Institute of Atomic Physics

Commissariat à l'énergie atomique et aux énergies alternatives

Plasma and laser processing of powders for nuclear applications (LAPART)

Project coordinators:

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2nd Joint CEA-IFA Symposium, 17-18th of October 2012, Magurele, Romania

Motivation and scope of the project

Issues :

- Dust incidence in fusion machines - tritium accumulation & high explosive potential of several hundreds of kg of dust - are expected to be produced in next-generation plasma fusion reactors -ITER;
- Toxiciy of powders: the health hazards posed by the handling and removal of such 'waste' are not known;
- Technological significance of particles and powders.

Scope of LAPART:

- To obtain particulates of materials related to nuclear technologies: focus on W, C, and Al or Mg as substitutes for Be; Tools: plasma and laser techniques;
- To investigate properties such as size, surface chemistry, structure, etc.
- To explore how to handle / modify / destroy and use such powders in toxicology studies, hydrogen isotopes adsorption studies, stabilization against the explosive potential.

Task sharing

Romanian partner:

- Fabrication of particles (W, C, Al) : plasma and laser techniques

- ablation of metal targets in liquids;
- etching of matrix in mixed materials;
- plasma cluster sources;

- **Particle characterization:** size, morphology, structure and chemical composition;

-Treatments by laser/plasma of particles/ powders aimed at changing their morphology, structure, surface chemistry.

French partner:

-Characterization of particles: particles size distributions, specific surface area;

-Toxicity studies: particles deposited in lung epithelium and toxicity analyzed;

-Adsorption, suspension, mobilization studies: analysis of deuteration and deuterium desorption; dust suspension study, dust mobilization methods;

-**Explosivity** – possible, a long term objective.

Results / first year

Particles generation / characterization:

• Setup and technique for obtaining metal particles embedded in a carbon matrix;

• Tungsten and Aluminum particles embedded in a carbon matrix via plasma;

• Experiments for plasma etching the carbon matrix, at low and atmospheric pressure;

• Setup and technique for particles production by pulsed laser ablation in liquids;

•Tungsten and Aluminum particles obtained by laser ablation in acetone and water;

• Size distributions determined – sizes from tens of nm to microns.

Exchanges: Christian Grisolia, Bucharest in April 2011;

Gheorghe Dinescu - participation to CIP 2011- Nantes, E-MRS - Nice.

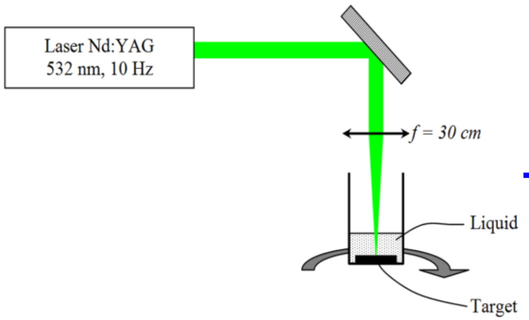
Publications: 3 contributions to International conferences;

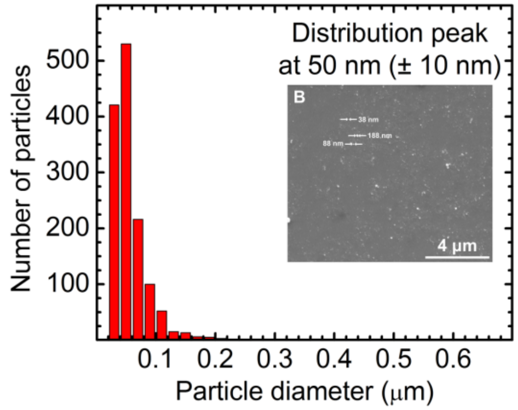
1 paper in work.

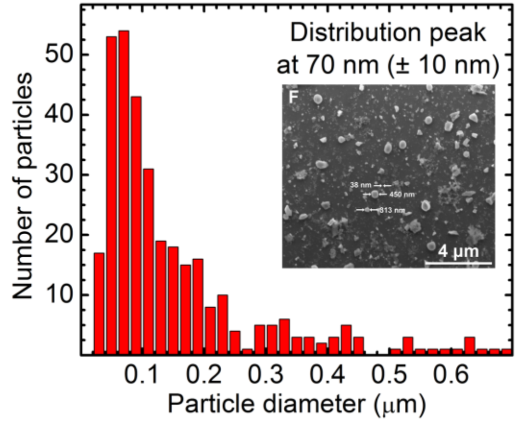
Scientific achievements 2nd year - outline

- Previous results;
- Setup and technique for fabrication of W particles by a plasma based cluster source;
- Setup for fluidization of powders by plasma jets at atmospheric pressure;
- Surface modification of powders in a fluidized bed plasma reactor;
- Future work, perspectives and benefits.

Previous work

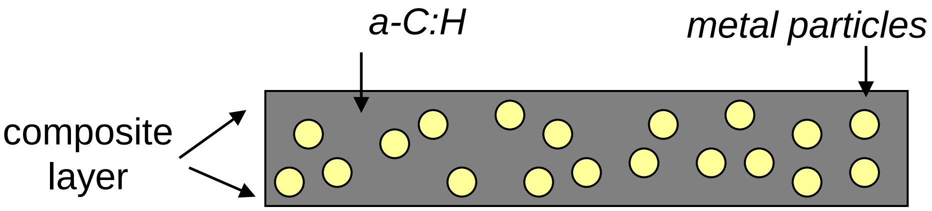




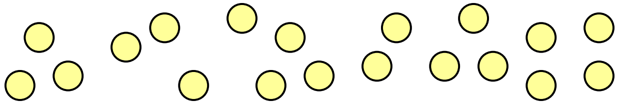


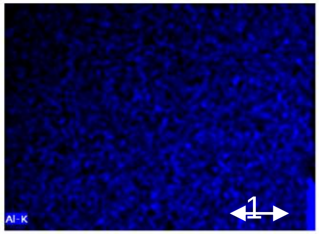
Successful!

Particles embedded in a-C;H matrix: PECVD and MS



plasma erosion: a-C:H matrix removal





EDX image: etched surface

- Particles (Al) visible in EDX;
- Removal of matrix at surface:

Successful!

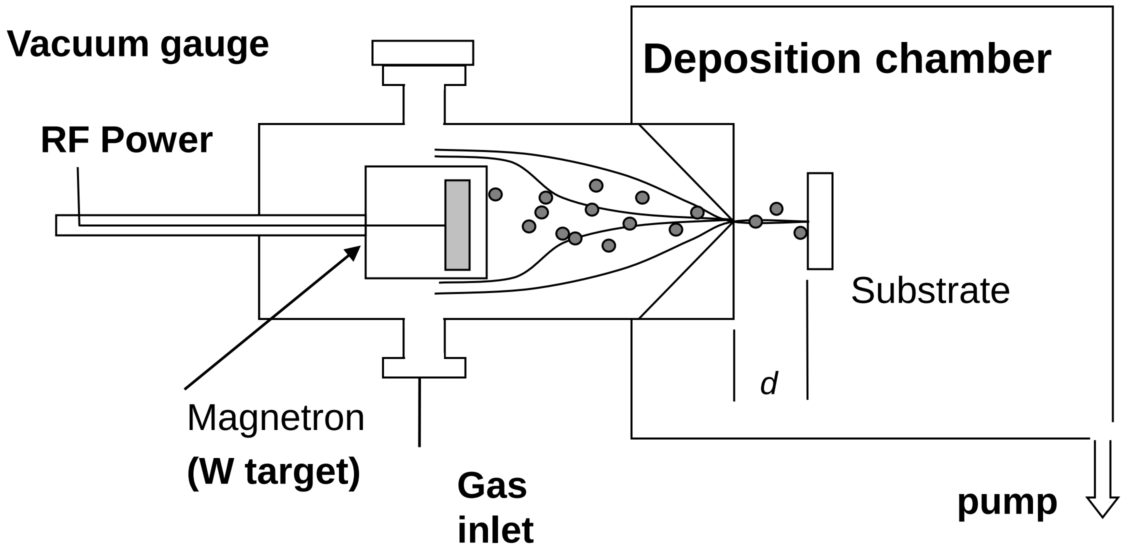
- Release of all particles:

Unsuccessful!

Particle production: plasma cluster source

Synthesis of W particles by a plasma based cluster source

Setup and technique



Parameters:

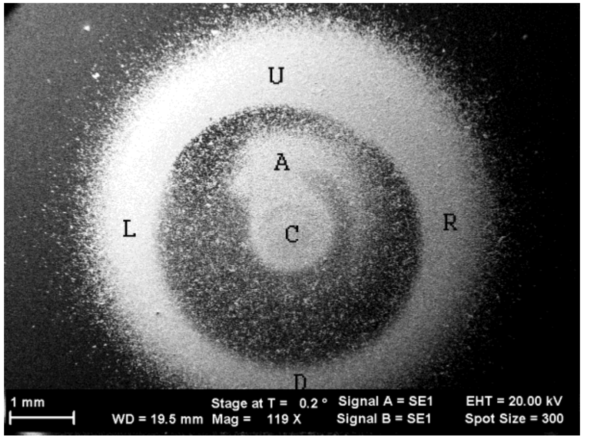
RF Power : 100W

Flow rate: 10 scmm Argon

Pressure in source: 8.5E-1 mbar

Pressure in chamber: 7.3×10^{-3} mbar

Deposition time: 30 min

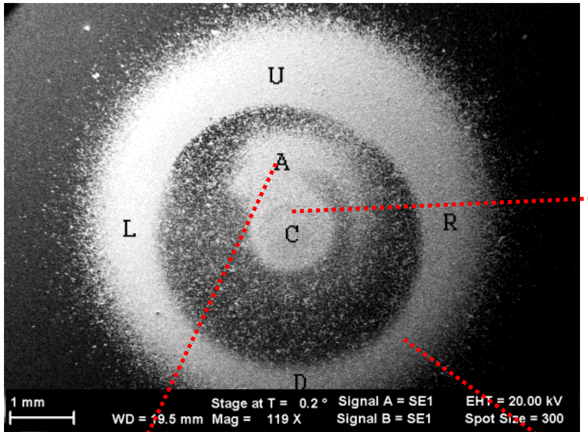


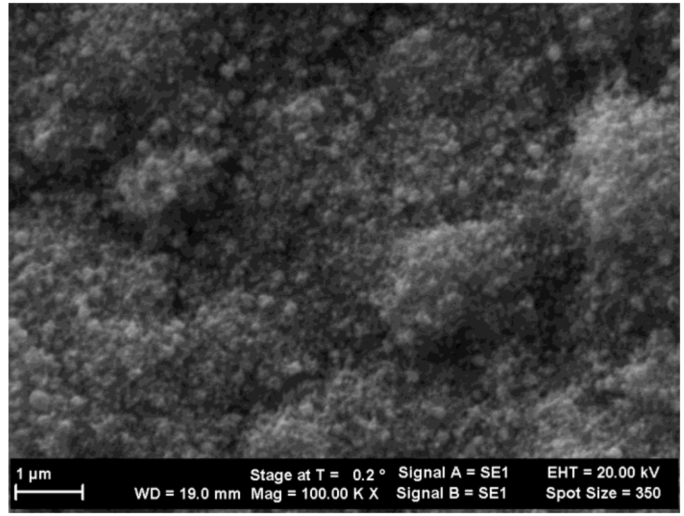
SEM image of the spot on the substrate

Setup components

- Aggregation chamber: enclosure with gas inlet, nozzle;
- Magnetron source;
- Collecting (deposition) chamber;

Particle morphology



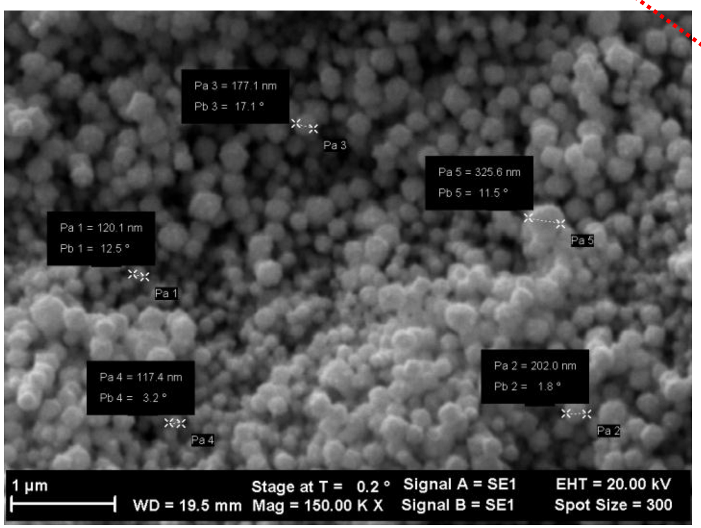


Central spot:

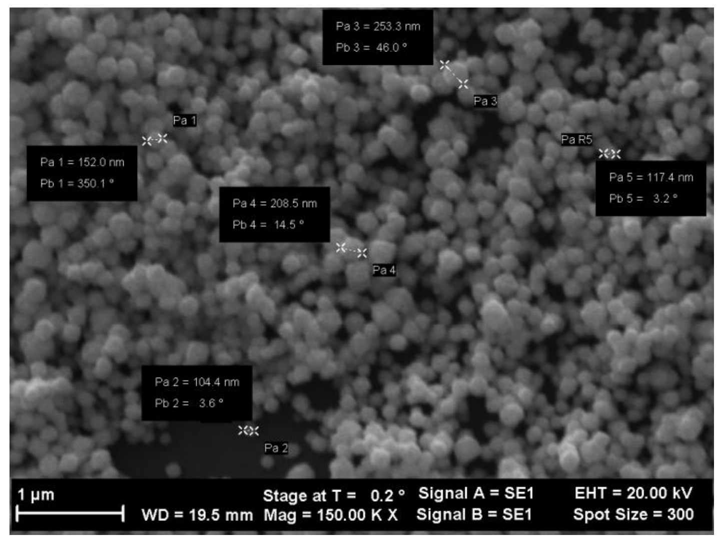
agglomerated

particles, small size,

also large size



Outer ring : apparently rounded particles, separated



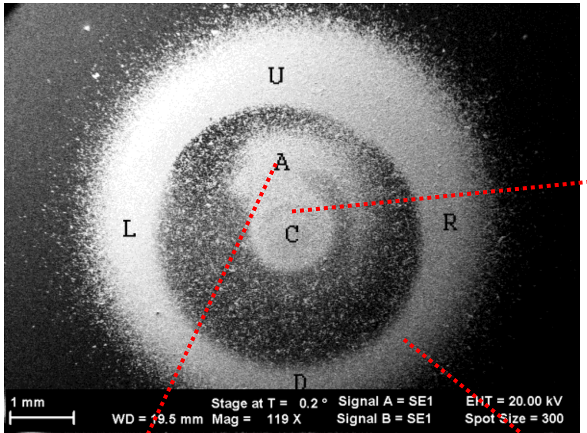
Lateral spot,

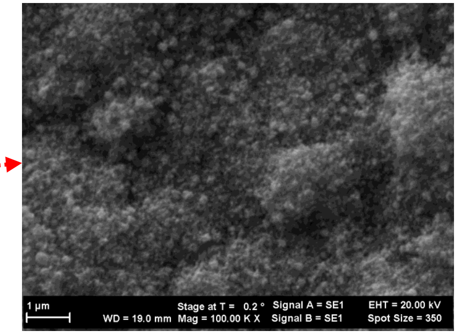
apparently rounded

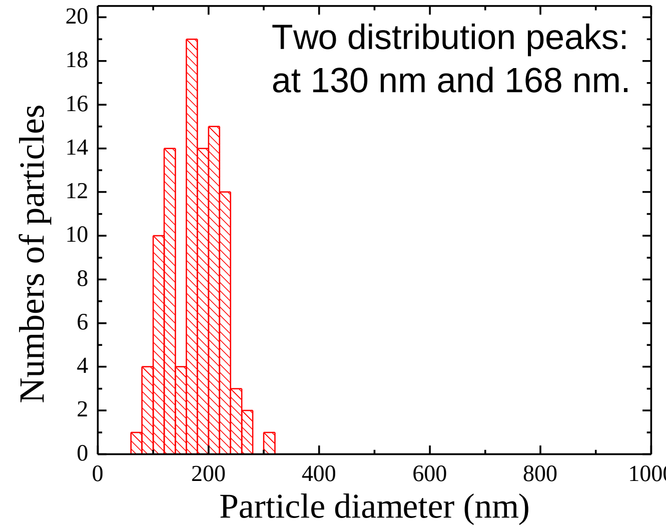
particles

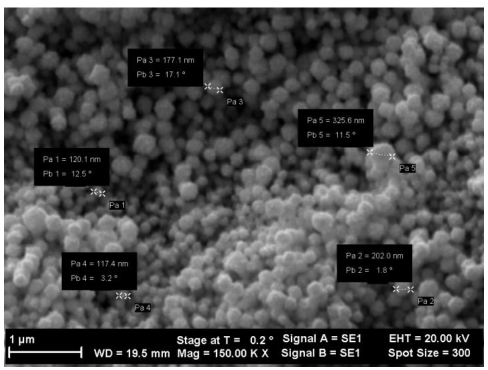
size around 200 nm

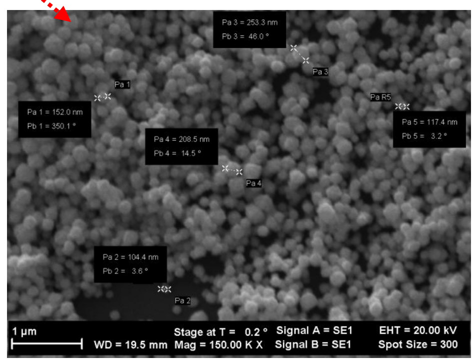
Size distribution

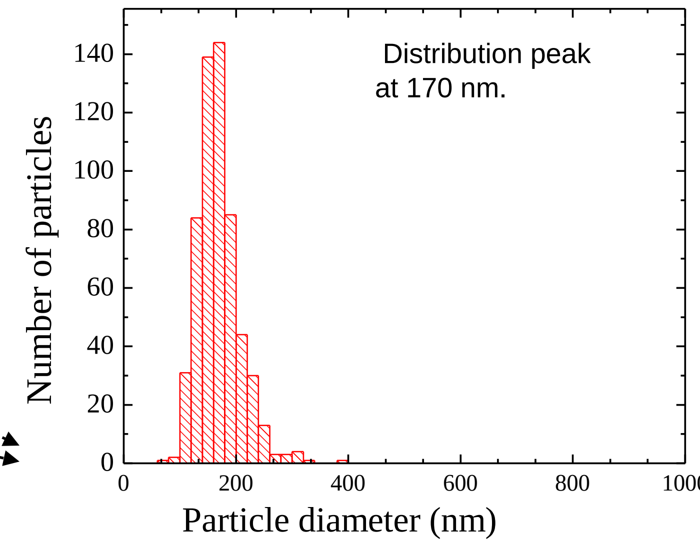






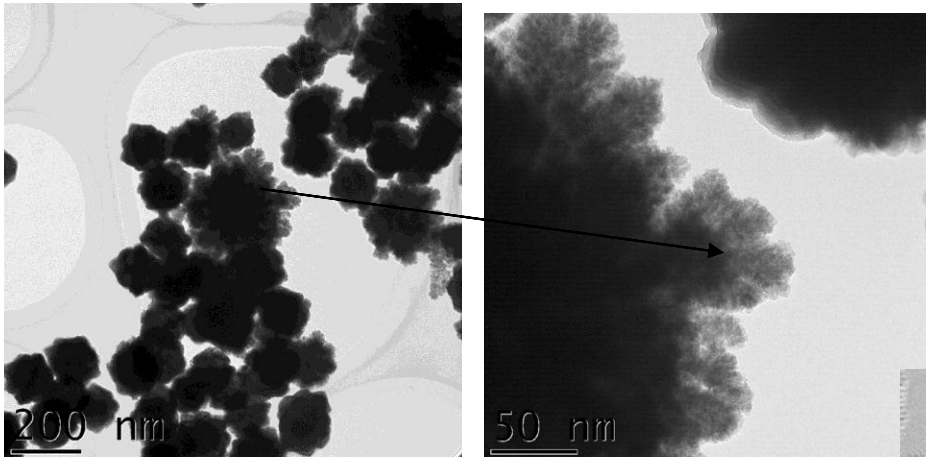




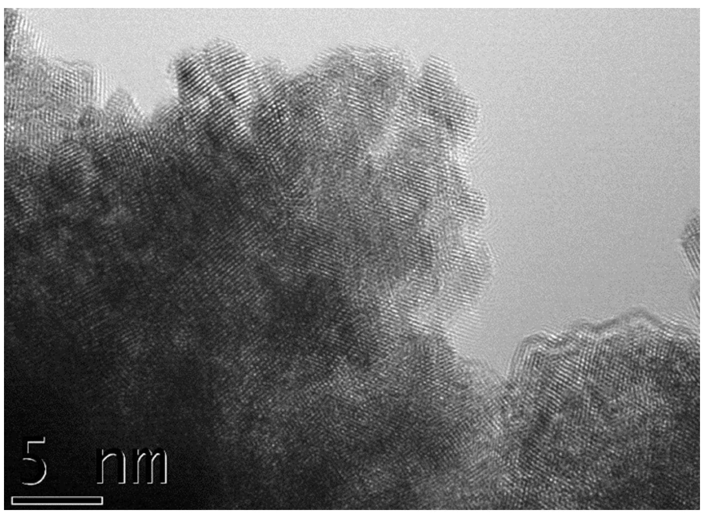


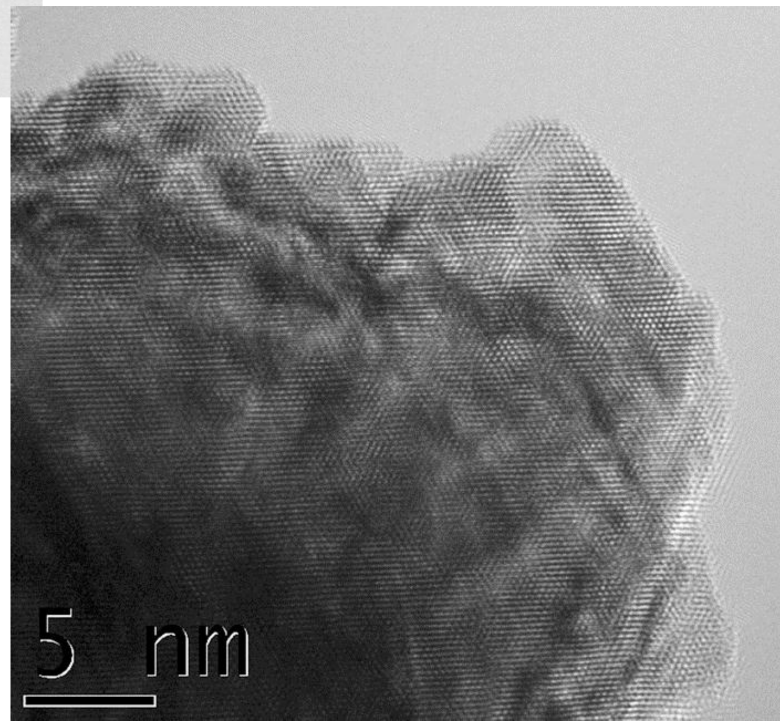
We succeeded to produce particles with size distribution peaked at around 170 -180 nm

Particle structure: TEM and HRTEM



- Two types of particles: a) showing facets; b) with dendritic branches
- Particles are formed from nanocrystals of 3-5 nm oriented in the faceted particles, and less oriented in the dendritic particles





Acknowledgments: Leona Nistor, INFM

Powder modification

Fluidization of W particles in a plasma jet

Problems in powders modification:

- make each particle to come in contact with plasma!
- Induce modification: oxidation, nitriding, structure?

Solution: Fluidization in a plasma jet

- contact: ensured by particles mixing with plasma
- modification: ensured by reactive plasma species

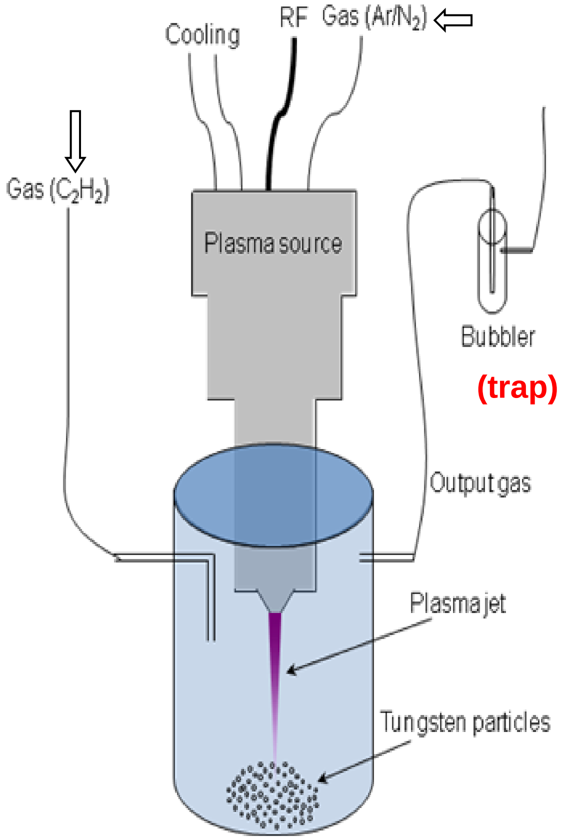
RF plasma jet - atmospheric pressure

Fluidization Designs

Previously realized in the frame of EFDA – EURATOM, collaboration with CEA

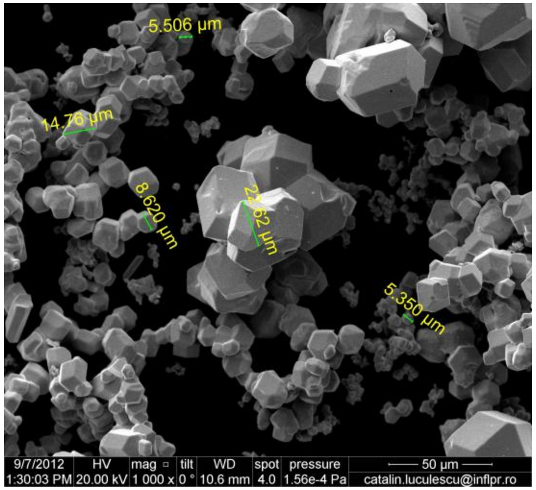
Advantage: atmospheric pressure, vacuum not needed!!!

Fluidization aspects (industrial W powder)



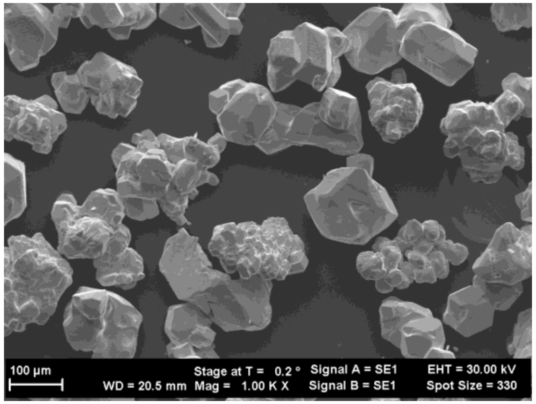
Fluidization effects: particles separation

Initial powder

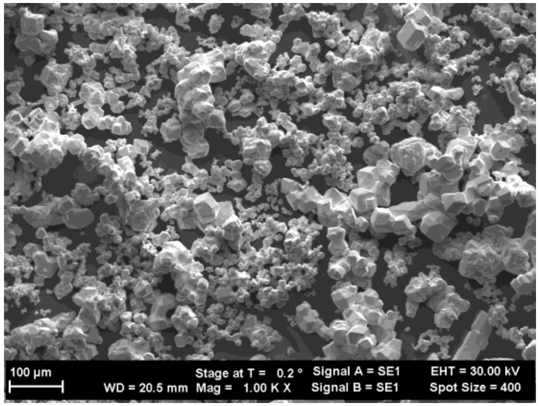


Large particles

Small particles

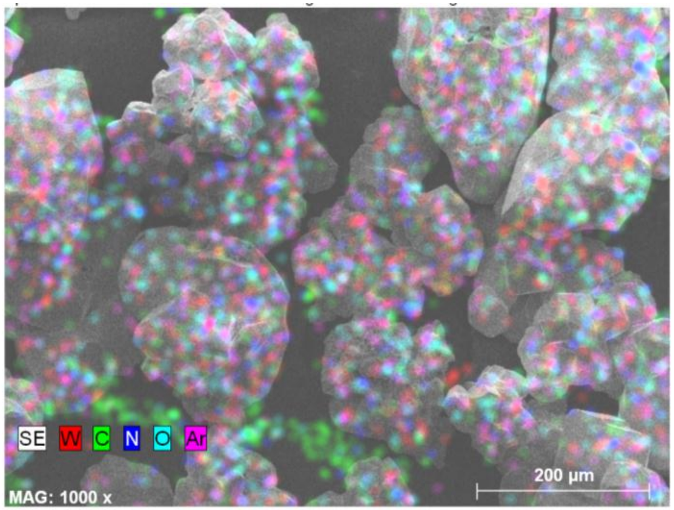


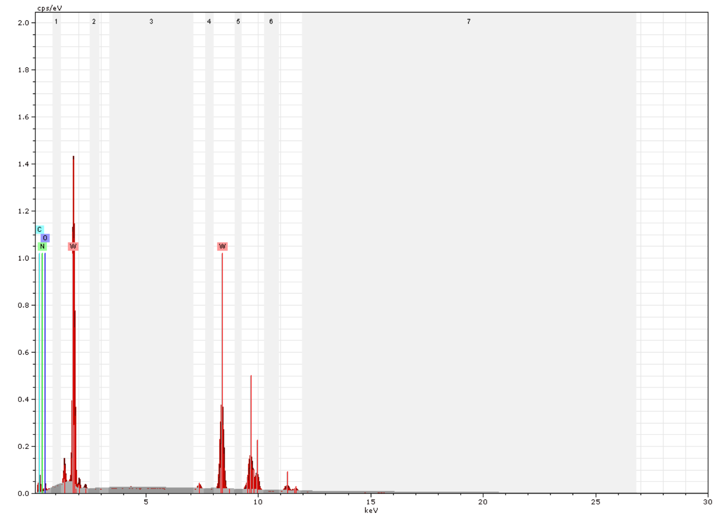
Powder remained in the reactor



Powder released in the trap

Modification of chemical composition -EDX

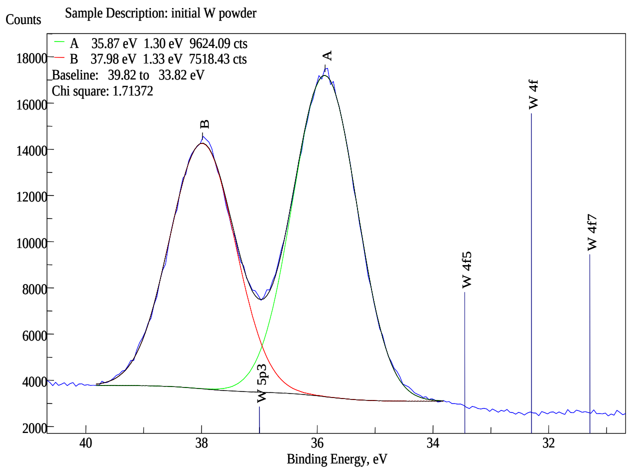


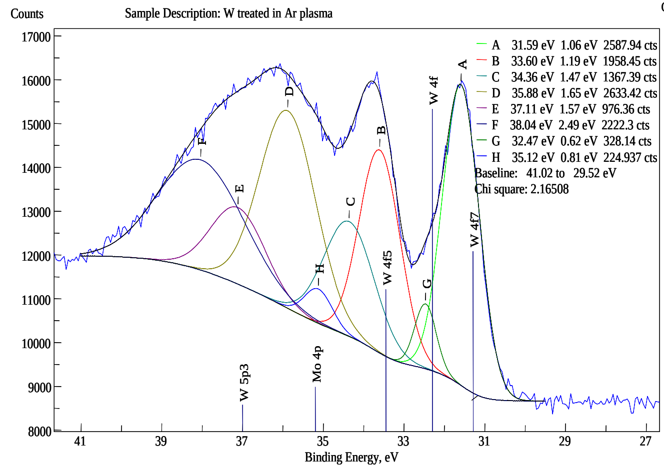


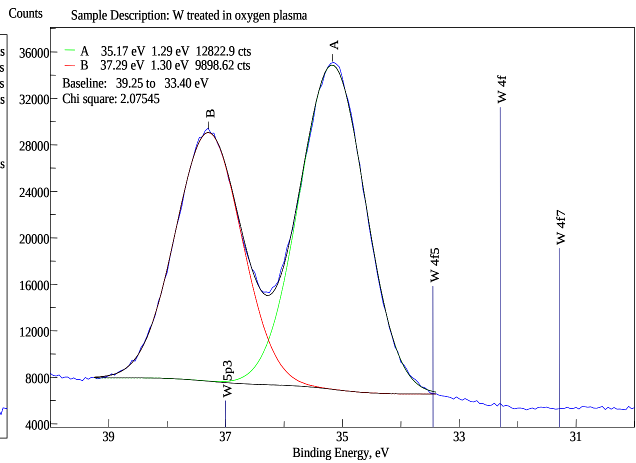
Element/ Sample Code	**W initial**	Ar plasma a (reactor)	Ar plasma B (trap)	Ar/O_2 plasma a	Ar/O_2 plasma b	Ar/N_2 plasma a	Ar/N2 plasma b
C	**47.80**	**19.37**	**29.80**	15.72	18.31	29.28	29.79
N	**23.01**	30.28	28.81	28.36	27.94	**32.94**	**28.35**
O	**15.34**	37.77	24.47	**42.86**	**42.58**	18.07	22.89
W	**13.85**	12.57	16.93	13.06	11.17	19.71	18.97
		Reduction of carbon content		**Important increase of oxygen content**		**Slight increase of nitrogen content**	

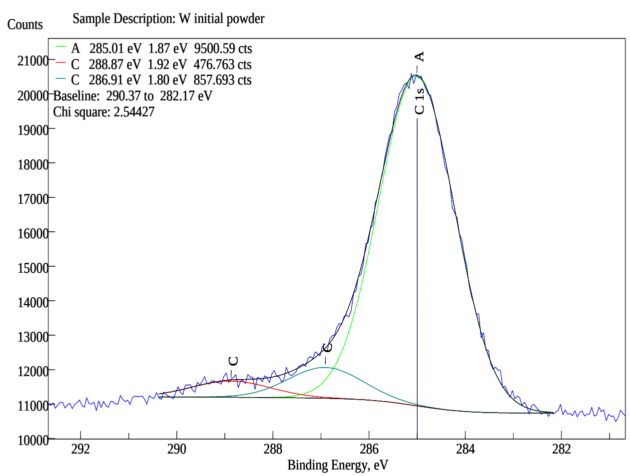
Modification of chemical bonding - XPS

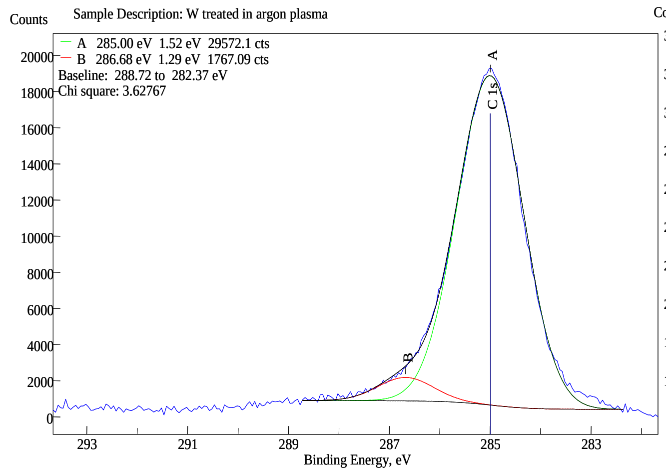
Initial W powder | Ar plasma treatment | Ar/O_2 plasma treatment

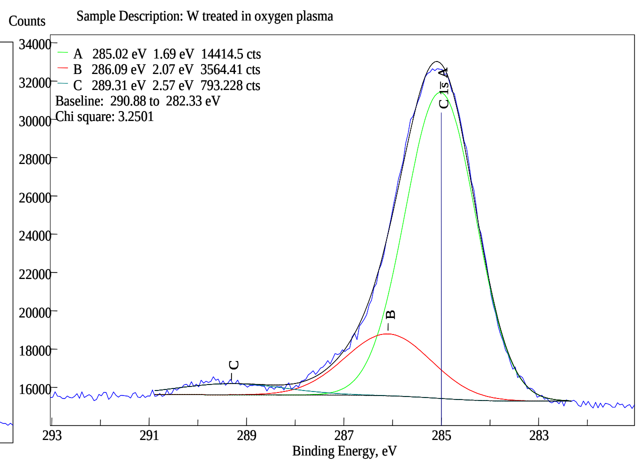












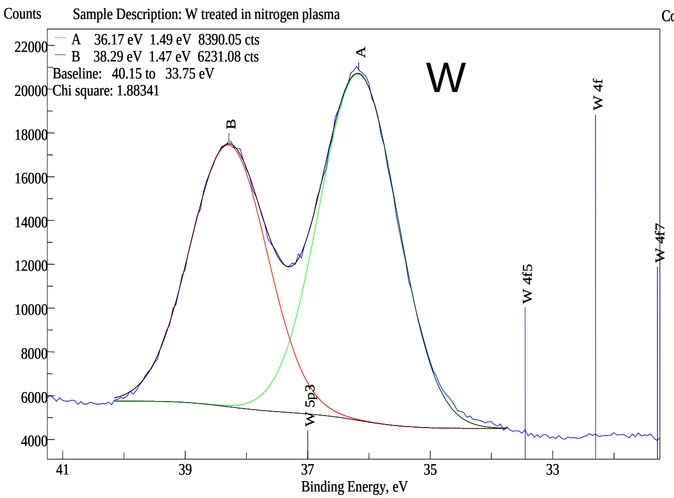
Completely oxidized

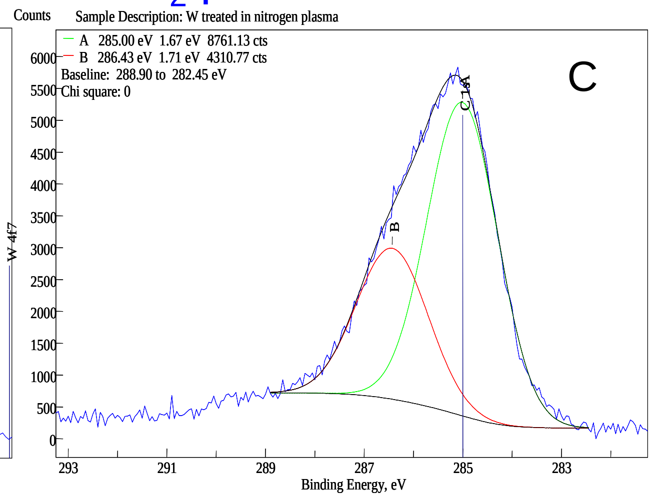
W metallic and in various oxidation states, no C=O

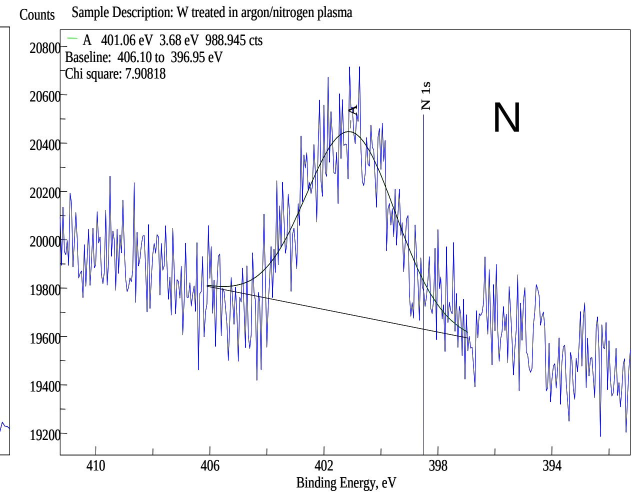
Completely oxidized W, C-O bonds increase, C=O

Modification of chemical bonding - XPS

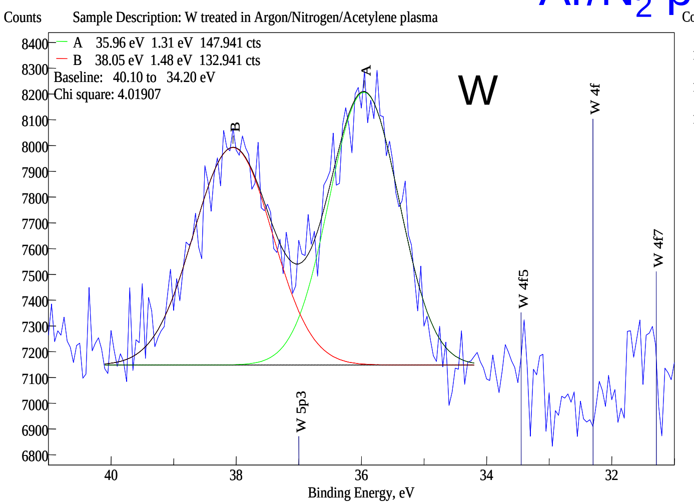
Ar/N_2 plasma treatment

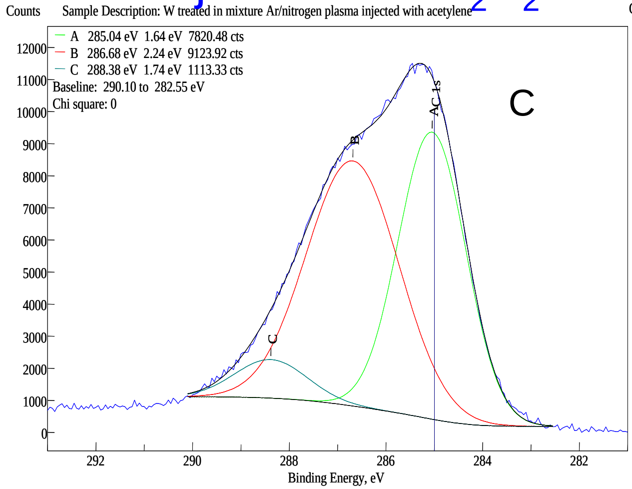


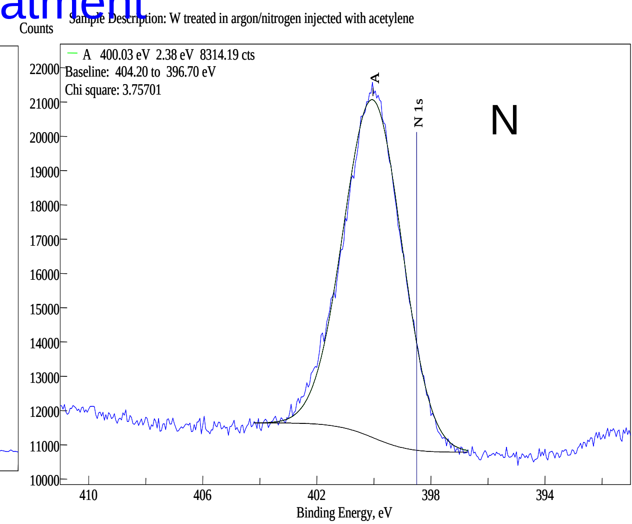




Ar/N_2 plasma injected with C_2H_2 treatment







Broadening of W peaks-nitridation, low signal-coating

Important contribution of C-N/O-C-N related peak

Formation of N-O related bonds

Conclusions

Results / second year

Particles generation / characterization/transformation:

•A cluster plasma source was elaborated and its ability of producing tungsten particles was proved;

•The size of the tungsten particles is in the range 160-200 nm;

•A fluidized bed reactor for particles modification was developed;

•Surface modification of industrial made tungsten particle was proved;

New projects and students:

• In preparation: a common project proposal in the frame of EURATOM;

• 1 PhD student starting October 2012 on a topic of particles in plasmas;

Exchanges: Christian Grisolia, Bucharest in Dec. 2011, Oct. 2012;

Publications: 1 paper published, 1 paper in work; 4 contributions to International conferences.

Project benefits and perspectives

Further work

• control of particle size distribution; more detailed characterization; production of larger quantities, deuteration of particles;

• study of the health and nuclear safety issues posed by the nanoparticles.

Benefits:

• *new research opportunities and projects*, good practice dissemination;

• *know-how* to produce metal nanoparticles by means of plasma and laser, powder modification techniques – also, industrial relevance;

• *research perspectives* in the field of nanomaterials with controlled properties, beyond those envisioned by the scope of the present project;

Thank you for your attention!

Acknowledgments: **Tomy Acsente**, for particle synthesis work
Bogdana Mitu, for particle characterization work