsten lines is more at times in compare with intensity of substrate lines.**

Results of XRD spectral analysis



Initial



W coating modified by QSPA plasma and irradiated by Ar^+ beam.

- **W lines are not recognized after sputtering of surface**
- **Intensity of substrate lines increased in compare with initial state.**

Content of tungsten (at %) in Eurofer as solid solution

	lattice spacing a_0, A	Content of tungsten
Initial	2.8714	0.8
Modified by plasma stream and sputtered by Ar beam	2.8676	0.8
W coatings sputtered by Ar beam	2.8717	1.3
Coated W, exposed by QSPA and sputtered by Ar beam	2.8703	0.85
Coated W, exposed by MPC and sputtered by Ar beam	2.8704	0.9

Results of XRD fluorescence studies

Element content (wt %) of Eurofer

	Cr	W	Fe
Initial	9.7	0.8	Base
Coated W and exposed by QSPA	1.3	85.8	Base
Coated W, exposed by QSPA and sputtered by Ar beam	8.9	1.1	Base
Coated W and exposed by MPC	9.7	1.1	Base
Coated W and sputtered by Ar beam	9.6	1.1	Base

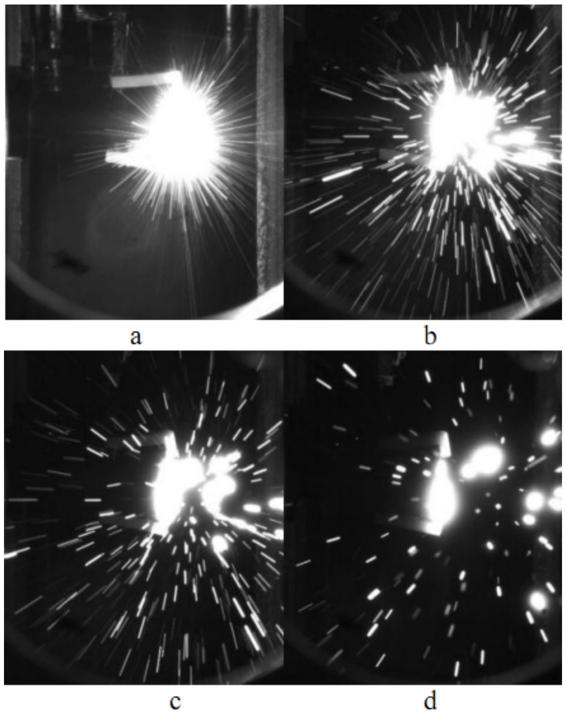
Microhardness by Vickers, kg/mm^2

Initial	Exposed QSPA	W coatings exposed QSPA	Exposed MPC	W coatings exposed MPC
186-213	310 (213)	260 (186)	270 (213)	295 (186)

Initial Microhardness is indicated in brackets for each samples
Plasma treatment leads to increases of microhardness
It is indicated on accumulation of elastic energy in the stressed surface layer.
As result of this the delamination of coatings developed

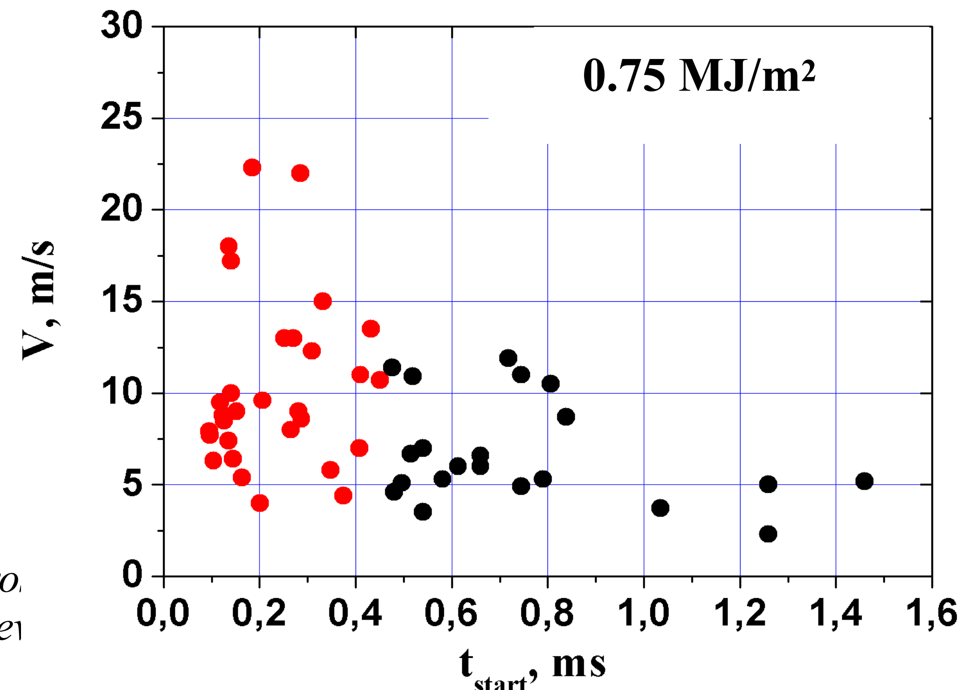
The delamination of coatings are also observed, especially when the surface is irradiated by short plasma streams.

SURFACE MODIFICATION OF TUNGSTEN COATINGS



Frames of the digital camera with the traces of ero[…] products (exposure time 1.2 ms). The camera's vie[…] parallel to the target surface:
a – 2.4 ms; b – 4.8 ms; c – 8.4 ms; d – 15.6 ms after the start of plasma-surface interaction

Velocity distribution of ejected particles vs. their start time from the surface



Plasma impacts with loads above the melting threshold cause the droplet/dust particles ejection from the surface of tungsten coating

The possible way to improve coatings resistance is application of several cycles of plasma treatment.

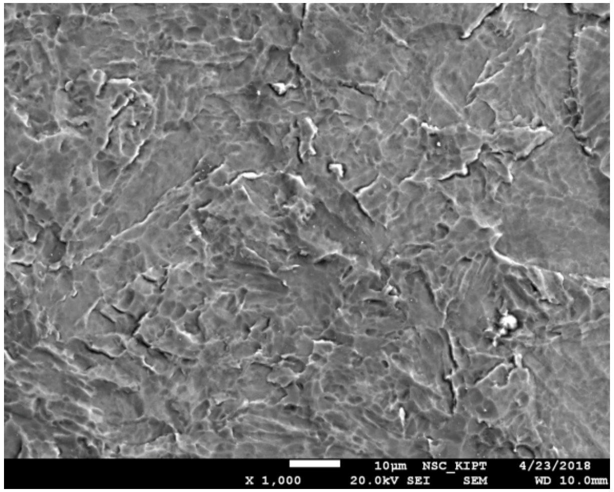
- One cycle consist of two stages

  - First stage: deposition of thin tungsten coating of 1-2 μm
  - Second stage the coated samples will processed with pulsed plasma.

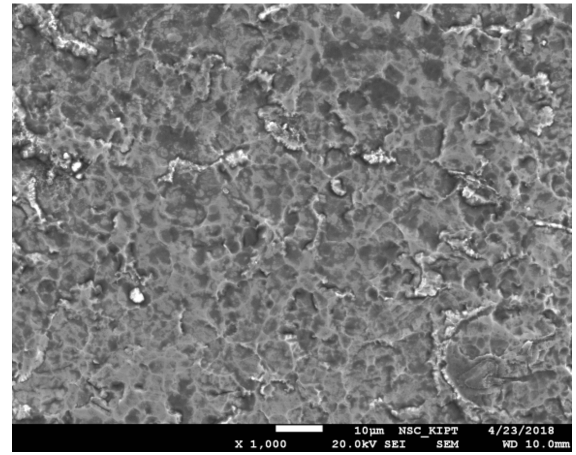
- Some decrease of coating thickness together with increasing of number of cycles of plasma treatment creates condition for penetration of alloying element in depth of substrate.

- **The samples of Eurufer and Rusfer were coated with W and irradiated by QSPA plasma stream.**
- **The sputtering tests of modified surfaces were also performed**

SEM images of Eurofer samples

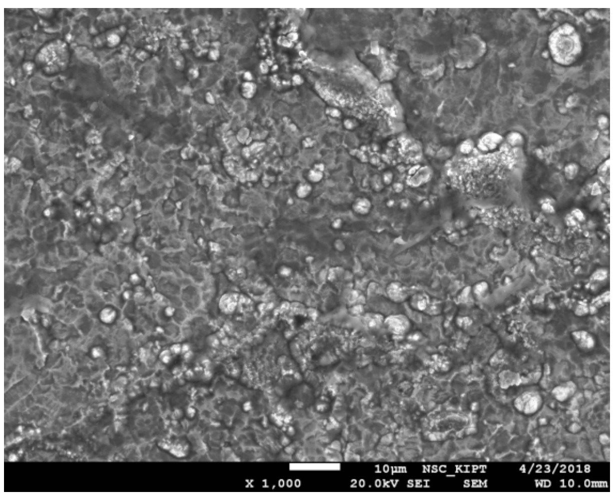


Initial



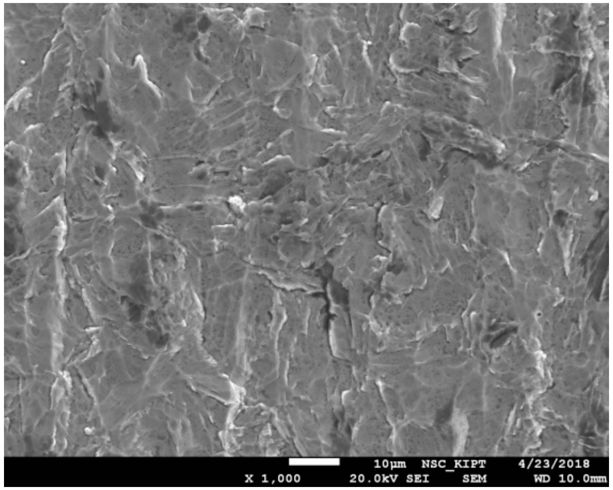
W coating modified by QSPA plasma

- Melted, re-solidified layer developed on exposed surfaces.
- Modified layer includes of material coating and substrate.
- Macro and micro cracks appear on exposed surfaces

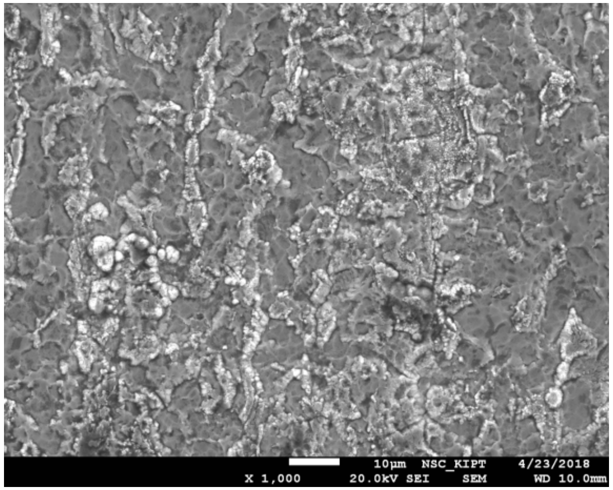


W coating modified by QSPA plasma and irradiated by Ar^+ beam.

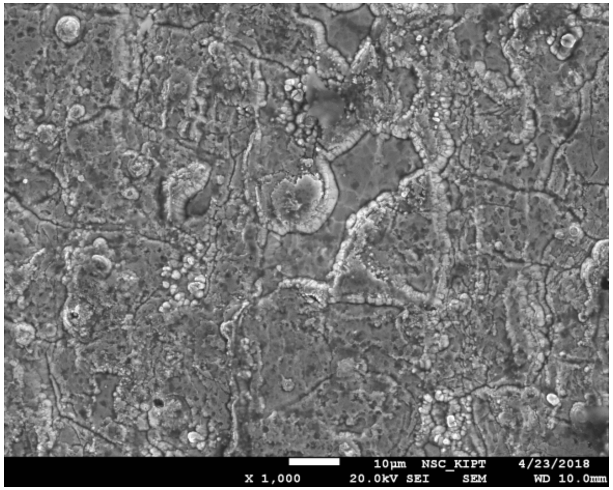
SEM images of Rusfer samples



Initial



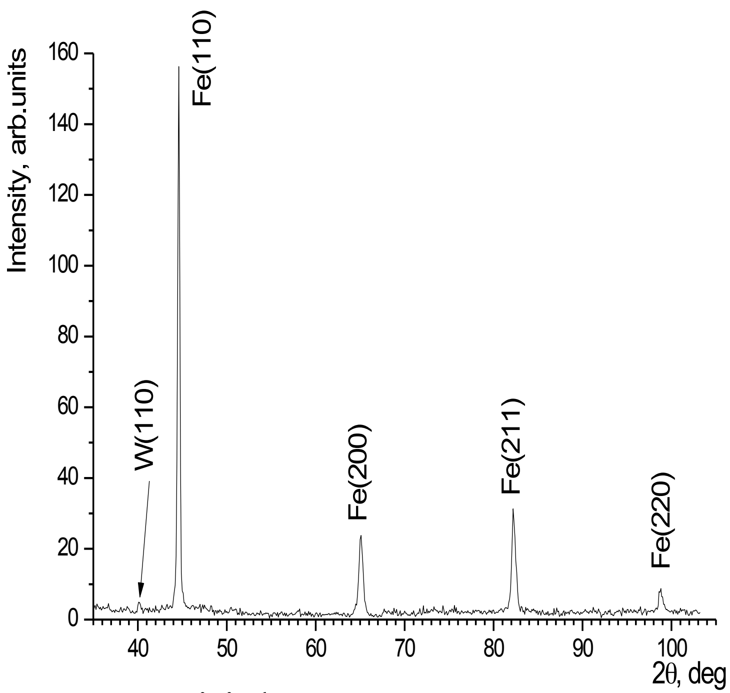
W coating modified by QSPA plasma



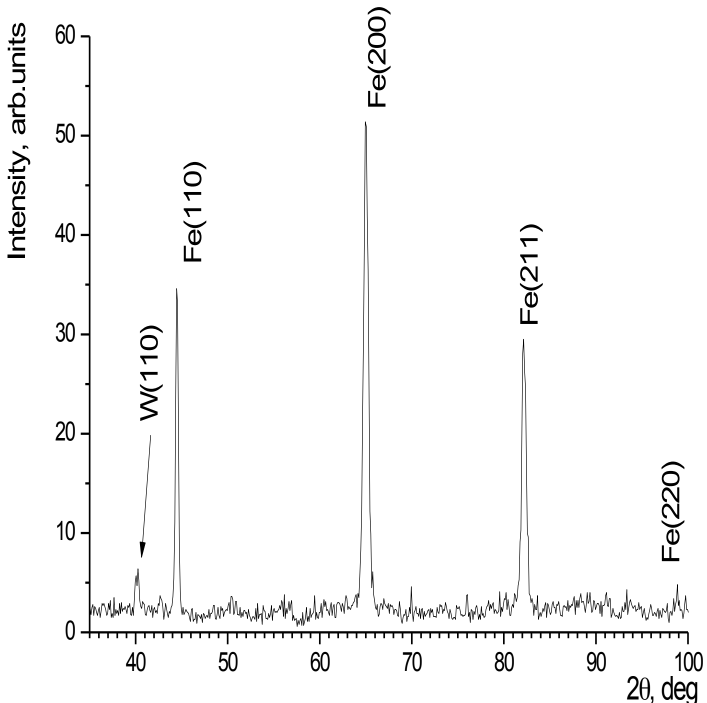
- Melted, re-solidified layer developed on exposed surfaces.
- Modified layer includes of material coating and substrate.
- Macro and micro cracks appear on exposed surfaces

W coating modified by QSPA plasma and irradiated by Ar^+ beam.

Results of XRD spectral analysis



Initial



W coating modified by QSPA plasma and irradiated by Ar^+ beam.

- **W phases are recognized together with lines of Fe phase on treatment surfaces.**
- **Intensity of tungsten lines is more at times in compare with intensity of substrate lines.**

Results of XRD fluorescence studies

Element content (wt %) of Eurofer

	Cr	W	Fe
Initial	9.7	0.8	Base
Single coated W and exposed by QSPA	1.3	85.8	Base
Twice covered W and exposed by QSPA	7.11	27.46	Base
Sputtered by Ar- beam	9.9	1.3	Base

The concentration of tungsten increased in 1.5 times

Results of XRD fluorescence studies

Element content (wt %) of Rusfer

	Cr	W	Fe
Initial	12.02	1.3	Base
Twice covered W and exposed by QSPA	8.5	37.37	Base
Sputtered by Ar- beam	9.4	2.3	Base

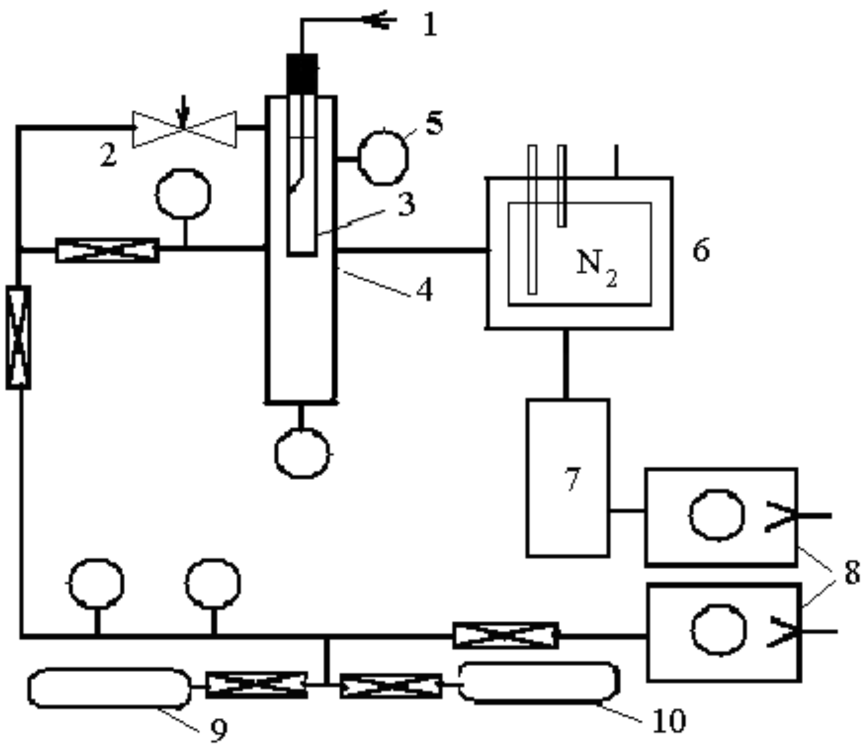
The concentration of tungsten increased in more 1.5 times

Mass losses measurements

	Δm, mg
Initial Eurofer	3.43
Modified Eurofer	2.70
Initial Rusfer	2.315
Modified Rusfer	2.20

The sputtering tests of modified surfaces were performed
Mass losses of modified Eurofer decreased by up to 20 %.

The experimental device GAS

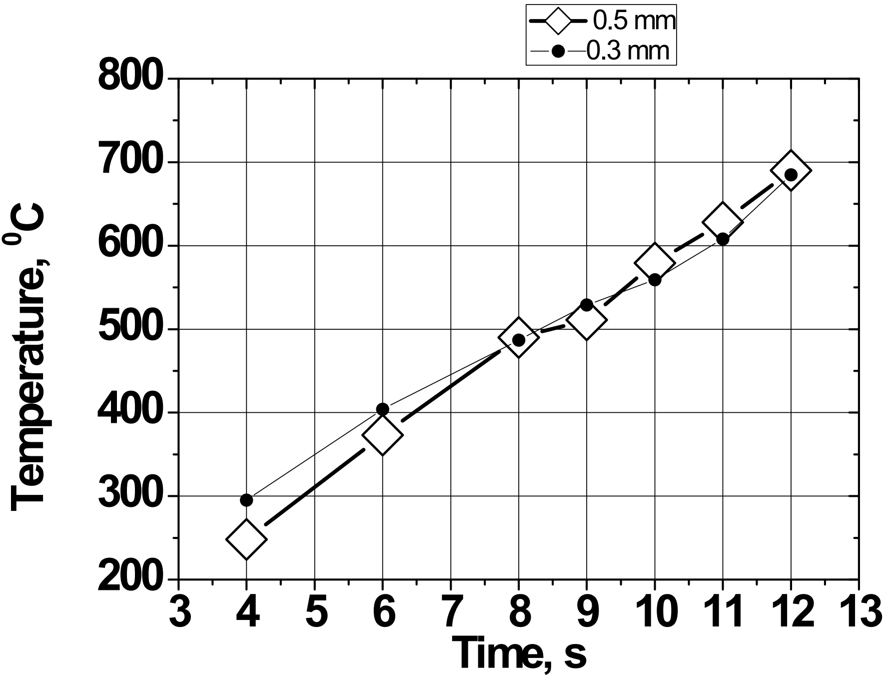


1 – W-Re thermocouple; 2 – flow; 3 – probe; 4 – vacuum chamber; 5 – mass spectrometer; 6 – nitrogen condensation pump; 7 – diffusion pump; 8 – for-vacuum pump; 9 – hydrogen balloon; 10 – helium balloon

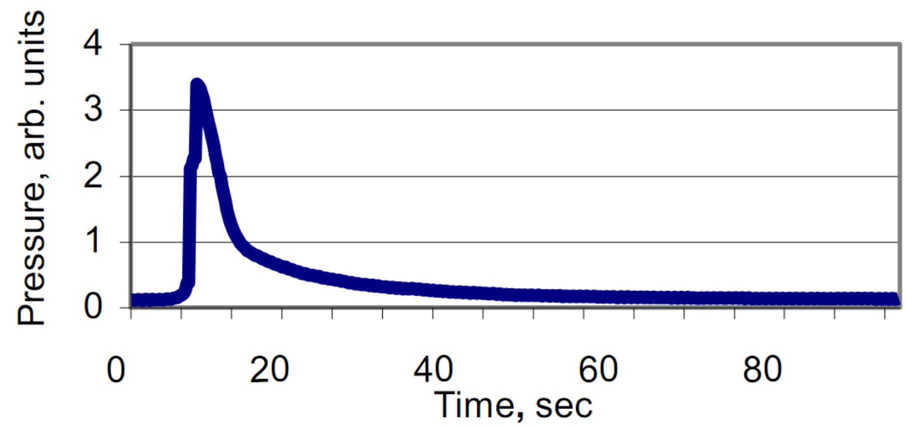
GAS used for the study of gas release, hydrogen sorption, hydrogen permeability

Direct current pulsed heating of the samples was applied

Pulsed heating of the samples



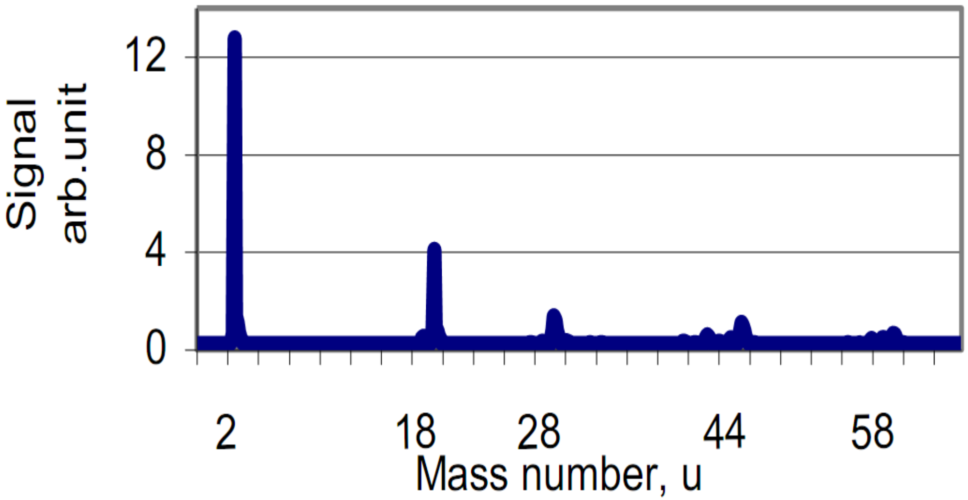
Temperature dependence on the heating time for SS samples with thickness of 0.3 mm and 0.5 mm



Apparatus signal during pulsed heating of the SS sample to the temperature of 500°C

Direct current pulsed heating of the samples was used during experiments in the GAS

Estimation of outgassing rate



Mass spectrum at a sample temperature of 500°C

Outgassing rate was calculated according to equation:

$q = (P - P_o)S/F$, [Torr ·L/s·cm²]

where

S[L/s] *pumping speed,*

F [cm²] the area of the probe surface heated to 500ºC.

P_0 [Torr]- *the initial pressure*

P [Torr]- *the maximum pressure after sample heating*

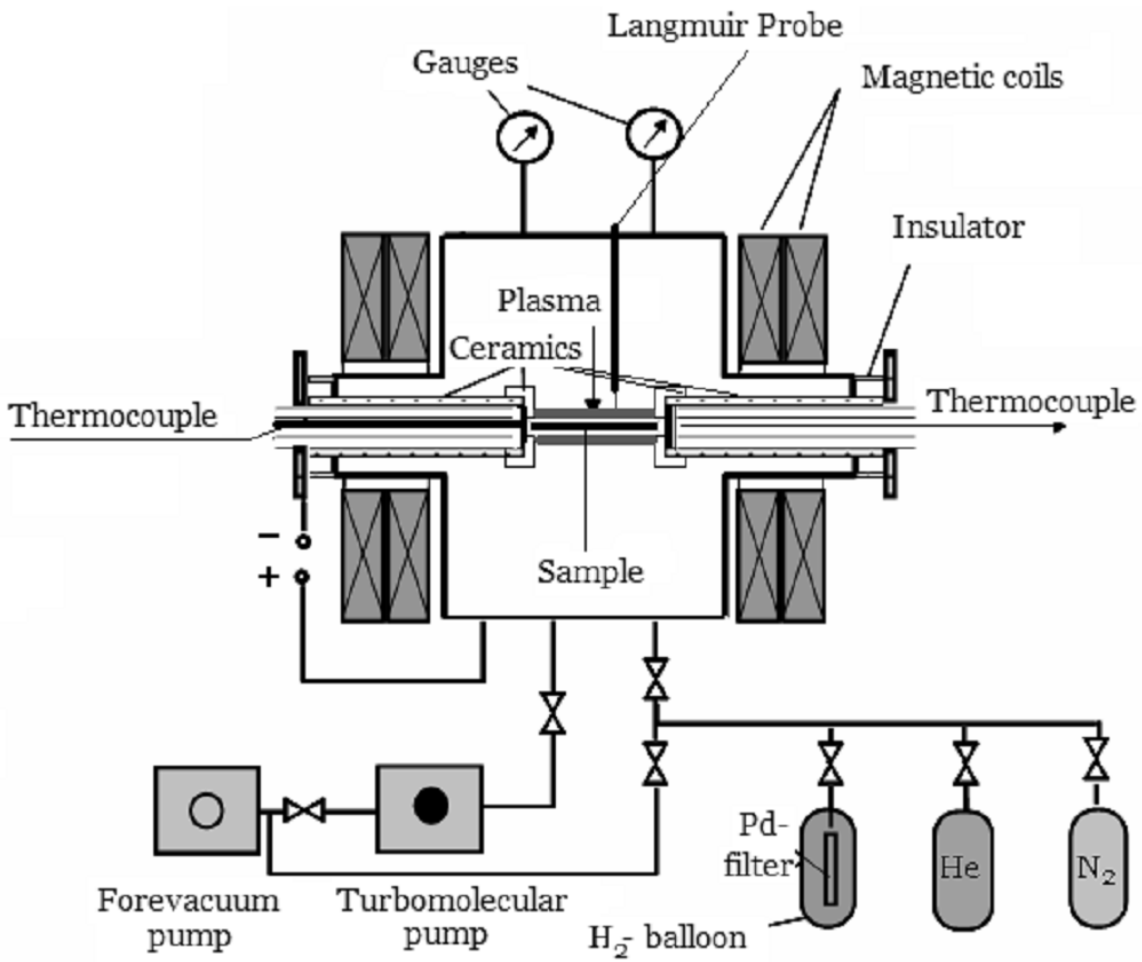
Mainly hydrogen desorbs from SS sample at temperature of *500°C*.

Samples for outgassing studies

Stainless steel (SS) 12Kh18N10T samples of 10x190x(0.3…0.5) mm were used for studies of outgassing.
Samples were studied after

1. Initial heated to *500°C*
   (initial specific rate of hydrogen outgassing (hydrogen elease)
2. saturated in a molecular hydrogen atmosphere at a pressure of ~10^{-2} Torr during 24 hours in GAS facility
3. irradiated by hydrogen plasma of stationary magnetron discharge in DSM-1
4. irradiated by hydrogen QSPA plasma with different heat loads.

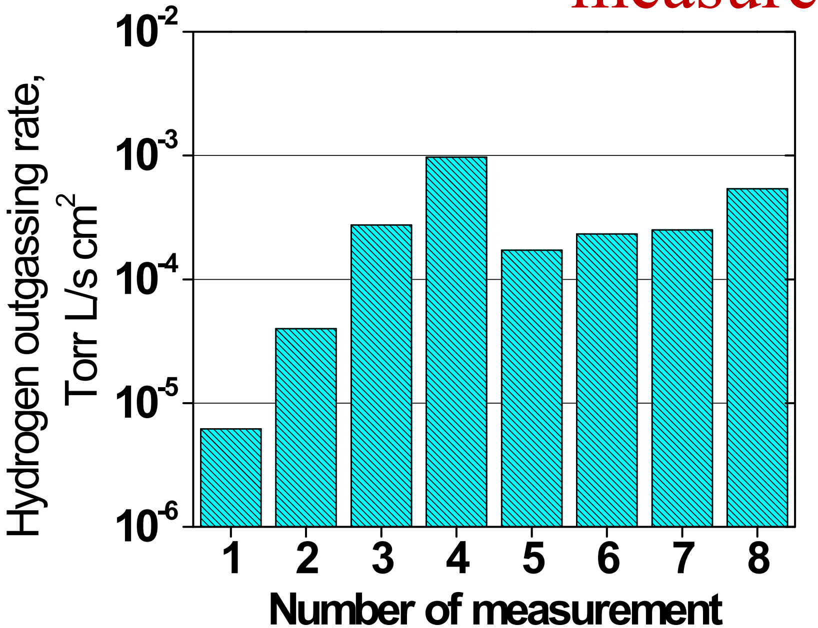
DSM-1 diagnostic stand of materials



DSM-1 is device with magnetron type discharges.
Two symmetrical cathodes were connected with one cylindrical cathode-sample.
The discharges were ignited in magnetic field of 0.05 T under work gas (hydrogen) pressure about 0.2 Pa.

DSM plasma
ion energy 0.7 keV
fluence $6\cdot10^{24}$ ions/m^2; up to 8 hours
QSPA plasma fluence $5\cdot10^{24}$ ion/m^2, pulse duration of 0.25 ms, ion energy 0.4 keV

First results of hydrogen **retention and** release rate measurements



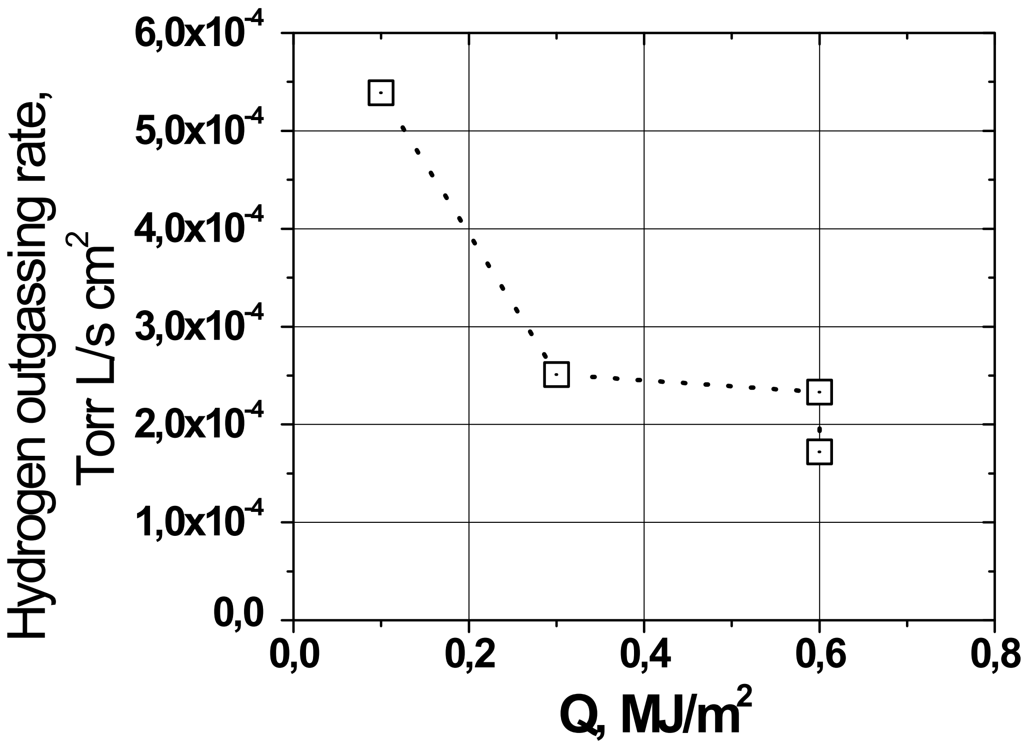
Rates of hydrogen release from SS samples at the temperature of 500°C

1 initial specific rate of hydrogen outgassing (hydrogen release)
2- saturated in a molecular hydrogen atmosphere at a pressure of ~10^{-2} Torr during 24 hours
3, 4- irradiated by hydrogen plasma of stationary magnetron discharge in DSM-1
5-8 irradiated by hydrogen QSPA plasma with different heat loads.

Hydrogen outgassing is close value for SS samples saturated in steady state discharges of DSM-1 and irradiated by plasma streams in QSPA Kh-50

Particle energy and fluence caused outgassing value.

Hydrogen release rate v.s. pulsed heat load on SS sample surface



Heat load of 0.6 MJ/m^2 caused pronounced melting of exposed surfaces

- Melted, re-solidified layer developed on exposed surfaces.

Heat loads below 0.3 MJ/m^2 not leads to surface melting.

Increase of pulsed heat load to surface (above SS melting threshold) leads to essential decrease of hydrogen saturation.

Conclusions

- **Experimental studies of surface modification of Eurofer and Rusfer samples covered by tungsten coatings have been performed with powerful plasma streams. The heat load on the surface was near the tungsten melting threshold. Tungsten coating was preliminary deposited by PVD method in facility of Bulat family. The sputtering tests of modified surfaces were also performed.**

- **The possibility of alloying of Eurufer and Rusfer surfaces with tungsten was demonstrated. The concentration of tungsten have been achieved several wt% in surface layer. The surface morphology is developed mostly by melting and re-solidification of a surface layer.**

- **The sputtering yield (mass losses) of samples modified by plasma streams decreased due to increasing of tungsten concentration in modified surface layer.**

- **Hydrogen outgassing from SS samples saturated in steady state discharges in DSM and in the plasma accelerator QSPA Kh-50 is close in value. Modified layer formation leads to essential decrease of hydrogen saturation.**

Summary

-The operational regimes of the plasma accelerators (QSPA Kh-50 and MPC) will be adjusted to achieve adequate variation of energy and particles loads to the exposed steel materials.

- Experiments on surface modification of different steels have been performed using multiples irradiation of material surfaces by quasi-steady and pulsed plasma streams with varied plasma loads.

-First experiments on hydrogen retention and release rate measurements of stainless steel modified by plasma screams have been performed..

Thank you for your attention