Modification and alloying of steel surfaces exposed by powerful plasma streams.

a part of the IAEA's Coordinated Research Project "F43022",
"Plasma–Wall Interaction with Reduced Activation Steel Surfaces in Fusion Devices"

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Outline

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- Experimental facility and samples
    - QSPA Kh-50
    - MPC
    - DSM-2
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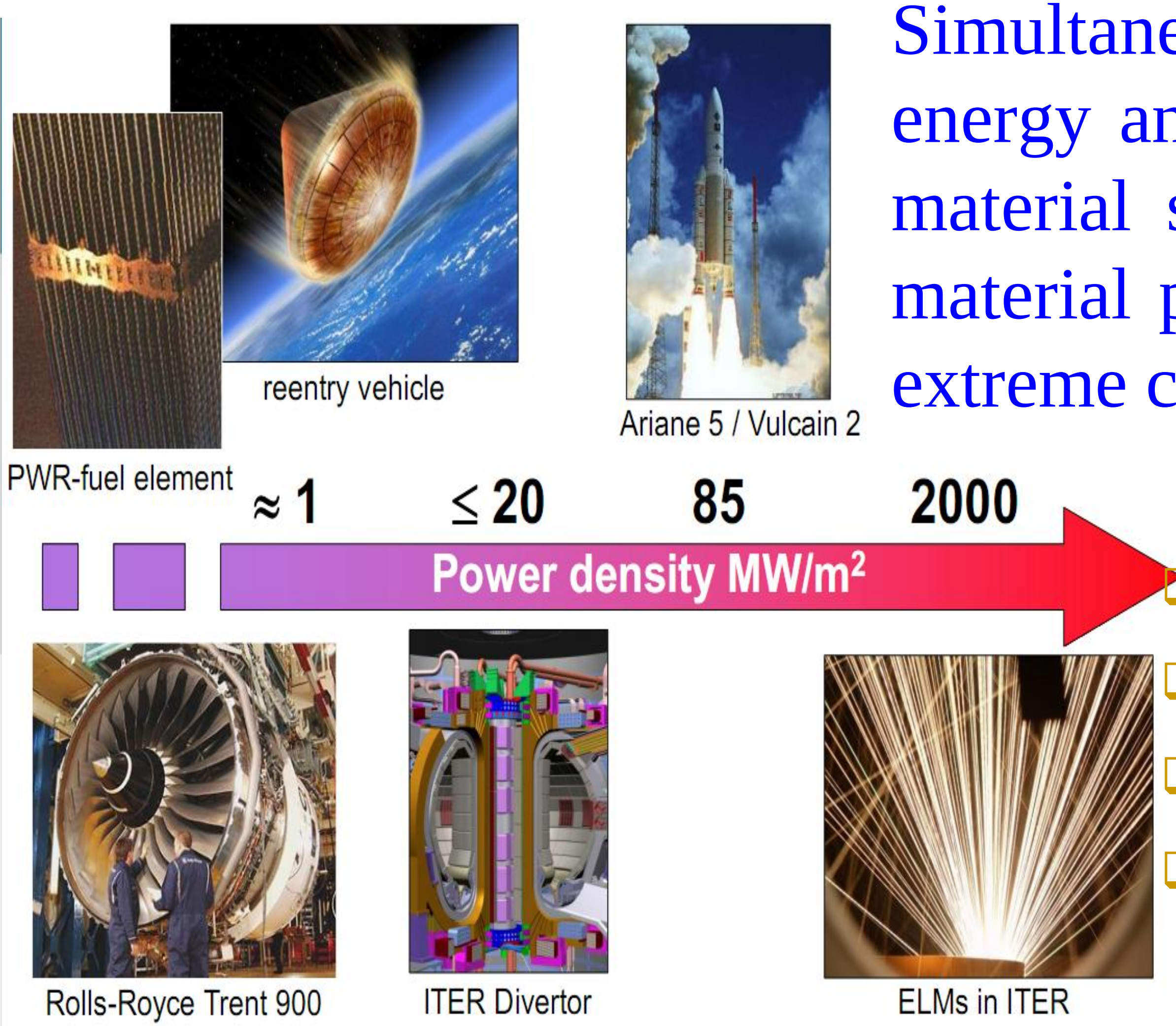
- Conclusions
- Collaboration established during a progress of CRP project
- Future plans as a part of project activity

Introduction

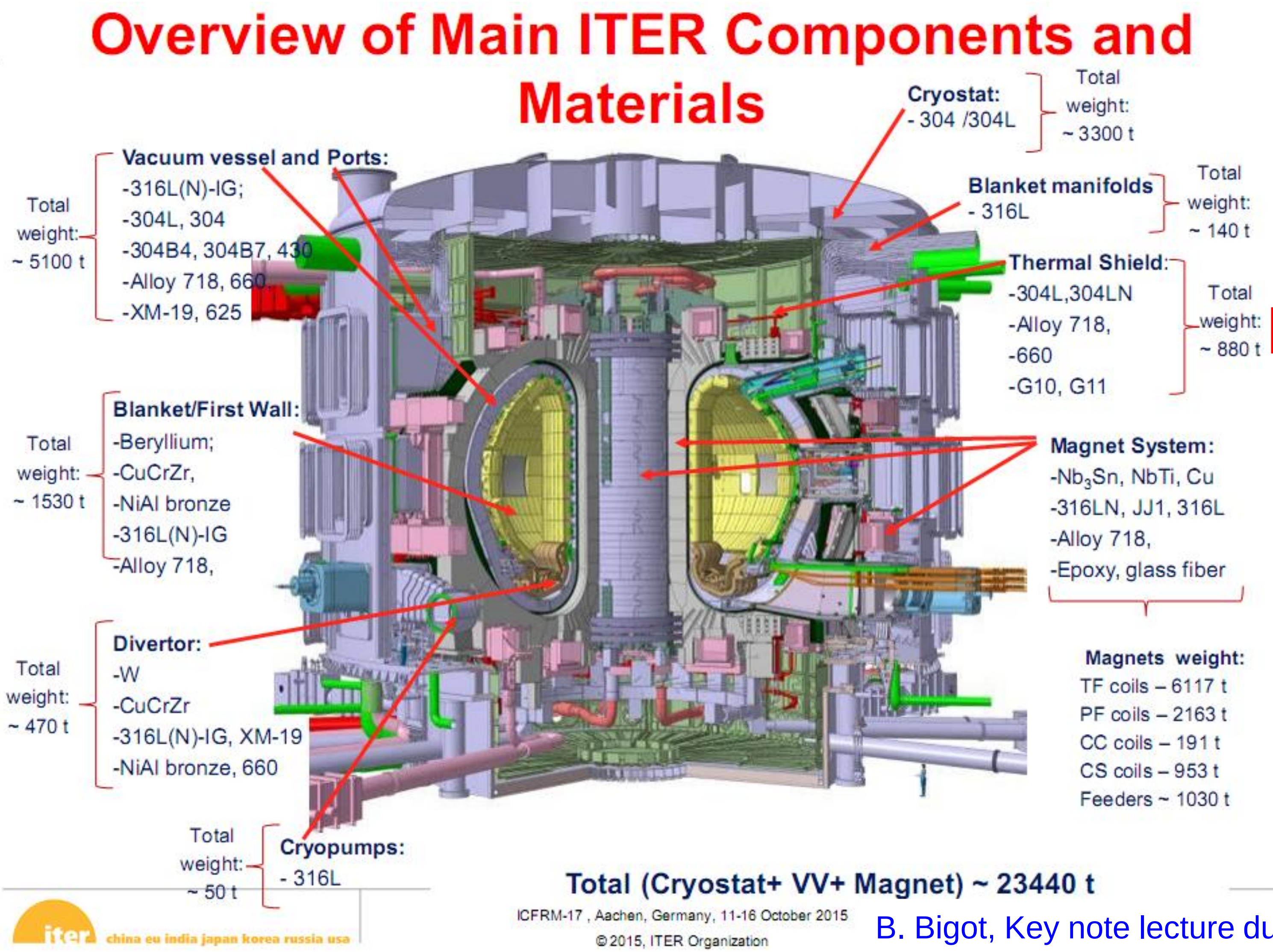
Simultaneous impacts of high energy and particle loads to the material surface are typical for material performance in various extreme conditions:



- turbines
- space apparatus,
- nuclear engineering
- fusion

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

Introduction



Materials issues are key factors for ITER success

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**

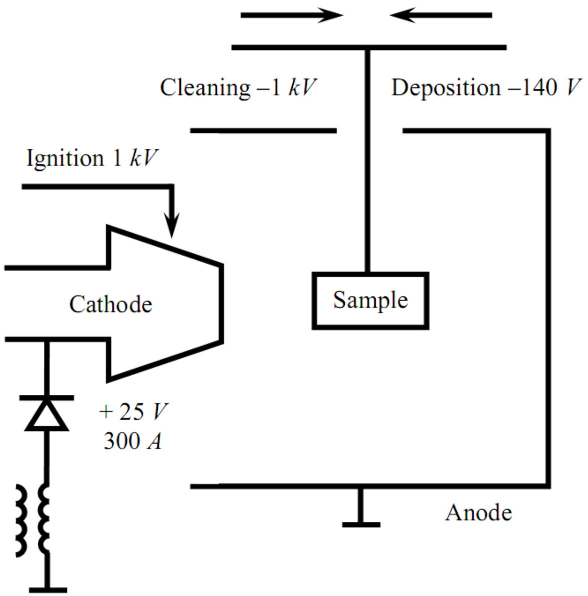
The detailed experimental studies of threshold values for the damaging processes under fusion reactor relevant loading scenarios are required for evaluation of the materials performance under high heat fluxes.

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

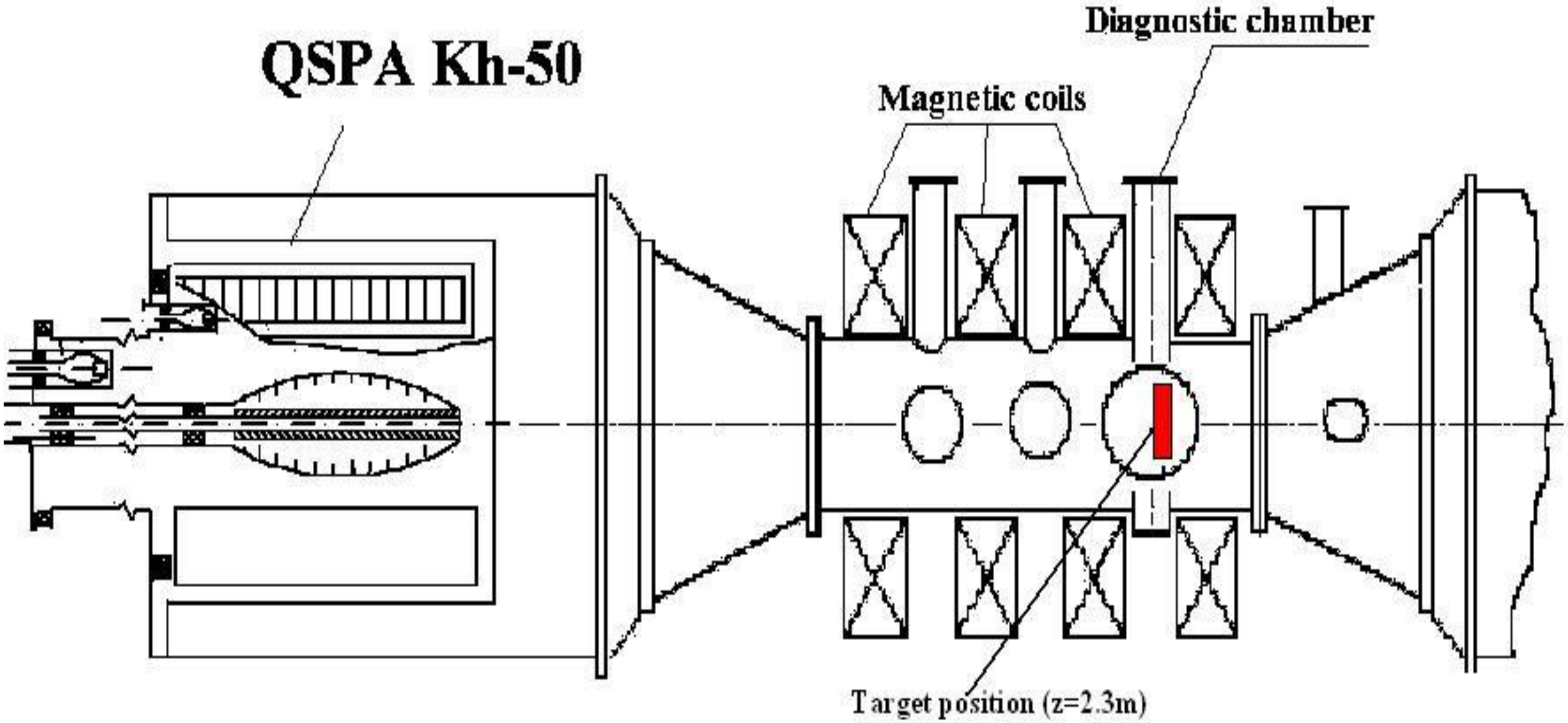
✓Material response to multiple exposures are studied with ion and electron beams, liner facilities, pulsed plasma guns and QSPA,

✓**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



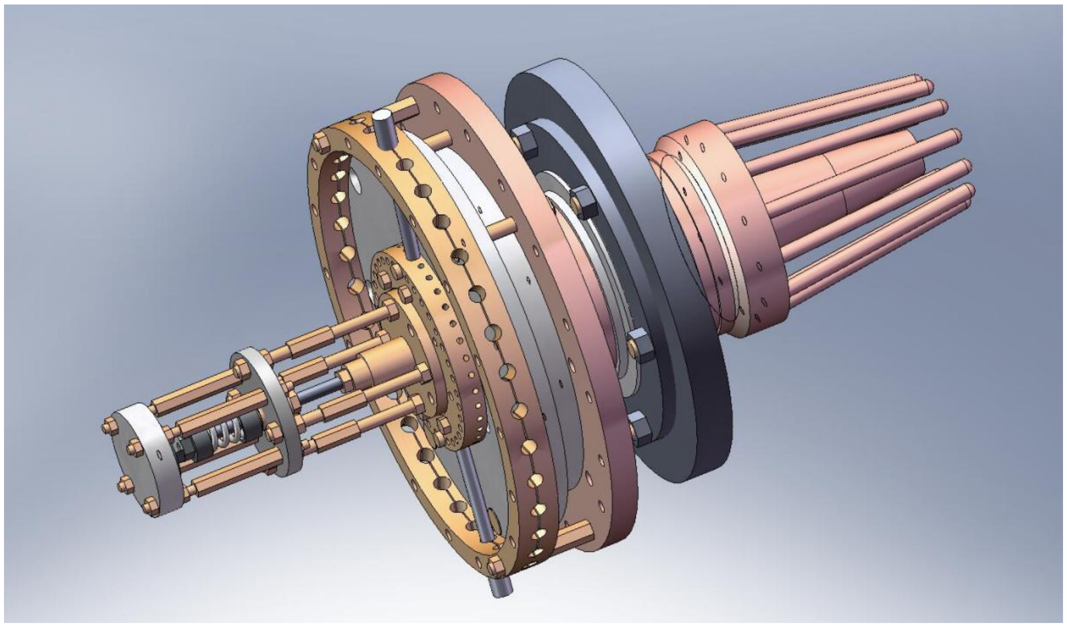
Energy density $\rho_w = (0.5…30)\ MJ/m^2$,
Plasma pulse duration $\tau \approx 0.25$ ms;
P_{max} = (3-18) bar, n = (0.2-5) $10^{16}\ cm^{-3}$; B_0 =0.54 T ($\beta \approx 0.3…0.4$);
Diameter of plasma stream- 15 cm

Examples of Plasma Parameters in Different Regimes QSPA Kh-50

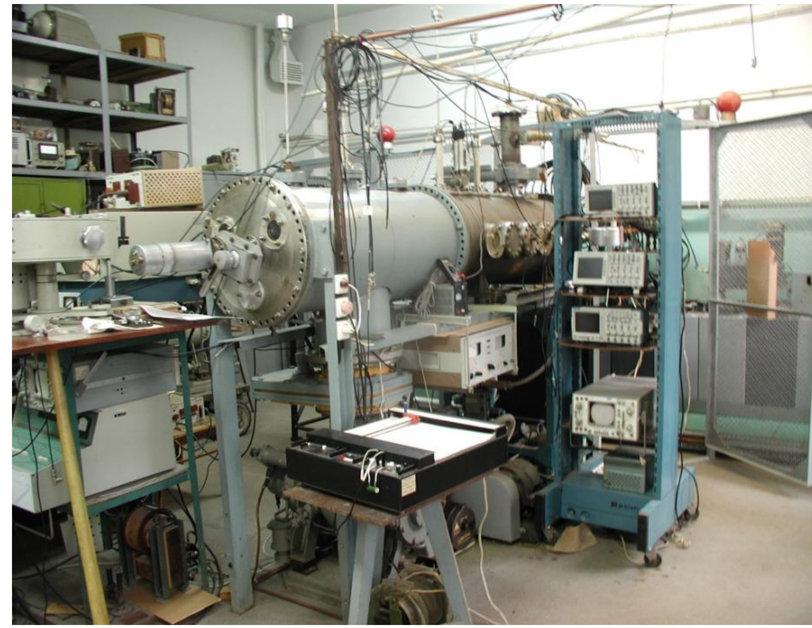
Parameters	ELM 0	ELM 1	ELM 2	ELM 3	Disruption
Plasma stream energy density [MJ/m^2]	0.4	0.9-1.0	1.2-1.5	2.4-2.5	24-30
Target Heat Load [MJ/m^2]	0.22	0.45	0.7-0.75	1-1.1	0.65-0.7 (strong vapor shielding)
Plasma load duration [ms]	0.25	0.25	0.25	0.25	0.2-0.25
Maximal dynamical pressure of plasma stream [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

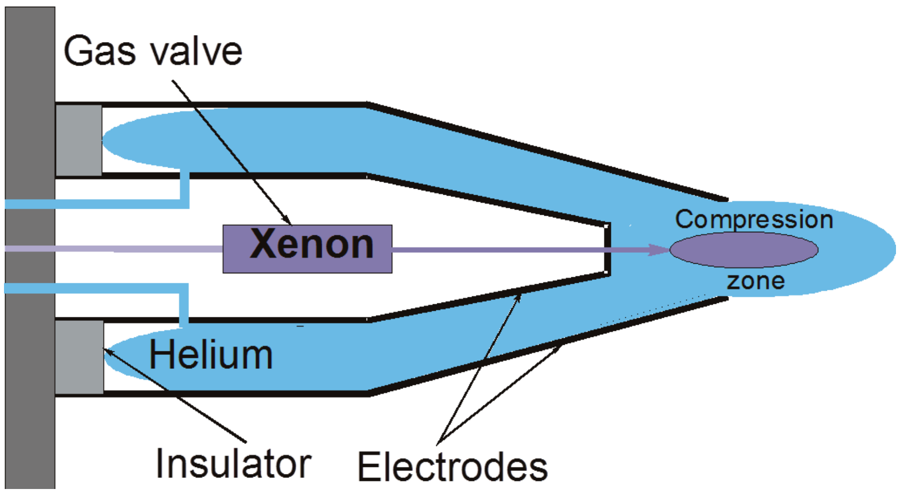


General view of MPC with diagnostics

∅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

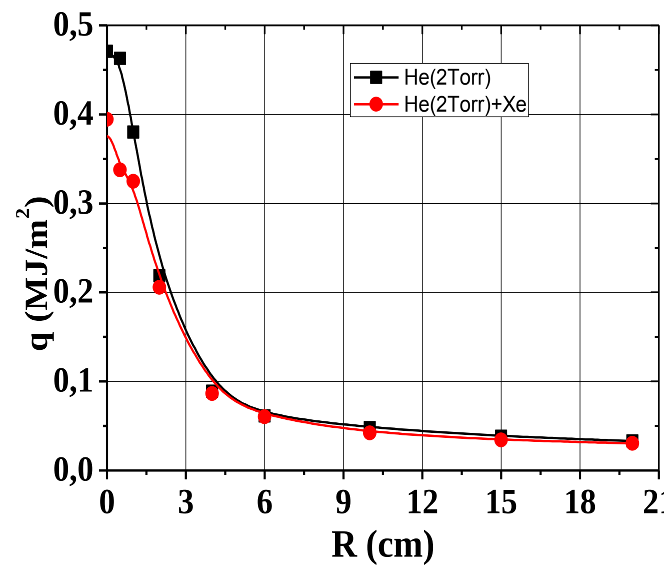
Operation modes of MPC



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

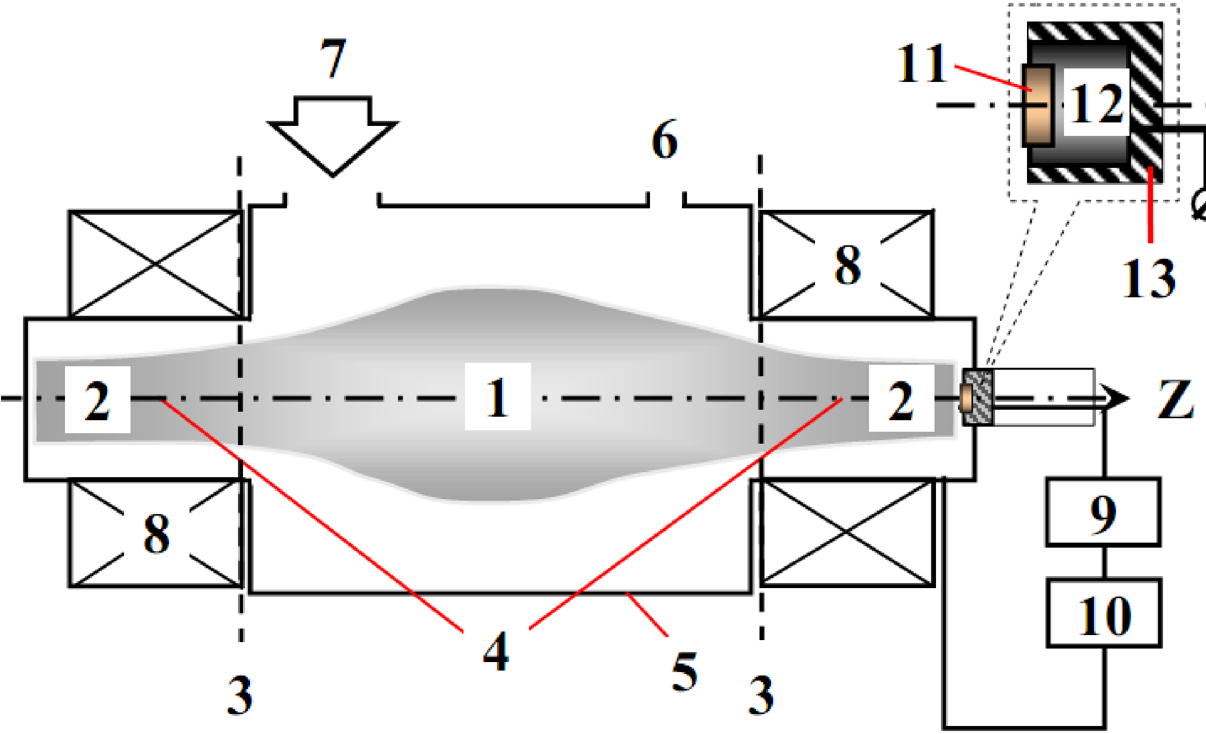
Maximal heat load to tungsten surface exposed to helium plasma achieved 0.39 MJ/m^2.
The heat load to tungsten surface exposed under additional injection of xenon is decreased to 0.33 MJ/m^2.

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.

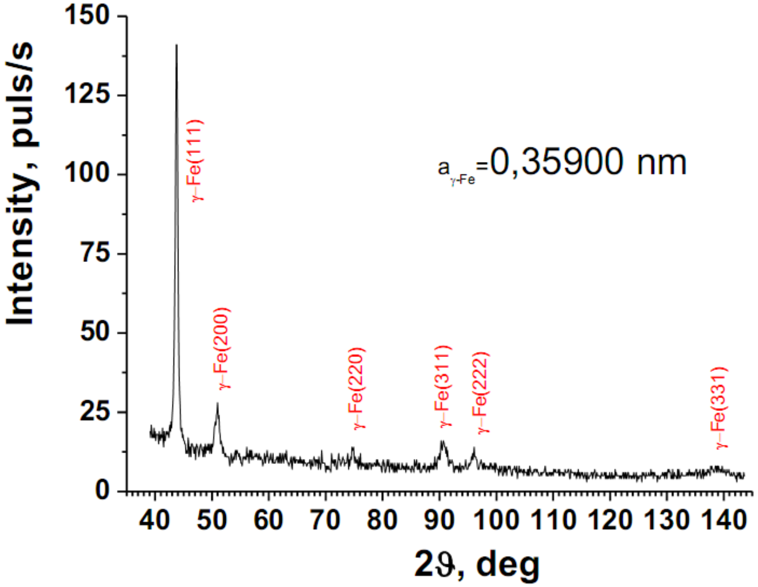
The asymmetry (δB) is attributed by the presence of complexes of point defects. The sign of δB is caused by the type of defects: vacancies ($\delta B>0$) or interstitial atoms ($\delta B<0$).

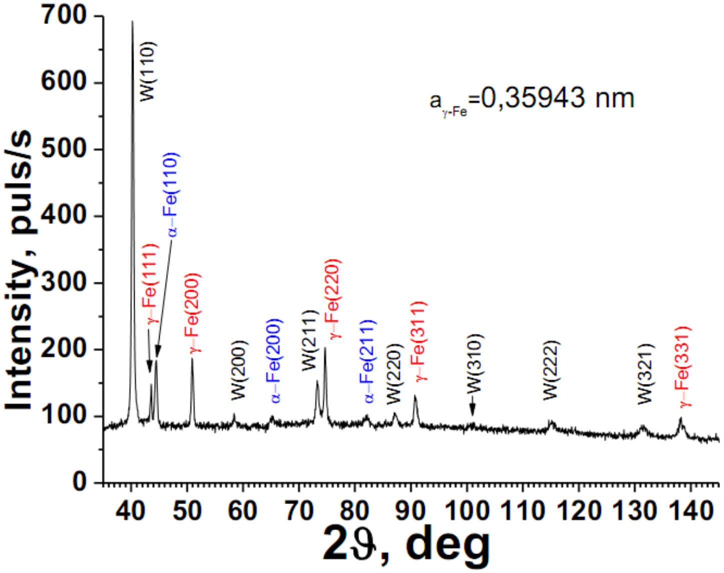
Coatings modification under heat load above melting threshold

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

Initial

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

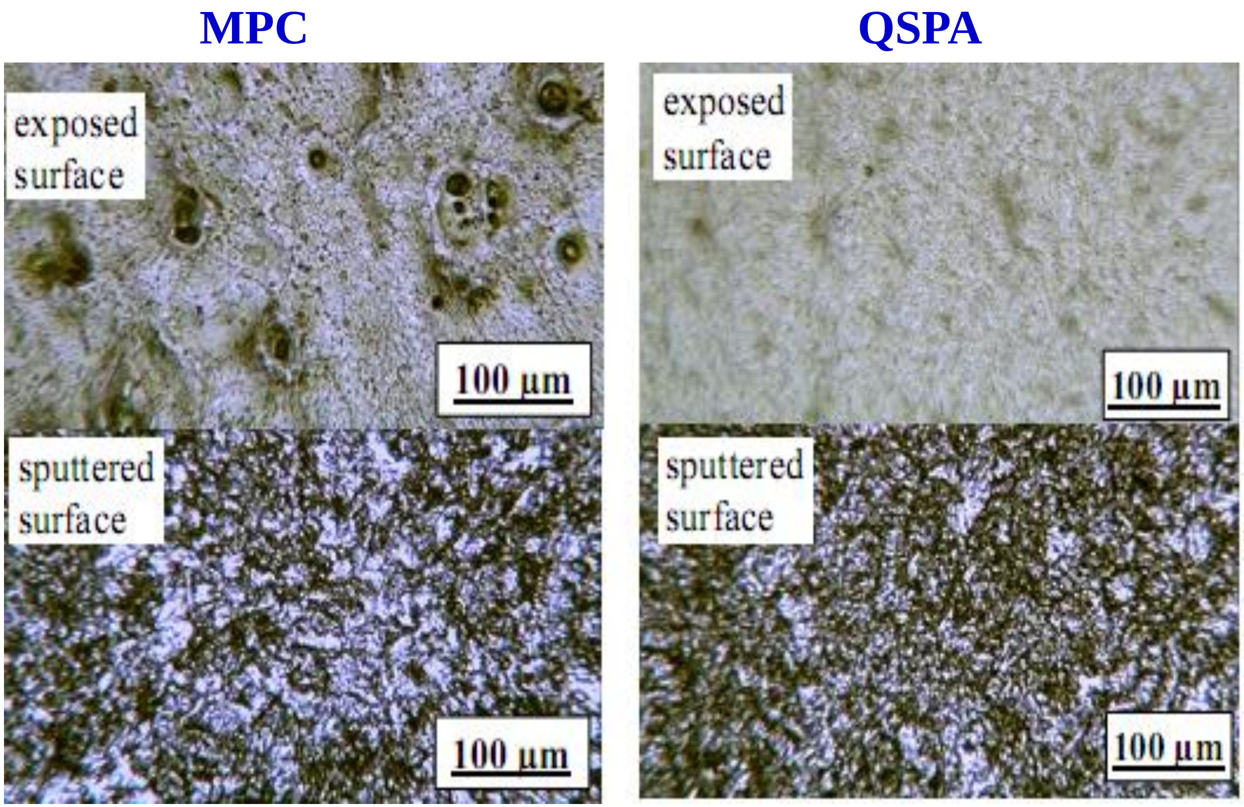




- α-Fe phase is recognized together with lines of γ-Fe phase and W on exposed surfaces.
- Lattice spacing of γ-Fe in stress free state increased from 0.359 nm up to $a_{\gamma\text{-Fe}}$= 0.35943 nm.
- The concentration of tungsten achieved 2 a.p. in modified layer.
- Tungsten penetrated up to 0.38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

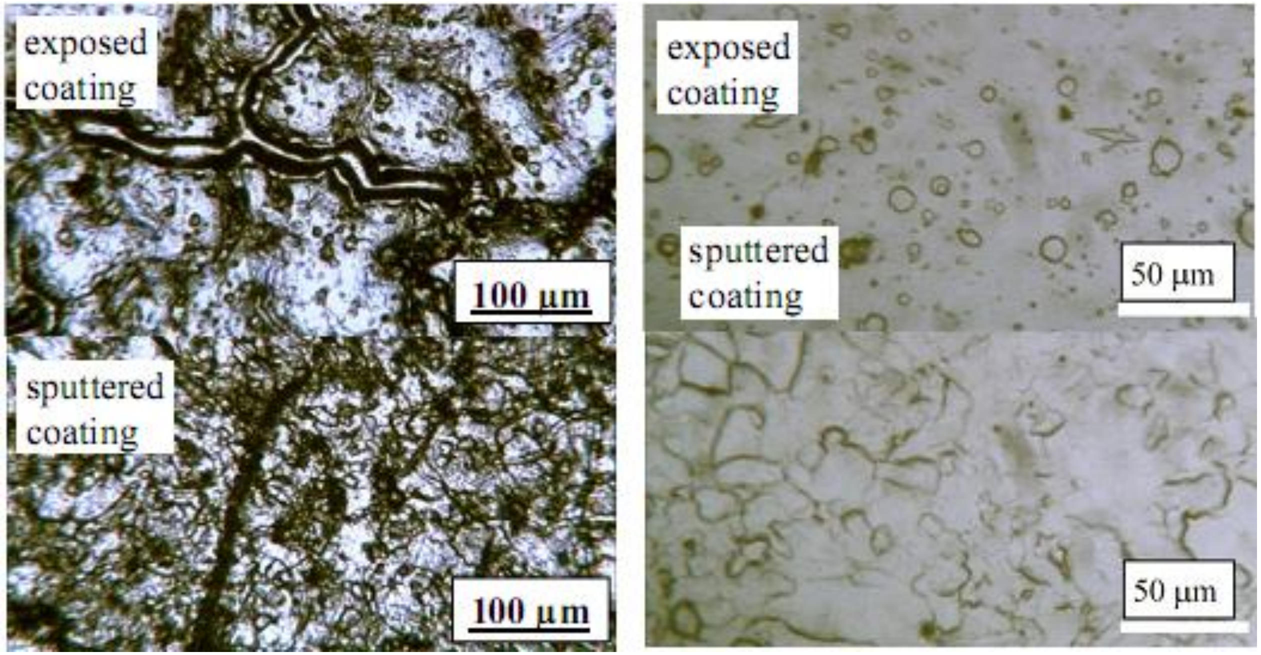
Modified substrate



- Melted, re-solidified layer developed on exposed surfaces.
- Macro and micro cracks appear on exposed surfaces

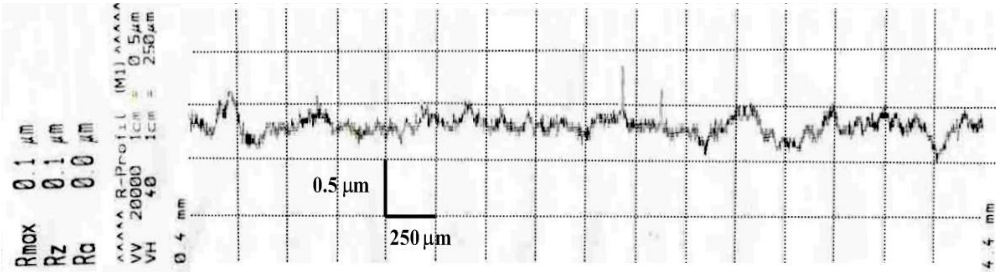
Modified tungsten coating

MPC **QSPA**

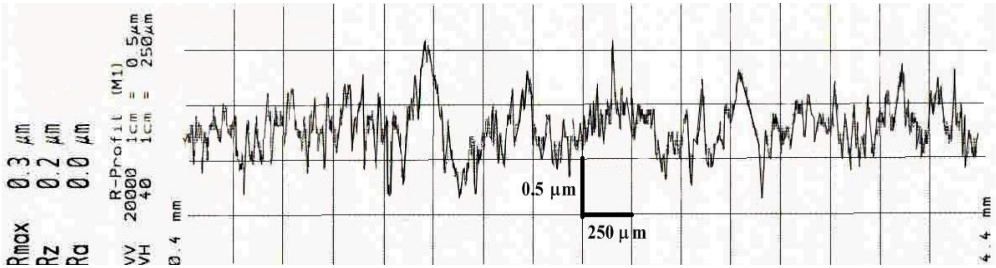


- Melted, re-solidified layer developed on exposed surfaces.
- Modified layer includes of material coating and substrate.
- Macro and micro cracks appear on exposed surfaces

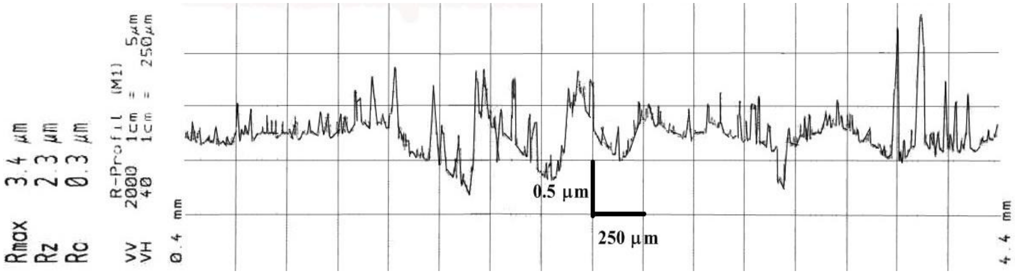
Profiles of Eurofer before and after plasma exposure



Initial



Irradiated by 5 QSPA plasma pulses;

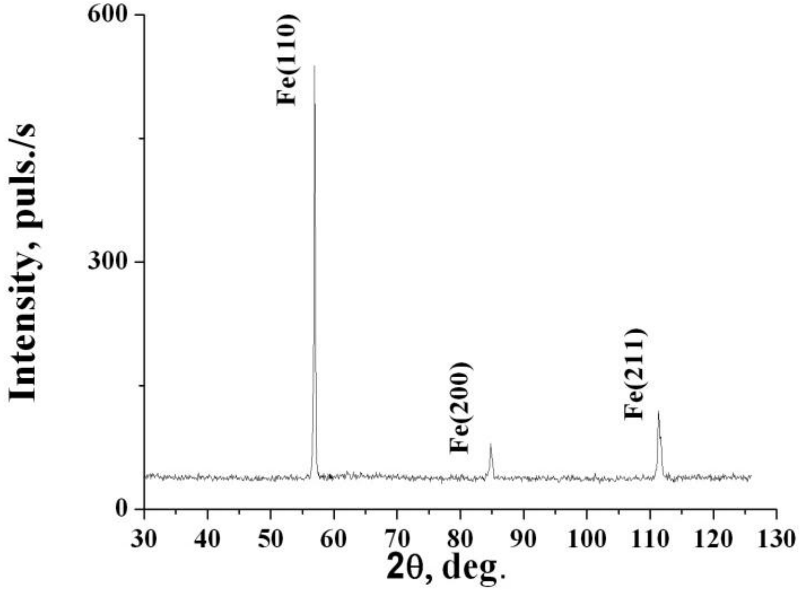


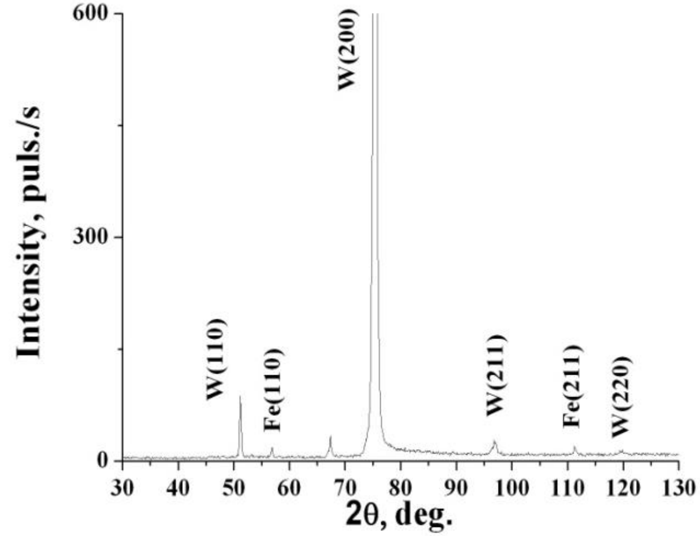
Surface covered by W and exposed to QSPA plasma

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





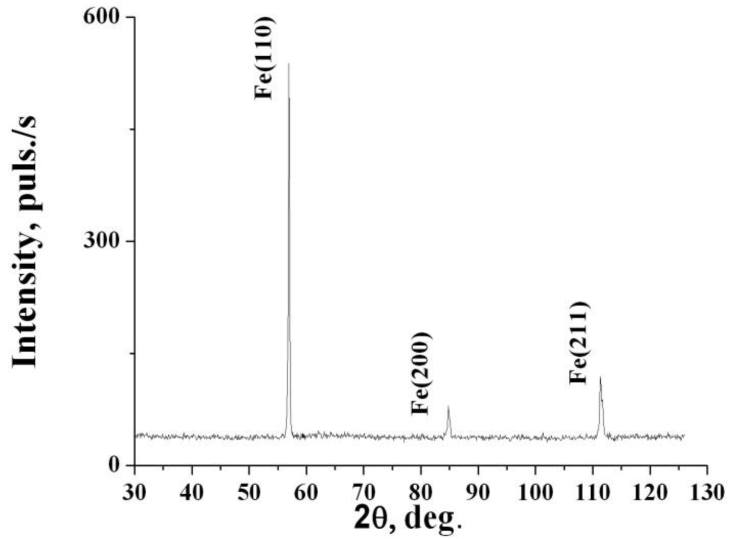
Initial

Surface coated by tungsten and exposed to QSPA plasma

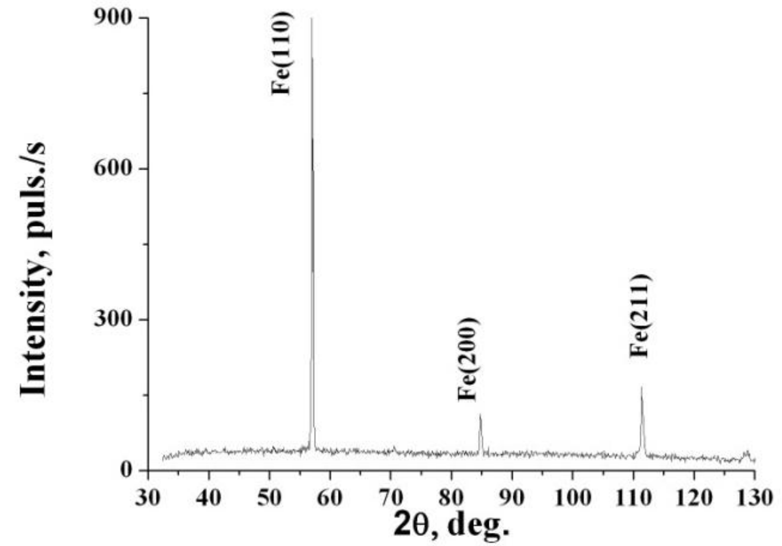
Diffraction patterns (Cu-Kα irradiation)

- **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.**

Results of XRD fluorescence studies

Element content (wt %) of Eurofer

	Cr	Mn	Fe	W
Initial	9.7	0.4	89.9	-----
Coated W and exposed by QSPA	1.3	----	12.95	85.8
Coated W, exposed by QSPA and sputtered by Ar beam	8.9	0.36	89.64	1.1
Coated W and exposed by MPC	9.7	0.4	88.8	1.1
Coated W and sputtered by Ar beam	9.6	0.4	88.9	1.1

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

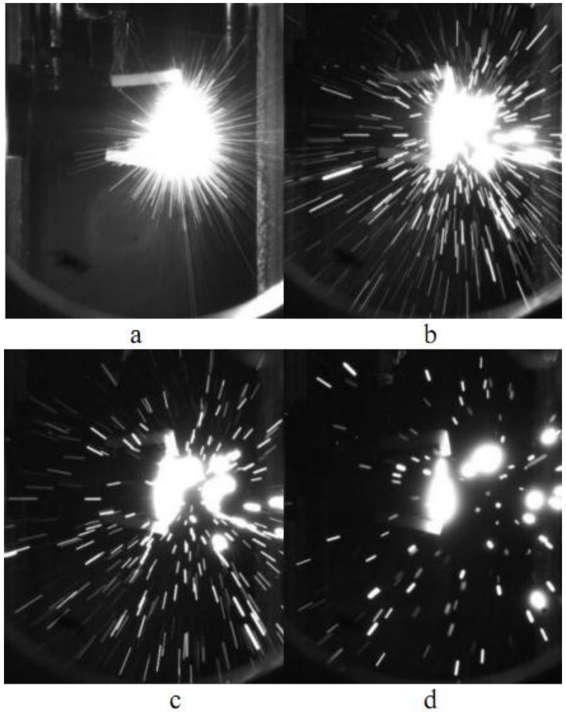
	lattice spacing a_0, A	Content of tungsten
Initial	2.8714	---
Modified by plasma stream and sputtered by Ar beam	2.8676	---
W coatings sputtered by Ar beam	2.8717	1.3
Coated W, exposed by QSPA and sputtered by Ar beam	2.8703	0.85
Coated W, exposed by MPC and sputtered by Ar beam	2.8704	0.9

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)

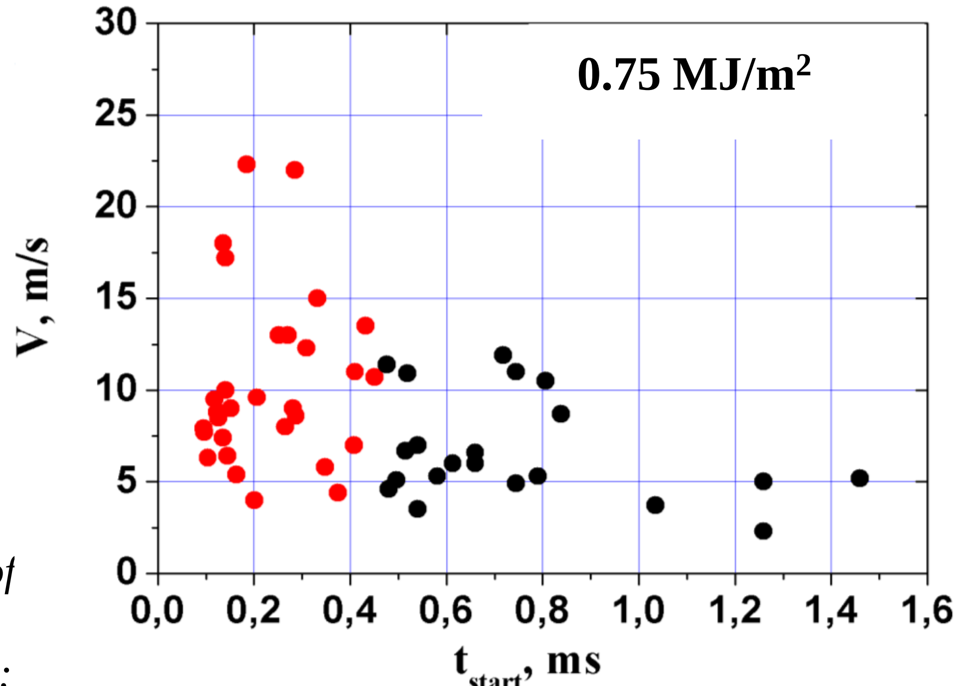
Initial Microhardness is indicated in brackets for each samples
Plasma treatment leads to increases of microhardness
It is indicated on accumulation of elastic energy in the stressed surface layer.
As result of this the delamination of coatings developed

SURFACE MODIFICATION OF TUNGSTEN COATINGS



Frames of the digital camera with the traces of erosion products (exposure time 1.2 ms). The camera's view is 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

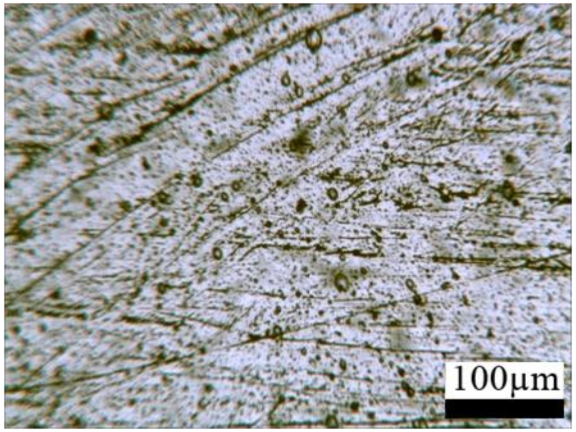
The possible way to improve coatings resistance is application of several cycles of plasma treatment.

- One cycle consist of two stages

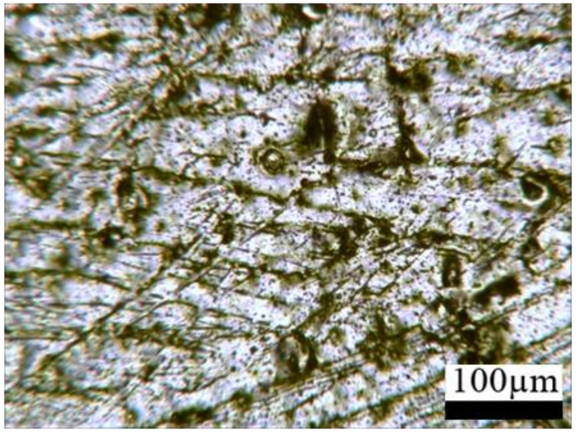
  - First stage: deposition of thin tungsten coating of 1-2 μm
  - Second stage the coated samples will processed with pulsed plasma.

- 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.

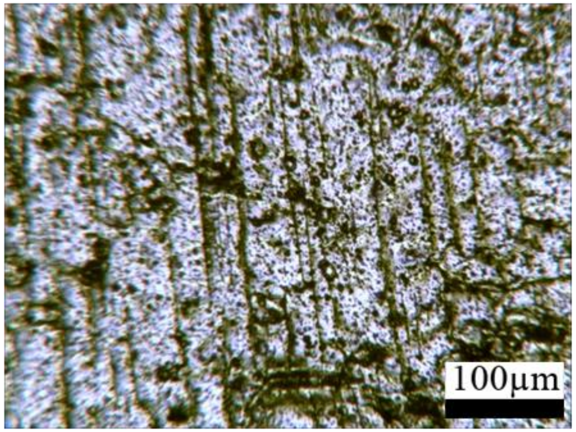
- The samples of Eurufer and Rusfer were coated with W and irradiated by QSPA Plasma stream



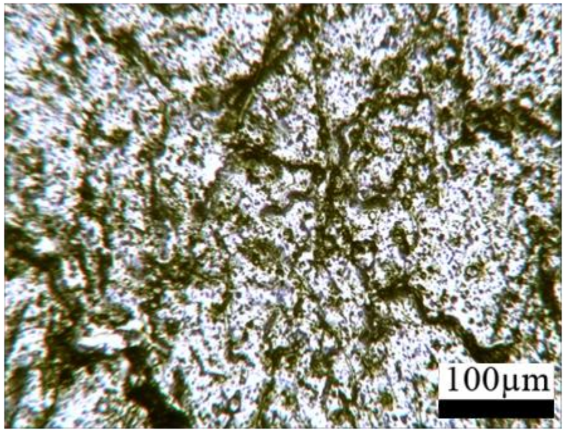
Eurofer coated by W;



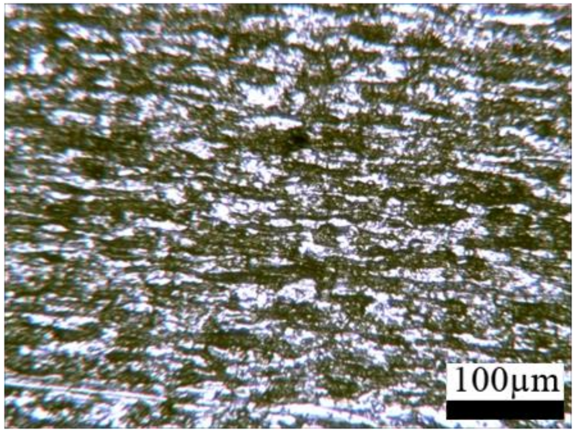
Eurofer coated by W and exposed by 5 QSPA plasma pulses of 0.6 MJ/m^2



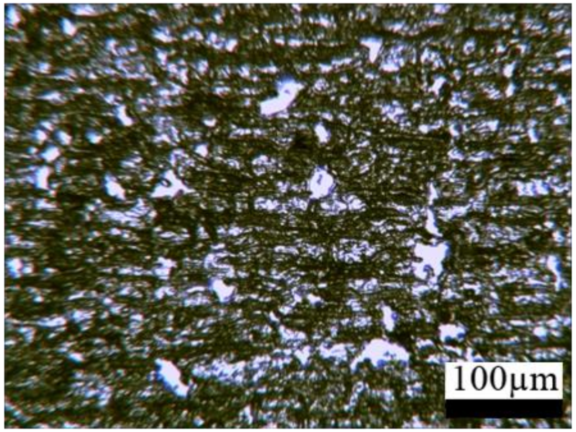
Eurofer coated by W additional W



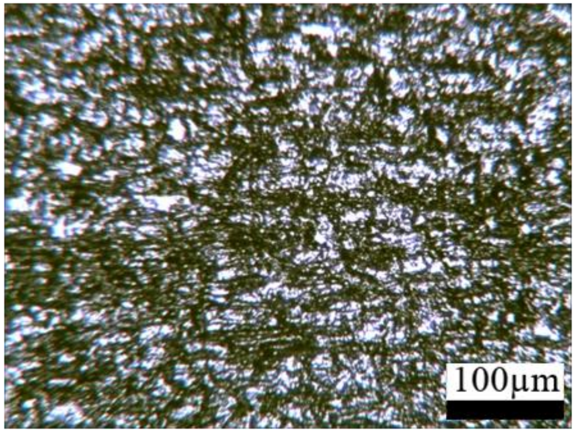
Eurofer coated by W additional W and exposed by 5 QSPA plasma pulses of 0.6 MJ/m^2



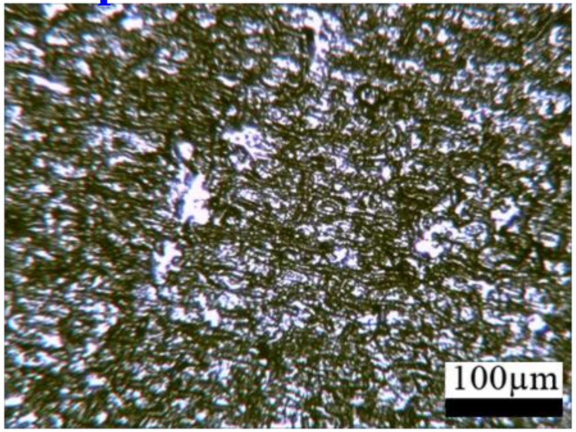
Rusfer coated by W;



Rusfer coated by W and exposed by 5 QSPA plasma pulses of 0.6 MJ/m^2



Rusfer coated by W additional W



Rusfer coated by W additional W and exposed by 5 QSPA plasma pulses of 0.6 MJ/m^2

Results of XRD fluorescence studies

Element content (wt %) of Eurofer

	Cr	Mn	Ni	Fe	W
Initial	9.7	0.4	-----	89.9	-----
Twice covered W and exposed by QSPA	7.11	0.54	0.68	64.21	27.46
Single coated W and exposed by QSPA	1.3	--	---	12.95	85.8

Element content (wt %) of Rusfer

	Cr	Mn	Ni	Fe	W
Initial	12.018	0.627	----	84.841	2.515
Twice covered W and exposed by QSPA	8.5	0.54	1.03	52.56	37.37

Conclusions

- **Experimental studies of surface modification of Eurofer and Rusfer samples covered by tungsten coatings have been performed with a quasi-stationary plasma accelerator QSPA Kh-50 and magneto-plasma compressor MPC . The heat load on the surface was near the melting threshold (i.e. about 0.6 MJ/m^2, pulse duration of τ= 0.25 ms for QSPA Kh-50 and about 0.4 MJ/m^2, pulse duration of τ= 20 µs for MPC). 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. Tungsten phase is recognized together with lines of Fe phase on treatment surfaces. The concentration of tungsten have been achieved several wt% in surface layer up to 4 µm.**

- **The surface morphology is developed mostly by melting and re-solidification of a surface layer. Macro and micro cracks appear also on modified surfaces. The some delamination of coatings are also observed, especially when the surface is irradiated by short plasma streams.**

- **The sputtering yield of samples modified by plasma streams does not so different from the yield of samples in initial state.**

Additional collaboration established during a progress of CRP project

10 sample of tungsten samples have been received together with Eurofer from Dr. Dmitry Terentyev of SCK-CEN Belgium Nuclear Research Centre, Mol, Belgium

5 samples of tungsten have been exposed each by 10 QSPA Kh-50 plasma pulses of heat load of 0.45 MJ/m2 (i.e. below tungsten melting threshold) and with different base temperature.

5 samples have been exposed by different number of QSPA Kh-50 plasma pulses with different base temperature.

Preliminary results of tungsten irradiation with plasma streams

Base Temperature	Sample	Number of pulses with heat load of 0.45 0.45 MJ/m^2	Surface effects
200ºC	W8	10	No cracks
300ºC	W11	10	No cracks
400ºC	W9	10	No cracks
500ºC	W7	10	No cracks
600ºC	W10	10	No cracks
300ºC	W16	10	No cracks
	W15	20	Single cracks
	W13	50	Network of cracks
	W14	70	Network of cracks
	W12	100	Network of cracks

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 will be initiated using multiples irradiation of material surfaces by quasi-steady and pulsed plasma streams with varied plasma loads.

-Cross-links have been started with other CRP members on joint diagnostics of exposed steel samples, material characterization, and preparation of Round Robin tests of reduced activation steel samples.

The next step of project will be focuses on 3 most critical issues relevant to plasma-facing materials of fusion reactor:

- (1) Continuation of studies on modification and alloying of steels under pulsed plasma treatment aimed to increase those sputtering resistance.
- (2) Characterization of various steel grades with respect to their response to sputtering by different kind of ions (hydrogen, helium etc.) and dust production under high flux stationary and transient plasma loads;
- (3) Comprehensive studies of hydrogen/helium retention (outgassing) in steels modified by pulsed plasma streams in comparison with virgin materials, i.e. without plasma treatment.