Positron annihilation lifetime measurement and X-ray analysis on energetic heavy ion beam irradiated polycrystalline tungsten

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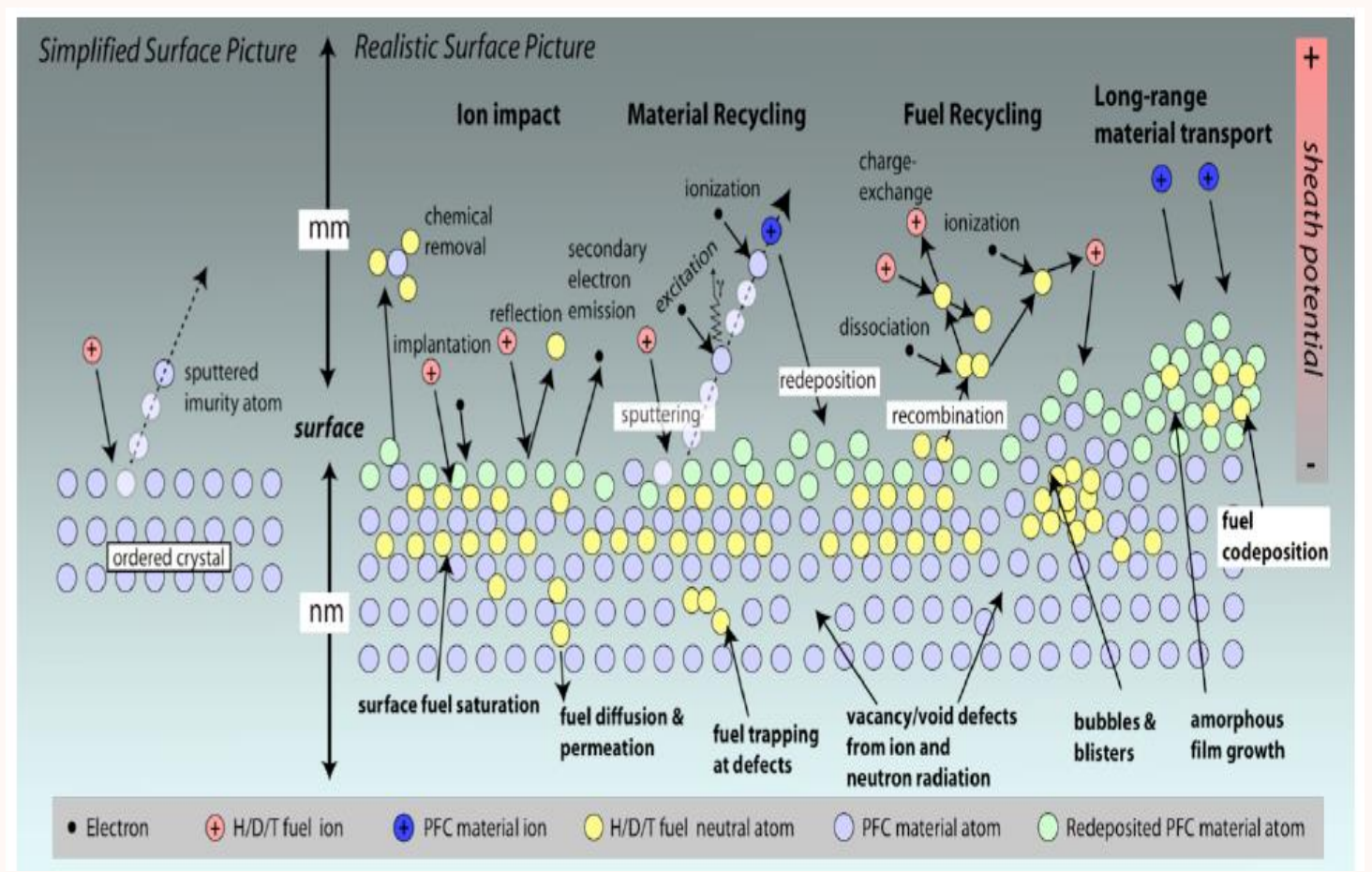
Plasma Wall Interaction



Source: http://web.mit.edu/aeroastro/labs/spl/pmi_overview.html

Motivation for Using Ion Irradiation as a surrogate for Neutron Irradiation

- The plasma facing materials in ITER Divertor will be subjected to: (i) irradiation damage due to fusion neutron of energy 14.1 MeV.
- The interaction of the fusion plasma with the neutron damaged plasma facing material lead to fuel trapping at defects.
- Subsequently, microstructural changes take place, which degrades materials performance.
- It is must to simulate radiation damages in materials for plasma –wall interaction studies.
- In order to simulate radiation damage in plasma facing materials, high energy heavy ion beams has been employed.
- The main advantage of employing heavy ion irradiation method to simulate radiation damage is high dose rates (typically 10^{-3} to 10^{-4} dpa/s) than under neutron irradiation (10^{-7} to 10^{-8} dpa/s). Ion irradiated samples have negligible radioactivity.

Simulation of Neutron Irradiation Effects with Ion Irradiation

- B.I. Khripunov *et al.* at Russian Research Center Kurchatov Institute

Developed an experimental approach aiming at the investigation of the combined effect of neutron irradiation and plasma bombardment on fusion reactor materials facing plasma.
High energy accelerated ion beam (4 MeV, α-particles) was used to produce radiation damage in tungsten (1-10 dpa).
Erosion in deuterium plasma was studied in simulated Tokamak SOL conditions on irradiated materials. Evidences of radiation damage effect on the erosion process have been studied.
J. Surf. Investigation. X-ray, Synchrotron and Neutron Techniques, March 2014, Volume 8, pp 229-233

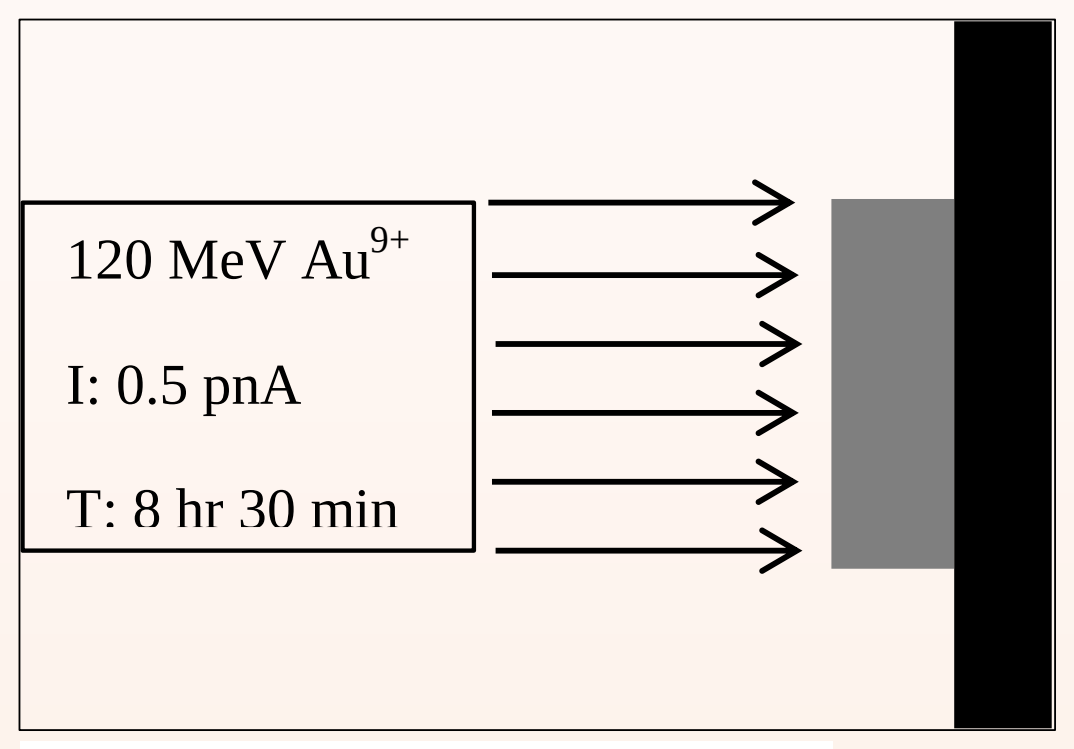
- D E J Armstrong *et al.* at University of Oxford

Ion implantation has been used to simulate neutron damage in W–5wt%Ta alloy. The damage levels of 0.07, 1.2, 13 and 33 dpa. The mechanical properties of the ion-implanted layer were investigated by nanoindentation.
J. Gibson, D. Armstrong, S. Roberts, Phys. Scr. T159 (2014) 014056; C. D. Hardie, C. A. Williams, S. Xu, S. G. Roberts, J. Nucl. Mater. 439 (2013) 33.

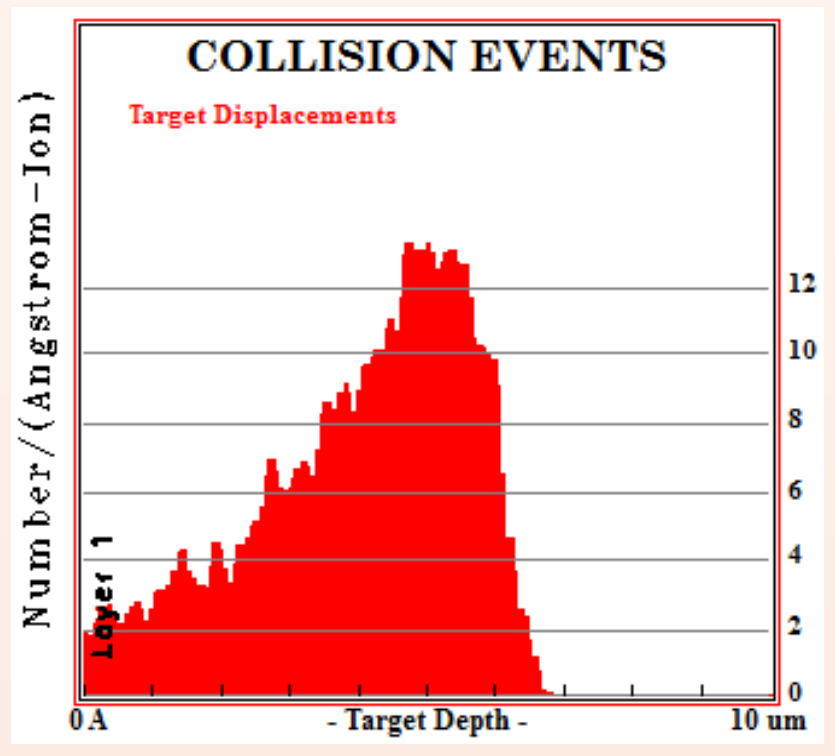
- Kirk *et al.* at Argonne National Laboratory

Attempts have been made to simulate neutron induced damages by employing Kr ions irradiation in Mo and further research is in progress to establish correlation between two methods.
M. A. Kirk, M. Li, P. Baldo, Microscopy and Microanalysis 15 (S2) (2009) 1348.

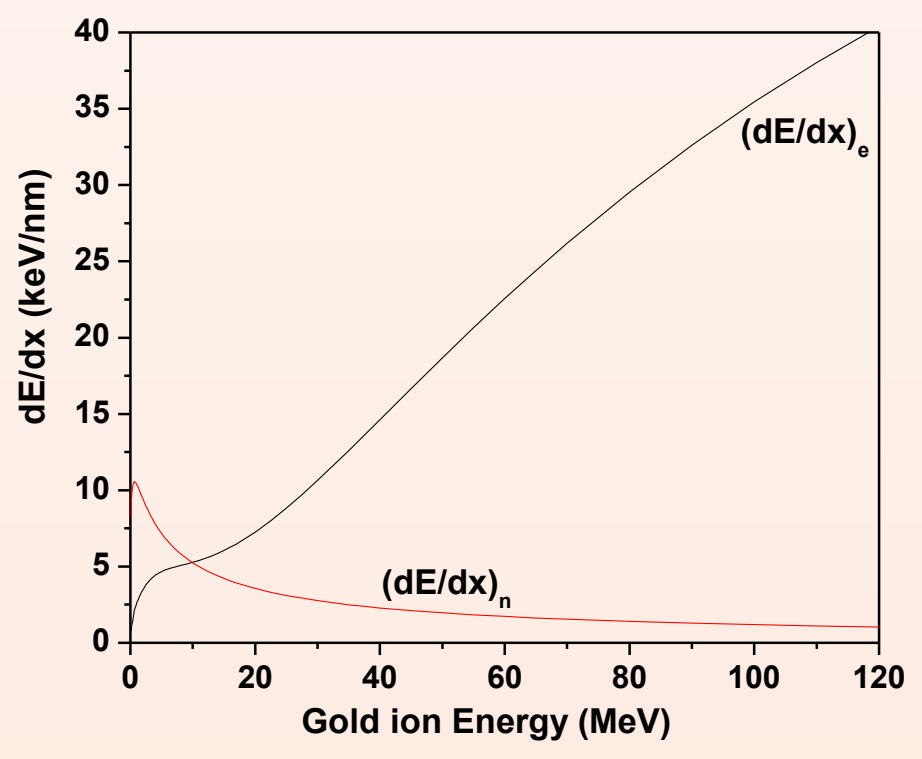
Experimental Details



Target element: Polycrystalline Tungsten (purity 99.97%, procured from Plansee);
Sample size: 8*8*3 mm;
Ion beam: Gold ions;
Charge state: 9^+ ;
Energy: 120 MeV;
Sample temperature: RT
Dose rate: $5*10^{-5}$ dpa/s



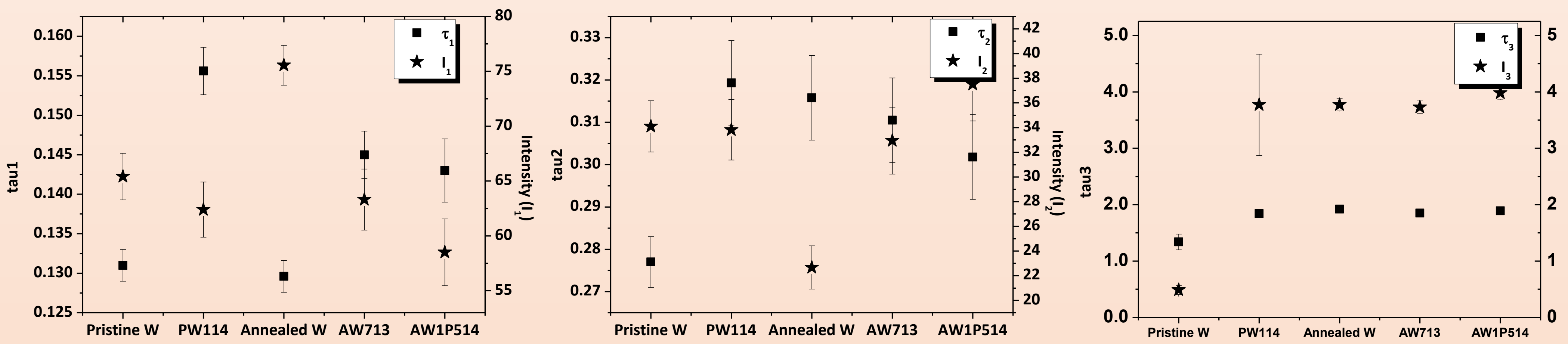
Damage profile in tungsten irradiated with Au ions of energy 120 MeV



Energy loss spectra of gold ions in tungsten obtained from SRIM software

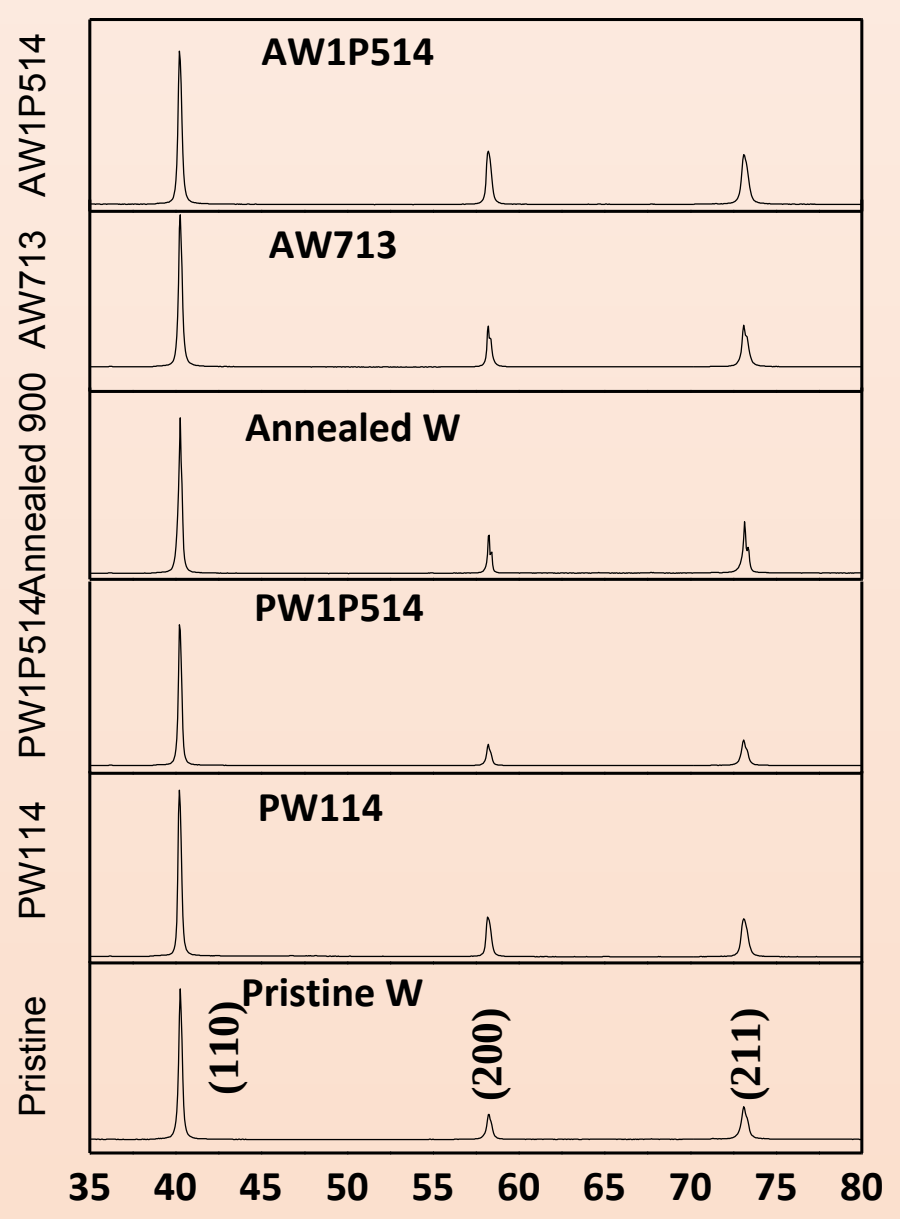
Sample name	Sample State	Fluence (ions/cm²)	Damage (dpa) (Obtained form TRIM simulations)
PW	Pristine W (As received)	0	0
PW114	Do	$1*10^{14}$	0.94
PW1P514	Do	$1.5*10^{14}$	1.42
AW	Annealed W (at 900C)	0	0
AW713	Do	$7*10^{13}$	0.66
AW1P514	Do	$1.5*10^{14}$	1.42

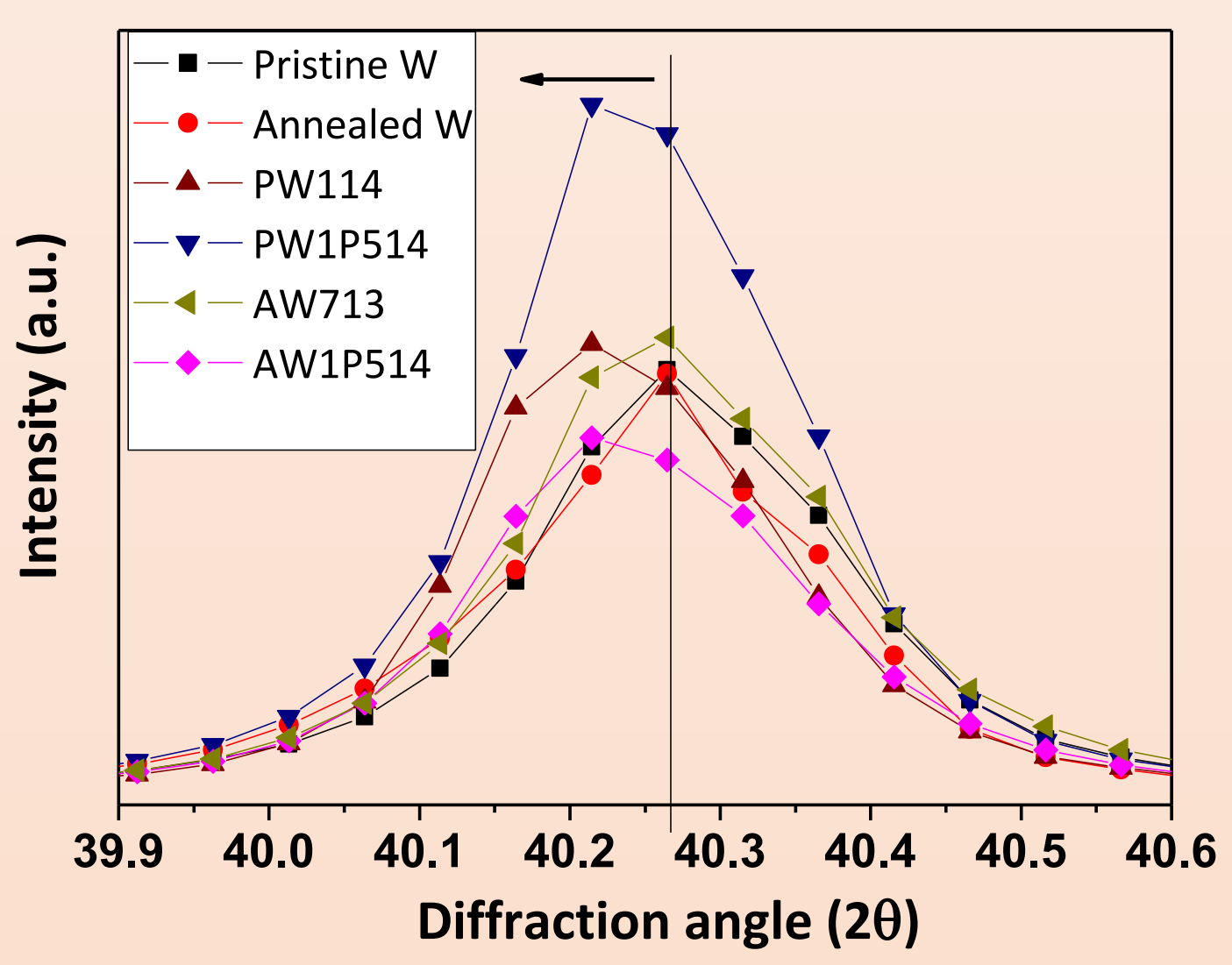
Positron annihilation lifetime measurement

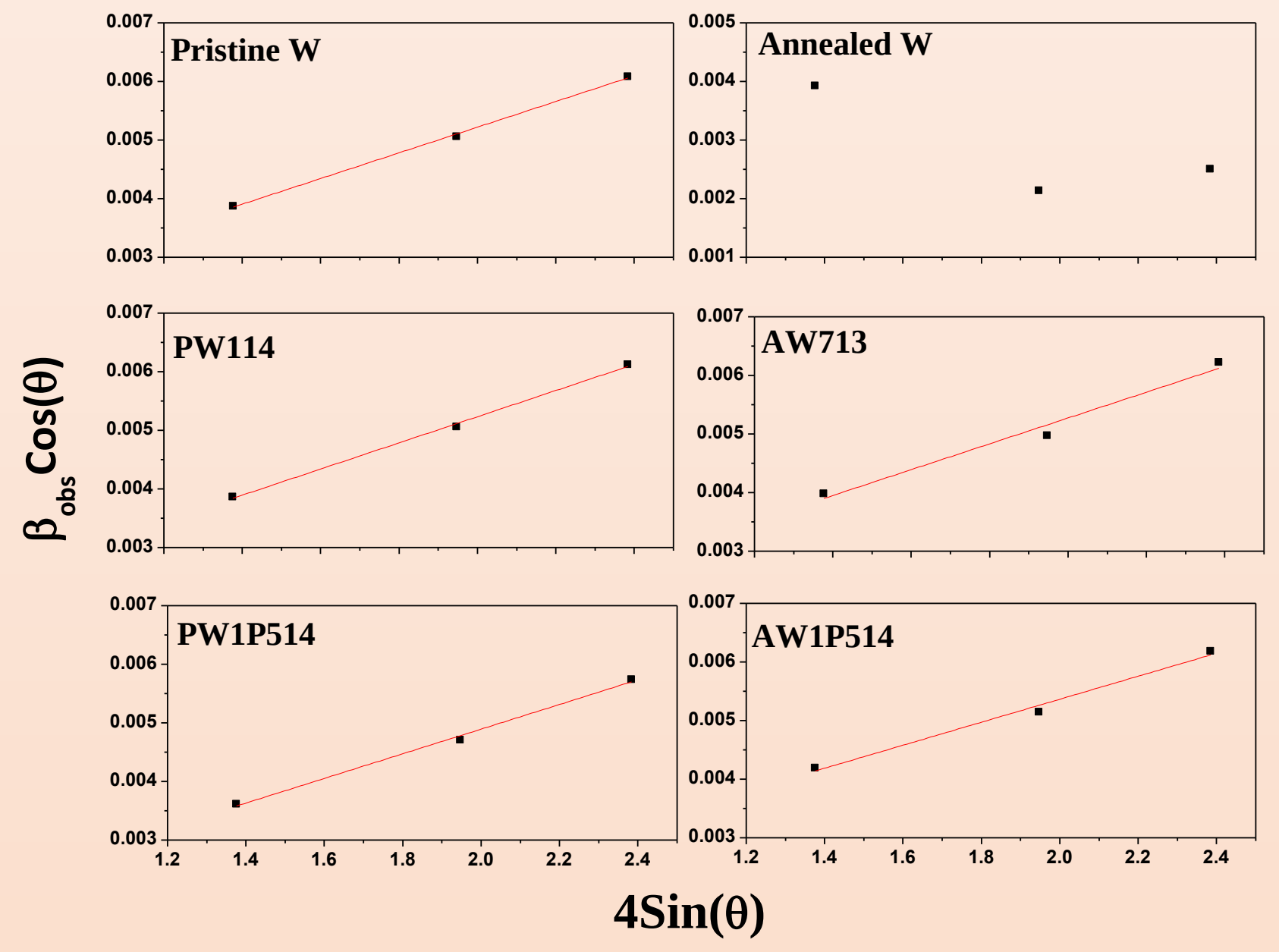


- Positron lifetime in a defect-free single crystal of tungsten is τ_f ~0.105ns and , the estimated positron lifetime in mono and di vacancies in tungsten are $\tau_{1v}=1.3\tau_f$ (~0.136ns) and $\tau_{2v}=1.5\tau_f$ (~0.157ns).
- Three positron lifetime components τ_1 (0.13-0.18ns), τ_2 (~0.28-0.38ns) and τ_3 (~1.9ns) are observed for different set of samples.
- **Pristine W:**
- The positron lifetime component τ_1 and τ_2 shows that pristine tungsten specimen is not defect-free.
- **Annealed W:**
- The intensity (I_1) for annealed tungsten sample is high as compared to that of pristine tungsten. It indicates annealed W has more "pure mono vaccancy" like defects.
- The positron lifetime (τ_2) for annealed W sample is higher as compared to that of pristine tungsten: It suggests migration of mono-vacancies during heat cycle through crystal lattice and forming larger defects.
- The ortho-positronium lifetime around 1.9ns with intensity around 4%, indicates presence of small pores. It confirms migration of larger vacancy like clusters during stage II recovery and forming small pores.
- **Irradiated Pristine W:**
- The increase in τ_1 from 0.13 to 0.15 and τ_2 from 0.27 to 0.31 indicates formation of larger vacancy clusters on heavy ion irradiation. This can be attributed to evolution of new vacancy clusters from damage cascades.
- The presence of τ_3 (corresponding to ortho-positronium lifetime) indicates presence of small pores, which can owe it origin to irradiation induced migration of smaller vacancy clusters and leading to formation of pores.
- **Irradiated Annealed W:**
- The τ_1 value in AW713 and AW1P514 is more than that in annealed W sample. This shows that AW713 and AW1P514 have mono as well as cluster of vacancies, where as un-irradiated annealed W has more "pure mono vacancy" like defects.
- The τ_2 components in AW713 and AW1P514, again show identical results. τ_2 generally corresponds larger size defects. In un-irradiated annealed W samples, intensity of τ_2 is less, which corresponds less number of larger size defects in un-irradiated annealed W sample.
- In all samples, except pristine W, the presence of third component tau3 indicates that all these samples have small pores

X-ray analysis







Sample Name	XRD peak position (2θ) in degree for (110) peak	FEHM of (110) XRD peak	Lattice strain ($\times10^{-3}$)	Crystallite Size (nm)
Pristine W (PW)	40.28	0.2368	2.19	164
PW114	40.24	0.2360	2.20	159
PW1P514	40.25	0.2208	2.10	200
Annealed W (AW)	40.25	0.2400	-	
AW713	40.27	0.2432	2.20	180
AW1P514	40.25	0.2560	1.96	96

- There is no evidence of major structural phase transformation in irradiated tungsten specimen. However, XRD of PW1P514 sample reveals improved crystallinity and on other hand XRD of AW1P514 sample indicates loss of crystallinity.
- **Pristine W before and after irradiation:**
- The XRD peak position for pristine W is observed at higher 2Θ value as compared to annealed W sample. It suggests that compressive strain is present in pristine W. In irradiated pristine W samples, the XRD peaks shift towards lower 2Θ value indicates that on irradiation, nature of strain is changing form compressive to tensile.
- The decrease in FWHM of (110) peak on irradiation for PW1P514 sample, indicates that non-uniform strain has decreased for irradiated pristine W samples at high ion dose. In addition grain growth is observed in case of PW1P514 sample. The localized damage caused by the energetic heavy ion irradiation in the vicinity of grain boundary is the likely driving force for grain growth. Decrease in internal lattice strain in case of PW1P514 sample, can be attributed to stress relaxation during grain growth.
- **Annealed W before and after irradiation:**
- Williamson-Hall plot shows that on annealing of as received W samples at 900C, lattice strain has got relaxed.
- Induction of lattice strain on heavy ion irradiation is observed. This can be attributed to generation of defects/dislocation loops etc. inside polycrystalline annealed tungsten samples.
- Grain refinement is observed AW1P514: which may owe its origin in formation of nanostructure on normal ion incidence as observed in other metallic systems.

Conclusions and future scope

- Positron annihilation lifetime estimation confirms generation of vacancy clusters inside polycrystalline tungsten.
- Irradiation induced migration of defects is observed, which has led to development of small pore under heavy ion irradiation.
- X-ray diffraction results suggest that there is no major structural phase transformation in irradiated tungsten specimen.
- Induction of lattice strain on heavy ion irradiation is observed. This can be attributed to generation of defects/dislocation loops etc. inside polycrystalline annealed tungsten samples.
- It appears that internal micro-stresses have played decisive role in defining microstructure of ion beam irradiated samples.
- It is worth to mention that defects have successfully been created within few hours, which is possible in days only, by using neutron irradiation method.
- The ion beam damaged W samples can further be utilized for plasma wall interaction studies.

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