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1 Strain profiles in ion implanted ceramic polycrystals: a new 2 approach based on reciprocal-space crystal selection

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19 20 **Abstract**

21 The determination of the state of strain in implanted materials is a key issue in the study of their mechanical
22 stability. Whereas this question is relatively easily solved in the case of single crystals, it remains a challenging
23 task in the case of polycrystalline materials. In this paper, we take benefit of the intense and parallel beams
24 provided by third generation synchrotron sources combined with a two-dimensional detection system to analyze
25 individual grains in polycrystals, hence obtaining “single crystal – like” data. The feasibility of the approach is
26 demonstrated with implanted UO₂ polycrystals where the in-depth strain profile is extracted for individual grains
27 using numerical simulations of the diffracted signal. The influence of the implantation dose is precisely analyzed
28 for several diffracting planes and grains. It is shown that, at low fluences, the development of strain is mainly

29 due to ballistic effects with little or no effect from He ions, independently from the crystallographic orientation.
30 At higher fluences, the evolution of the strain profiles suggests a partial and anisotropic plastic relaxation. With
31 the present approach, robust and reliable structural information can be obtained, even from complex
32 polycrystalline ceramic materials.

33

34 **Main Part**

35 Ion implantation has found many applications in material science encompassing for example the optimization of
36 the mechanical properties of steel¹, doping² or spatial organization of nano-objects^{3,4} for microelectronics, and
37 the study of nuclear materials under irradiation^{5,6}. The alteration of the crystal lattice (i.e. defect creation) due to
38 ion implantation leads to significant strain, which has been extensively studied in the last decades with the view
39 to investigate mainly the mechanical stability of implanted systems (i.e. margins with respect to fracture). In the
40 standard case where grain size is much larger than the implanted thickness⁷, it is shown that the component of
41 the strain tensor normal to the sample surface (referred as to ε_{zz} in the following), is the most important one. ε_{zz}
42 is mostly confined in a subsurface layer which thickness depends on the ion energy, and is often heterogeneously
43 distributed along the surface normal.

44 In the case of single crystals, the strain profile can be determined using dark field electron holography⁸ or
45 straightforwardly using high-resolution X-ray diffraction (XRD) on laboratory equipment. In the latter case, the
46 measured high-resolution XRD patterns exhibit strong oscillations in the vicinity of the diffraction peak resulting
47 from the interferences between iso-strain regions from which the phase of the diffracted amplitude (hence the
48 strain profile) can be retrieved^{9,10,11,12,13,14}.

49 For polycrystals, powder diffraction with focused X-ray beam is widely used to collect the diffraction pattern.
50 While such an approach, combined with a structural refinement analysis based, for instance, on the Rietveld
51 method^{15,16}, is efficient for retrieving averaged quantities (average strain, root-mean-squared strain for instance)
52 it is absolutely inefficient to determine the spatial variations of these quantities, although the knowledge of the
53 spatial gradients is essential for the understanding of underlying physical mechanism responsible for structural
54 modifications in implanted materials.

55 In this paper, we demonstrate that the in-depth strain distribution $\varepsilon_{zz}(z)$ can be accurately determined in
56 individual grains of a polycrystal using synchrotron radiation based XRD coupled with numerical simulations.
57 The potential of this approach is illustrated with He implantations in uranium dioxide (UO₂) polycrystals. This
58 system has been widely investigated within the framework of spent nuclear fuel storage in dry conditions^{17,18,19}.

59 Under such conditions, He is produced as a result of the α -decay of radionuclides created during in-reactor
60 irradiation.

61 The UO₂ polycrystals considered for this study are disks of 8 mm in diameter and about 1 mm thick. They
62 exhibit a 9 μm grain mean size and do not show any preferred crystallographic orientation^{7, 20}. A virgin sample
63 has been kept as a reference and three others have been implanted with 60 keV ⁴He⁺ ions with fluencies ranging
64 from 1 up to 6×10^{16} ions/cm². This corresponds to damages from 0.4 up to 2.3 displacements per atoms (dpa) as
65 determined using the SRIM Monte-Carlo simulation with 40 and 20 eV displacement energies for U and O
66 atoms respectively²¹. The thickness of the implanted layer is about 0.23 microns. For fluences up to 2×10^{16}
67 ions/cm², strains in the implanted layer have been extensively characterized and a mechanical model has been
68 validated using Laue micro-diffraction measurements^{7, 22}.

69 XRD patterns were collected on the BM02/D2AM diffraction beamline at the ESRF (Grenoble, France) with a
70 8.3 keV monochromatic and 1×0.3 mm² (horizontal \times vertical) quasi parallel incoming X-ray beam (divergence
71 was 9 and 5 mrad in the horizontal and vertical directions respectively)²³. Diffraction data were recorded using a
72 XPAD3 2D detector (560×120 pixels with a 130 μm pixel size)²⁴, mounted on the delta arm of the kappa
73 diffractometer 1 m away from the sample.

74 This experimental set-up offers the possibility to isolate the Bragg spots coming from individual grains (see
75 Figure 1(a)), the angular resolution of the set-up being much higher than the misorientation between grains. This
76 approach has been applied to our implanted samples around four different UO₂ Bragg reflections (i.e. (220),
77 (311), (222) and (400) covering the elastic anisotropy range). For each reflection, the sample is rocked through
78 the Bragg's law setting over a range wide enough to include all the scattering from the implanted layer. With the
79 2D detector used in this work, a complete 3D reciprocal space map (RSM) is obtained in a few minutes only,
80 potentially containing the diffraction from several *well separated* grains (see Figure 1(b)). 1D intensity profiles
81 normal to the surface are obtained for several grains by extracting the intensity along a one pixel wide line
82 parallel to the out-of-plane component of the scattering vector (Q_z). These 1D patterns have been then simulated
83 to determine strain profiles using a procedure initially developed for ion implanted single crystals. Briefly, this
84 methodology is based on the dynamical theory, uses B-spline function to model the strain profile and a simulated
85 annealing algorithm as an optimization procedure¹³. The comparison between the measured and simulated 1D
86 patterns is given by Figure 2 for a (220)-oriented grain and increasing fluence. It can readily be observed that the
87 agreement between the model and the data is close to perfect over the whole intensity dynamic range. The
88 corresponding strain profiles are displayed in the inset. For each Bragg reflection we analyzed 4-6 different

89 grains in order to check the reliability of the retrieved strain profiles. The ε_{zz} strain profiles (for grains oriented
90 along (220), (222) and (400)) are shown in Figure 3(a). For each crystallographic orientation, the maximum
91 deviation observed over all grains analyzed (represented as error bars in Figure 3(b)) turns out to be limited. It
92 can hence be concluded that the other characteristics of a given grain (size, thickness²⁵) do not significantly
93 influence the ε_{zz} strain profile.

94 Comparing strain profiles measured on grain with different orientations, it can firstly be mentioned that they all
95 follow the same general trend: sharp increase in the first 0.05 μm (below the sample surface) up to plateau that is
96 observed up to about 0.27 μm then a decrease of ε_{zz} down to zero at about 0.45 μm . For depth higher than 0.45
97 μm , ε_{zz} is systematically zero. Secondly, however, the values of strains in the [0.05-0.27 μm] depth range
98 strongly depend on grain orientation. This is consistent with previous XRD measurements^{20,22} on these samples.
99 This behavior can be rationalized by considering the linear free swelling instead of the ε_{zz} strain²². The free
100 swelling corresponds to the isotropic swelling induced by the implantation without any mechanical reaction from
101 the non-implanted substrate, which can be calculated on the basis of the elastic constant of the material. The free
102 swelling profiles are compared in Figure 3(b). An excellent quantitative agreement can be seen, within the
103 experimental error bars, which proves that the swelling is independent of the crystallographic orientation. Finally
104 these profiles are compared with the calculated damage and He concentration profiles. Conclusions are two-fold.
105 Firstly this suggests that the swelling is not caused in this case by the presence of implanted hexogen ion
106 (contrary to Si implanted with H^+ ions^{26,27} for instance), this maximum of the He concentration profile being
107 shifted by about 0.05 μm deeper in the UO_2 implanted matrix with respect to the free swelling profile. Secondly
108 the similarity between the damage profile and the free swelling profile (namely for depth in the [0.2; 0.4 μm]
109 range) suggests that the latter mainly originates from ballistic effects, since only this energy loss mechanism is
110 taken into account in the damage profile calculation. Moreover a contribution to the swelling of the electronic
111 energy loss mechanism in the [0; 0.2 μm] depth range can not be excluded.

112
113 We now consider the materials implanted at higher fluencies i.e. 4 and 6×10^{16} He ions/ cm^2 (i.e. 1.2 and 2.3 dpa
114 respectively). Conventional powder diffraction evidenced a dramatic decrease of the intensity diffracted by the
115 implanted layer²⁸, which prevented any accurate strain analysis. Debelle *et al.* observed a similar behavior using
116 HR-XRD on a (100) UO_2 single crystal implanted in very close conditions and concluded to a plastic relaxation¹⁹
117 with no residual elastic strain. On the contrary, in the case of UO_2 pellets doped with short lived α -emitter
118 (²³⁸Pu), Wiss *et al.* suggested that the linear free swelling increases with damage up to a saturation (at 2.5 dpa),

119 i.e. no relaxation was found²⁹. This discrepancy is even more puzzling considering that all three studies provide
120 very similar variations of free swelling with damage for lower damage values (below 0.8 dpa)²². Using the same
121 experimental approach, ϵ_{zz} strain profiles were determined for the four orientations ((220), (311), (222) and
122 (400)). Figure 2 shows the evolution of (220) reflection for increasing fluence. Their comparison confirms our
123 previous laboratory characterizations i.e. a strong decrease of the diffracted intensity especially for the highest
124 strained part of the implanted layer which corresponds to the lowest 2θ values. However in these synchrotron
125 characterizations, because of the much higher signal to noise ratio, oscillations are clearly observed (see Figure
126 2). Three main conclusions can be derived from this figure. Firstly with increasing implantation fluence, ϵ_{zz}
127 keeps on increasing to reach locally very high values of 1.86 and 3.15% (for 4 and 6×10^{16} He ions/cm² final
128 implantation dose respectively). The maximal strain values are observed at a depth of about 0.2 μm which also
129 corresponds to the maximum of the calculated damage profile. Moreover it seems that the increase in ion
130 implantation dose mainly affects this depth (located at 0.2 μm below the surface) and not the full implanted
131 layer: the strain levels in the other parts of the implanted layer are less affected. Secondly it must be mentioned
132 that the thickness of the implanted layer does not significantly vary with ion implantation dose: ϵ_{zz} remains zero
133 for depth higher than 0.45 μm . This room temperature behavior seems to be specific to UO_2 or at least different
134 from what has been observed in ion implanted zirconia for example³⁰. Finally, it can be shown that the elastic
135 mechanical model developed for low ion implantation dose (below 0.8 dpa) is not valid for these higher ion
136 implantation doses: it turns out that the free swelling profile do not superimpose for the different crystallographic
137 orientation probed (see Figure 4). In particular, pseudo-free swelling values measured in the (222) oriented
138 grains, are systematically significantly higher than the one measured for (400) and (220) grains. Moreover such
139 an anisotropic strain could explain the embrittlement observed in aged self-irradiated $^{238}\text{PuO}_2$ pellet, in which
140 incompatible grain deformation could have occurred³¹.

141 To sum-up on the mechanical behavior of He implanted UO_2 polycrystals at high doses (higher than 0.8 dpa),
142 this work contradicts the hypothesis of a full plastic relaxation¹⁹: high local elastic strains are found to remain
143 (together with a strong increase of structural disorder, data not shown here) at high fluences. Moreover it is also
144 found that a free swelling cannot be deduced anymore for ion implantation experiments in single or polycrystals
145 which makes impossible any discussion regarding the saturation of free swelling measured in UO_2 pellets doped
146 with ^{238}Pu ²⁹.

147 To conclude on this work, a very promising methodology has been proposed to characterize out-of-
148 plane strains in ion implanted ceramic polycrystal with an excellent accuracy. The intense and parallel beam of

149 synchrotron radiation permits the analysis of single grains, hence providing the same type of information as
150 studies conventionally performed on single crystals. This approach has been validated on UO₂ polycrystal
151 implanted with He ion up to an intermediate ion dose (0.4 dpa) and has enabled an accurate definition of strain
152 profiles at higher doses (1.2 and 2.3 dpa), which was not possible using standard analyses.

153 This methodology should find a wide range of applications: it shows that strain profile analysis does not require
154 systematic use of single crystals. This is all the more interesting that ceramic single crystals often exhibit a
155 mosaïcicity .i.e. they are made a large domains with a slight (below 1°) misorientation. If the interest of the work
156 is demonstrated by the analysis of 1D XRD patterns, the analysis of the full reciprocal space scattering (2D³² and
157 ultimately 3D) should be very helpful for determining the size of extended defects in polycrystals implanted in
158 severe conditions (high energy ions³³ and/or high damage values³⁴). Finally this work can be considered as first
159 step towards the use of coherent X-ray diffraction to characterize strains in ion implanted polycrystals³⁵.

160

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164 committees for beamtime provision (MA-2069 and 02-02-822).

165

166 **Figure Legends**

167 **Figure 1: High resolution XRD data collected on a single grain of UO₂ polycrystal implanted**
168 **with 10¹⁶ He/cm².** (a) Schematic representation of the experiment set-up. (b) 3D reciprocal space
169 maps of 8 different Bragg reflections measured on 8 different (400) grains within a single data
170 collection.

171 **Figure 2: Measured and calculated high resolution XRD study of a single (220) grain within**
172 **three UO₂ polycrystals implanted with 1, 4 and 6×10¹⁶ He/cm² respectively.** The inset shows the
173 associated profiles of ϵ_{zz} out-of-plane strain.

174 **Figure 3: Influence of grain orientation on the profiles of both strains (a) and free swelling (b).**

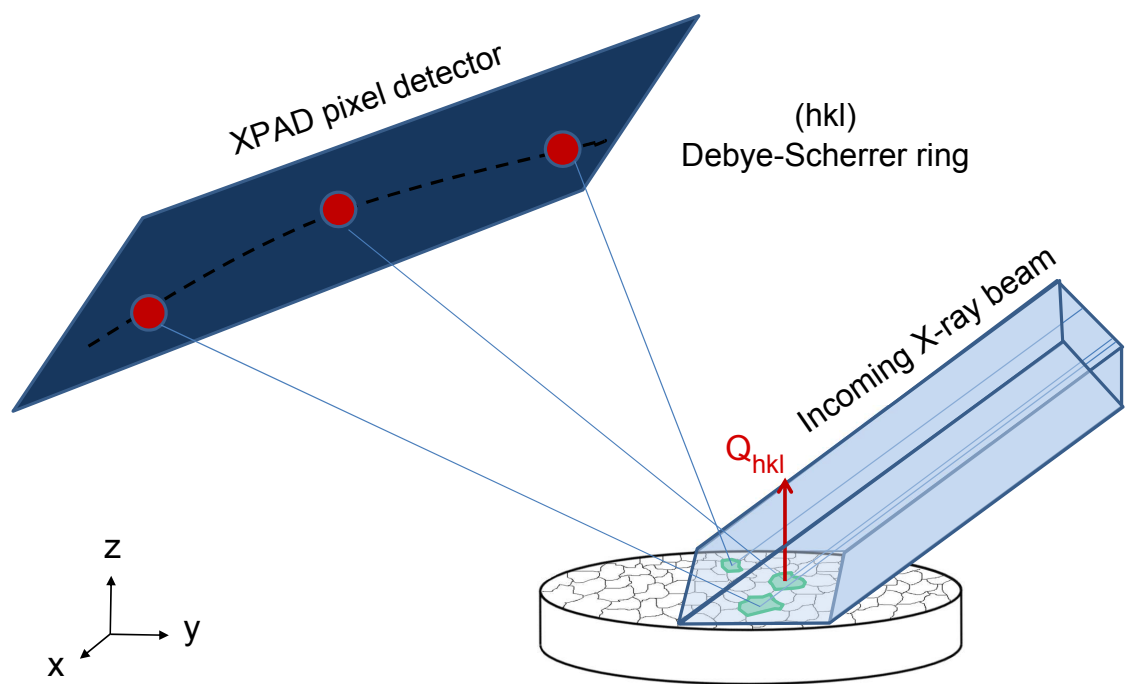
175 **Figure 4: “Pseudo” linear free swelling variations with grain orientation at high implantation**
176 **dose (i.e. 4 and 6×10¹⁶ He/cm²) for a UO₂ polycrystal.** The word “pseudo” is used to underline the
177 impossibility to obtain a single free swelling value relevant for all grain orientations inside a given
178 UO₂ polycrystal implanted at such high doses.

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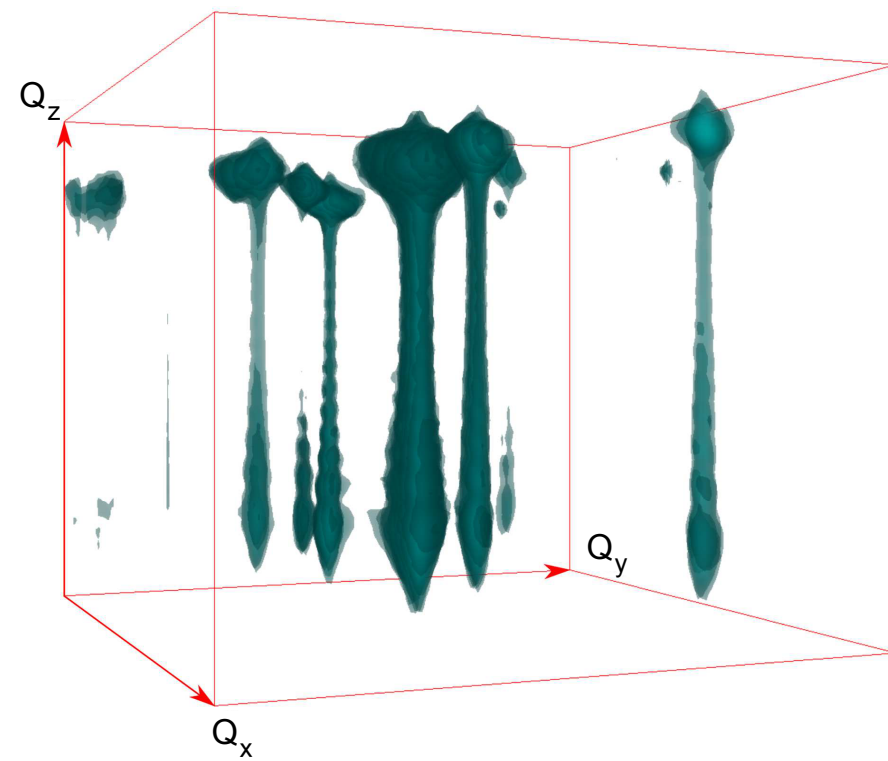
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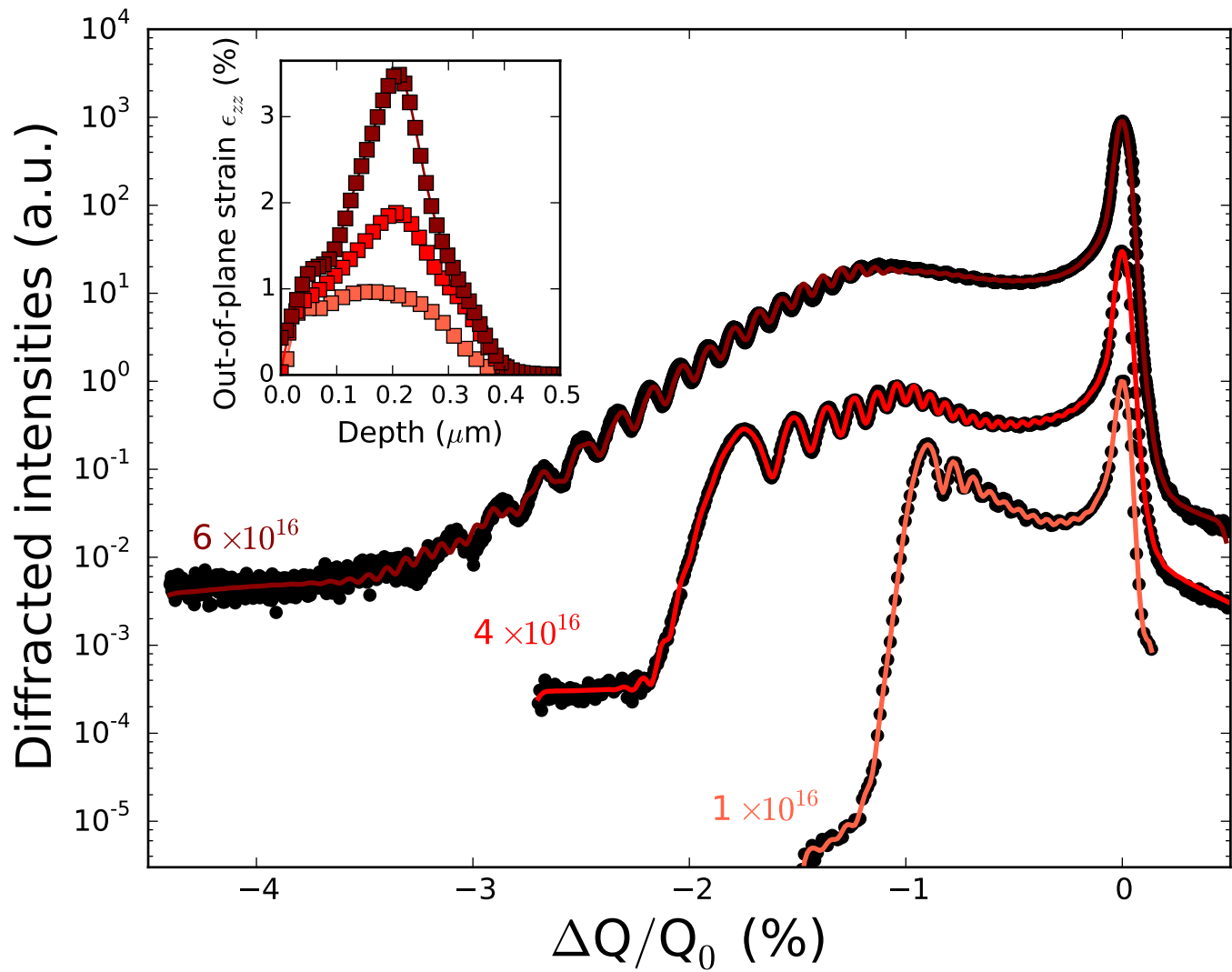
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(a)

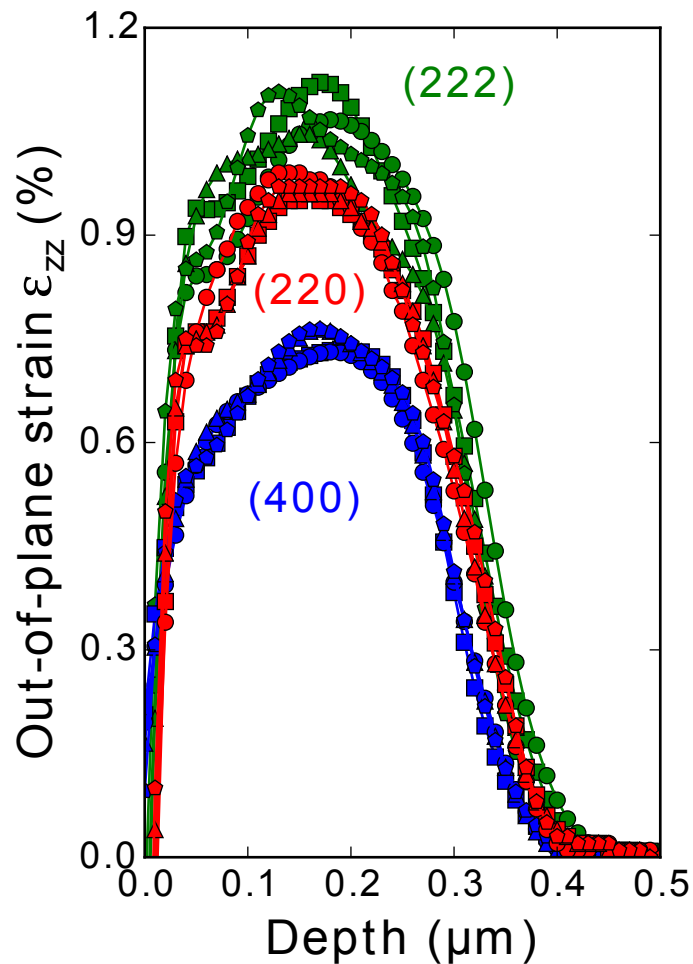


(b)





(a)



(b)

