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Depth-Localized Ion Irradiation-Induced Compressive Stress Relaxation in W Thin Film Revealed by Cross-Sectional X-ray Nanodiffraction

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Abstract

The influence of ion irradiation on residual stress and microstructure of thin films is not fully understood. Here, 5 MeV Si^{2+} ions were used to irradiate a 7 µm thick tungsten film prepared by magnetron sputtering. Cross-sectional X-ray nanodiffraction and electron microscopy analyses revealed a depth-localized relaxation of in-plane compressive residual stresses from −2.5 to −0.75 GPa after the irradiation, which is correlated with the calculated *displacements per atom* within a ~2 µm thick film region. The relaxation can be explained by the irradiation-induced removal of point defects from the crystal lattice, resulting in a reduction of strains of the 3rd order, manifested by a decrease of X-ray diffraction peak broadening, an increase of peak intensities and a decrease of lattice parameter. The results indicate that ion irradiation can be used to perform residual stress design in thin films at particular depths.

Keywords: tungsten thin film, ion irradiation, residual stress, microstructure, synchrotron X-ray diffraction

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1. Introduction

An understanding of the influence of post-deposition ion irradiation on the microstructure, stress state and physical properties of thin films is important for their further technological application in microelectronics, space components and fusion reactors [1][2][3]. Employing various ions types and fluences, ion irradiation has been extensively used to modify especially the films' microstructure, residual stresses, biocompatibility and electronic properties. One of the motivation was to relax and/or to design residual stresses in as-deposited films and to understand the microstructural changes caused by ions [4][5].

Tungsten and tungsten thin films have been used in microelectronic and space components, optical and sensor applications due to their particular physical properties like high melting point of ~3700 K, good thermal conductivity, high solar wind absorption and diffusion barrier characteristics [2][4][5][6]. The films prepared by physical and chemical vapor deposition techniques (PVD, CVD) usually exhibit *tensile* or *compressive* residual stresses, which may enhance the functional properties of the components or result in degradation phenomena like cracking and loss of adhesion. In order to perform post-deposition stress engineering in tungsten and also other types of thin films, ion irradiation has been used to modify (mainly tensile) stresses [4][7][8]. For instance, Snoeks *et al*. [4] used B and P ions to relax *tensile* stresses of 260 MPa in 0.34 µm thick W films and observed an exponential decrease as a function of the applied fluence, accompanied also by the formation of minor structural features like defects loops and small crystalline inclusions, revealed by transmission election microscopy (TEM). During a continuous irradiation with He, Ne, Ar and Kr ions, Pranevicius *et al*. [5] observed the formation of *compressive* stresses in growing W/Cu multilayers, followed by a time-dependent stress relaxation after the deposition had finished. The relaxation was interpreted as a structural rearrangement in the multilayer, caused by mobile structural defects resulting in the formation of a new microstructure with a lower energy [5]. Similarly, Fu *et al.* [7] used He ions to reduce *tensile* stresses in Cu thin films from 757 to 452 MPa, which was interpreted as an incorporation of excess atoms at the Cu grain boundaries. It should be noted, however, that in the majority of the previous ion irradiation experiments on thin films, changes in volume-averaged stresses were evaluated primarily using X-ray diffraction (XRD) and/or wafer curvature techniques and therefore, no detailed information on depth localization of the stress changes could be provided. The only exception to this is the recent work of Du *et al*. [9], which showed a correlation between the concentration of implanted He atoms and *tensile* strain formation in an epitaxial 285 nm thick Nb film on MgO substrate.

Because of its unique physical properties, tungsten is considered also as a very promising plasma-facing material in divertors of fusion reactors [10][11]. During service, high heat and radiation fluxes of H isotopes, He ions and/or neutrons are expected to impact the W surface and to induce severe microstructural and stress changes, resulting in materials porosity and/or embrittlement, which may be a critical factor for the fusion plant's service life [11][12]. In the majority of previous studies, He-ion and self-implantation were used to simulate radiation damage in W and W alloys. Hofmann *et al.* [13] used μ-Laue diffraction and *ab initio* calculations to analyze lattice swelling and reported out-of-plane lattice *tensile strain* of ~1.5 % down to a depth of ~4 μm in a W-1 at.%-Re alloy. In the most recent study, Phillips *et al.* [14] used self-implantation to mimic neutron irradiation damage in W and analyzed lattice swelling using multi-reflection Bragg coherent diffractive imaging (MBCDI) and μ-Laue diffraction. The swelling was interpreted as out-of-plane *tensile* strain of ~1 % reaching a depth of ~1 μm in a tungsten cylinder with a diameter of ~1 μm.

Most of the previous experimental studies on thin films (and bulk materials) indicated that ion irradiation induces complex micro- and nano-scopic changes to microstructure and to residual stresses. The previous studies on residua stress gradients were performed however on monocrystalline Nb thin film using TEM [9] and single-crystalline tungsten grains using XRD [13][14]. Currently, however, it is still not known how ion irradiation modifies *locally* already existing stress gradients, which were present in *polycrystalline thin films* before the irradiation experiment. Therefore, the aim of this experimental work is to investigate changes to a pre-existing gradient of *compressive stress and microstructure* in a nanocrystalline tungsten thin film after the irradiation with silicon ions. Tungsten was chosen for this study because of its relevance to microelectronics and also in fusion reactors.

2. Experiment

In this work, a tungsten film with a columnar-grained morphology was grown on a WC substrate by direct current pulsed magnetron sputtering using two sputter sources with 3 inch W targets operated at 400 W. Film growth was carried out in an argon atmosphere with a total pressure of 0.5 Pa and at a deposition temperature of 150 °C. In order to reach a thickness of ~7 µm, the deposition time was set to ~190 min. The morphology and density of the material was tuned by a power-controlled RF bias of 20 W, in order to reach high compressive stress in the film.

The as-deposited film was irradiated at the Tandetron Laboratory of the Nuclear Physics Institute of the Czech Academy of Sciences employing a 3 MV Tandetron MC 4130 accelerator [15] to induce radiation damage using Si^{2+} ions with an energy of 5 MeV at room temperature. A fluence of $2\text{x}10^{16}$ ions/cm^2 was achieved, with a beam current density of 168-279 nA/cm^2 on the target and an irradiation time of 8 hours. The anticipated radiation damage was predicted using the software package *The Stopping and Range of Ions in Matter* (SRIM) [16]. Using the approach of Kinchin and Pease [17], this software is capable to calculate the average number of displacements per atom (DPA) inside the material. In this particular case, a simulation was performed for 5000 Si^{2+} ions of 5 MeV energy to assess the damage and penetration depth. The maximal DPA of ~6 was calculated at a depth of ~1.1 µm below the surface, as indicated in Fig. 1.

Cross-sectional lamellae with a thickness of ~50 µm were prepared from both the as-deposited and the irradiated samples. The lamellae were investigated using cross-sectional X-ray nanodiffraction (CSnanoXRD) [18]. The as-deposited sample was characterized at the Nanofocus Endstation of P03 beamline of the PETRA III synchrotron source in Hamburg (D), using a photon energy of 15.0 keV and an X-ray beam cross-section of 0.5 x 2.0 µm^2. The implanted sample was investigated at the ID13 beamline of the ESRF synchrotron source in Grenoble (F) using a photon energy of 15.2 keV and a beam size of 0.1 x 2.0 µm^2. The experimental setup is presented in Fig. 2. Both films were scanned along their cross-sections using the X-ray beams and two-dimensional detector images capturing the W 110 and 200 Debye-Scherrer rings were collected at each scanned position. The W 110 rings were used to evaluate the intensities and full widths at half maxima (FWHM), as well as the residual stresses as a function of film thickness, using the methodology presented in our previous works [18][19]. For the stress evaluation, X-ray elastic constant (XEC) for W of $S_1^{\mathrm{W},110} = -7.29 \times 10^{-4}\mathrm{GPa}^{-1}$ and $\frac{1}{2}S_2^{\mathrm{W},110} = 3.302 \times 10^{-3}\mathrm{GPa}^{-1}$ were used. Bright-field scanning TEM analysis was performed using a JEOL 2200 FS system operated at 200 kV. The spot size was set to 0.7 nm. TEM lamella was carefully prepared using a FEI Helios NanoLab 660 focused ion beam (FIB) microscope and a standard lift-out technique applying accelerating voltages of 30 to 2 kV and currents of 30 nA to 50 pA.

3. Results

In Fig. 3, a TEM micrograph showing the microstructure of the near-surface region of the irradiated tungsten film's cross-section is presented. The TEM analysis does not indicate any significant microstructural changes within the sample, like the presence of precipitates or bubbles [1][10]. These were also not observed in high-resolution TEM mode. The columnar-grained microstructure was preserved within the film after the irradiation and no changes in crystallites diameter or length have been observed. This can be explained by the relatively small dose and the particular type of implanted ions, which were apparently not sufficient to induce any more remarkable microstructural changes.

In Figs. 4a and b, results from CSnanoXRD show the depth evolutions of the W 110 reflection across the as-deposited and irradiated films, respectively. In the case of the as-deposited film (Fig. 4a), the position of the reflection does not change significantly as a function of film thickness, whereas in the case of the implanted film, one can observe an abrupt change in the reflection's position at a depth of ~2 μm below the surface, as indicated by the arrow (Fig. 4b). Additionally, the intensity of the W 110 reflection in the film's surface region (Fig. 4b) is also significantly increased. Please note that the difference in average Bragg's angles of the W 110 reflections in Figs. 4a and b is caused by the differing photon energies used at the PETRA and the ESRF beamlines, which were used to characterize the as-deposited and irradiated samples, respectively.

The elliptical distortion of Debye-Scherer rings was used to evaluate in-plane X-ray elastic strains and stresses using the XECs of tungsten. Furthermore, the strain-free direction is also given by the XECs and thus lattice parameter changes could be measured [18]. In Fig. 5, in-plane and out-of-plane X-ray elastin strain depth-dependencies obtained from W 110 reflection indicate a respective relaxation of compressive and tensile strains down to a film depth of ~2 μm. Similarly, the (unstressed) lattice parameter of the irradiated film decreased in this region (Fig. 4d), in agreement with the W 110 peak shift visible in Fig. 4b. The magnitudes of the observed strains (Fig. 5) are in the same range as the results in Ref. [13].

In the next step, the X-ray elastic strains from both films were used to evaluate *in-plane residual stress gradients*, which are presented in Fig. 4c. Across the entire thickness of the as-deposited film and at depths of $2-7$ μm within the irradiated film, there are relatively constant compressive stresses present, at a level of approx. -2.5 GPa. Down to a depth of ~ 2 μm, however, compressive stresses in the irradiated sample relaxed to a relatively constant value of about -0.75 GPa. This stress relaxation is accompanied by a decrease in FWHM and an increased intensity of the W 110 reflection at the same depths (cf. Fig. 2d). The diffraction peak intensity increase close to the film's surface within the irradiated sample is presented also in Suppl. Fig. 2, along with the intensity depth-profile of the as-deposited sample, documenting the significant intensity increase in the irradiation-affected film region.

4. Discussion

The comparison of the DPA data from Fig. 1 and the CSnanoXRD data from Fig. 4 allows obtaining the correlation between the applied irradiation conditions on one side and localized residual stress as well as microstructure changes on the other side. The correlation between the observed *in-plane compressive stress relaxation* (Fig. 4c) and the *SRIM DPA profile* (Fig. 1) down to a depth of ~ 2 μm within the irradiated tungsten film is presented in Fig. 3. The results document that the irradiation by 5 MeV Si^{2+} ions caused a relaxation of in-plane compressive strains and stresses within the irradiated region subjected to a relevant level of DPA. Since the TEM micrograph in Fig. 3 does not show any significant irradiation-induced morphological changes, the decrease of lattice parameter, the decrease of FWHM and the increase of the diffraction intensity (*cf*. Fig. 4d) can be interpreted as irradiation-induced changes in the atomistic structure of the sputter-deposited tungsten. It is commonly accepted that the thermodynamically non-equilibrium magnetron sputtering process is accompanied by the formation of point defects in growing films, such as incorporation of self-interstitials and vacancies [20][21]. These defects can be removed from the crystal structure by thermal treatment, as indicated in our previous studies [22], which is always also accompanied by a decrease of compressive intrinsic stresses, XRD peak FWHM and lattice parameter as well as an increase in XRD peak intensity. It appears also in the present case that similar *atomistic structure recovery processes* were induced in the sputtered tungsten film during the irradiation with Si^{2+} ions. It seems plausible that the kinetic energy of the ions, transferred to the film during the irradiation, resulted in the activation of point defects which were thus removed from the crystal structure by diffusion-controlled mechanisms. In this way strains of 3rd order were consequently also removed from the tungsten crystal lattice, as indirectly indicated by the observed FWHM decrease and intensity increase of the XRD peaks.

The relaxation of overall compressive stresses was reported already in the work by Pranevicius *et al.* [5] , where He, Ne, Ar and Kr ions were used to irradiate a W/Cu multilayer. The present results indicate that this relaxation is a localized effect and can be correlated with the energy, irradiation time and the type of the ions resulting in a specific DPA depth-profile (Fig. 1). In contrast to the results from MBCDI and μ-Laue diffraction on almost defect-free tungsten *single crystals* by Hofmann *et al*. [13] and Phillips *et al*. [12], the present data were measured in originally very defect-rich and *nanocrystalline* tungsten films. According to our view, the build-up of tensile out-of-plane strain in the irradiated volume reported in their work actually represents a lattice parameter increase due to the incorporation of point defects in the W lattice, while in this work, the amount of point defects present in the crystal lattice was diminished as a result of the irradiation. Furthermore, it should be noted, that in-plane and out-of-plane residual strains (Fig. 5), as well as in-plane residual stresses (Figs. 3 and 4c) presented here, are evaluated by a methodology very different to MBCDI and μ-Laue diffraction. The present approach allows for the decoupled observation of lattice swelling/shrinkage (3rd order strains) and "macroscopic" in-plane stresses of 1st order. The present results, compared with the mentioned previous single crystal studies thus also suggest that the presence of atomistic defects and pre-existing intrinsic residual stresses could be used to tune the material's response to irradiation.

5. Conclusion

Cross-sectional X-ray nanodiffraction and TEM analyses on a magnetron-sputtered W thin film showed a depth-localized relaxation of compressive residual stresses from approx. −2.5 to 0.75 GPa after the irradiation with 5 MeV Si^{2+} ions. The irradiation process is accompanied by an apparent atomistic structure recovery which was attributed to the removal of crystal lattice point defects and relaxation of strains of 3rd order, manifested by a decrease of FWHM and an increase of intensity of XRD peaks, respectively, as well as a lattice parameter decrease.

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References

[1] I.J. Beyerlein, A. Caro, M.J. Demkowicz, N.A. Mara, A. Misra, B.P. Uberuaga, Radiation damage tolerant nanomaterials, Mater. Today. 16 (2013) 443–449. doi:10.1016/j.mattod.2013.10.019.

[2] G. Vijaya, M.M. Singh, M.S. Krupashankara, M.R. Srinivas, R.S. Kulkarni, Development and Analysis of Tungsten Thin film Coating for Solar Absorption, in: Mater. Today Proc., Elsevier Ltd, 2018: pp. 2555–2563. doi:10.1016/j.matpr.2017.11.039.

[3] I.P. Jain, G. Agarwal, Ion beam induced surface and interface engineering, Surf. Sci. Rep. 66 (2011) 77–172. doi:10.1016/j.surfrep.2010.11.001.

[4] E. Snoeks, K.S. Boutros, J. Barone, Stress relaxation in tungsten films by ion irradiation, Appl. Phys. Lett. 71 (1997) 267–269. doi:10.1063/1.119515.

[5] L. Pranevičius, K.F. Badawi, N. Durand, J. Delafond, P. Goudeau, Relaxation of residual stresses in highly stressed multilayers initiated by ion irradiation, Surf. Coatings Technol. 71 (1995) 254–258. doi:10.1016/0257-8972(94)02321-G.

[6] J. Martan, N. Semmar, C. Boulmer-Leborgne, P. Plantin, E. Le Menn, Thermal Characterization of Tungsten Thin Films by Pulsed Photothermal Radiometry, Nanoscale Microscale Thermophys. Eng. 10 (2006) 333–344. doi:10.1080/15567260601009189.

[7] E.G. Fu, Y.Q. Wang, M. Nastasi, Mechanisms for ion-irradiation-induced relaxation of stress in mosaic structured Cu thin films, J. Phys. D. Appl. Phys. 45 (2012) 495303. doi:10.1088/0022-3727/45/49/495303.

[8] A. Jain, S. Loganathan, U. Jain, Reduction of stresses in thin films by high energy ion beams, Nucl. Instruments Methods Phys. Res. Sect. B Beam Interact. with Mater. Atoms. 127–128 (1997) 43–45. doi:10.1016/S0168-583X(96)00850-6.

[9] J.L. Du, Y.H. Qiu, J. Zhang, J.C. Huang, Z.M. Wu, X.F. Zhang, Y.H. Wang, J.K. Baldwin, Y.Q. Wang, Y.G. Wang, E.G. Fu, The alleviation of radiation-damage on Nb/MgO film driven by strain gradient in He ion irradiation, Appl. Surf. Sci. 465 (2019) 1014–1018. doi:10.1016/j.apsusc.2018.09.174.

[10] S. Kajita, W. Sakaguchi, N. Ohno, N. Yoshida, T. Saeki, Formation process of tungsten nanostructure by the exposure to helium plasma under fusion relevant plasma conditions, Nucl. Fusion. 49 (2009) 095005. doi:10.1088/0029-5515/49/9/095005.

[11] R.W. Harrison, N. Peng, R.P. Webb, J.A. Hinks, S.E. Donnelly, Characterisation of helium ion irradiated bulk tungsten: A comparison with the in-situ TEM technique, Fusion Eng. Des. 138 (2019) 210–216. doi:10.1016/j.fusengdes.2018.11.024.

[12] N.W. Phillips, H. Yu, S. Das, D. Yang, K. Mizohata, W. Liu, R. Xu, R.J. Harder, F. Hofmann, Nanoscale lattice strains in self-ion implanted tungsten, Acta Mater. 195 (2020) 219–228. doi:10.1016/j.actamat.2020.05.033.

[13] F. Hofmann, D. Nguyen-Manh, M.R. Gilbert, C.E. Beck, J.K. Eliason, A.A. Maznev, W. Liu, D.E.J. Armstrong, K.A. Nelson, S.L. Dudarev, Lattice swelling and modulus change in a helium-implanted tungsten alloy: X-ray micro-diffraction, surface acoustic wave measurements, and multiscale modelling, Acta Mater. 89 (2015) 352–363. doi:10.1016/j.actamat.2015.01.055.

[14] B.M. Steinetz, T.L. Benyo, A. Chait, R.C. Hendricks, L.P. Forsley, B. Baramsai, P.B. Ugorowski, M.D. Becks, V. Pines, M. Pines, R.E. Martin, N. Penney, G.C. Fralick, C.E. Sandifer, Novel nuclear reactions observed in bremsstrahlung-irradiated deuterated metals, Phys. Rev. C. 101 (2020) 044610. doi:10.1103/PhysRevC.101.044610.

[15] O. Romanenko, V. Havranek, A. Mackova, M. Davidkova, M. Cutroneo, A.G. Ponomarev, G. Nagy, J. Stammers, Performance and application of heavy ion nuclear microbeam facility at the Nuclear Physics Institute in Řež, Czech Republic, Rev. Sci. Instrum. 90 (2019) 013701. doi:10.1063/1.5070121.

[16] J.F. Ziegler, J.P. Biersack, The Stopping and Range of Ions in Matter, in: Treatise Heavy-Ion Sci., Springer US (1985) 93–129. doi:10.1007/978-1-4615-8103-1_3.

[17] G.H. Kinchin, R.S. Pease, The displacement of atoms in solids by radiation, Reports Prog. Phys. 18 (1955) 1–51. doi:10.1088/0034-4885/18/1/301.

[18] J. Keckes, R. Daniel, J. Todt, J. Zalesak, B. Sartory, S. Braun, J. Gluch, M. Rosenthal, M. Burghammer, C. Mitterer, S. Niese, A. Kubec, 30 nm X-ray focusing correlates oscillatory stress, texture and structural defect gradients across multilayered TiN-SiOx thin film, Acta Mater. 144 (2018) 862–873.

doi:10.1016/j.actamat.2017.11.049.

[19] M. Stefenelli, J. Todt, A. Riedl, W. Ecker, T. Müller, R. Daniel, M. Burghammer, J. Keckes, X-ray analysis of residual stress gradients in TiN coatings by a Laplace space approach and cross-sectional nanodiffraction: A critical comparison, J. Appl. Crystallogr. 46 (2013) 1378–1385. doi:10.1107/S0021889813019535.

[20] I. Petrov, P.B. Barna, L. Hultman, J.E. Greene, Microstructural evolution during film growth, J. Vac. Sci. Technol. A Vacuum, Surfaces, Film. 21 (2003) S117–S128. doi:10.1116/1.1601610.

[21] I. Petrov, L. Hultman, U. Helmersson, J.E. Sundgren, J.E. Greene, Microstructure modification of TiN by ion bombardment during reactive sputter deposition, Thin Solid Films. 169 (1989) 299–314. doi:10.1016/0040-6090(89)90713-X.

[22] H. Köstenbauer, G.A. Fontalvo, M. Kapp, J. Keckes, C. Mitterer, Annealing of intrinsic stresses in sputtered TiN films: The role of thickness-dependent gradients of point defect density, Surf. Coatings Technol. 201 (2007) 4777–4780. doi:10.1016/j.surfcoat.2006.10.017.

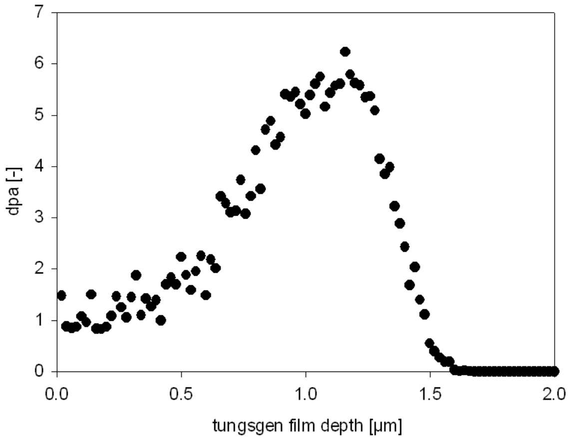

Figure 1 Calculated damage in displacements per atom (DPA) across the tungsten film depth caused by the implantation with Si^{2+} ions with an energy of 5 MeV.

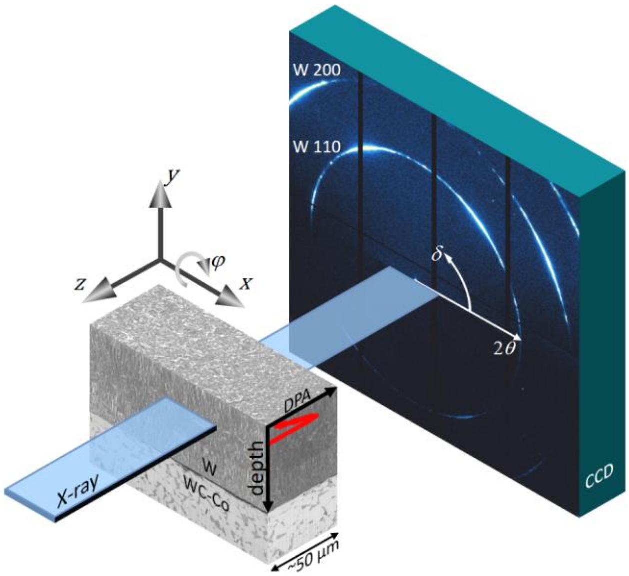

Figure 2 A schematic sketch of the cross-sectional X-ray nanodiffraction experiments on ~50 µm thick sample lamellae. The 7 µm thick W films were scanned along the vertical direction (y) and W 110 and 200 Debye-Scherer rings were collected using two-dimensional detector. Pencil beams with dimensions of 0.5 x 2.0 µm^2 and 0.1 x 2.0 µm^2 were used to analyze as-deposited and irradiated samples at the beamlines P03 and ID13, respectively. The figure shows schematically on the side of the sample, that the maximal DPA was located about 1.1 µm below the surface.

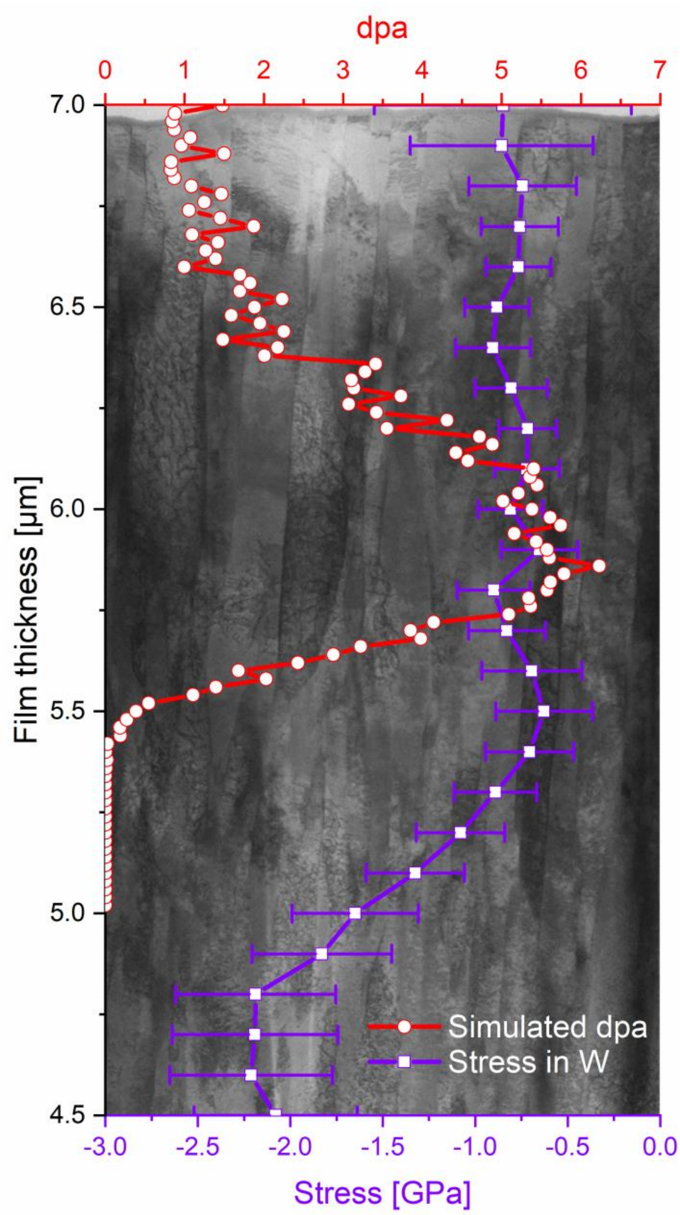

Figure 3 The distribution of calculated DPA and the observed in-plane compressive residual stress distribution, featuring a relaxation from approx. -2.5 to -0.75 GPa, is superimposed onto a TEM bright-field micrograph, which shows the columnar microstructure of the upper region of the tungsten film, apparently unaltered by the irradiation.

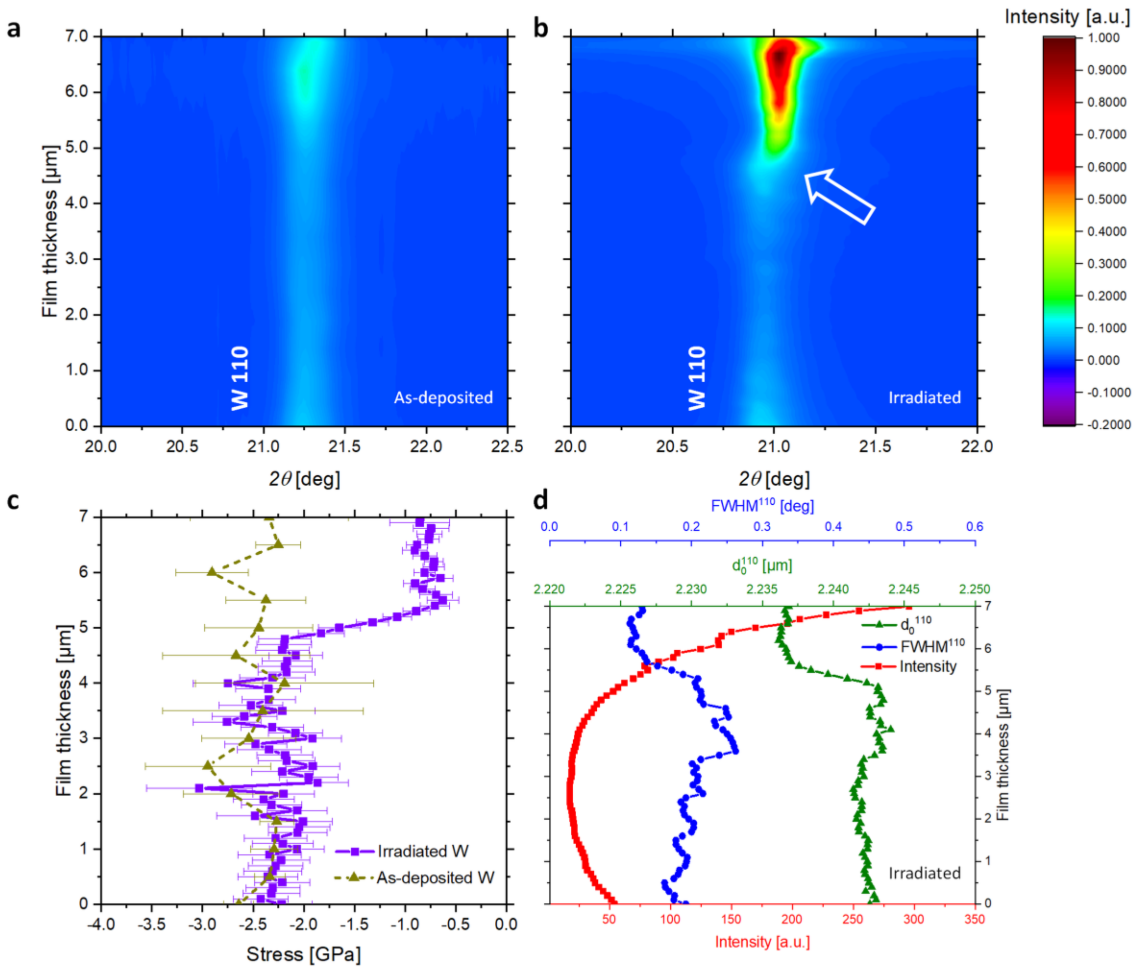

Figure 4, CSnanoXRD data, showing the evolution of the W 110 reflection as a function of film thickness in the as-deposited (a) and irradiated (b) films. The arrow in (b) indicates the lower border of the irradiation-affected film region. Depth-dependencies of residual stresses in (c) show a nearly constant level of approx. −2.5 GPa in the as-deposited film and a stress relaxation to −0.75 GPa in the irradiated film, down to a depth of ~2 µm. The supposed structure recovery in the irradiated film is supported by a decrease in FWHM and an increase in peak intensity of the W 110 reflection, as well as a lattice parameter decrease (d).

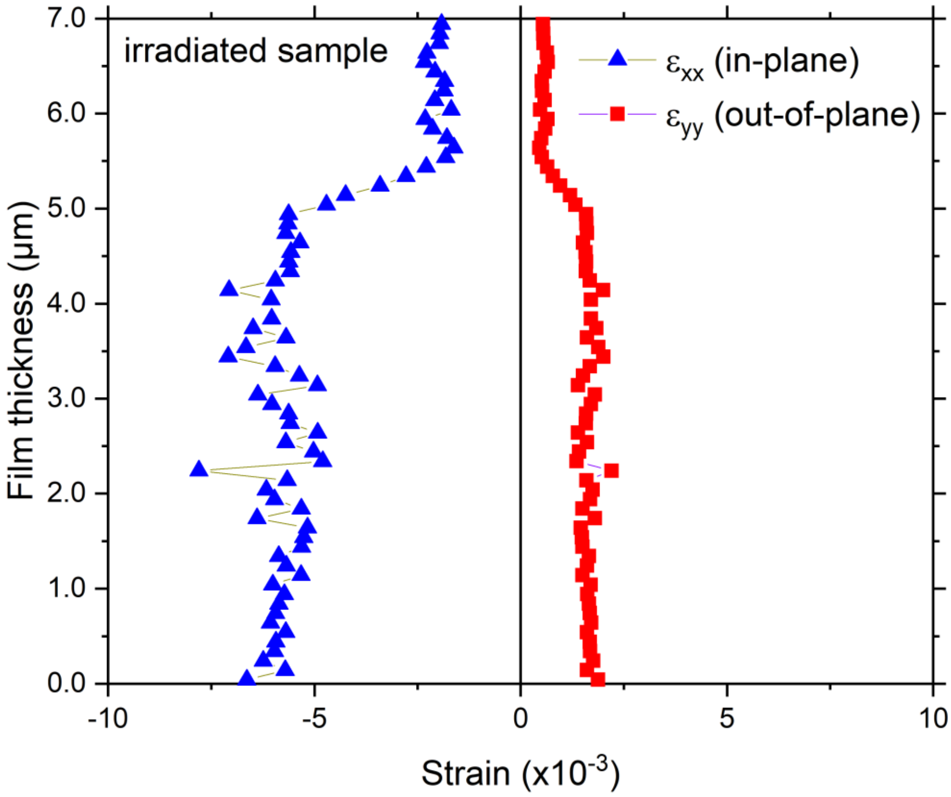

Figure 5 Depth profiles of X-ray elastic strains across the implanted film thickness indicate a decrease of compressive in-plane strains and tensile out-of-plane strains in the near-surface film region.