Radiation-induced Ductility Enhancement in Amorphous Fe and Al2O3+TiO2 Nano-structured Coatings under Fast Neutrons

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BROOKHAVEN NATIONAL LABORATORY

a passion for discovery

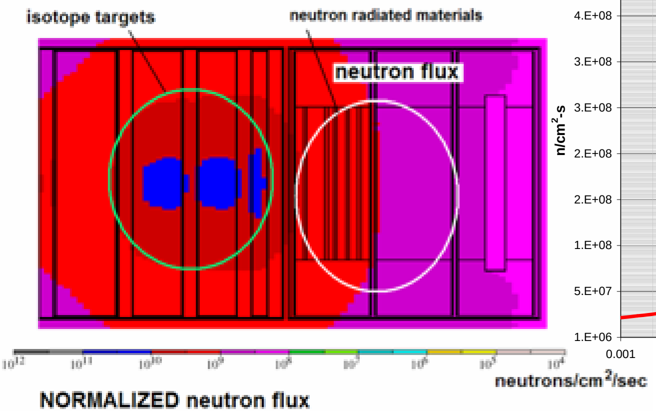
Webinar - August 20, 2013

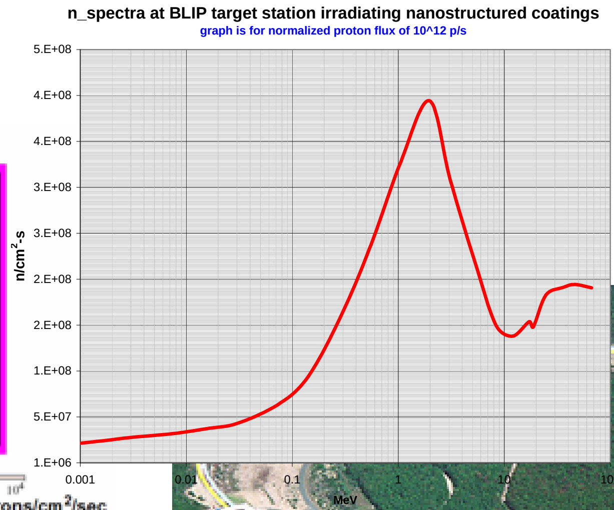
Office of Science
U.S. DEPARTMENT OF ENERGY

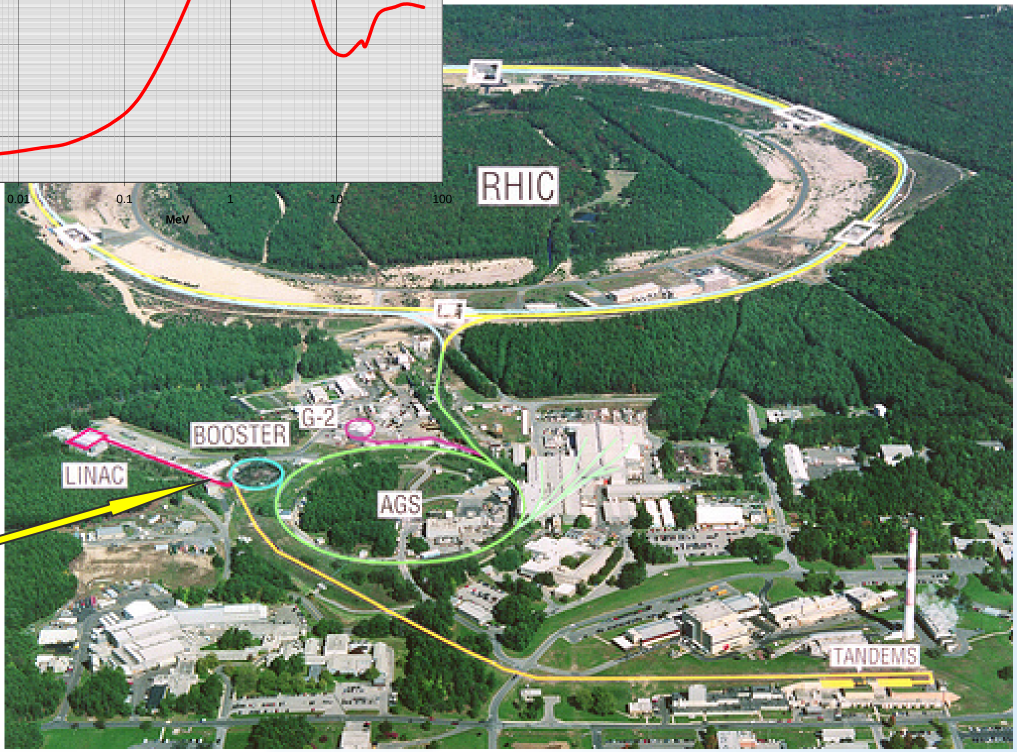
Project Description

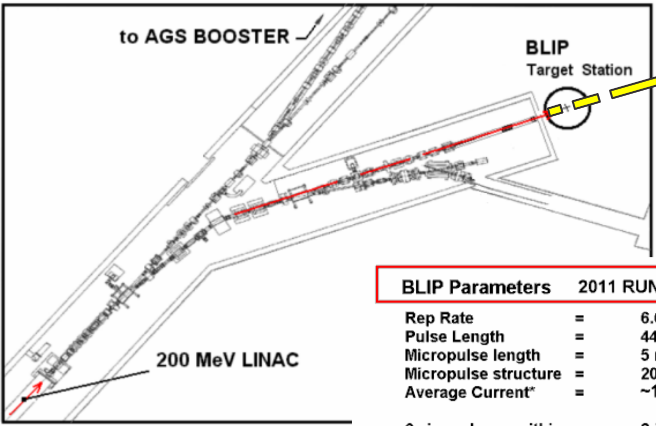
- Explore the protective performance of Fe-based amorphous and ceramic (Al_2O_3 and Al_2O_3+TiO_2) nanostructured coatings under extreme temperatures, neutron irradiation and aggressively corrosive environments
- Study the effects of fast neutrons
- Study resistance to ductility loss of the composite structures (coating + substrate) and dimensional changes for Ti and steel alloy substrates
- Explore oxidation resistance of the nano-structured coatings
- Study their micro-structural evolution (amorphous to crystallization) up to 1100 ^{o}C

Assess whether significant technological impact can be realized by providing the reactor materials program with a new, high-performance class of metal/ceramic composite materials for use in critical reactor components









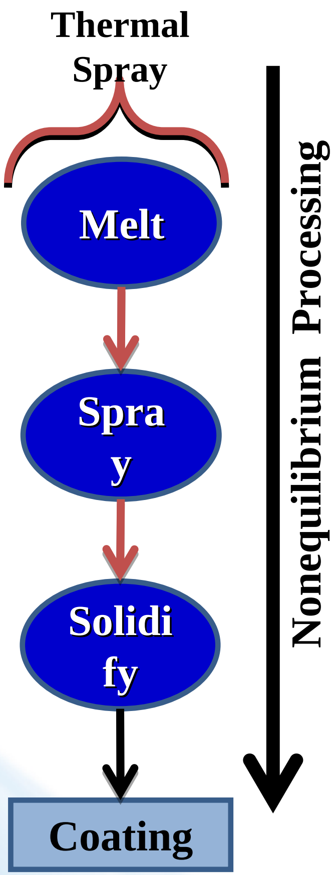
BLIP Parameters		2011 RUN
Rep Rate	=	6.67 Hz
Pulse Length	=	440 micro-secs
Micropulse length	=	5 ns
Micropulse structure	=	200.25 MHz
Average Current*	=	~105 micro-A
6 sigma beam within	=	2-inch diameter
Beam Gaussian	==>	1 sigma = 4.233 mm

Linac proton flux of 5.93 e+14 p/s is typical

Project Status

- Fast neutron irradiation of amorphous Fe and ceramic coatings (ongoing) at BNL accelerator complex (irradiation through spallation)
- Temperature effects (annealing, microstructural changes, thermal expansion of substrates and composite structures)
- Mechanical testing (stress-strain, 3-point bending)
- Characterization (x-ray phase/strain mapping)
- Microscopic analysis and amorphous-to-crystallization transition
- Oxidation resistance assessment (experimental and thermodynamic analysis)

Why interest in Fe-based amorphous coating?

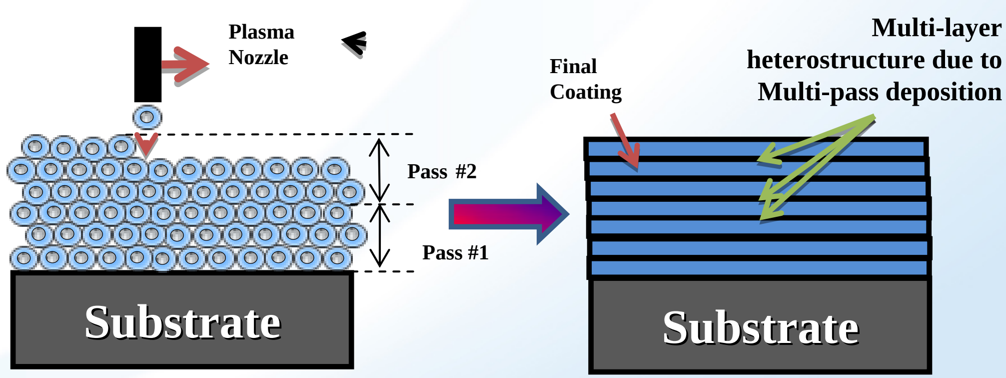


Purpose:
Deposit a single phase coating of a thermodynamically metastable but kinetically stabilized single phase amorphous-Fe alloy by melting-cooling type non-equilibrium processing.

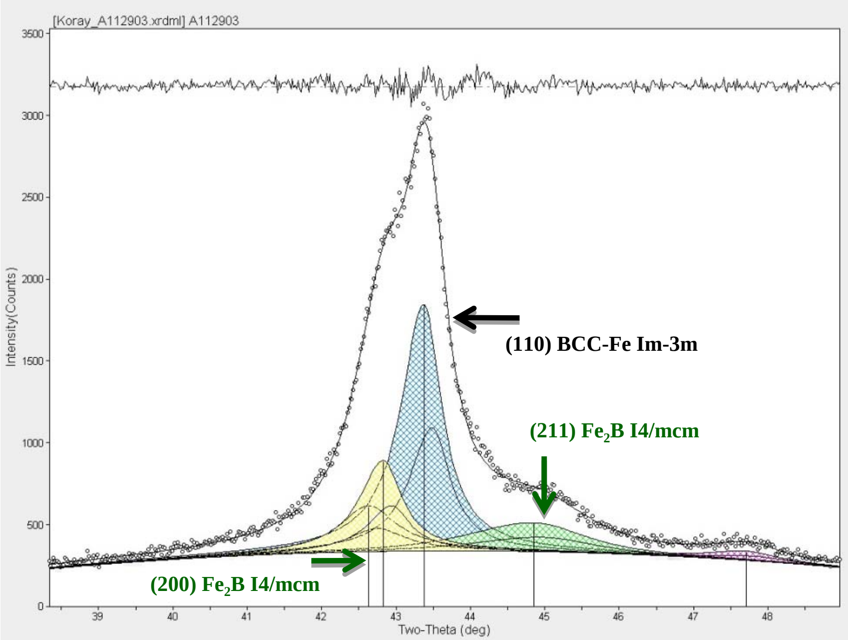
Desired Properties:
High strength and superb tribological properties
Superb oxidation and corrosion resistance

Challenge:
Preserve metastability during processing to obtain an amorphous metallic coating.

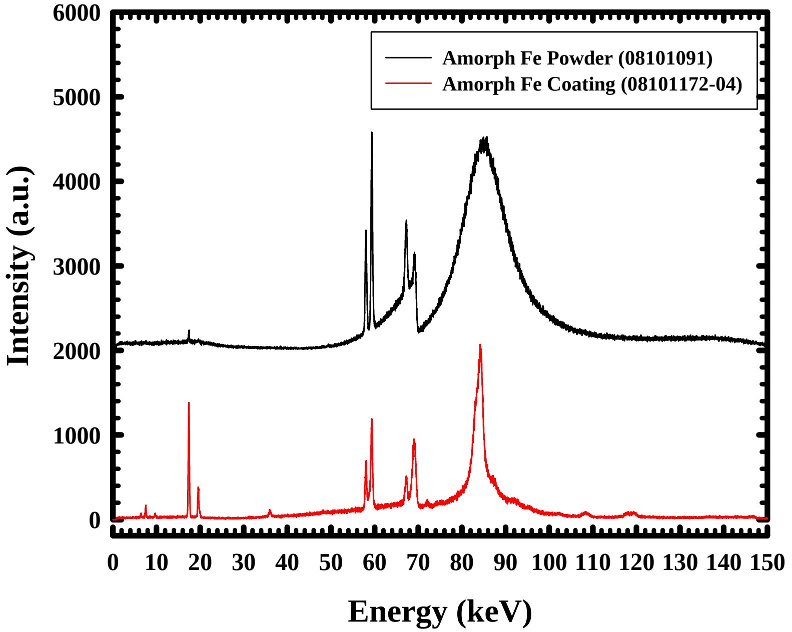
Phenomena	Desired
Melting	100% Melting
Solidification	Fast enough to prevent crystallization
Phase Separation	None. Homogenous, single phase coating preferred
Oxidation	High oxidation resistance of Amorphous Fe
Distribution of Alloying Elements	No carbide formation & homogenously distributed in Fe coating
Compositional Fluctuation	None. Homogeneous coating desired



Qualitative & Semi-Quantitative Phase Analysis



The spectrum for the coating pertains to the scan which was taken at an approximate depth of 80 μm.



The spectrum of powder was shifted by 2000 counts upward to superimpose it onto the spectrum of the coating.

Assessment of Fe-based amorphous coating in un-irradiated state (Rutgers U.)

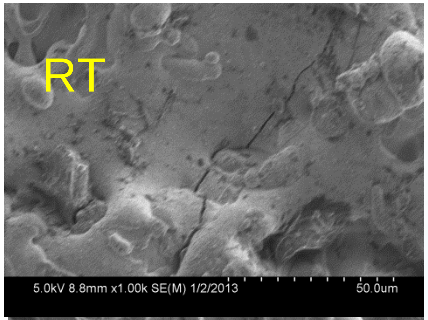
The coating of interest is a 4-phasic composite consisting of BCC-(Fe-B-C) alloy, $(Fe,Cr,Mo)_3O_4$, $(Fe,Cr)_2B$ and an amorphous (Fe-B-C) phases.
Hence, it is a metal-ceramic-metallic glass composite.

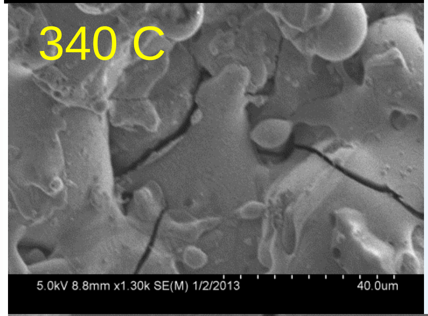
The amorphous phase should be considered as the leftover of the incomplete melting during the thermal spraying due to limited residence of particles in the plasma.

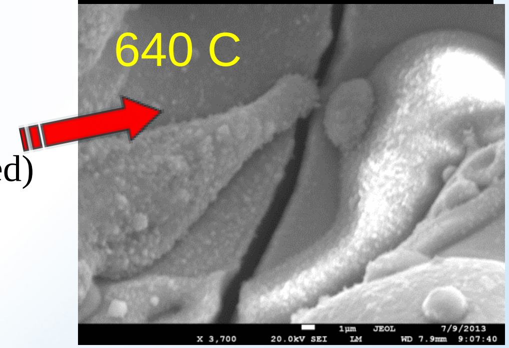
Phase separation, crystallization and oxidation observed in the coating, indicating the feed material is not stable to any of these processes under the processing conditions used. The cooling rate of the coating and atmosphere control during thermal spray during cooling are the main processing parameters to control.

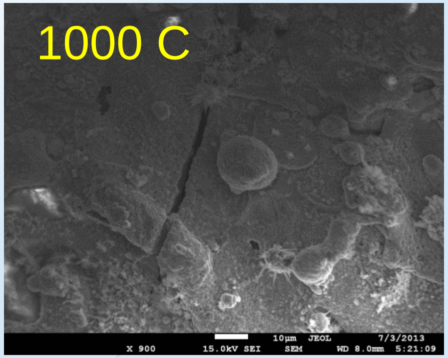
Poor mechanical behavior expected/attributed to oxide (Fe_3O_4-based) and boride (Fe_2B-based) formation. **Both phases as hard and brittle.** The oxide phase is expected to form at grain boundaries as per known oxidation mechanisms in polycrystalline metals.

Poor corrosion (and oxidation resistance) of Fe is expected/attributed to redistribution of alloying elements among BCC-Fe, Amorphous-Fe, $(Fe,Cr,Mo)_3O_4$ and $(Fe,Cr)_2B$.

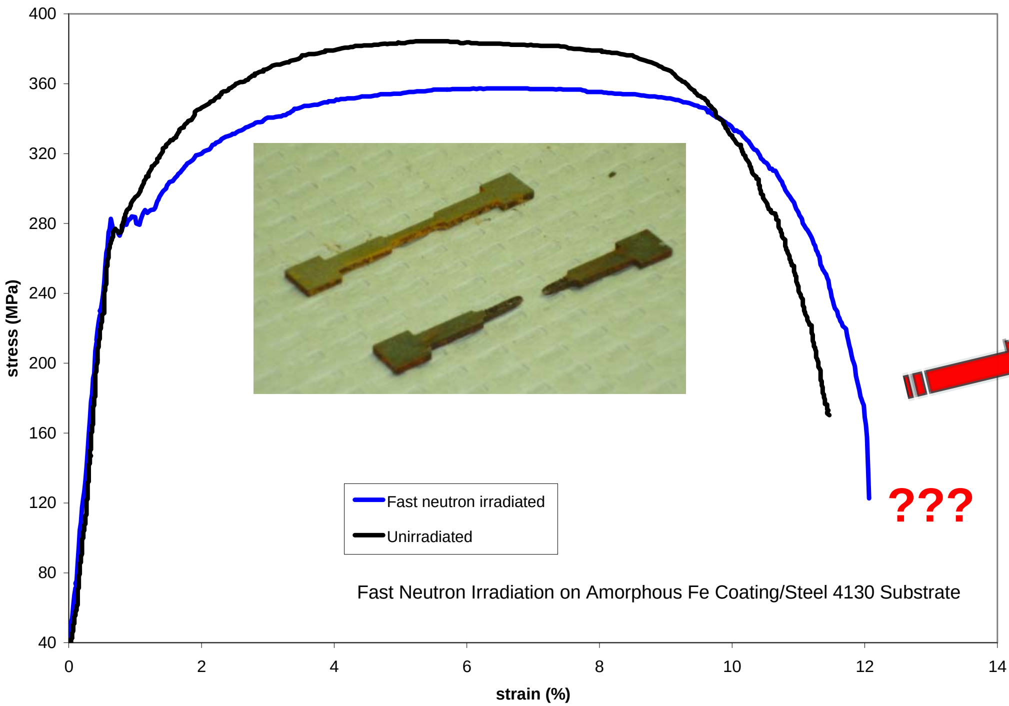






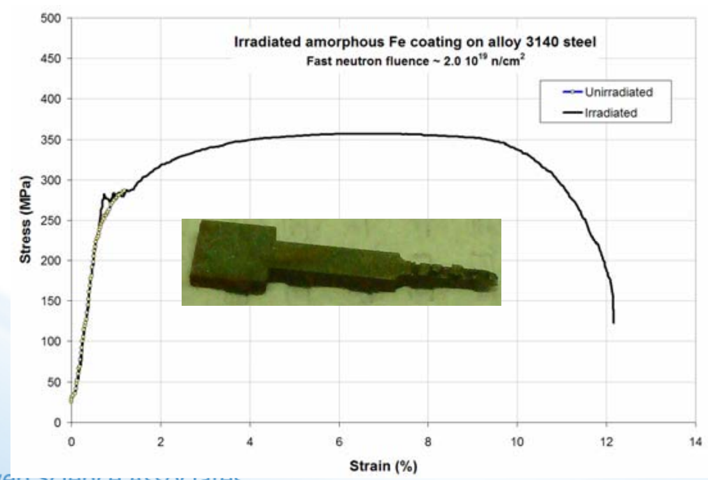


Amorphous Fe nanostructured Coating on 4130 Alloy Steel

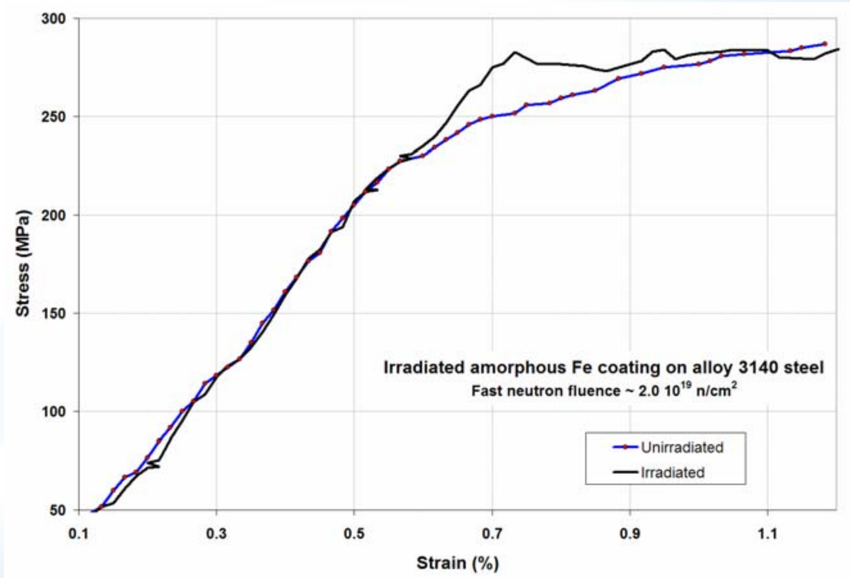


???

Excess free volume from irradiation-induced swelling

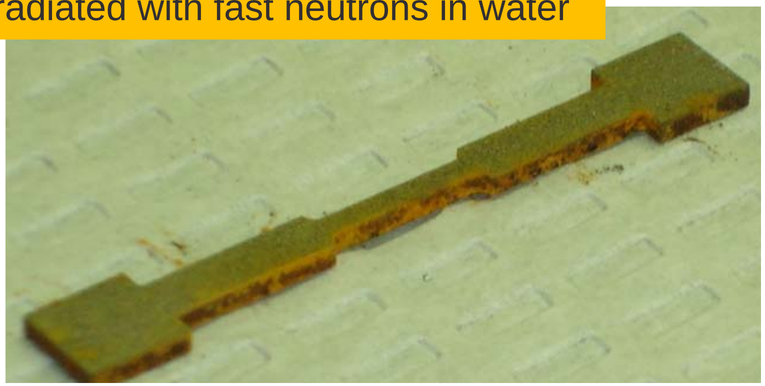


KNE-like behavior in TENSION !!!



Amorphous Fe nanostructured Coating on Steel

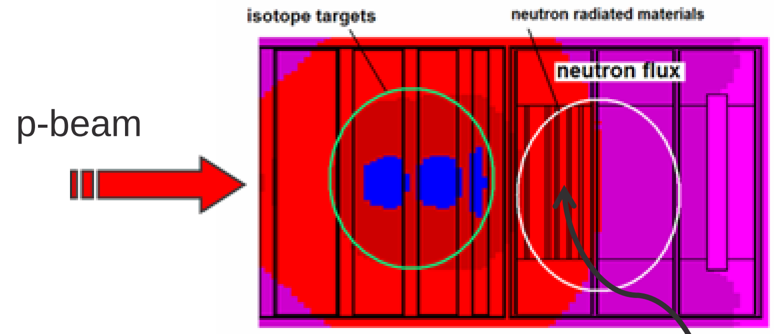
Irradiated with fast neutrons in water



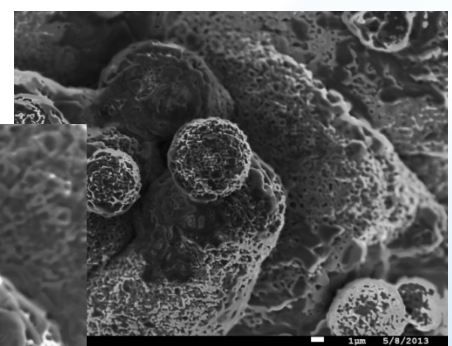
Corrosion resistance of the coating might very well arise from Fe_2B

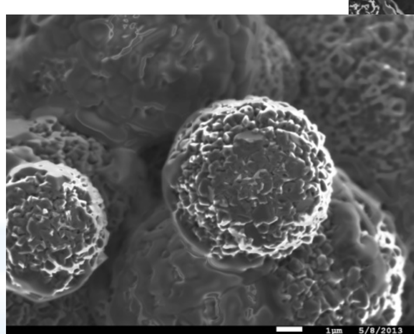
(after E.K. Akdogan, Quantitative Phase Analysis Of Amorphous-Fe Thermal Spray Coating;
Stability, electronic and mechanical properties of Fe2B
B. Xiaoa, et al, Physica B 403 (2008) 1723–1730)

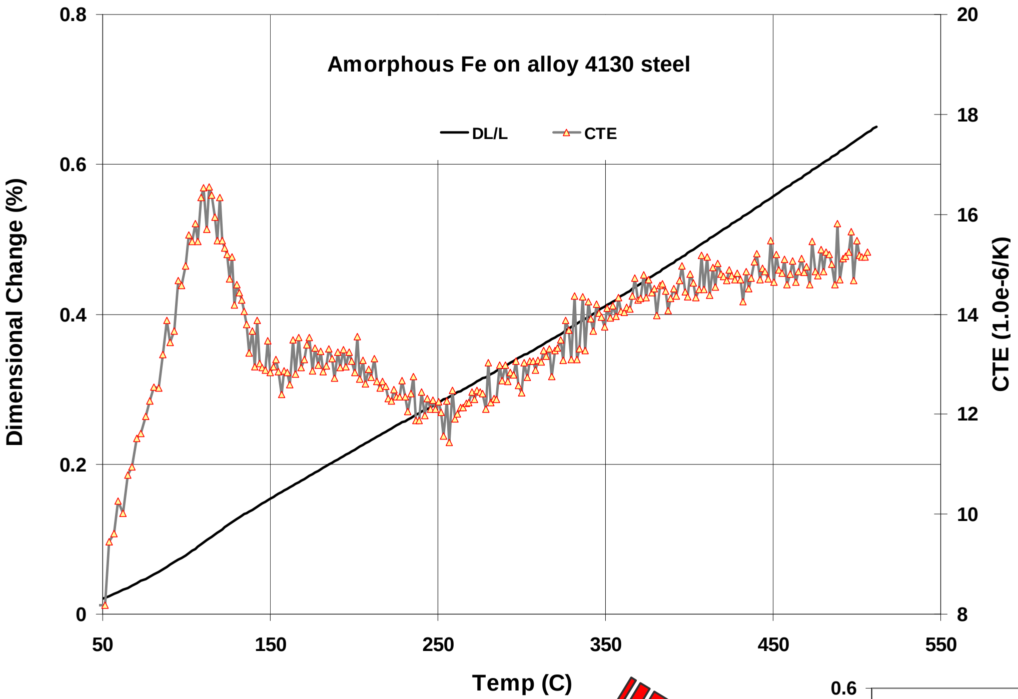
On-going Irradiation/Oxidation Test

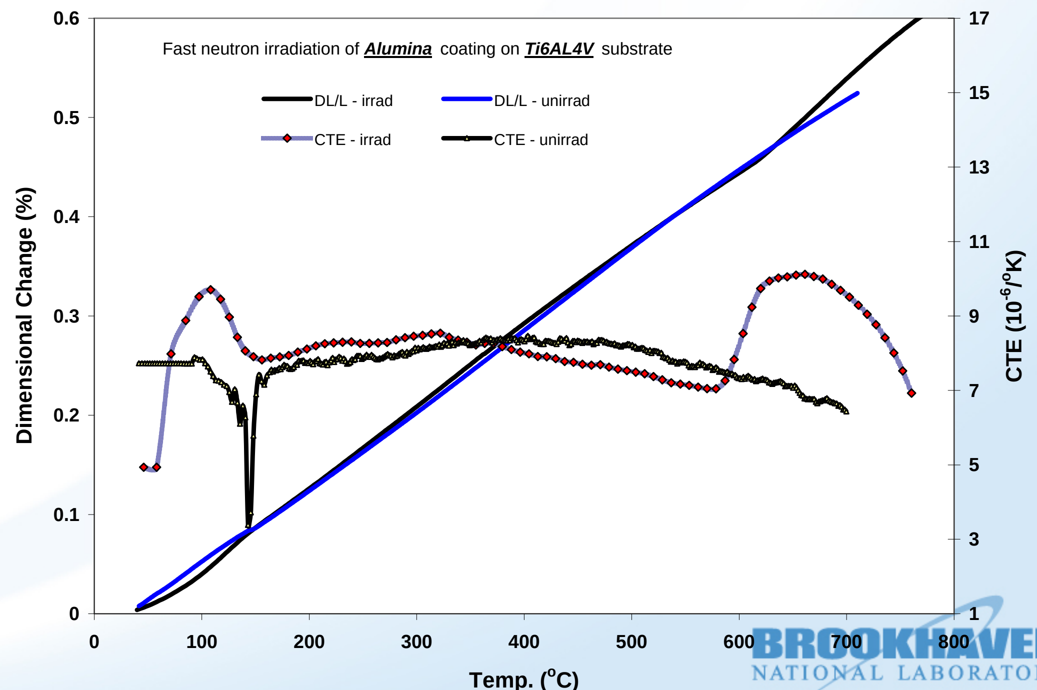


Annealed in oxidizing atm at 700 °C





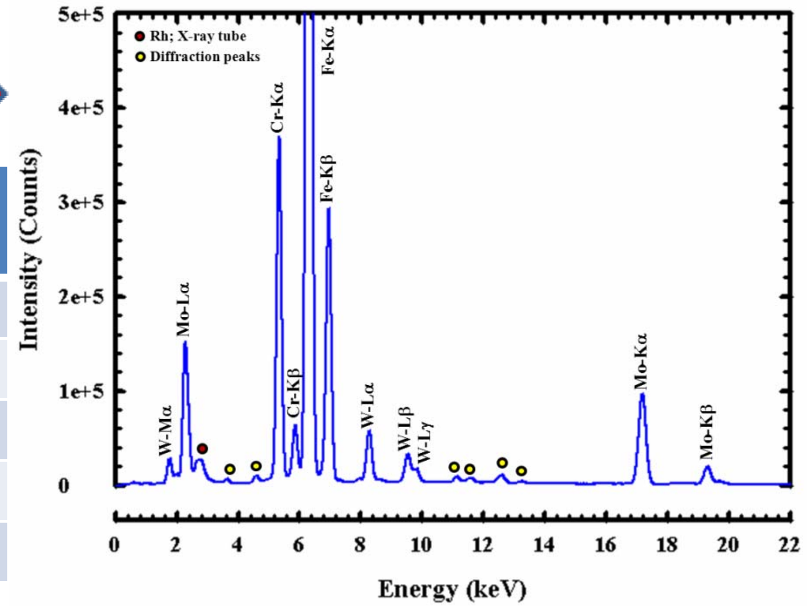

Amorphous Fe on alloy 4130 steel
DL/L
CTE
Dimensional Change (%)
CTE (1.0e-6/K)
Temp (C)
0
0.2
0.4
0.6
0.8
8
10
12
14
16
18
20
50
150
250
350
450
550


Fast neutron irradiation of Alumina coating on Ti6AL4V substrate
DL/L - irrad
DL/L - unirrad
CTE - irrad
CTE - unirrad
Dimensional Change (%)
CTE ($10^{-6}/^oK$)
Temp. (^oC)
0
0.1
0.2
0.3
0.4
0.5
0.6
1
3
5
7
9
11
13
15
17
0
100
200
300
400
500
600
700
800

XRF Spectrum of the Amorphous-Fe Feed Powder

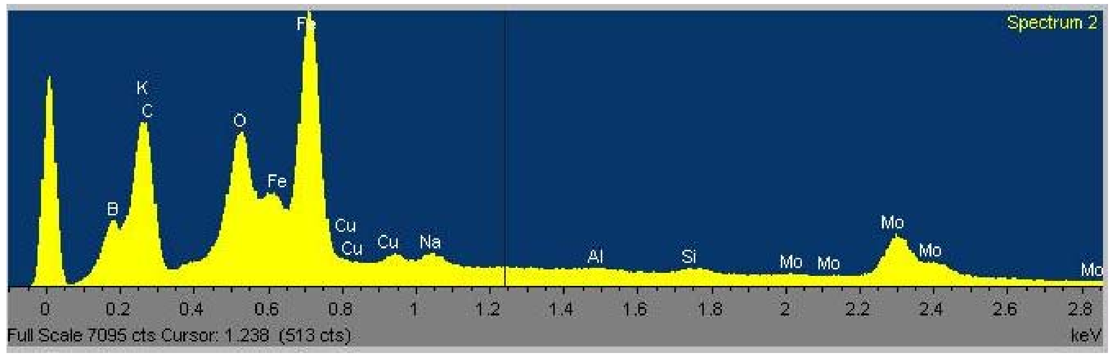
Note: The XRF analysis is not able to detect the presence of B and C.

Element	Net	Wt%	At%	I-Error %	Background
Cr-K	915.69	9.55	11.49	0.25	37.30
Fe-K	4698.59	69.59	77.97	0.11	36.16
W-L	112.08	9.83	3.35	0.79	16.74
Mo-K	212.49	11.03	7.19	0.53	8.45
Total	-	100.00	100.00	-	

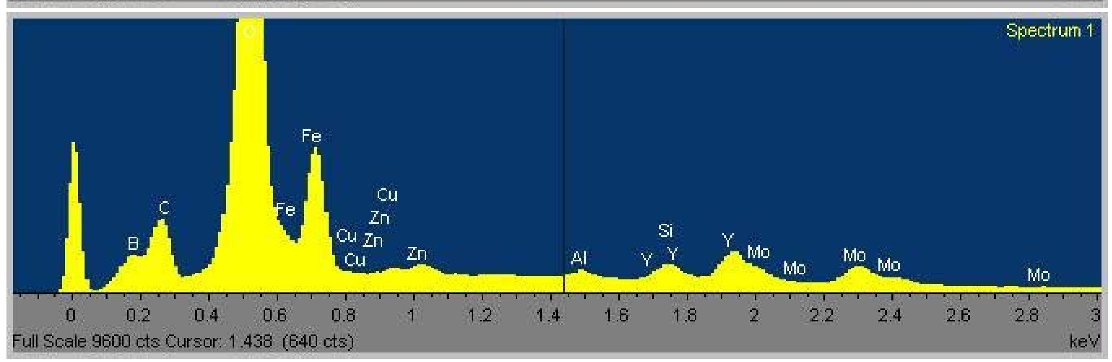


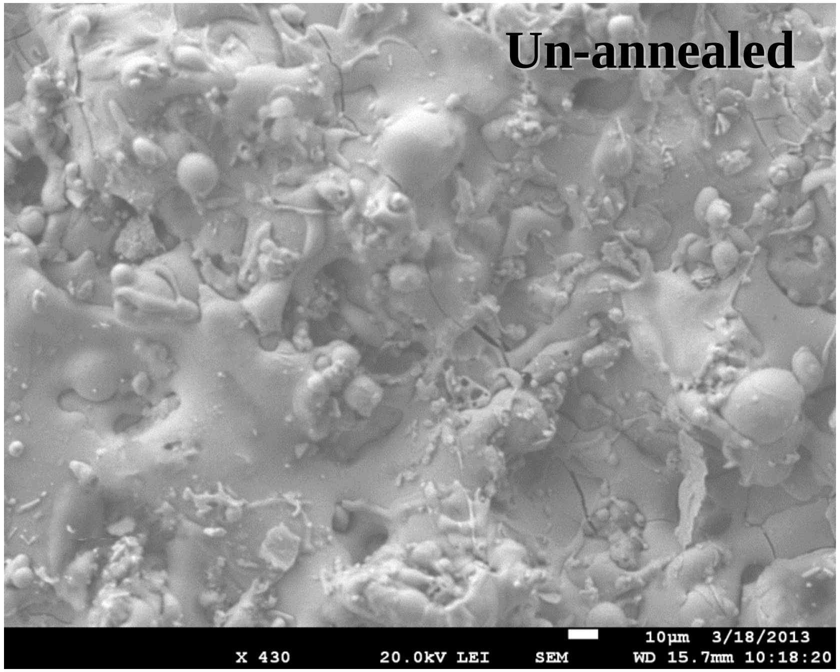
EDS Analysis at CFN

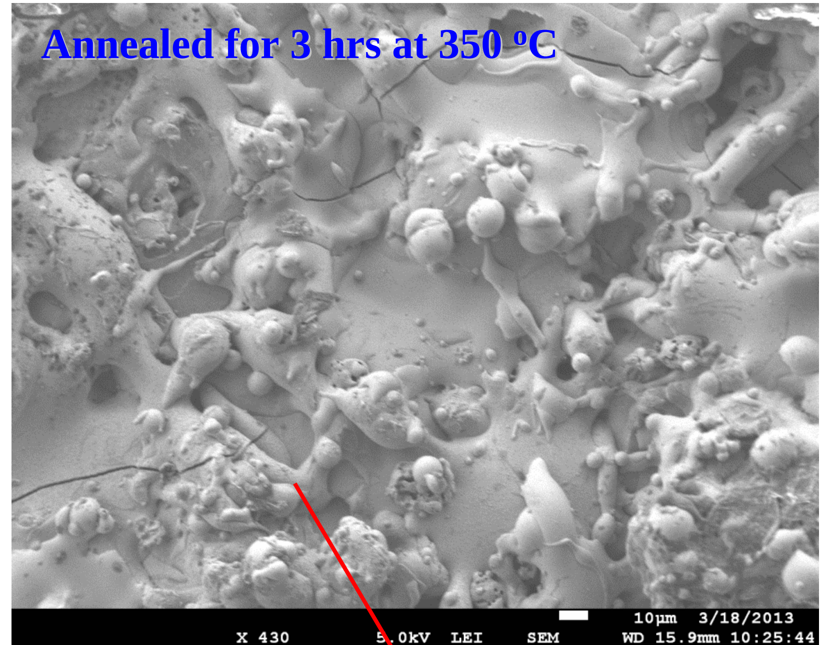
Amorphous Fe nano-structured coating on steel following annealing at 350 ºC



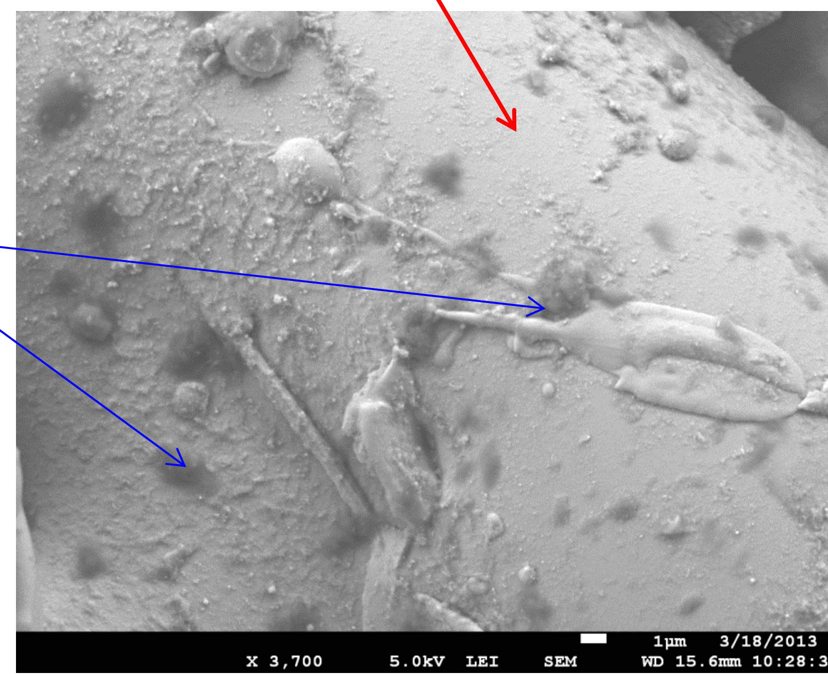
Amorphous Fe nano-structured coating on steel following annealing at 700 ºC





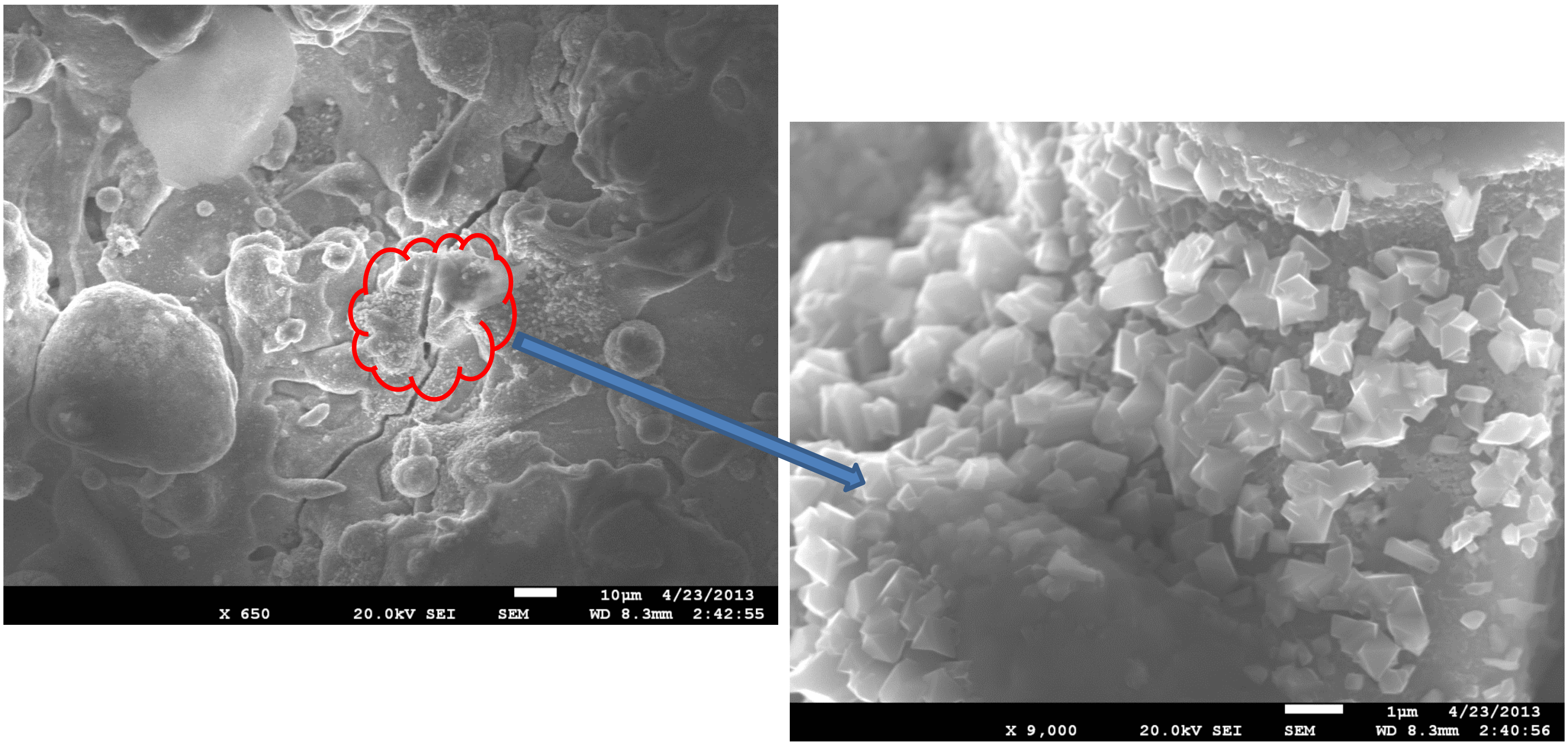


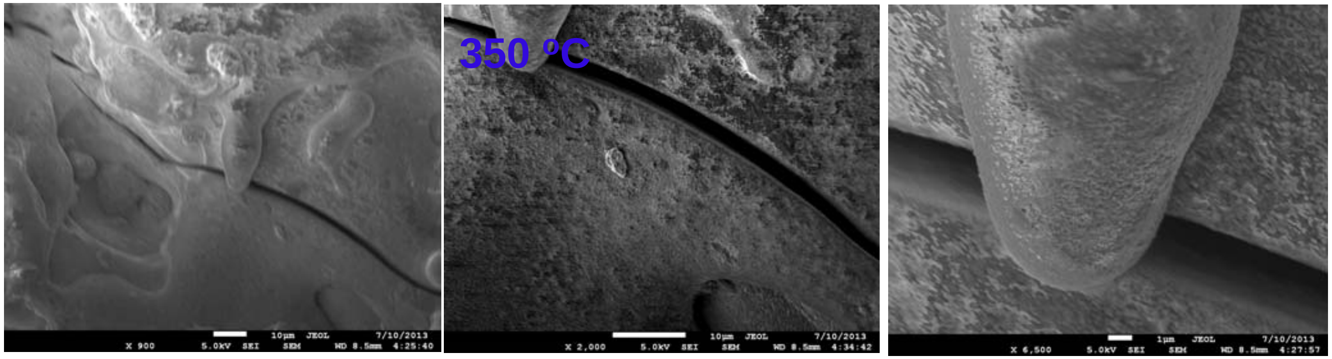
Annealed at 350 ºC –
appearance of features

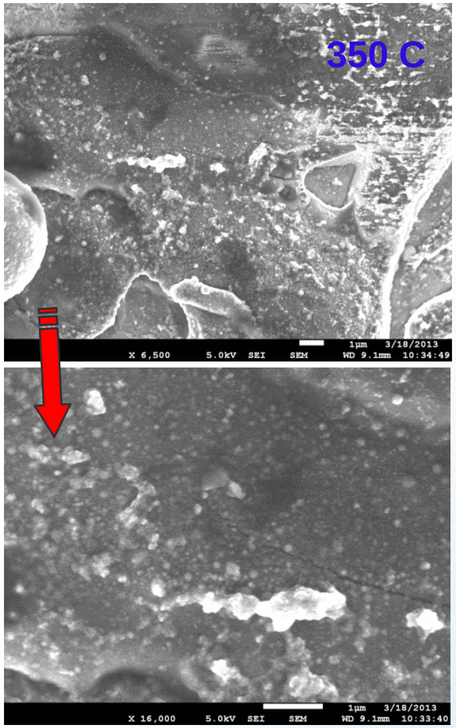


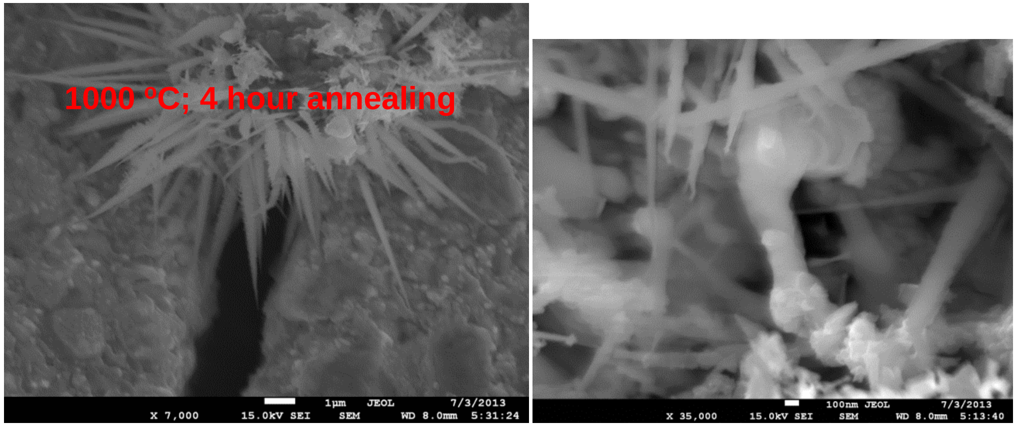
Amorphous-Fe Coating (un-irradiated) following annealing for 4hrs at 700 °C

CLEARLY, 700 °C is above the crystallization temperature threshold as seen in the SEM micrographs

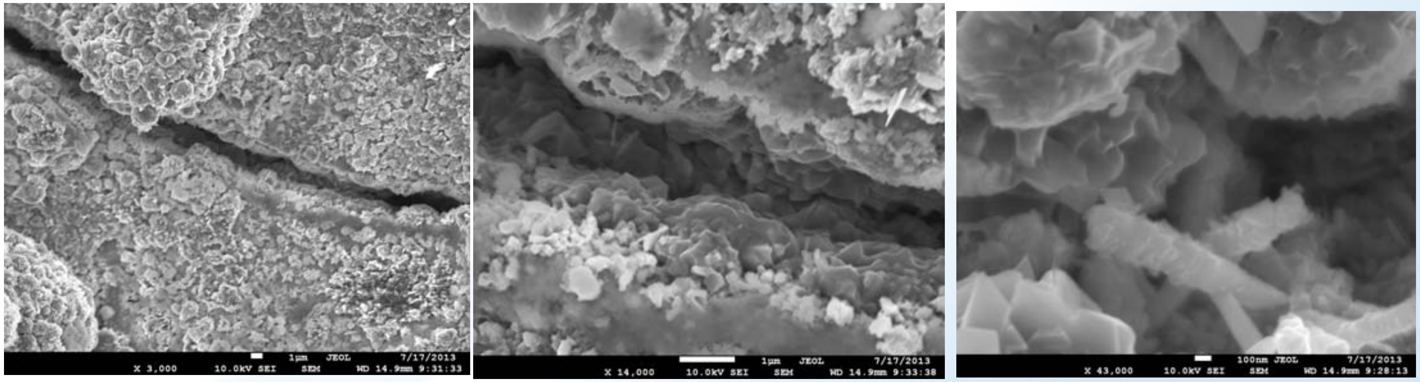






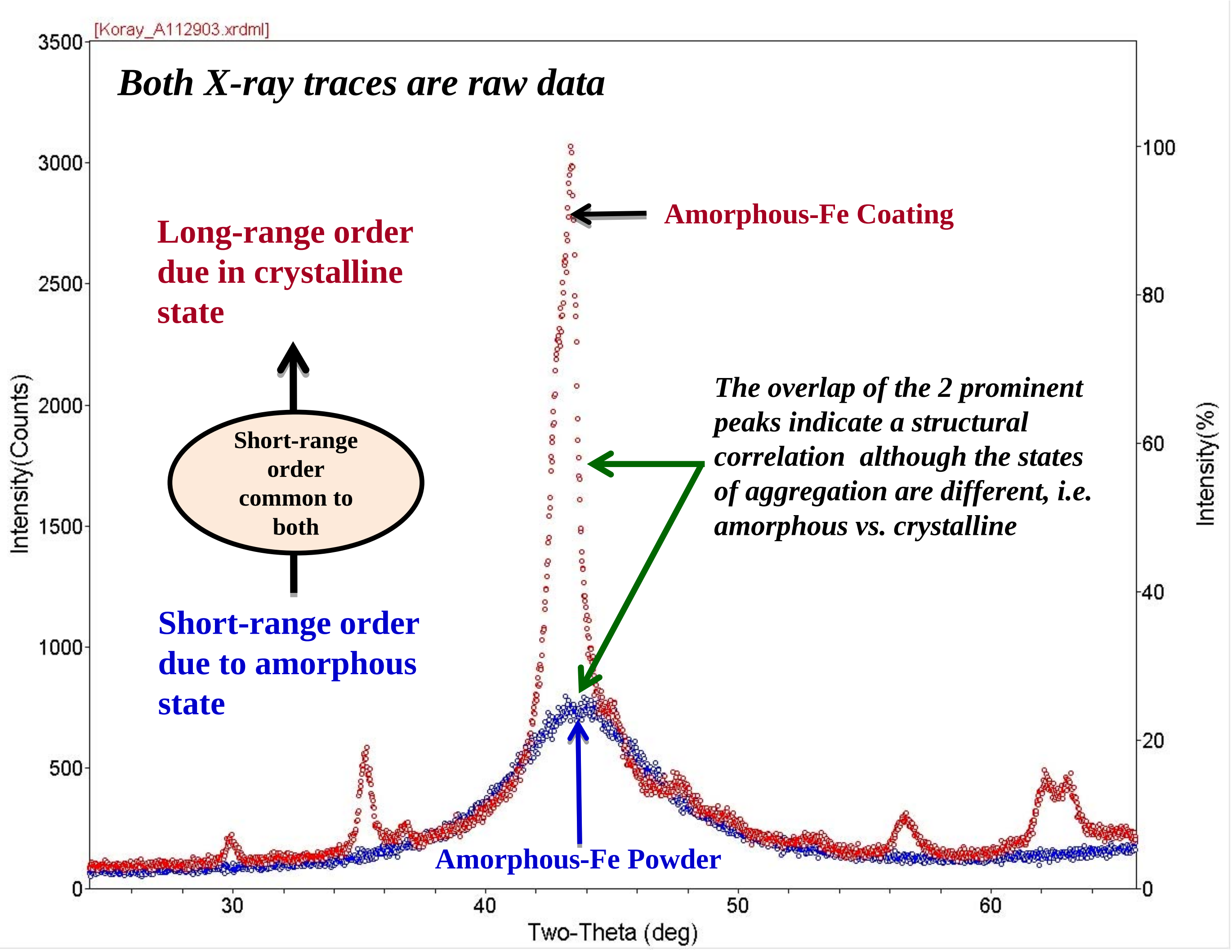


1000 °C; 6 hour annealing



What will neutron irradiation do to the evolution?

Comparison of the Diffraction Patterns of Amorphous Fe Powder & Coating



Al_2O_3 + TiO_2 on Ti-6Al-4V substrate

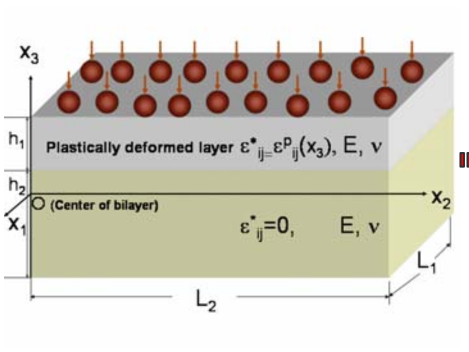
Matrix of coatings/substrates

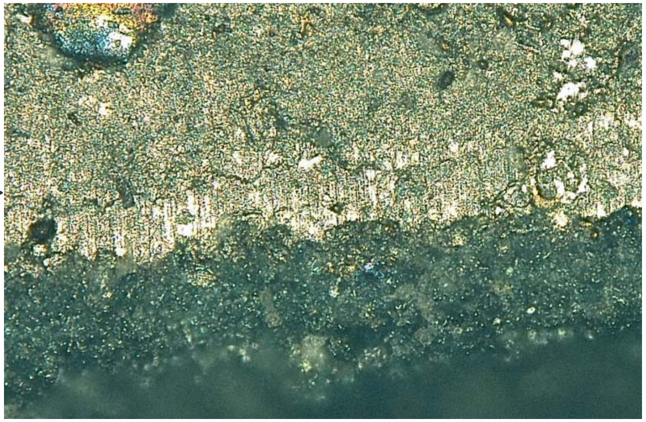
- Amorphous Fe on 4130 steel

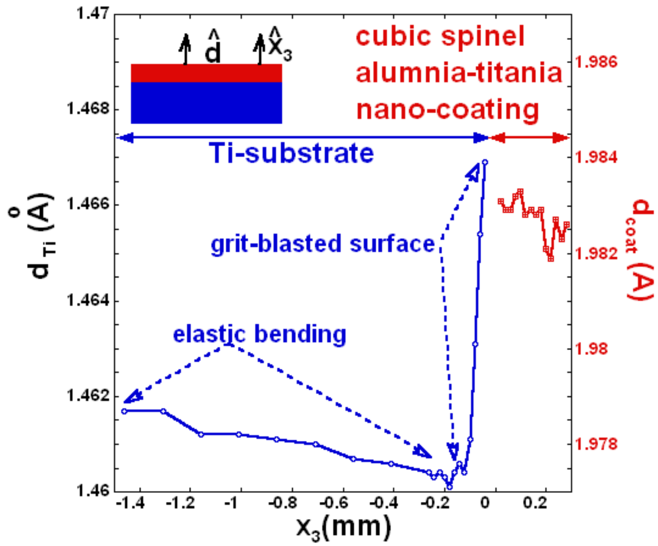
- Micron Alumina+Titania (87/13) on 4140 steel
- Micron Alumina+Titania (87/13) on 1020 steel (2-pass coating, thin)
- Micron Alumina+Titania (87/13) on 1020 steel (over-coating, thick ~500 um)
- Micron Alumina+Titania (87/13) on 1020 steel (typical coating, grit blast + bond coating))

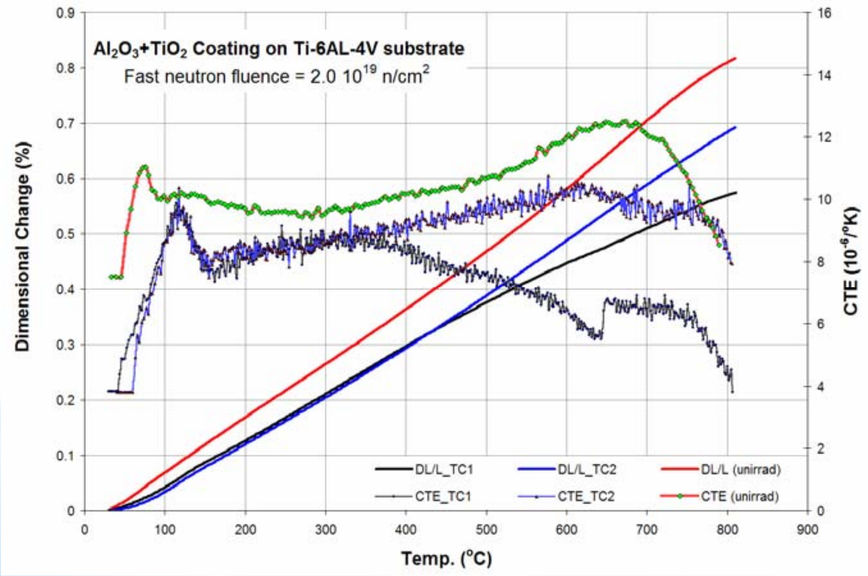
- Nano Alumina/Titania (87/13) on 1020 steel
- Nano Alumina/Titania (87/13) on 1040 steel

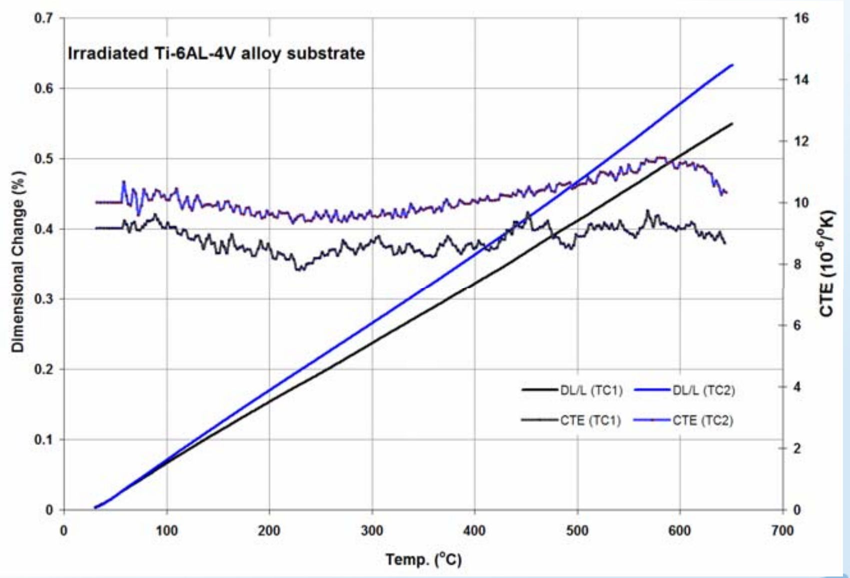
$Al_2O_3 + TiO_2$ on Ti-6Al-4V substrate



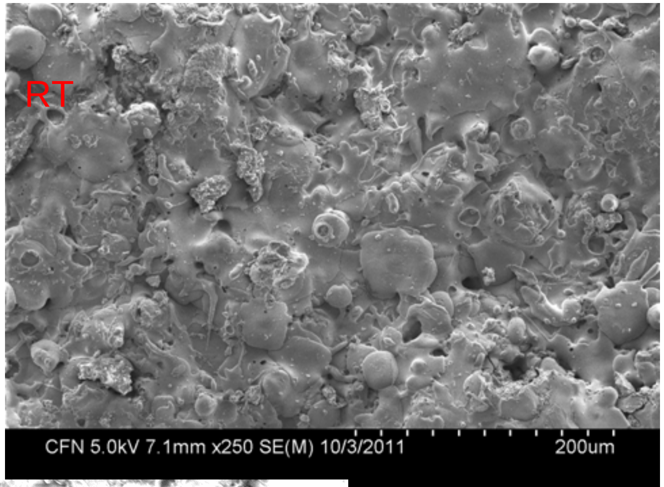


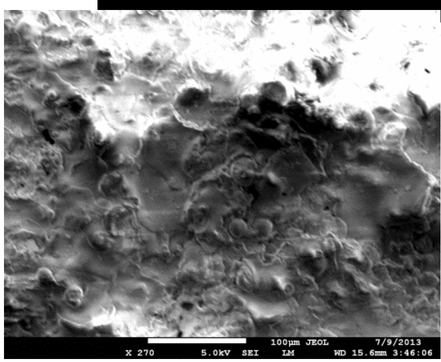


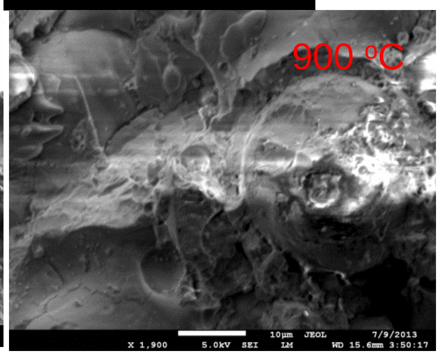


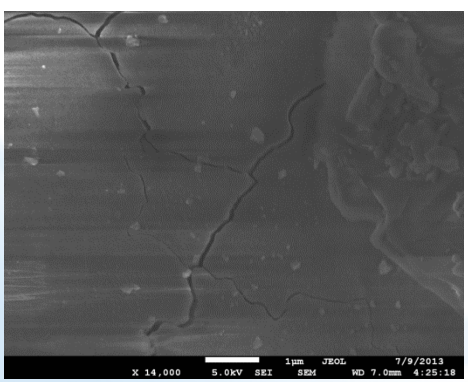


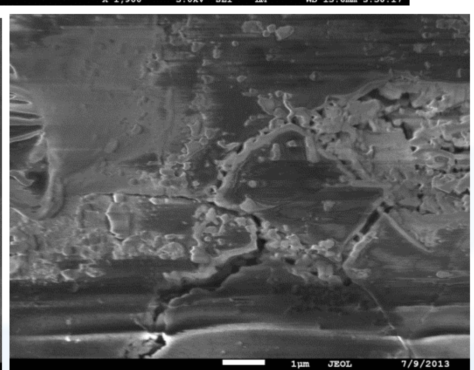
Al_2O_3 + TiO_2 on Ti-6Al-4V substrate

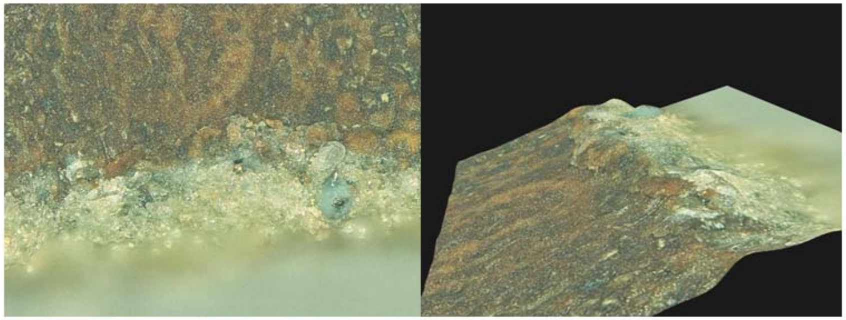




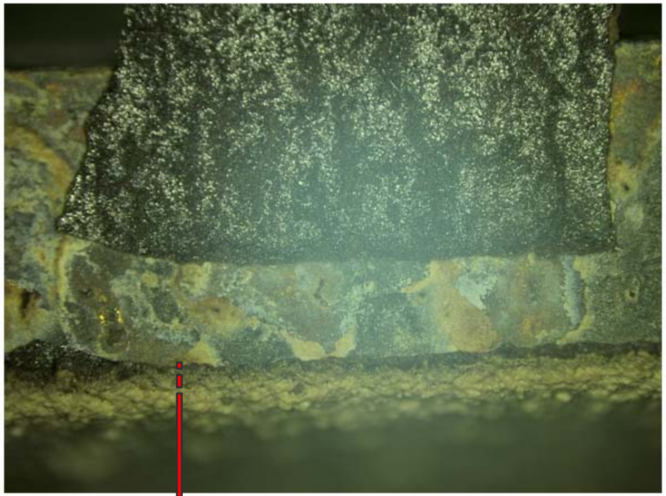




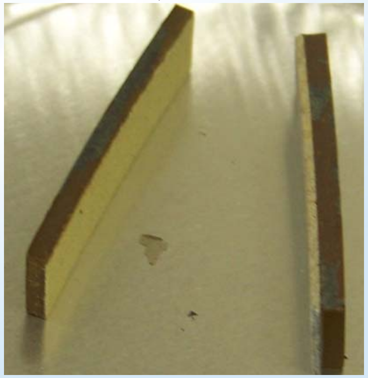




900 °C

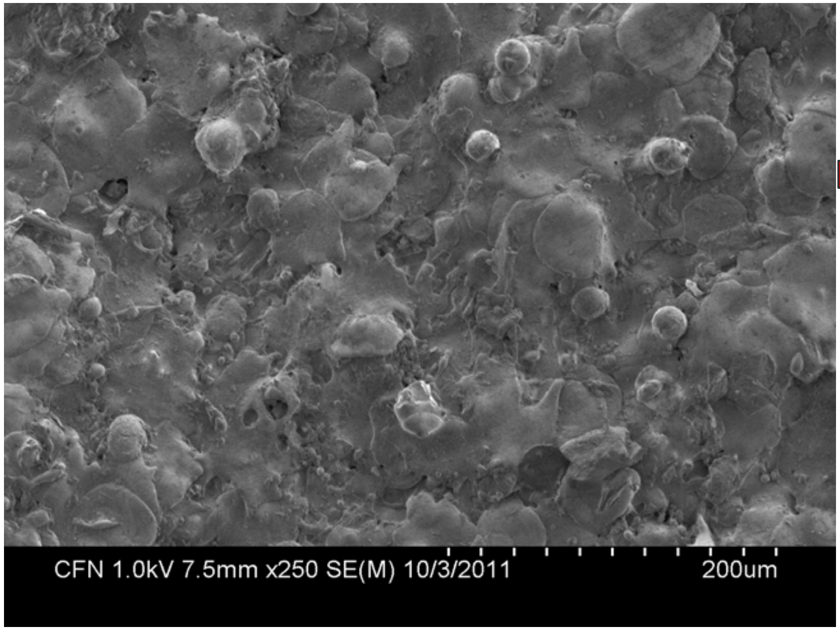


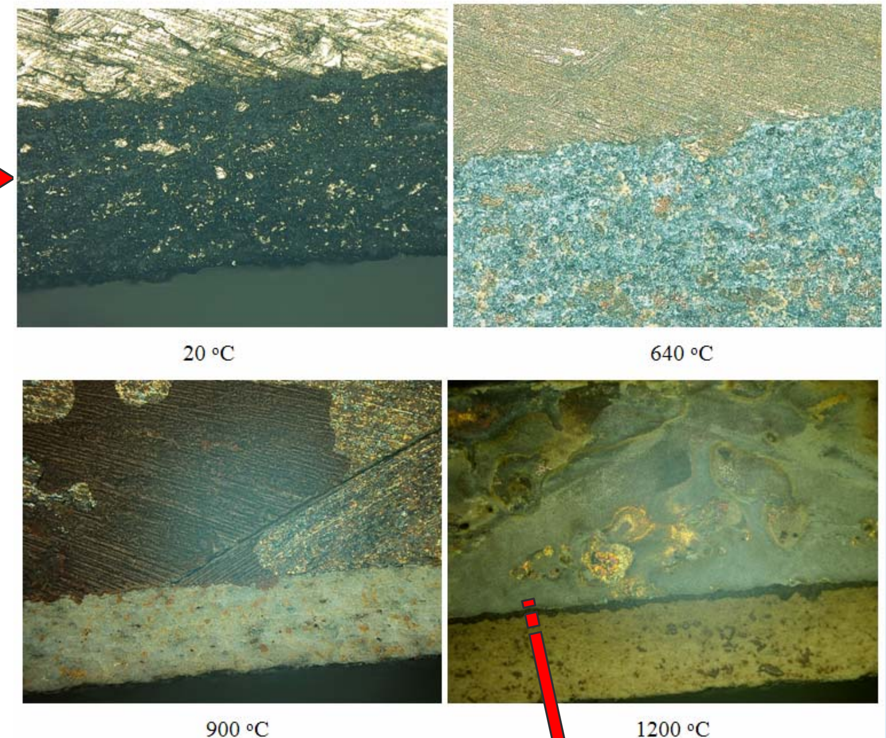
1200 °C

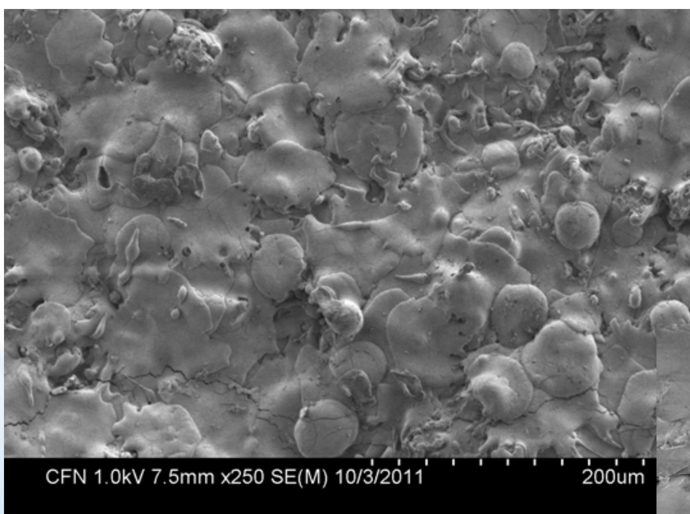


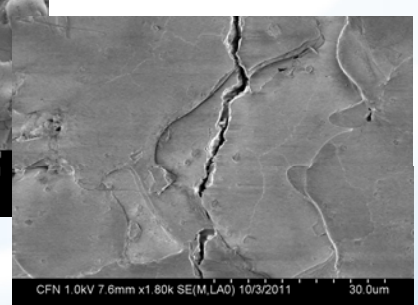
600 µm-thick Al_2O_3 on Ti-6Al-4V substrate

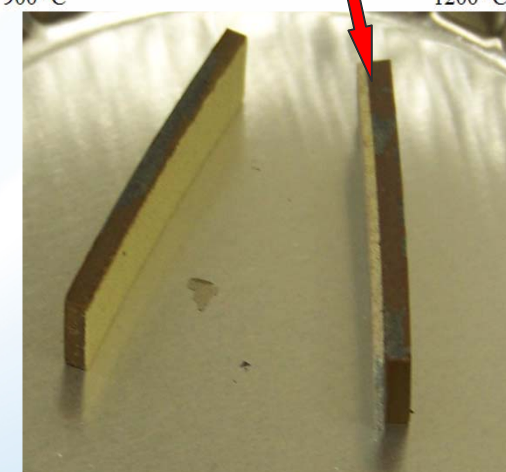
600 µm-thick Al_2O_3 on Ti-6Al-4V substrate



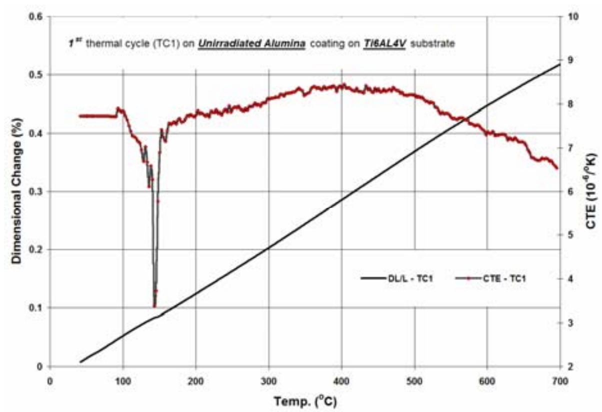


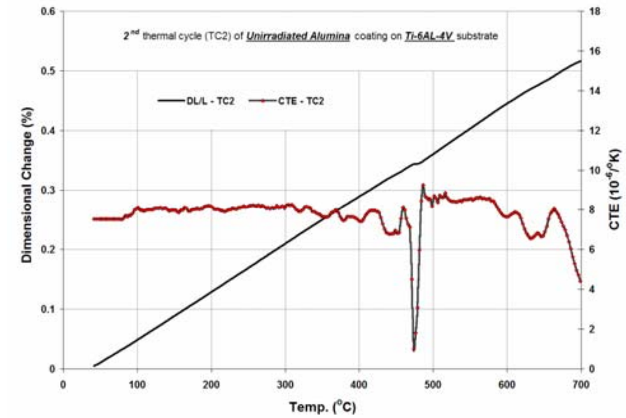


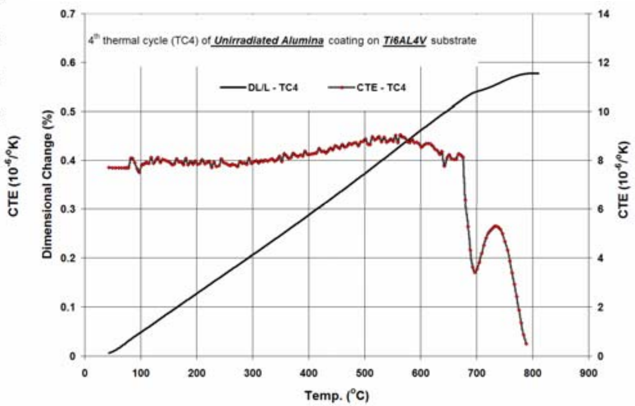


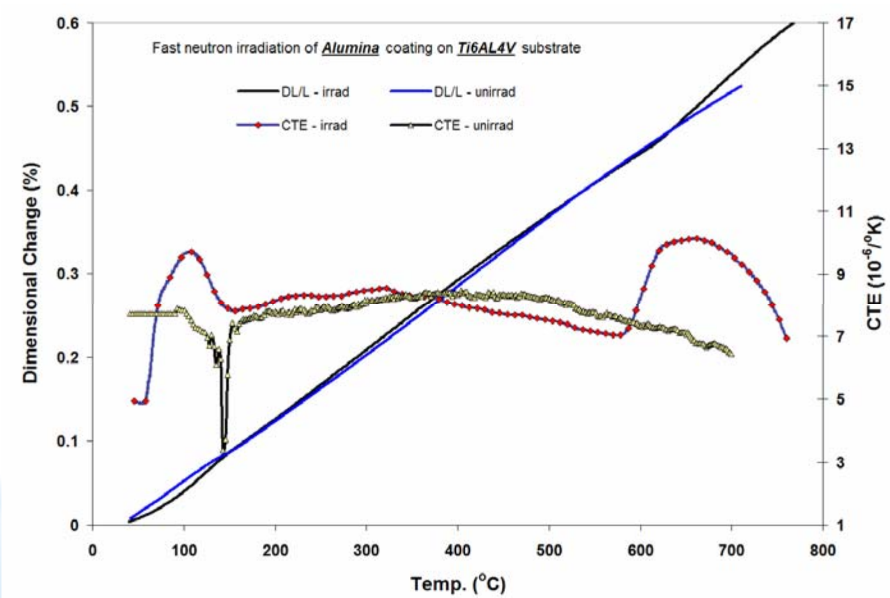


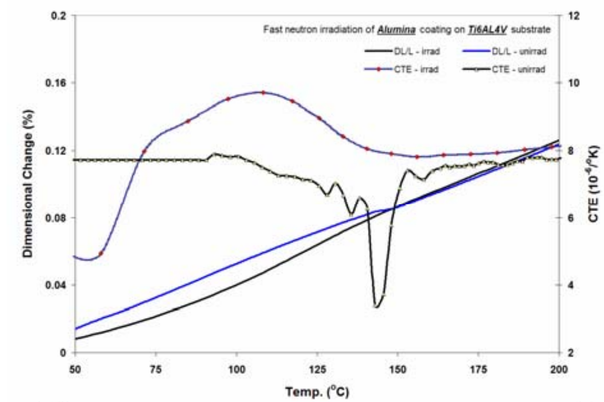
600 µm-thick Al_2O_3 on Ti-6Al-4V substrate

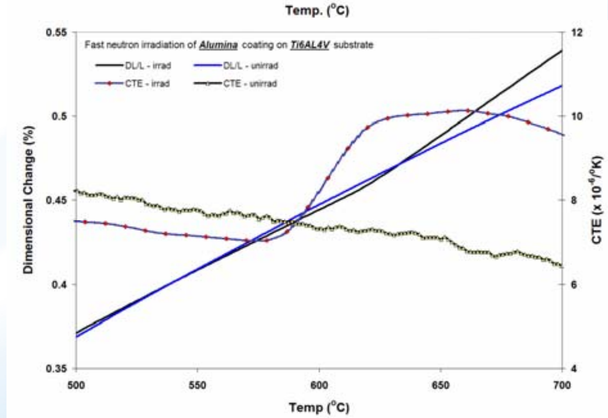




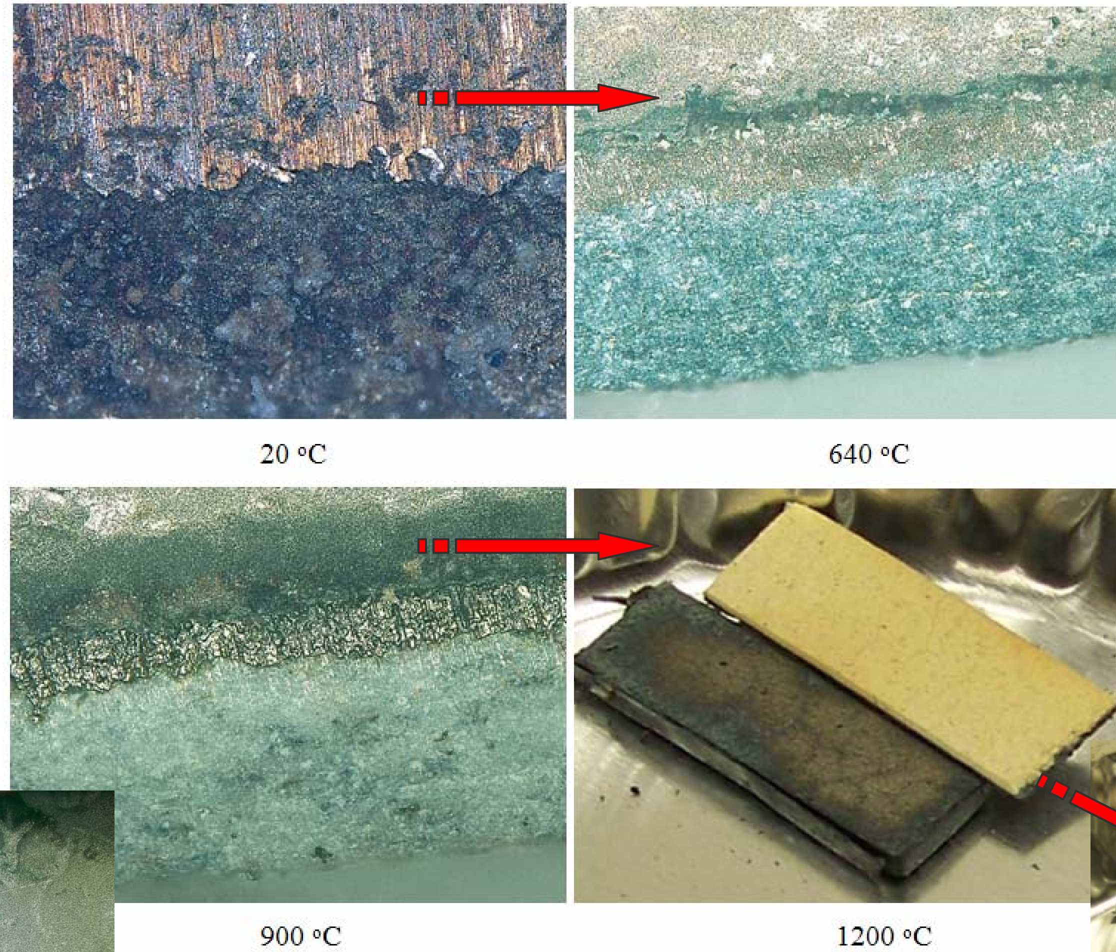


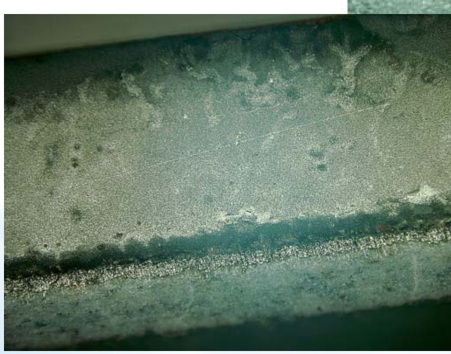






600 um-thick Al_2O_3 on Steel





On-going & Future Work

FY-2013

- Irradiation and annealing effects on ductility (stress-strain and 3-point bending tests) for amorphous Fe and ceramic coatings
- Protective performance of amorphous Fe as a function of temperature/crystallization
- ➔ appearance/disappearance of micro-cracking
- Oxidation performance assessment

FY-2014

- Irradiation and annealing effects on ductility (mechanical testing)
- Continue neutron irradiation and temperature vs. ductility studies
- Post-irradiation phase and strain mapping (x-ray diffraction at NSLS)
- Post irradiation SEM characterization (nanocoating and interfaces)

FY-2015

- Continue irradiation and annealing effects on ductility
- Continue post-irradiation phase and strain mapping (x-ray diffraction)
- Exploration of nanostructure coating nanoparticle, thickness and substrate variation
- Explore other amorphous nanostructures that remain amorphous up to 1100 C
- MD calculations