HIGH-RESOLUTION MICRODIFFRACTION STUDY OF NOTCH-TIP DEFORMATION
IN Mo SINGLE CRYSTALS USING X-RAY SYNCHROTRON RADIATION

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Abstract

A new technique is presented for the determination of strain fields in single crystals, based on the simultaneous recording of the energy and position of a diffracted beam, with a resolution of 25 micrometers under current experimental conditions. The technique can be profitably used for perfect to highly deformed crystals, in materials as highly absorbing as Molybdenum, and allows a spatial resolution of one part in 10^4 . Indications are given as to possible refinements and improvements of the method.

I. INTRODUCTION

The determination of the strain field in the proximity of discontinuities and stress concentrators with high spatial resolution is a topic of traditional interest in materials science. The study of many phenomena, such as fatigue behaviour, crack microplasticity or the dislocation-free zone, heavily rests on the theoretical tools of continuum mechanics. These approaches, however, tend to be of dubious applicability in the dimensional ranges where the phenomena themselves originate. More modern approaches, such as computer simulations, are forced, on the other hand, to attack the problem at a scale probably too fine to be of technological interest. There is a whole range of characteristic dimensions, from a few hundred angstroms to approximately ten microns, where theoretical prediction of mechanical behaviour is extremely arduous. The importance of experimental methods capable of sampling phenomena occurring on this scale is therefore quite relevant. Several techniques are presently capable of providing information about the deformation field in stressed materials. We have devised a novel one, which seems quite promising both from the spatial resolution point of view, and for its applicability to technologically relevant materials and conditions. This new method avails itself of the high intensity and brightness available at x-ray synchrotron installations, and of the recently developed technologies which have made available pinholes in the micron range.

The aim of the present study is to show that, by coupling x-ray synchrotron radiation and micron-range pinholes, it is possible to achieve high-resolution determination of strain fields in highly deformed single crystals, with a sensitivity of one part in 10^4 . The experiments described in this study probed the bulk properties of technologically relevant materials (Mo), which had up to now proven ill-suited for x-ray investigations because of their high

absorption coefficients.

In order to put the existing strain-field mapping techniques in perspective, and to clarify the advantages and limitations of the new method, a brief outline of the currently available experimental methods to measure deformation fields with high spatial resolution will be given below.

II EXPERIMENTAL

1. Existing experimental techniques for high-resolution mapping of strain fields.

Several techniques are presently available for high-resolution mapping of strain fields. The most straightforward approach to measuring the strain field in a single crystal is a point-by-point rocking curve mapping. By use of a double-crystal diffractometer lattice misorientations as small as 10 arcsec can be analyzed. The work of Liu et al. (Liu, Weng and Weissman, 1982) is a typical example of this kind of experiment. With standard x-ray sources, however, the maximum brightness (flux/mm² rad²) obtainable is such that collimating devices with apertures not smaller than 10⁴ micron² have to be used for the rocking-curve measurements (Stock et al. 1987, Ice, 1986). Present limitations in x-ray optics, on the other hand, make it impossible to create demagnified images smaller than approximately 100 microns. With this spatial resolution, the crucial information about phenomena such as notch micro-plasticity is almost completely integrated over the sampled volume. To give a concrete example, in the above-referred-to work by Liu et al. (Liu et al, 1982), the spatial resolution obtained was of 300 microns, more than ten times larger than the resolution achieved in the present work.

As for traditional topographic methods, single-crystal and double-crystal

techniques should be mentioned, amongst the direct methods. For x-rays the angular width of reflecting range around a typical reciprocal lattice point is of the order of 10^{-5} , whereas for 100 KeV electrons it is approximately 10^{-2} , which explains why the electron microscopy technique, unrivalled as far as spatial resolution is concerned, has very poor strain sensitivity. Focussing attention, for concreteness, on the displacement field of a dislocation, which falls off asymptotically as the inverse of the distance from the core, regions of the crystal farther and farther from the dislocation core will produce image contrast as the reflecting range becomes narrower and narrower. The rocking curve width, in turn, is inversely proportional to the scattering factor. As a result, high-order weak reflections have a very narrow reflecting range (high-sensitivity to lattice misalignments) and will give rise to wide dislocation images. Single-crystal techniques, apparently sensitive to strains of the order of 10^{-5} , are in reality quite insensitive (Tanner, 1976) to slow variations in the lattice parameter, since the angular divergence of the x-ray beam causes the diffracted intensity to be equivalent to an integrated intensity. The image is therefore not observed to change until the lattice parameter variations become of the order of one part in 10^3 . Double crystal techniques (Bond and Andrews, 1952, Bonse and Kappler, 1958) in the (+,-) parallel setting (Tanner, 1976) become extremely sensitive to rotations, or misorientations, of the diffracting planes, since the angular range over which simultaneous diffraction occurs can be as low as 0.1 arcsec. Using this technique, Hart (Hart, 1968) found, in the canonical Si, variations of lattice parameter of the order of one part in 10^7 . However, for high sensitivity it is essential that the same material should be used for the reference and the specimen crystal, and that the relative twist between the reflecting planes of the two crystals should be as low as possible. The images of the long-range displacement field of dislocations in this kind of topograph are very broad (poor spatial resolution) and even with

dislocation-free crystals the image arises from both the reference and the specimen crystal. For this reason samples with dislocation densities higher than 10^5 dislocations/cm² are not suited for this kind of technique. It should be remembered that it is quite common that, for many materials of technological relevance, this concentration is already exceeded in the crystal-growth process, even prior to any deformation. Thus for materials of technological interest, so-called indirect topographic methods are needed, such as absorption-edge analysis or equi-inclination contours. (Stock, 1987) With the latter techniques, monochromatic radiation is used to map the position of an equi-inclination volume as a function of the rotation of the crystal with respect to the incoming beam. Similar contours can be produced with white radiation orienting the deformed crystal in such a way that it can diffract a range of wavelengths on either side of the absorption edge of an element present in significant amounts in the specimen crystal.

As far as equi-inclination contours are concerned (Stock, 1987), the volume of the crystal that will give rise to narrow bands of enhanced intensity is constituted by that portion of the crystal whose misalignment is of the order of the intrinsic rocking-curve width ($\Delta k/k = 10^{-4}$ for a typical strong reflection). This sets an upper limit for the strain sensitivity. The best spatial resolutions obtained so far are of the order of 40 microns, in the most favourable cases.

The technique reported in the present work has its domain of applicability in the study of perfect to highly deformed crystals, with a spatial resolution at present of 25 microns, but susceptible to be pushed down into the ~ 1 micrometer range and with a strain sensitivity comparable to the equi-inclination contours method. When implemented at synchrotron installation it is suited for the study of virtually bulk specimens

($\mu t = 9.5$), where μ is the mass absorption coefficient and t the sample thickness. This last characteristic should render it particularly interesting for experimental observation of the strain field at a dislocation-free zone (Ohr, 1982), up until now only studied in very thin samples via transmission electron microscopy.

2. Description of the White Beam High Resolution Synchrotron Micro-Diffraction Technique

a) General

Given a general strain field in a crystal, its dilational ($\partial u_i / \partial x_i$) and rotational ($\partial u_i / \partial x_j$) components can be obtained from three independent crystallographic reflections. For an incident white beam, in particular, the change in lattice spacing of the diffracting planes will only have the effect of varying the energy of the resulting reflection, without affecting its angular orientation, whereas the rotational component will, in general, modify both the spatial location and the energy of the diffracted beam. If, after choosing a reference point in the crystal with respect to which deformations will be measured, one records the energy and spatial location of the beam diffracted from this point, and one also measures the variation in energy and position of the reflection as a function of location in the sample, both the dilation and the rotation of the diffracting planes can be mapped on a point-by-point basis. Alternatively, instead of choosing a fixed reference point, one can perform a dynamic experiment, by looking at the differential change in energy and position of the diffracted beam before and after, say, the application of a load, again on a point-by-point basis. The azimuthal and 2θ angles (where θ is the Bragg angle,) can be easily measured by means of a suitable slit system, or of a position-sensitive detector. From the

measurement of the wavelength of the reflection one can then obtain the variation in lattice spacing via simple manipulations of Bragg's law:

$$d^* - d_0 = n/2 (\lambda^*/\sin \theta^* - \lambda_0/\sin \theta_0) \quad (1)$$

$$d^*/d_0 = \lambda^*/\lambda_0 (1 + \cotan(\theta_0)\Delta\theta - 1/2 \Delta\theta^2) \quad (2)$$

where superscripted starred quantities refer to the loaded condition, n is the reflection order, and $\theta^* = \theta^0 + \Delta\theta$.

Three different kinds of resolution must be considered in the evaluation of the potentialities of the method: i) energy resolution, (ii) resolution in the location of the position of the diffracted spot, and (iii) collimation of the incoming beam as to sample the smallest possible volume of the sample crystal. If, as described later, one attains a resolution of one part in 10^4 in the energy detector, a similar precision in the spatial location of the Laue spot can be attained if the position of the diffracted beam is determined to within 200-500 microns for a specimen-to-detector distance of approximately 50 cm. The most stringent requirement is set by the collimation of the incoming beam. As discussed at greater length later, this is achieved by means of suitable pinholes that intercept the white beam with apertures in principle only limited by the brightness of the source, and by the Rayleigh criterion. Whereas the first two kinds of resolutions are responsible for the sensitivity of the method - roughly of the order of one part in 10^4 both for the dilational and the rotational component of the strain tensor - it is the third type of resolution that determines the dimension scale over which the quantities of interests will be sampled. What makes the technique particularly attractive is the fact that, although present technological constraints have limited this resolution to 25 microns, the intrinsic

limitations of the technique are in the sub-micron range. For as bright a source as the beam available as a synchrotron installation, in fact, the limit is set by Rayleigh criterion,

$$E \geq 0.122 \lambda \text{ (Å) mrad} \quad (3)$$

where E is the aperture-limited emittance in milliradian micron, and λ is the wavelength in Å. For 1 Å radiation this sets a limit for the spatial resolution of ~ 1 micrometer. Even smaller microprobe beams can be achieved by going to higher x-ray energies (shorter wavelengths); at the Brookhaven NSLS installation 19-C, appreciable signal was detected up to 90 keV.

The technological problems that have, in the present experiments, limited the spatial resolution to 25 microns are connected with the preparation of the pinholes. The following section is devoted to the discussion of this crucial point, and to the description of the experimental set up.

b) Experimental set-up

The intense white-beam radiation available at the Synchrotron Topography Project Hutch X-19C at the NSLS at BNL was collimated in front of the specimen by means of either two pinholes, described in what follows as pinholes 1 and 2. (see Figure 1).

Pinhole 1 had a diameter of 70 microns, and had been laser-drilled in a 250 micron foil; pinhole 2 had an opening of 25 microns, and had been obtained by mechanically drilling a 200 micron Ta foil. Since for both pinholes the aspect ratio was of the order of 1:10, they were mounted in front of the specimen on a double-head goniometer that allowed rotation of the

cylindrically-shaped hole to maximize the output intensity. This angular collimation was very important, because the white beam intensity transmitted through the foil in which the pinhole was drilled was not negligible compared with the intensity through the pinhole itself. The situation could be substantially improved by i) obtaining pinholes through thicker, more highly absorbing materials, and ii) choosing a geometrical arrangement (higher 2θ angles) that would allow the diffraction of softer wavelengths, more effectively absorbed by the Pt or Ta foils. (This second possibility was made impossible in our case by the geometrical constraints imposed by the in-situ tensile stage).

The pinhole on the double-head goniometer was then mounted on a computer-controlled x-y stage, capable of movements down to a tenth of a micrometer. This x-y stage allowed movement of the pinhole in a plane parallel to the surface of the specimen, which, in turn, was oriented in the Laue (transmission) geometry approximately perpendicularly to the incoming beam. With this configuration, the diffracted beam averaged over the entire thickness of the specimen. This should not constitute a problem for experiments, like the present, where the strain field is expected to be uniform over the thickness of the specimens.

The specimen itself was mounted on an in-situ tensile stage positioned right in front of the pinhole, see Figure 2. The specimen was so oriented as to have one of the highest intensity Laue beams produced by the incident white radiation directed into the window of a Si/Li detector. The signal from the detector was then sent to a Multi-Channel Analyzer (MCA). The total intensity reaching the detector originated from the diffraction Laue spot itself, from the fluorescence due to the Mo sample and from the fluorescence caused by any structural material intercepted by the incident or diffracted beams on their

- 11 -

pathways. This total intensity was so high that, in order to ensure linearity in the detector, the incoming white beam radiation had to be attenuated by interposing as much as 2 mm of lead. This lead filtering had the effect of selectively letting through only the harder wavelengths, thus worsening the signal to noise ratio due to the radiation penetrating through the Ta or Pt foil. Since, again, this limitation is strictly determined by the type of detector used, and not of fundamental character, this is another reason to believe that the present spatial resolution is susceptible to significant improvements. The x-y stage allowed scanning of the pinhole over each point of two-dimensional grid on the surface of the specimen. For each of these points, the energy and the spatial location of the diffracted beam was than recorded before and after the in-situ application of a load in the elastic range. As for the determination of the position of the spot, two different systems were used with the two pinholes. Given the higher intensity through pinhole 1, it proved possible to record the position of the diffracted spot at the detector before and after the application of the load by means of double exposure of a high-speed Polaroid film. Since the specimen-to detector distance of approximately 50 cm required, for a determination of the plane rotation with a precision of 1 part in 10^4 , a spatial resolution no better than half a millimeter, the modest resolution of the high-speed Polaroid film (50-100 microns) was perfectly adequate for the purpose. Exposure times of five to ten minutes were sufficient for each spot.

The smaller diameter of pinhole 2 increased the necessary exposure times by almost a factor ten, thus rendering this double-exposure technique impracticable. The location of the Laue spots reaching the detector was therefore determined by a system of L-shaped slits, (see Figure 1), placed immediately in front of the detector window. The slits were computer-controlled by the same kind of stepping motors used for the x-y

stage. Since, at the necessary specimen-to-detector distance, the spot had considerably 'blurred', in order to determine its position unambiguously an intensity profile was constructed as a function of the position of one slit at a time. One such typical profile and the fit obtained are shown in Figure 4. With the slits completely opened, an intensity profile was also recorded as a function of the x-y coordinates of the stepping motors controlling the displacement of the pinhole, in order to construct a map of the position of the specimen with respect to the incoming beam. The geometry of the experimental set-up is depicted in Figures 1,3, which also show the orientation of the diffracting planes (211) for specimen A and (110) for specimen B) with respect to the incoming beam.

Specimen A was pulled under conditions of constant load, whereas specimen B experienced constant-strain loading ($\Delta L/L = 1.6 \cdot 10^{-4}$); both conditions were well within the elastic limit.

c) Sample preparation

Two Mo single crystals, denoted in what follows as crystals A and B, of respective thicknesses of 200 and 500 microns were grown by secondary recrystallization technique. The original polycrystal line foil from which crystal A was to be obtained was (99.99 + pure) placed in an Ultra-High-Vacuum chamber at an initial pressure of 8×10^{-9} torr, and then heated to 2475-2525 °K for two hours. Crystal B had been obtained by the same secondary recrystallization technique. The pressure at which the heating took place was considerably higher (1×10^{-6} torr) and the recrystallization time was approximately one hour.

Synchrotron X-ray transmission topographs of the specimen taken after growth

showed a much more homogeneous matrix for crystal B. Crystal A, in particular, showed a grain of different orientation, with respect to the matrix, of radius of approximately 100 micrometers, close to the position where a notch was introduced.

A notch in sample B was produced after the recrystallization process using an electric discharge machine (EDM). As for sample A, an indentation produced during the growth process by contact with one of the tungsten wires was used as a stress raiser. The shape of the indentation was highly irregular. A relevant plastic zone arose in the vicinity of this notch. As for crystal B, not only was the extent of the plastic zone around the stress raiser much more limited, but the shape of the notch (approximately parabolic) was also more regular.

III. RESULTS

From the spectra and the intensity profiles collected as described in the previous section, the rotation (2θ and azimuthal angle) and dilation of the diffracting planes could be obtained using expression 1 and 2. The positions of the maximum of the energy peak was determined by numerical fitting of the experimental profile to a Pearson-VII distribution (see, eg Naidu and Houska 1982), as described in the Appendix.

For the set of data collected with the 25-micron pinhole 2, the intensity profiles for the location of the diffracted beam were fitted to a Fermi-like function, and the position of the inflection point was taken to represent the position of the spot. A gradient search method was utilized to optimize the four parameters. Figure 6 shows a map of the variation of lattice parameter in the vicinity of the crack for specimen A, obtained using pinhole 1 (70 micron diameter). It should be remembered that, for both specimens, a significant

- 12 -

extent of permanent plastic deformation was present prior to the elastic loading, and the geometry of the stress raiser is quite different from the elliptic or hyperbolic notch that constitutes the classic model of continuum mechanics calculations. A direct comparison with continuum mechanics calculations of displacement fields cannot therefore be accomplished except at a qualitative level. At this level, as discussed at greater length later on, good agreement was in fact found.

What is of special relevance is the fact that, with this spatial resolution, changes in variation of lattice spacing over contiguous grid cells ($\delta\Delta a$) as large as 300% could be detected (specimen A, pinhole 1), with a maximum overall variation of 600%. The variation in the rotation of the lattice planes as a function of position on the specimen rarely exceeded 100%. Although, at least for elastic loading of moderately pre-deformed specimens, the major variation occurs for the dilational component of the strain tensor, in the determination of the variation of lattice spacing (Equations 1 and 2) of the diffracting planes the greater role is played by the rotational components of the deformation. The contribution to the d^*/d^0 term in equation (2) arising from the displacement of the energy peaks in fact is five to ten times smaller than the contribution from the lattice rotations. This conclusion can be profitably compared with the general assumption of predominance of lattice rotations with respect to dilation commonly enforced in the different case of unloaded plastically deformed specimens. In the present case the rotations and dilation reported always refer to the unloaded condition, irrespective of whatever permanent deformation might be present in the crystal. In this type of experiment, therefore, one does not have to worry about an a-priori determination of an undeformed reference state, as the case has been for recent equi-contour experiments (Stock et al. 1987). For specimen A, which presented as described above a grain of different

orientation of approximate diameter of 150 micron in the proximity of the notch, a sharp increase in the variation of lattice rotation was observed in the proximity of the grain. In this neighbourhood the rotations of the diffracting planes with respect to the unloaded condition become extremely accentuated, and are seen to be of both signs over the volume sampled. These points are not displayed in Figure 6 since it proved impossible to give an unambiguous value to the displacement of the diffraction spot. Presence of rotations of both signs indicates that the sampled volume is so large that the significant quantities characterizing the deformation are effectively integrated over the 70 x 70 micrometers.

In going from the more complex crystal A to the 'cleaner' sample B, and by pushing the resolution from 70 to 25 micron this problem disappears: the quality of the peaks is better and double peak structures are never encountered. The results obtained with pinhole 2 and specimen B are shown in figure 7. Several interesting features should be noted: the diffracting planes that, it will be remembered, are not perpendicular to the mode-I tensile axis, neither experience dilation at every sampled point, nor display rotation (2θ angle) always in the same direction. One can actually distinguish two regions, an upper one of dilation and rotation towards smaller two theta angles, and a lower one where these features are reversed. Those points showing a contraction of the reflecting planes in general also display opposite rotation with respect to the dilated planes. A qualitative explanation of this behaviour follows from the orientation of the diffracting planes, tilted by 34° with respect to the tensile axis: in Figure 3 equipotential lines have been drawn in the neighbourhood of the notch, which reflect the fact that normal stresses must vanish at a free surface. One can see that the stresses, always constrained to be perpendicular to the equipotential lines, will be acting almost parallel to the planes themselves

in the lower part of the notch region, whereas the orientation of the stresses is almost perpendicular in the upper half. Below the apex of the notch the diffracting planes therefore experience a Poisson-modulus contraction, which is actually found in the high resolution measurements. This effect is strictly local, since, as one moves away from the notch, the asymptotical dilational behaviour must be recovered. This is exactly what is observed in our case. This interesting effect would have been lost by a resolution even only twice as coarse. It should also be pointed out that there is a high correlation between regions of the crystal with contraction instead of dilation, and regions with rotation opposite to those observed in the upper part of the specimen. It should also be noted that, for crystal B, if one neglects the variation of the 2θ angle in the calculation of the dilational component of the strain tensor, the values obtained for the variation of the lattice spacing are almost an order of magnitude too small. This consideration seems to be of general character. By looking at equation 2, one can see that the importance of the rotational term in the determination of the variation in the spacing of the diffracting planes linearly depends on the cotangent of the diffracting angle 2θ . Since the construction of the intensity profiles is the most time-consuming part of the experiment, it could be useful to choose a geometrical setting (high 2θ angles) as to make the cotangent term negligible. This increase in λ would also be beneficial for the absorption in the pinhole Pt or Ta foils. If, in future experiments, the need could be eliminated for the construction of the intensity profiles, the saving in experimental time would be substantial, of the order of ten to twenty times. Information about the rotation components of the strain tensor would however be lost.

IV. CONCLUSION AND DISCUSSION

We have presented a novel technique for high resolution determination of

strain fields in single crystals, based on the simultaneous recording of the energy and position of a diffracted beam. The technique proved to be suitable for the investigation of perfect to highly deformed crystals, with a strain sensitivity of one part in 10^4 . The method was implemented at the synchrotron ring of NSLS at BNL in order to make use of the high intensity and brightness of synchrotron radiation.

It was also shown that, for the displacement field present at the tip of an elastically strained notch, the change in lattice spacing of the diffracting planes cannot be determined by simply recording the change in energy position of the Bragg peaks, at least for the geometrical setting chosen for this experiment. This finding is analogous to the frequently enforced assumption (Liu et al. 1982) that the state of deformation in a plastically deformed crystal can be almost completely ascribed to the planes rotation.

The best spatial resolution attained was 25 micrometers. This limit is presently set by pinhole technologies and by detector characteristics, and, therefore, is not of a fundamental nature. As for the collimation, future experiment will benefit from new techniques just now being explored. Research is in fact under way to achieve multi-layer focussing optics with demagnification of 500:1 (Thompson et al. 1986). Although presently limited by spherical aberrations, recent experiments have achieved probe sizes of 8×10 micrometers. In principle this technique can achieve focussed images below one micrometer (Thompson, private communication). Another promising method for achieving small spots is the use of the so-called inverse pinholes. These devices are elliptical posts, which intercept and reflect only a micron-sized cross section of the incident beam. By making use of multilayer coated reflective surfaces, 10% bandwidths have been obtained. Both techniques are suitable for producing microprobe beams for the present experiments and avoid the limitations of normal pinholes.

A new generation of detectors has recently become available with system throughput (ratio of output to input events) almost twice as high as the Si/Li detector employed in the present experiment. Since these new detectors could work in linear range for twice as high intensities, the need would be reduced, or eliminated, for the lead filtering described above. This would significantly improve the signal to noise ratio through the pinhole foil.

Another possible improvement to the experiment is the use of an area detector to identify quickly the diffracted beam position. Since only small diffracted beam displacements are tracked, an active area of a square centimeter would be adequate. A transmission proportional counter would be particularly suited, since it could sort out the harmonics, and allow simultaneous monitoring of the diffracted beam energy by a solid state detector. The spatial resolution of 50 to 100 micrometers routinely attainable with position sensitive detectors would be more than adequate for the purpose of beam location. As previously cited above, reducing the time of this part of the experiment would allow substantial increase in the number of collected points.

APPENDIX

For a given detector-multi-channel-analyser system, the energy resolution is solely determined by the precision and reproducibility of the fitting procedure. Special care was therefore given to the choice of the most suitable fitting curve.

The Pearson-VII (henceforth referred to as P-VII) distribution (see eg Naidu, Huska 1982) with four adjustable parameters was used to fit the energy profiles. The P-VII distribution is given by

$$P-VII(x) = a_1 \frac{1}{(1+y)a_4} \quad (A)$$

$$y = (x - a_2)/a_4 a_3^2 \quad (A2)$$

An attractive feature of the P-VII distribution is that, by continuous variation of the parameter a_4 , it describes a Lorentzian ($a_4 = 1$), a Voight ($a_4 = 1.7-2$) or a Gaussian distribution ($a_4 = \infty$).

A value of a_4 equal to 20 is sufficient to represent a Gaussian shape. For a P-VII distribution the peak is centered at $x_0 = a_2$, the peak maximum is given by $P-VII(x_0) = a_1$, and the full-width-half-maximum (FWHM) can be obtained by solving for $x_{1/2}$: from $P-VII(x_{1/2}) = \frac{1}{2} a_1$, one gets

$$FWHM = 2 a_3 f(a_4) \quad (A3)$$

$$f(a_4) = a_4 (2^{1/a_4} - 1) \quad (A4)$$

Since $f(a_4)$ is a slowly varying function of a_4 and it is almost equal to one, the FWHM can be approximated by $2 a_3$.

The P-VII distribution was utilized in combination with a multi-peak non-linear least-square numerical algorithm (gradient method), (Bevington, 1969).

The accuracy and reproducibility of the procedure was tested by recording several spectra corresponding to given locations on the sample and load conditions (4 different positions and two different load conditions were analysed). The resulting best-fit values of the location of the maxima of

the peaks were found to differ within each group, at most, by 3-4 eV. This value is consistent with the overall precision and sensitivity of the method, of the order of a few parts in 10^4 .

The intensity profiles for the location of the diffracted beam were fitted to a four-parameter Fermi-like function.

$$Y = \frac{A}{1 + \exp (x - B)/C} + D$$

with A giving the step height, C the step width, B the location of the inflection and D the background. Examples of the fits thus obtained are given in Figure 4. The same gradient-search least-square numerical algorithm was utilized for the optimization of the parameters.

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Figure 1. = Schematic of the micro-diffraction experiment:

the x-y stage with the pinhole;

the L-shaped Ta block for the determination of the position of the diffracted beam;

the Si/Li detector.

Figure 2. The in-situ tensile stage.

Figure 3. Geometry of the experimental set-up (specimen B) with the diffracting (211) planes (dashed lines). Qualitative 'equipotential' lines are also drawn in the proximity of the notch (see text for discussion). The thick arrows indicate the direction of traction, tilted by 34° with respect to the normal to the diffracting planes. The thin arrows in the proximity of the notch indicate the local direction of tension.

Figure 4.

- a) Fitting to the experimental points for the diffraction peak; photon energy in keV.
- b) Fitting to the intensity profile for the location of the diffracted beam; counts/60 secs on the y axis, and arbitrary distance units along the x axis.

Figure 5. Local variation of lattice spacing (Eqn.1) in the proximity of the notch for specimen A in units of $\text{\AA} \cdot 10^{-4}$, with cell size $70 \times 70 \mu\text{m}$.

Figure 6. Ratio of the deformed to undeformed lattice spacing (Eqn.2) for specimen B. Underlined values correspond to grid cell with an overall contraction of the lattice spacing, with cell as indicated $25\mu\text{m} \times 30\mu\text{m}$.

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Tensile Stage for In-situ X-ray Topography

$\sigma \uparrow$

21	22	21	25	24	23	23
			15	16	15	15
			5	5	5	6

$70\mu\text{m}$

$70\mu\text{m}$

$\sigma \downarrow$

σ $\uparrow\uparrow$

								1-0116	1-0119
		25 μ m							1-0164
			1-0055	0-9960	1-0033	1-0028	1-0060		
			1-0058	1-0037	0-9967	0-9996	1-0100		
									1-0044
			0-9991	0-9976	0-9957	0-9908	0-9862	1-0000	
		30 μ m							
			1-0021	1-0036	0-9992	1-0041	0-9979		

$\uparrow\uparrow$ σ

END

**DATE
FILMED
5/14/92**