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To cite this version:

Peng Li, Nicholas Phillips, Steven Leake, Marc Allain, Felix Hofmann, et al.. Revealing ‘invisible’ defects in implanted material with 3D Bragg ptychography. 2020. hal-02925871
Revealing ‘invisible’ defects in implanted material with 3D Bragg ptychography

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Abstract

Small ion-irradiation-induced defects can dramatically alter material properties and speed up degradation. Unfortunately, most of the defects irradiation creates are below the visibility limit of state-of-the-art microscopy. As such, our understanding of their impact is largely based on simulations with major unknowns. Here we present a novel x-ray crystalline microscopy approach, able to image with high sensitivity, nano-scale 3D resolution and extended field of view, the lattice strains and tilts in crystalline materials. Using this enhanced Bragg ptychography tool, we study the damage helium-ion-irradiation produces in tungsten, revealing a series of crystalline details in the 3D sample. Our results show that few-atom-large ‘invisible’ defects are likely isotropic in orientation and homogeneously distributed. No defect-denuded region is observed close to the grain boundary. These results open up exciting perspectives for the modelling of irradiation damage and the detailed analysis of crystalline properties in complex materials.
Introduction

Atomic defects play a fundamental role in controlling the mechanical and physical properties of crystalline materials, resulting in critical hurdles for advanced applications, as epitomized in e.g., energy generation (nuclear\(^1\) or photo-voltaic\(^2\)), energy storage,\(^3\) aerospace,\(^4\) micromechanics\(^5\) and semiconductor miniaturization.\(^6\) Native or induced defects localise distortion of the crystal lattice and thereby reduce the overall strain energy.\(^7\) Conversely, the behaviour of defects is strongly dependent on the microstructural environment, which provides fantastic potential for tuning material properties.\(^8\) Understanding and exploiting defects in crystalline materials requires probing the material structure from atomic- to macro-scale. At near atomic resolution, transmission electron microscopy (TEM) is an essential method, which allows the direct visualisation of lattice defects in two dimensions (2D) and even three dimensions (3D).\(^9,10\) While it is also sensitive to the associated lattice strains, the investigations of these are restricted to 2D and only possible for a small subset of samples (e.g., straight dislocations of edge character\(^11\)). This is detrimental for the understanding of the defect-defect interplays, since defects interact via their 3D strain fields and resulting stresses.\(^12\) In inherently thin TEM samples, defects may also be lost to nearby surfaces that act as strong defect sinks, thus reducing the apparent defect density.\(^13\)

A further challenge concerns the visibility of small defects: In structures containing more than a few thousand atoms, TEM is insensitive to defects smaller than ~1.5 nm.\(^14\) However crystals that contain a population of rather small defects with broad size distribution constitute a large class of materials, such as the ones resulting from intense irradiation exposure (materials for future fusion and fission technologies,\(^1\) accelerator targets\(^15\) or modified surfaces for biocompatibility\(^16\)). Defects produced by irradiation range from single atom defects to clusters of tens of nanometres in size, where the number density of defects is typically linked to the defect size by a power law with a negative exponent, i.e., the vast majority of defects are below the visibility limit.\(^17\) Although small, these “invisible” defects can dramatically change the mechanical properties of the material\(^18\) or its thermal transport,\(^19\) and may lead to irradiation-induced dimensional change.\(^20,21\) Recent simulations have successfully predicted the presence and evolution of large populations of “invisible” defects during irradiation.\(^22\) However, these predictions remain unverified by experiments. As such there is an urgent need for techniques able to determine the “invisible” defect number density and spatial distribution.
A promising alternative is to infer the concentration of those small defects by measuring the distortions (i.e., strains) they cause in the crystal lattice. This idea has been successfully demonstrated using micro-beam Laue diffraction. However the spatial resolution of this technique (~0.5 μm in 3D) is insufficient to resolve the nano-scale spatial heterogeneities and defect clustering predicted by simulations.

In this context, we foresee that x-ray lens-less microscopy could play a major role. Since its first demonstration in 2001, x-ray Bragg coherent diffraction imaging has been proven as a powerful method to investigate crystalline properties of materials in various sample environments. X-ray Bragg ptychography (BP), a recently developed method, combines sensitivity to the atomic displacements of lattice planes and outstanding imaging performances of ptychography. Thereby, it provides the means to image, in 3D, extended crystalline materials, with high sensitivity to weak crystalline displacements and strong robustness to large strain fields, those specifications being mandatory to cope with the distinctive crystalline features of strain induced defects and native strains of e.g., irradiated materials. To date, BP with 3D spatial resolution of 10 - 50 nm, and strain resolution on the order of 10^{-4} has been demonstrated and applied to complex problems in material science, e.g., anti-phase domain boundaries in metallic alloy, stacking faults in semiconductor quantum wire, and crystalline domains in biomineral. The advent of 4th generation synchrotrons, where BP will become available at several beamlines, should provide a broader access to this microscopy method.

X-ray BP makes use of a series of 3D Bragg diffraction intensity data sets, measured in the vicinity of a Bragg reflection, obtained by scanning the sample across a localised illumination beam. The sample motion is designed to ensure a significant amount of probe overlap, the redundancy in the data set allowing retrieving the otherwise inaccessible phase of the diffracted field from the set of diffracted intensities. As long as the probe is known, the phase recovery is performed with iterative algorithms, which further give access to the sample 3D image. An effective complex-valued electron density, specifically designed to account for the Bragg geometry, is used to describe the sample as a function of \( r \), the 3D spatial coordinate. Its amplitude \( |\rho(r)| \), provides information about the morphology (or density) of the scattering crystal domain. The spatially varying phase \( \phi_{hkl}(r) \) associated with a specific hkl Bragg vector \( Q_{hkl} \) is linked to the atomic displacement field in the crystal, \( u(r) \), by \( \phi_{hkl}(r) = Q_{hkl} \cdot u(r) \).
However, current BP approaches suffer from substantial limitations. They require the acquisition of an extended data set, which relies on the stability of the experimental set-up over long measurement times (typically 6-12 hours). This necessitates the samples be resistant to radiation damage, a particular challenge for biologically relevant materials. Moreover, BP requires the 3D probe to be known prior to the sample electron density reconstruction. Although some probe pre-characterisations can be performed via ptychography of a test pattern (or similar) in the transmission geometry, the final BP image quality fully depends on the amount of uncertainties introduced by the lack of detailed knowledge on the probe used during the BP experiment. In forward direction ptychographic imaging, this limitation has long been recognised, and simultaneous retrieval of the probe and object is now the universal measurement standard. The additional complexity of the Bragg geometry has thus far prevented this refinement in BP.

Here, we used an advanced BP approach to investigate TEM-invisible defects in a tungsten-rhenium alloy sample implanted with helium ions. To enable these measurements, we develop a simultaneous probe refinement strategy that makes it possible to improve the retrieved image sensitivity and map a much larger field of view than previously attainable. Notably, this was achieved without increasing the amount of collected data (and therefore the total acquisition time). This approach allows us to view the extended sample and directly compare implanted and non-implanted regions, highlighting details of the crystalline structure, which include lattice damage from helium irradiation, dislocations and sample preparation damage. The results are discussed in the context of understanding irradiation damage processes within tungsten and their potential impacts for its use in the design of future fusion reactor components.

**Results**

A tungsten, 1% rhenium alloy was manufactured by arc melting, producing a polycrystalline material with grain size of a few hundred microns as determined by scanning electron microscopy (SEM). The addition of rhenium mimics neutron-irradiation-induced transmutation alloying. The material was further implanted with helium ions that modified a ~3 μm thick surface layer (see Methods Sec. 1) and generate neutron-like collision cascade damage. Note that, during fusion reactor operation, helium is generated by transmutation and also diffuses from the plasma. Our use of helium ion implantation effectively mimics
these two effects. Finally, using focussed ion beam (FIB) milling, a cross-section sample containing a grain boundary was extracted from the bulk and a region of $15 \times 8 \, \mu m^2$ was thinned to $\sim 0.45 \, \mu m$ thickness. The resulting sample is shown in Fig. 1a.

BP microscopy was performed at a third generation synchrotron beamline, schematically presented in Fig. 1b and described in detail elsewhere. An 8 keV coherent x-ray beam was focused down to about 200 nm at the sample position with a set of Kirkpatrick-Baez (KB) mirrors. The sample was placed on a three-axis translation stage, allowing for nano-positioning and nano-scanning. The stage was mounted on the top of a goniometer cradle, used to orientate the sample to the Bragg conditions and to map the 3D components of the scattered intensity distribution, by angularly scanning the so-called rocking curve. The sample was scanned across the beam propagation direction, in steps sufficiently small to collect partially redundant information. For each probe-to-sample position and each angle along the rocking curve, coherent diffraction patterns were recorded on a 2D pixelated detector, placed at an exit angle of twice the Bragg angle, at a sufficiently large distance to ensure far-field regime detection. For our measurements, the (220) specular reflection of the right-hand grain of Fig. 1a was probed. A region of $2 \times 2 \, \mu m^2$ (enclosed in the red rectangle in Fig. 1a) near the grain boundary and the implantation layer was selected for the ptychography scan, so that both implanted and non-implanted areas could be imaged. Full experimental details are provided in Methods Sec. 2.

As an example representative of the collected data, a 2D coherent diffraction pattern from the He-implanted region is shown in Fig. 1c. Its asymmetry and extent are strong signatures of the presence of strain and rotation fields within the illuminated sample volume. A set of four patterns are further presented, corresponding to the intensity distribution obtained when integrating the 2D patterns along the rocking curve, for each of the extreme positions of the raster scan (Fig. 1d). One observes clearly the motion of the main peak towards lower Bragg angles, resulting from the strain in the implanted region (i.e., on the $20^{th}$ and $400^{th}$ positions). This suggests that the implantation results in a crystalline lattice swelling, in line with previous reports. Moreover, the 3D representation of the intensity distribution allows the investigation of the fringe structure. They arise from the interference from the two surfaces of the sample foil and present a period of about $14 \, \mu m^{-1}$. This provides an initial estimation of the sample thickness of $\sim 0.45 \, \mu m$, in excellent agreement with the thickness expected from SEM.
To go further in the sample structure analysis, the whole data set is inverted to retrieve the 3D sample electron density. This requires a detailed modelling of the scattering process: Under the kinematic scattering approximation, the Bragg diffraction intensity distribution $I_j(q)$ at the $j^{th}$ given probe position $r_j$ is given by:

$$I_j(q) = \left| \int_0^\infty P(r - r_j) \rho(r)e^{iQ_{hkln}(r)}e^{iq\cdot r}dr \right|^2,$$

where $q$ represents the 3D coordinates reciprocal spaces, $P$ is the probe function. For the numerical implementation, the direct and reciprocal spaces are introduced. To preserve the data information, the reciprocal frame follows the measurement scanning space ($q_1$, $q_2$, $q_3$). Using a recently developed formalism\(^{39}\), this sampling depicts a direct space frame ($r_1$, $r_2$, $r_3$). Fig. 2a shows a schematic representation of both space coordinate frames. Those are the spaces into which the inversion is performed before the final object reconstruction is plotted in the orthogonal $(x, y, z)$ sample frame (Fig. 1b).

Accessing the phase of the sample scattering function from intensity measurements requires one to overcome the loss of the scattered field phase. In forward ptychography, the 3D problem can be decomposed in a series of 2D problems, each of them being solved in the plane which contains the two scanning ptychographic directions.\(^{40}\) BP inversion is more intricate because it aims at solving a problem that is intrinsically 3D. Indeed, Eq. 1 cannot be simplified into a series of 2D ptychographic problems, by e.g., separating the probe and the object in the integral. However, only two scanning ptychographic directions are available, transverse to the probe propagation direction (note that along the beam direction, the weak focussing power of x-ray lenses produces an extremely elongated depth of focus). Therefore, while forward ptychography allows the simultaneous retrieval of both probe and sample functions,\(^{35}\) 3D BP requires strong a priori knowledge of the probe.

The structure and size of the probe also have a major impact on the 3D intensity acquisition in BP. To better illustrate this issue, we present the probe used during the experiment (see the cross-section and calculated profile along the beam propagation direction, in Fig. 2b and c). This probe function was characterized prior to our measurement (see Methods Sec. 2). The cross-section presents a rather structured distribution, which includes a central spot of about 400×200 nm\(^2\) (intensity FWHM) along the horizontal and vertical planes, respectively and some beam tails and secondary maxima that extend to a much bigger area, of about 4×4 \(\mu\text{m}^2\). These features have to be considered when defining the rocking curve angular steps. Indeed
the numerical analysis of the data set, which is based on the use of the fast Fourier transform, connects reciprocal and direct space through Fourier conjugation relations. Precisely, the sampling angle must be chosen such that the illuminated volume (green line) is fully contained in a cuboid defined in the \((r_1, r_2, r_3)\) reconstruction space (orange line) as shown in Fig. 2a. Depending on the probe structure and on the criterion used to define the probe size, the angular sampling \(\Delta \theta\) required along the rocking curve varies substantially, as illustrated in Fig. 2d, for vertical-plane diffraction geometry. This plot presents the sampling condition as a function of the considered (i.e., vertical) probe width \(W\) and the sample thickness \(T\), for a Bragg angle \(\theta_B\) of 43.9° (see Methods Sec. 3 and Eq. 2 for further details). For our sample, the required angular sampling varies from a few 0.01° when only the probe central lobe is accounted for, to about 0.001° when the full probe extent is considered (interestingly, this behaviour is rather marginally affected by the sample thickness). While 0.001° angular steps are mechanically accessible at Bragg diffraction optimized beamline set-ups, those small steps are still challenging and result in a detrimental linear increase of the total measurement time. To mitigate issues caused by long data collection, a series of compromises are usually made, such as choosing an angular step only accounting for the central lobe sampling condition and/or fewer ptychographic positions and/or smaller angular range, which reduce the field of view and/or degrade the image quality. Furthermore, the structure of the probe secondary maxima that develop far from the central lobe, i.e., corresponding to high frequencies, is more prone to optics instabilities, limiting the use of prior knowledge of the probe for faithful reconstruction. All these underline the need for retrieving the full probe distribution function and the sample scattering function simultaneously, a novel strategy we have implemented and applied in this work, to produce high-fidelity maps of lattice strains and rotations.

To succeed with the simultaneous retrieval of the 3D sample scattering function and illumination function, additional information needs to be brought to the inversion problem. Considering the generally small numerical aperture of x-ray optics, which results in a self-similar probe along the beam direction, a straightforward constraint can be derived on the probe invariance. This inversion strategy allows recovering not only the information in the close vicinity of the central probe lobe, but also the information arising from much larger distances where only the tails (the series of secondary maxima) of the probe illuminate the sample. According to the sampling principles described above and illustrated in Fig. 2d, this would imply scanning the rocking curve in extremely small angular steps (in the order of a
few 0.001°). We circumvented this issue by further up-sampling the rocking curve and retrieving the information in the missing planes (see Methods Sec. 4). This operation corresponds to virtually inserting angular sampling points in-between the measured ones, and to retrieving their diffraction patterns based on the intensity information in the measured ones. Using this strategy, the field of view was increased and the image quality was improved, revealing unprecedented structural details, as shown hereafter. The whole reconstruction strategy and inversion details are given in Methods Sec. 4.

The iso-surfaces of the retrieved 3D electron density and the phase \( \phi_{220}(\mathbf{r}) \) associated with the displacement field \( \phi_{220}(\mathbf{r}) = \mathbf{Q}_{220} \cdot \mathbf{u}(\mathbf{r}) \), with \( \mathbf{Q}_{220} \) being the Bragg vector for the (220) reflection), are shown in Fig. 3. For sake of clarity, only the relevant part of the retrieved image is shown, using a mask applied on the all presented maps (see Supplementary information S2 for the mask definition). The presence of a strong edge (seen from the top left corner to the bottom middle) in the electron density map corresponds to the expected crystal grain boundary. While the sample density is rather homogenous in most parts of the retrieved field of view, some fluctuations are visible. The pipe-like ones (see black arrows in Fig. 3c and 3d) are well known features, which can be understood when considering the phase map.

Here, phase vortices are co-localised with the pipes of missing intensity, a characteristic feature of dislocations in noise-limited BCDI data.\(^{27,41-43}\) Similarly, additional density oscillations appear in regions, where the phase wraps rapidly, e.g., at the top right corner.\(^{33}\) A few density voids are observed in the vicinity of the grain boundary, corresponding to cavities in the crystal. Finally, at the limit of the raster scan, where both the counting statistics and overlap constraint are reduced the reconstruction quality is degraded. The probe function retrieved simultaneously from the BP data set (cross-section profile shown in Fig. 3e) exhibits the expected characteristic features (e.g., the central lobe with size of 450×190 nm\(^2\), intensity FWHM), in excellent agreement with the profile obtained separately and previously shown in Fig. 2b. The missing triangular region at the bottom left of the probe reconstruction is due to the limited extent of the crystal set by the grain boundary. Note the extent of the field of views, of about 0.6×6×6 \( \mu m^3 \) (z×y×x) for the 3D sample and 5×4 \( \mu m^2 \) (p\(_1\)×p\(_2\)) for the probe cross section. If the intensity information were not limited by the grain boundary (non-scattering crystal), we estimate the field of view along x could be as large as 7.8 \( \mu m \). Finally, the spatial resolution of this 3D reconstruction is estimated as 37×40×39 nm\(^3\) along z, y and x respectively (see Supplementary information S3).
As a matter of comparison, the same data set was used to perform a reconstruction using the formerly employed approach,\textsuperscript{29,44} i.e., no angular up-sampling and using a reconstructed result of the probe based on transmission ptychography. The 3D reconstruction numerical volume was directly designed according to the conjugation relation applied to the experiment parameters, in particular the angular step. The final results are shown in Figs. 3f-h. The reduction in the field of view is particularly evident along the x direction (maximum field of view of 4.6 $\mu$m compared to 7.8 $\mu$m for the angular up-sampled approach), which is the direction mostly impacted by the angular sampling along the rocking curve. Although the main focal spot is fully contained in the probe function, the beam tails are clearly truncated. This impacts the quality of the object image, where the grain boundary is still visible, but artefacts, such as fluctuations in both electron density and phase, are more abundant. The previously evident dislocations are barely visible. This direct comparison underlines the merit of the new inversion strategy we have proposed.

The high-quality extended reconstruction was used to analyse the structural features present in the implanted crystal. To this aim, we used 3D phase map to extract the lattice strain projected along $Q_{220}$ and the lattice rotations about the x and y axes, hereafter referred to as $\varepsilon_{zz}$, $\omega_x$ and $\omega_y$, respectively (see Methods Sec. 5). The top edge of the reconstructed volume corresponds to the former sample surface during ion-implantation (note that, the strain increase observed at the very top surface is an artefact due to a bit of aliasing resulting from the probe extent). The 3D strain map (Fig. 4 (a) and (b)) clearly shows the He-implantation induced strain of about $3 \times 10^{-4}$, evident to a depth of approximately 2.8 $\mu$m. In the vicinity of the two FIB-processed surfaces, large positive strains, corresponding to a lattice expansion, can be seen. These strains extend to similar depths (about 150 nm for the top surface and 100 nm for the bottom surface) and have similar magnitude ($\sim 1.5 \times 10^{-3}$ and $\sim 1.1 \times 10^{-3}$ for the top and bottom surfaces, respectively). Several dislocations are also observed in both implanted and non-implanted layers. Besides these, $\varepsilon_{zz}$ presents homogeneous behaviour in the whole implanted layer including in the vicinity of the grain boundary. Finally, we would also encourage the reader to view the Supplementary Videos as these provide an interactive way to visualise the multi-dimensional dataset. SV1 shows the object iso-surface, whilst SV2 and SV3 each show a perpendicular plane propagating through the object with the $\varepsilon_{zz}$, $\omega_x$ and $\omega_y$ components displayed.

To further illustrate the interest of our BP result, characterisation using more routinely applied strain microscopy methods, namely high-resolution electron back scattering...
diffraction (HR-EBSD) and x-ray micro-beam Laue diffraction measurements, were performed for the same region of the sample. These results are shown in the Supplementary information S4. Whilst these methods are considered a mainstay of analysis for strain and tilt distributions in polycrystalline materials, their limitations are obvious in the present case. HR-EBSD presents a lateral spatial resolution of $\sim 100 \text{ nm}^{45}$ and is only surface sensitive (down to a depth of about 10-20 nm) due to the short mean free path of backscattered electrons.$^{46}$ On the other hand, micro-beam Laue diffraction is bulk sensitive, but the obtained 2D maps represent the integration of the strain and tilts information along the whole thickness. Its transverse spatial resolution, which integrates the size of the focused beam, the scanning precision and the step size, builds up to about 0.5 $\mu$m in the present case. Some indication of positive lattice strain along the upper edge of the sample (where the implanted layer is known to be) was recorded by HR-EBSD and micro-beam Laue diffraction. However, neither approaches were able to definitively capture the presence of the implanted layer with a high level of clarity or any indication of the dislocations and the fine details of the strains as was possible using BP.

**Discussion**

The success of our approach relies on the capability of our new method to retrieve the far-field information in planes, whose intensity distributions are not experimentally measured. Of course, this process has a limit and a dedicated analytical work is on the way to further derive it mathematically. It was numerically determined for this experiment (Supplementary information S5). Qualitatively, the limit can be understood by considering that the numerical aperture of the focusing optic broadens the signal along the corresponding directions in the reciprocal space, in particular along the rocking curve direction. Therefore, the intensity information obtained in a given detector plane carries additional features arising from the broadening of the signal along the direction perpendicular to the detector plane. Interestingly, we observed that the reconstruction quality using 7 angular measurements (up-sampling ratio of 18 and angular step of 0.03°) is comparable to the quality of the image produced by the former reconstruction strategy, using 42 angles. From this comparison, considering the gains with respect to the measurement time ($\times 6$) and to the achieved field of view ($\times 1.4$ considering the limits imposed by the crystalline grain size or $\times 1.7$ considering the whole accessible field of view), a total gain of about 8-10 is estimated. This significant
improvement brought by our approach should further benefit from the advent of 4th generation synchrotron sources and their expected gain (of about 100) in coherence flux.

Regarding the crystalline properties imaged in the sample, we note that the 3D lattice strain and rotation maps obtained using our new approach are not accessible in any other way and reveal important new features: In the vicinity of the top and bottom sample surfaces, large positive strains, corresponding to a lattice expansion, can be seen. The strains extend to similar depths (~120 nm), have similar values (~1.1-1.5×10⁻³) and are present over the entire sample surfaces. Their spatial distribution and location are consistent with the effects of residual FIB-induced damage. FIB imaging and machining are known to be able to cause dramatic material changes such as introduction of lattice defects,⁴⁷ large lattice strains,⁴⁸ amorphization,⁴⁹ and formation of Ga intermetallic.⁵⁰ From electron microscopy, FIB damage is often indistinguishable from the intrinsic defects and damage features of interest in the sample.⁵¹,⁵² In the present case, it is worth noting that FIB-induced damage remains after employing preventative and mitigating measures, routinely employed in fabricating strain microscopy samples for electron microscopy including sacrificial capping, glancing ion incidence angle and the removal of surface material via low energy polishing (see Methods Sec. 1 for fabrication details). We observe that even these residual effects lead to large lattice distortions. However, the beauty of our approach is that sample regions affected by FIB damage can be unambiguously identified and then excluded from further analysis. This dramatically simplifies data interpretation and gives certainty that features observed in the sample are not artefacts of the preparation. In the following only sample volumes more than 150 nm from sample surfaces, and thereby unaffected by FIB, are considered.

The ε_{zz} lattice strain map (Fig. 4a and 4b) shows a substantial strain with an average value of 3×10⁻⁴ in the helium-implanted layer. This is in good agreement with low-resolution microbeam Laue measurements of helium-implantation-induced lattice strain,²¹ estimating strains of about 2.4×10⁻⁴. Whilst there is a clear change in lattice strain from the He-implanted layer to the un-implanted material beneath, there is no discernible, abrupt change in lattice rotation at the boundary between the implanted and un-implanted material (Fig. 4c-4f). This is a very important evidence, since, in principle, a shear deformation of the lattice during ion implantation could lead to rotations about the x and z axes. Such a shearing would indicate the preferential formation of defects with specific orientation.⁵³ In tungsten, helium implantation is expected to predominantly produce Frenkel pairs that are prevented from recombining as He occupies the vacancies.²⁰,⁵⁴,⁵⁵ The lack of sharp change in lattice
orientation at the un-implanted/implanted interface shows that defects are randomly oriented, leading to a purely volumetric strain. This important result should greatly simplify the simulation of irradiation-induced strain in reactors, which will be one of the main mechanisms driving in-service degradation of fusion reactor armour.

Across the layer, the homogeneous behaviour of $\varepsilon_{zz}$ indicates a uniform distribution of defects, at least within the resolution and sensitivity limit of the present measurements. Previously, the formation of a zone denuded of large irradiation-induced dislocations loops was reported close to grain boundaries (within 20 to 50 nm) in self-ion irradiated material.

Our results suggest that for small defects and defect clusters, such as those created by helium ion-irradiation, this is not the case, limiting the potential effectiveness of grain size reduction as a means of reducing irradiation defect accumulation.

Finally, the quality of our crystalline image allows us to consider in more detail the dislocations visible in the reconstructed sample. An iso-surface rendering of the electron density, as well as the associated phase variation in the sample mid-plane are shown in Fig. 4h and Supplementary information S6 for dislocation #1 (implanted layer) and #2 (un-implanted region), respectively. Dislocations in tungsten have predominantly $b=1/2\langle111\rangle$ Burgers vector, although dislocations with $b=\langle100\rangle$ have also been reported. For a given Bragg reflection, only dislocations for which $Q_{hkl}\cdot b$ is non-zero are visible. For the present (220) reflection, this means that the observed dislocations may have the following Burgers vectors: $1/2[111]$, $1/2[11-1]$, [100] or [010]. Dislocation #2 shows a central pipe of missing intensity. The phase variation around the dislocation line shows a vortex with a total phase increase of $4\pi$. The phase variation agrees well with the simulated phase for a dislocation in the thickness direction, as shown in Supplementary information S6. In contrast, dislocation #1 shows a more complex and surprising structure: Here two separate pipes of reduced intensity are clearly identified, suggesting that in fact this dislocation corresponds to two parallel dislocations, each with an associated phase vortex of $2\pi$. This is the signature expected from two partial dislocations linked by a stacking fault on the (-110) plane. The presence of dissociated dislocations in tungsten is surprising since bcc metals have comparatively high stacking fault energy. However, recent ab-initio calculations show that in fact, the addition of Re can substantially reduce the stacking fault energy in tungsten, a scenario possibly at play here.
Conclusions

Thanks to the development of an upgraded BP approach, we obtain quantitative 3D maps of lattice strain and rotation in a He-implanted Tungsten crystal. These maps, of unmatched quality and extent, reveal numerous, otherwise inaccessible, crystalline features. Beyond a FIB-damaged layer, which can now be unambiguously discarded from the analysis, we observed strains and lattice rotations caused by ‘invisible’ helium-implantation-induced defects, identified to be of random orientation. Surprisingly, we do not find a defect-denuded region in the vicinity of the crystal boundary. These results provide new insights, essential for predicting the effects of ion-irradiation on crystals. Moreover, they pave the way for highly detailed investigation of complex next-generation crystalline materials, e.g., refractory high-entropy alloys for extreme environments.60,61
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Methods

1 - Sample preparation

The sample was produced from a bulk polycrystalline tungsten-1wt% rhenium alloy, manufactured by arc melting from high purity elemental powders. The sample surface was mechanically ground and further polished using diamond paste. A final chemo-mechanical polishing step (with 0.1 μm-colloidal silica suspension) was used to produce a high-quality surface finish. The grain size in the polycrystal was ~500 μm. The sample was implanted with helium ions to produce a 2.8 μm thick implanted layer. To achieve a relatively uniform damage level (0.02 ± 0.003 dpa between 0 μm and 2.8 μm depth) and injected helium concentration (310 ± 30 appm between 0 μm and 2.8 μm depth), implantation was performed using a number of different ion energies up to 2 MeV. The details of the implantation are provided elsewhere.21 For BP measurements, a small specimen (~25×14×0.5 μm³) was extracted from the bulk using a focused ion beam (FIB) lift-out process. This preparation method is adapted from that developed for the preparation of isolated micro-crystals for BCDI.43 In order to minimise the effects of lattice damage induced during the lift-out process a combination of protective capping, glancing angle milling, and low energy polishing was used. Specifically, the surface of the bulk sample was first covered with carbonaceous platinum, to further protect the tungsten from normal incidence angle milling ions, using an electron-beam, which does not introduce any damage in the implanted material. This produces a thin layer of about 0.1 – 0.2 μm, which appears as a dark line in the SEM picture (Fig. 2a). On top of this layer, 2 μm of carbonaceous platinum are further deposited using the gallium-ion-beam, which achieves a balance of sputtering and deposition. This is faster than the electron-beam but more intrusive, justifying the prior deposition of the electron-beam assisted deposition layer. The gallium-deposited platinum appears with a different contrast at the top of the sample. Trenches on either side of the lift-out sample were made using FIB, this was then undercut and attached to a micro-manipulator. The sample was finally attached to a copper TEM-lift-out holder as 90 degrees to its original orientation i.e., the walls of the trench became the top and bottom surfaces of the sample for all measurements. As the sample was still rather thick at this point (1 – 2 μm), FIB was again used to further thin down the sample. This was performed at 30 keV, with a current in the order of 240 pA or slightly lower. Finally, the obtained lamella was polished with a 2 keV gallium ion beam to remove most of the damage from the previous FIB steps. It results in a ~0.35 μm thinned region
around the implantation layer region, in the vicinity of the grain boundary. The implanted layer starts from the dark line downwards (Fig. 2a). The (220) lattice planes of the interested grain (further away from the copper support end in Fig. 2a) are parallel to the two lift-out surfaces.

2 - Experimental details – Bragg ptychography

The x-ray beam of the ID01 beamline (ESRF) was filtered with a Si-111 double crystal monochromator to provide a monochromatic beam with energy of 8 keV (or wavelength $\lambda$ of 1.54 Å) with a bandwidth of about $1 \times 10^{-4}$. Kirkpatrick-Baez mirrors were used to focus the beam onto the sample, horizontally (H) and vertically (V). A pair of slits placed upstream the mirrors were set to a width of $60 \times 200 \ \mu m^2 (H \times V)$, selecting a spatially coherent beam for the experiment. From a dedicated (forward ptychography) beam characterization, the beam profile was extracted and the central spot size (intensity FWHM) was measured as $400 \times 200 \ \text{nm}^2 (H \times V)$. The resulting numerical aperture (NA) was estimated to be $1.9 \times 10^{-4}$ and $3.7 \times 10^{-4}$ (H×V) using the extent of the calculated over-focused beam and the depth of focus to $1950 \times 540 \ \mu m^2 (H\times V)$. More details are given in the following Methods sections.

For the BP measurements, the specular (220) Bragg reflection of the grain away from the copper support was chosen. It corresponds to a Bragg angle $\theta_B$ of 43.9°. The intensity patterns were measured with an area detector (Maxipix, 516×516 pixels, pitch size of $55 \times 55 \mu m^2$) located at 1.4 m away from the sample. A total of 42 angular positions, with an angular step size of 0.005°, were taken along the rocking curve. At each of these angles, a set of $20 \times 20$ positions, with a step size of 100 nm, was used for the raster scan. It corresponds to an area of $2 \times 2 \ \mu m^2$. The exposure time per frame was set to 0.2 seconds.

Immediately after BP measurements, the experimental geometry was changed to the forward geometry to perform a conventional transmission ptychographic scan with a Siemens star pattern, in order to retrieve the illumination profile. An Archimedean spiral scan of 513 positions, with a step size of 100 nm, was performed and for each position the detector was exposed for 1s to measure the diffraction pattern. A sub-region of $334 \times 334$ pixels, centered around the forward beam, was used to retrieve the probe profile at the focal plane using standard approaches based on the extended ptychographical iterative engine algorithm (ePIE). The initial guess for the probe was a simulated beam profile based on the
experimental setup, i.e. calculated via the Fourier transform of a rectangle that is the numerical aperture (or NA) of the beam. The initial guess for the object was simply a uniform matrix of 1s. The gap between the different sensor modules was masked and left free. The reconstruction was run for 100 iterations, where the difference between the measured diffraction patterns and the calculated ones is very small and further iterations provide very little further convergence.

3 – Angular sampling along the rocking curve for 3D Fourier transform based Bragg coherent diffraction imaging

Due to the use of a 3D discrete Fourier transform to describe the propagation between real and reciprocal spaces, the angular sampling pitch $\Delta \theta$ and the window size $R_3$ of the illumination function along $r_3$ are linked via $\Delta \theta = \lambda / 2R_3\sin(\theta_B)\cos(\theta_B)$, according to the sampling relations,\textsuperscript{39,63} where, considering the geometry depicted in Fig. 1a, $R_3 = T\cos(2\theta_B)/\sin(\theta_B) + W/\tan(\theta_B)$. Here $T$ is the sample thickness and $W$ is the beam width along $p_1$. This leads to the following relation between the beam width $W$ and the sampling pitch $\Delta \theta$:

$$W = \lambda/2\Delta \theta[\cos(\theta_B)]^2 - T\cos(2\theta_B)/\cos(\theta_B)$$

(2)

For the used angular step size of 0.005° with a sample thickness of 500 nm, $W$ is about 1.7 $\mu$m. It is big enough to encompass the focal spot, but not the secondary maxima in the probe tails. Those strong beam tails, which extend beyond the window size, result in an aliasing artefact according to the Nyquist sampling theorem.\textsuperscript{64} Therefore, with this angular step size, the quality of the reconstruction is limited.

4 – Reconstruction strategy

The overall reconstruction strategy stems from previous works in 3D Bragg ptychography\textsuperscript{28,33,63} with some pivotal adaptations that are required to obtain the high quality, stable reconstruction shown in Fig. 3. The reconstruction is performed via the following cost-function to minimized over all the probe positions

$$\mathcal{L}(\rho, P, b) = \sum_{j=1}^{J} \mathcal{L}_j(\rho, P, b)$$

(3)
where $\rho$, $P$ and $b$, respectively, the 3D electronic density (i.e., the sample), the 3D probe function and the incoherent intensity background. Individual cost-functions are defined by\textsuperscript{28,33}

$$\mathcal{L}_j(\rho, P, b_j) := \left\| w(q) \times \left( I_j^{1/2}(q) - h_j^{1/2}(q) \right) \right\|^2 + \mu_j \sum_{r \in S} |\rho(r)|^2$$  \hspace{1cm} (4)

where $I_j(q)$ and $h_j(q) = |\Psi_j(q)|^2 + b(q)$ are the measured and predicted photon counts for the $j$-th probe position, respectively and $\Psi_j(q)$ is the Fourier transform of the exit field. The detector mask $\omega(q)$ is designed to discard hot or dead camera pixels but also to provide the expected angular up-sampling factor (described in details below). The second, quadratic term is a thickness-support regularization applied to the retrieved sample preventing the density of the sample to build-up in $\mathcal{S}$, the set of points outside the sample support. The regularization parameters $\mu_j \geq 0$ adjust how the final solution should comply with this support constraint; in the Bragg geometry, we note that this regularization is pivotal in dealing with the intrinsic lack of diversity of the beam along the propagation direction.\textsuperscript{63} The reconstruction strategy aims at retrieving simultaneously $\rho$, $P$ and $b$ from the set of measurements $\{I_j(q)\}_{j=1}^J$ via the minimization of Eq. 3.

1. Presentation of the reconstruction algorithm

The reconstruction algorithm was derived from the ePIE\textsuperscript{62} approach, with specific adaptations that allow the probe function to be accurately retrieved in the Bragg geometry. More specifically, a probe and object updates associated with the current (updated) position $j \in \{1 \ldots J\}$ are given by

$$\rho(r) \leftarrow \rho(r) + \lambda_P^{-1}(r) \times \partial_{\rho,j}(r),$$  \hspace{1cm} (5a)

$$P(r) \leftarrow B_{k_i} R_{k_i} \left( P(r) + \lambda_P^{-1}(r) \times \partial_{P,j}(r) \right),$$  \hspace{1cm} (5b)

where $\partial_{\rho,j}$ and $\partial_{P,j}$ are the gradients of $\mathcal{L}_j$ with respect to $\rho$ and $P$, respectively

$$\partial_{\rho,j}(r) = P^*(r - r_j) [\psi'_j(r) - \psi_j(r)] - \mu_j [1 - S(r)] \rho(r)$$  \hspace{1cm} (6a)

$$\partial_{P,j}(r) = \rho^*(r) [\psi'_j(r) - \psi_j(r)].$$  \hspace{1cm} (6b)

The projection and back-projection operators $R_{k_i}$ and $B_{k_i}$, respectively, are acting altogether along the direction of the incoming beam $k_i$; the pair of operators is basically enforcing, in the probe update (Eq. 5b), the invariance of the probe along the direction $k_i$ (described in
The spatial extension of the probe is directly connected with the angular sampling step-size \( \Delta \theta \). From the numerical point of view, as shown in Fig. 1a, this step-size is driven by the

\[
\lambda_{p,j}(\mathbf{r}) := (1 - \beta)|P(\mathbf{r} - \mathbf{r}_j)|^2 + \beta|P(\mathbf{r} - \mathbf{r}_j)|_{\max}^2 + \mu_j(1 - S(\mathbf{r})) \tag{7a}
\]

\[
\lambda_{p,j}(\mathbf{r}) := (1 - \alpha)|\rho(\mathbf{r})|^2 + \alpha|\rho(\mathbf{r})|_{\max}^2 \tag{7b}
\]

where \( 1 \geq \alpha, \beta \geq 0 \) are constant parameters whose tuning is left to the user. In Eq. 6

\[
\psi_j(\mathbf{r}) = P(\mathbf{r} - \mathbf{r}_j)\rho(\mathbf{r}) \text{ is the exit wave-field and } \psi'_j(\mathbf{r}) \text{ is the updated/corrected exit wave-field, whose Fourier transform is denoted } \Psi'_j(\mathbf{q}), \text{ and the support function } S(\mathbf{r}) \text{ vanishes whenever } \mathbf{r} \in \mathcal{S} \text{ and is equal to one otherwise. Finally, the incoherent intensity background } b(\mathbf{q}) \text{ is jointly estimated, thanks to a multiplicative update that enforces the positivity of the background (as long as the initial estimate is positive) and reads for } j \in \{1 \cdots J\}
\]

\[
b(\mathbf{q}) \leftarrow b(\mathbf{q}) \times \left(1 - \gamma + \gamma \left[ \frac{f_j(\mathbf{q})}{\bar{h}_j(\mathbf{q})} \right]^{1/2} \right)^2 \tag{8}
\]

with \( \gamma \geq 0 \). This background helps in accounting for parasitic scattering and small instabilities of the set-up. The updating relations Eq. 5 and Eq. 8 are at the core of the iterative joint reconstruction strategy used in this work.

2 - Probe retrieval: invariance property and spatial extension

The invariance of the probe function along the incident beam direction \( \mathbf{k}_i \) is pivotal as it reduces greatly the solution space for the probe and therefore realizes the actual, simultaneous reconstruction of the probe and the object. To motivate this assumption, we note that typical NA for a focusing optics is a few \( 10^{-4} \) at hard x-ray energies, hence resulting in typical depth-of-focus (DOF) of a few 100 \( \mu \text{m} \), as given by the relation

\[
\text{DOF} = \lambda / (2\text{NA}^2).
\]

In Bragg ptychography applications, the maximal thickness of the sample is about 2 \( \mu \text{m} \), a limit set by the longitudinal coherence length of the x-ray source. Along the beam direction, the length \( L \) of the probe-object intersection volume, enclosed by the dashed green parallelogram in Fig. 1a, is

\[
L = T / \sin(\theta_B) + W / \tan(\theta_B), \text{ which is on the order of a few microns. At this scale, the probe invariance along its propagation direction can be safely assumed. In each iteration, this beam propagation constraint is enforced in the current probe estimate, by the projection/back-projection operator pair shown in Eq. 6b.}
\]

The spatial extension of the probe is directly connected with the angular sampling step-size \( \Delta \theta \). From the numerical point of view, as shown in Fig. 1a, this step-size is driven by the
window size in the reconstruction frame \textit{(i.e.}, the orange rectangle, determined by the sampling in the measurements), which has to be bigger than the intersection volume (the green lines), see Fig. 2a. Therefore, using Eq. 2, the angular step $\Delta \theta$ has to meet the following requirement:

$$\Delta \theta \leq \frac{\lambda}{2 \cos(\theta_B) \left[ T \cos(2\theta_B) + W \cos(\theta_B) \right] }$$  \hspace{1cm} (9)

However, in practice, the angular step-size can be designed so that it is bigger than the sampling requirement $\Delta \theta$ along the rocking curve, without losing information. In this case, to preserve the real to reciprocal space conjugation relation, one can numerically insert virtual angular sampling points between the measured angles to access an effective angular step that meets the Nyquist sampling requirement. Only the measured angles are then used for the intensity constraint during the reconstruction, and the virtual ones are left free. Using this approach, one can build a \textit{corrected} field $\Psi'_j$ for all diffraction planes, from the following equation\textsuperscript{63}

$$\Psi'_j(q) = \left[ |\Psi_j(q)| + w(q) \times \left( I_j^{1/2}(q) - |\Psi_j(q)| \right) \right] \frac{\Psi_j(q)}{h_j^{1/2}(q)}$$  \hspace{1cm} (10)

where the mask function $\omega(q)$ is set to 1 for the measured angles and 0 for the virtual angles, \textit{i.e.}, the algorithm is left to retrieve the diffracted field for those virtual angles. In our data-set, the angular up-sampling implemented \textit{via} Eq. 10 was used, with an angular up-sampling ratio set to 3, which corresponds to the insertion of two virtual angles between every two angular measurements within the reconstructed dataset. This gives an effective angular step of 0.0017° and an effective window size of 5 $\mu$m for the probe reconstruction along $p_1$. From a computational viewpoint, we note that the updates (Eq. 5 and Eq. 8) were implemented \textit{via} a modified 3D Fourier transform recently developed.\textsuperscript{39,67} This transform allows the unknown quantities to be retrieved in an orthogonal frame determined by the detector plane and the exit beam direction $k_f$ while still preserving the statistics of the measured signal, \textit{i.e.}, there is no need to interpolate the measurements acquired in a non-orthogonal frame (see Fig. 2a).

3 - Inversion parameters

For the reconstructions, a sub-region of 200×160 pixels, centered around the Bragg peak, was cropped out from the full-size measurements. In the reconstruction frame, it results in pixel sizes of 13.6×24.6×42.3 nm$^3$ ($r_1\times r_2\times r_3$). When converted into the object frame, the pixel size was equalized to 18.9 nm \textit{(i.e.,} the pixel size along $z$). In the detector plane, the gap between
different sensor modules was masked out. We set the support-thickness regularization to
\[ \mu_j := \mu \times |P(r - r_j)|^2_{\text{max}} \] with \( \mu = 0.02 \) and a first set of 1000 updates were performed with
of Eq. 5 and Eq. 8, where we set \( \alpha = 1, \beta = 0.5 \) and \( \gamma = 0.1 \). The initial guess was a flat
field with a constant value of 0.1 for the background, and a pre-characterised illumination
obtained from forward ptychography for the probe; we note that this initial probe is not a
necessity for the convergence of the joint reconstruction (alternatively, other illumination
functions were used as an initial guesses and successful reconstructions could be robustly
produced, see Supplementary information S1). As a commonly used strategy, the probe was
kept unchanged for the first 5 iterations. Since the illumination has a restricted NA easily
estimated from the sum of all the forward ptychographic diffraction patterns, we also
constrained the probe reconstruction in the Fourier space with a rectangular mask of 1.5 times
the estimated NA size for each iteration (until 1000 iterations). The initial guess for the
sample was a uniform slab. As the surfaces of the tungsten crystal foil were not parallel to the
translational scan plane, the slab was tilted by \( 5.5^\circ \) around \( x \) axis and by \( -2^\circ \) around \( y \) axis
with right-handed rule (angles are estimated from some preliminary reconstructions). With
the angular tilt applied, during the reconstruction, the slab thickness was enlarged in three
successive steps: the thickness was set to \( 0.5 \mu m \) for the first 100 iterations, then increased to
\( 0.7 \mu m \) for another 100 iterations, and finally increased to \( 0.9 \mu m \) for a third set of 100
iterations. This strategy helps in the early convergence of an accurate probe. An increasing
thickness also accounts for any wrong estimation of the angular tilt and the irregular (larger)
thickness of the crystal at the bottom part along \( y \). Eventually, this also ends-up with a
support too large in many places, which is detrimental in the end. This problem was solved
with a final support refinement, similar to shrink-wrap.\(^{68}\) from 300 to 500 iterations, the
voxels in the object whose amplitude is lower than 15% of the maximum amplitude of the
object reconstruction were set to zero every 5 iterations. After 500 iterations, the support
function is fixed to the end of the reconstruction. Finally, a last step was performed, with 500
updates of the maximum likelihood algorithm with Gaussian noise model.\(^{33}\) The whole
inversion is performed on a NVIDIA high power computer (4 GPUs) and lasts for about 24
hours.
For the reconstruction using a known, fixed probe, we followed the same procedures as
above, except that the probe was not updated, and the angular up-sampling was not
introduced.
The strain and tilts can be extracted from the 3D phase profile. They are derived as:

\[
\begin{align*}
\varepsilon_{zz}(\mathbf{r}) &= |\mathbf{Q}_{220}|^{-1} \times \frac{\partial \phi_{220}(\mathbf{r})}{\partial r_z} \\
\omega_x(\mathbf{r}) &= \arcsin\left[|\mathbf{Q}_{220}|^{-1} \times \frac{\partial \phi_{220}(\mathbf{r})}{\partial r_x}\right] \\
\omega_y(\mathbf{r}) &= \arcsin\left[|\mathbf{Q}_{220}|^{-1} \times \frac{\partial \phi_{220}(\mathbf{r})}{\partial r_y}\right].
\end{align*}
\] (11)

where \( \varepsilon_{zz} \) is the strain along the \( z \) axis, which is coincident with \( \mathbf{Q}_{220} \), \( \omega_x \) is the lattice rotation about \( x \) axis and \( \omega_y \) the lattice rotation about \( y \) axis. As seen in Fig. 3, the extracted phase values are wrapped between \([-\pi, \pi]\). This wrapping would cause discontinuities when calculating the phase derivatives \( \partial \phi_{220}(\mathbf{r})/\partial \mathbf{r} \). To avoid this, the extracted phase needs to be unwrapped. However, phase unwrapping is problematic with the presence of dislocation defects due to their characteristic phase vortices.\(^{27,42}\) Hence, the phase derivative was calculated using\(^{69}\)

\[
\frac{\partial \phi_{220}(\mathbf{r})}{\partial \mathbf{r}} = \arg\{\exp[i \phi_{220}(\mathbf{r})] \times \exp[-i \phi_{220}(\mathbf{r} - \mathbf{r}^*)]\}/r^*,
\] (12)

which does not require the phase to be unwrapped.

Supplemental Figures S1 to S7 are provided as Supplementary information.

Supplementary Videos SV1 to SV3 are provided as separate files.

Acknowledgments: The BP-CDI experiment was performed on beamline ID01 at the European Synchrotron Radiation Facility (ESRF), Grenoble, France. This work received funding from the European Research Council (European Union’s Horizon H2020 research and innovation program grant agreements No 724881 and No 714697). The authors acknowledge use of characterisation facilities within the David Cockayne Centre for Electron Microscopy, Department of Materials, University of Oxford. Micro-beam Laue Diffraction experiments used the Advanced Photon Source, a US Department of Energy (DOE) Office of Science User Facility operated for the DOE Office of Science by Argonne National Laboratory under Contract No. DE-AC02-06CH11357. Ion implantations were performed at the Ion Beam Centre at the University of Surrey as part of United Kingdom Engineering and Physical Sciences Research Council grant EP/H018921/1. We are grateful to Hongbing Yu for help with sample preparation and Edoardo Zatterin for help during experiments. Vincent Favre-Nicolin is warmly acknowledged for his help during experiments and for fruitful discussions.

Author Contributions
VC designed the project. PL developed the inversion strategy, wrote the code, inverted and analysed the data. FH and NP designed and prepared the samples and the synchrotron based experiments. All authors were involved in the coherent diffraction experiment at ESRF.
Micro-Laue and EBSD characterisation were performed by FH and NP. VC and PL wrote the manuscript with the help of all co-authors.

**Author Information**

The authors declare no competing financial interest.

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**Code availability**

Matlab routines for Bragg ptychography are available on demand by contacting the corresponding author.

**Data availability statement**

The experimental data that support the plots within this paper are available on demand by contacting the corresponding author.
Figures and Figure captions

Figure list:

Figure 1 - X-ray diffraction microscopy of the He-implanted tungsten foil
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Figure 1 – X-ray diffraction microscopy of the He-implanted tungsten foil. (a) Scanning electron microscopy images of the tungsten foil sample. The platinum protection layers, helium-implanted layer and the grain domain boundary are indicated. The red rectangle shows the extreme probe positions used during the ptychography raster scan (noted as ‘1’, ‘20’, ‘381’ and ‘400’, in agreement with their order within the full scan), while the dashed yellow rectangle corresponds to the area finally imaged with BP. (b) Experimental setup for BP at the synchrotron beamline, detailing the main components used to condition the beam and detect the diffraction signal. The sample frame (x, y, z) is defined. (c) Example of a diffraction pattern, obtained at the maximum of the Bragg peak, at position 20. (d) A series of four diffraction intensity patterns integrated over all angles along the rocking curve, plotted for the four extreme positions of the raster scan. For (c, d) the used logarithmic colour scale is indicated.
Figure 2 - Bragg ptychography and sampling principles. (a) A finite size beam illuminates a crystalline thin film sample in Bragg geometry, while a 2D detector collects the coherent diffracted pattern, so that the incident and exit wave vectors (\(k_i\) and \(k_f\), respectively) fulfill the Bragg condition. The 3D information is acquired by rotating the sample in the vicinity of the Bragg angle \(\theta_B\), along the rocking curve, i.e. at the \(\theta_1 \ldots \theta_N\) positions. The intensity information is recorded as a function of \((q_1, q_2, q_3)\), the components of the wave-vector transfer \(Q\). Using a recently developed formalism\(^{39}\), this sampling depicts a direct space frame \((r_1, r_2, r_3)\). It implies some sampling rates along the three directions, to ensure that the illuminated volume (delimited by the green line) is contained within the numerically retrieved direct space (shown as an orange rectangle). The probe frame is also defined as \((p_1, p_2, p_3)\). (b) A typical hard x-ray probe profile presented in its focal plane and (c) numerically propagated around the focal plane (see Methods Sec. 2). The dashed rectangle corresponds to the accessible region based on the parameters used during the acquisition of the BP data set. The full extent of the probe, visible outside this rectangle, is only retrieved through the implementation of the angular up-sampling approach. Along the propagation direction, note the probe invariance over distance as large as a few hundreds of micrometres. (d) Sufficiently small sampling steps of \(\Delta \theta\) are needed to fulfill the numerical sampling relation. This relation is depicted in the plot, as a function of the probe size \(W\) and the sample thickness \(T\).
Figure 3 – Extended field of view retrieved image. (a) 3D density and (b) phase plots, retrieved with the proposed BP approach. The grey volume represents an iso-surface based on the recovered object density, while the slices show the internal structure of density and phase, respectively. The field of view extends over 0.6 x 6 x 6 μm³. (c, d) Cross sections of the crystal density and phase, respectively, shown over the plane indicated in red in (a) and (b), and (e) the associated retrieved probe. (f, g) 3D density and phase reconstructions, respectively, obtained from the same data set, using the former BP approach. Note the strong reduction of the field of view (evidenced by the dashed rectangle) and the degradation of the image quality. (h) Probe cross-section used for this second reconstruction, limited along p₁ according to the conjugation relations applied to the experimental parameters. As the probe is kept fixed during this process, the probe profile is extracted from another separated and dedicated probe reconstruction (Methods Sec. 2). The colour scale for the density, phase and probe (hue rendering) are indicated on the plots.
Figure 4 - Strain and tilts revealed in the He-implanted polycrystalline tungsten foil. (a) 3D iso-surface density plot overlaid with the $\varepsilon_{zz}$ strain map. Three planes used for further investigations are indicated as red rectangles. (b) Cross-sections of the $\varepsilon_{zz}$ map extracted over the planes indicated in (a). (c) 3D iso-surface density plot overlaid with the $\omega_y$ lattice rotation around $y$-axis. (d) Cross-sections of $\omega_y$ extracted over the planes indicated in (c). (e, f) Same as (c, d) for the $\omega_x$ lattice rotation around $x$-axis. (g) Left zoomed-in 3D iso-surface density plots of the dislocation #1 highlighted by a white rectangle in the implanted layer, shown in (b, d, f), middle 2D cross-section map of the $\phi_{220}$ reconstructed phase and right estimated phase variation resulting from simulation (see Supplementary information S6). (h) Zoomed-in view of the $\varepsilon_{zz}$ strain map extracted from the implanted region in the vicinity of the grain boundary (as shown in (b)). (i, j) One-dimensional cross sections of the $\varepsilon_{zz}$ strain map, along the implantation direction and across the film thickness, respectively, as indicated in (b). In (i) the grey area corresponds to a region where $\varepsilon_{zz}$ is slightly degraded due to a bit of parasitic aliasing along the $y$ direction. All scale bars and angular colour scales in radian are indicated on the plots.
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\[ \varepsilon_{ZZ} \]

\[ \omega_y \]

\[ \omega_x \]

\[ h_i \]

\[ 3 \times 10^{-4} \]

\[ \pi \]

\[ 0.2 \mu m \]

\[ 0.2 \mu m \]