| ESRF | Experiment title: Statistical study of the mechanical deformation of nanocrystals | Experiment number : MA-3056 | | | | | | |
|--|---|---|--|--|--|--|--|--|
| Beamline : | Date of experiment: | Date of report: | | | | | | |
| ID01 | from: 30/09/2016 to: 05/10/2016 | 14/03/2017 | | | | | | |
| Shifts: | Local contact(s): | Received at ESRF: | | | | | | |
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REPORT

The purpose of the experiment was to perform a statistical study of the microstructure of gold nanocrystals before and after nanoindentation. We used the high throughput of ID01 for Coherent X-ray Diffraction (CXD) measurements and of the nanoindentation facility of our lab, also based in Grenoble, to measure a large number of crystals by CXD *before* and *after ex situ* nanoindentation, during the same experimental run.

The experiment went almost as initially planned and was very successful.

The gold nanocrystals were prepared by solid state dewetting of a gold thin film on a sapphire substrate. The substrates were covered with a patterned mask in order to obtain well isolated crystals aligned on a square lattice (Figure 1). The large separation is needed for the CXD measurements because the focused beam has weak but large tails that can cause the measurement to fail by illuminating nearby crystals. The template is used to identify the crystals individually during the nanoindentation and the CXD measurements. SEM prior to the experiment allowed do identified the best crystals, i.e. the ones that display a shape as close as possible to the Winterbottom equilibrium shape. This characteristic is important to ease the modelling, and allows to select a set of crystals with external shapes as similar as possible, in order to make statistics.

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| • | • | • | | • | • |
| 2 µm ⊣ | Mag = 2.00 K X EHT = 3.00 kV | Signal A = SE2 WD = 6.9 mm | Signal B = SE2 Mixing = Off Signal = 1.000 | Date :16 Sep 2016 Time :14:28:43 Stage at X = 76.011 mm Stage at Y = 54.259 mm | INPG CMTC |

Figure 1: typical SEM view of the nanocrystals.

We prepared 2 substrates hosting slightly different types of crystals: they were obtained by dewetting films of different thickness (45 nm and 60 nm), leading to nanocrystals with slightly different average sizes. The influence of the size on the mechanical behaviour is an open debate in the community.

As initially planned, a large number of crystals on both substrates were sequentially measured in their initial state, then indented in our lab, then measured again at ID01. The full list of crystals is given in appendix. The beamtime was fully used, by measuring one of the samples while indenting the other one. In addition, due to the larger through-put of CXD than nano-indentation, a large number of crystals were measured only by CXD, for future investigation of their mechanical response: the initial microstructure is relevant to understand it. Characterisation of the final microstructure, if needed, can be done later (beamtime will be required).

Different parameters of indentation were used. Some crystals were loaded with a "flat punch" and others were properly indented with a "cube corner" tip (Figure 2). In all cases, the load-displacement curves, which characterize globally the mechanical test, were recorded, and the crystals were loaded/indented at various load levels and depths.



Figure 2: Example of nanoindentation with a cube corner tip.

The CXD experiment was performed with the standard set-up, i.e. at 8 keV with Fresnel Zone Plate focusing (the sample was placed 1 mm out of focus in order to match the beam size with the crystals size). The vicinity of the 111 Bragg reflection was measured in 3D by rocking the sample and recording the diffraction pattern with the Maxipix4 detector. Sufficient oversampling in the 3 dimensions was ensured by the detector to sample distance (~1m) and the rocking curve step size (0.005°).

We intend to recover real space images of the crystals by using phase retrieval algorithms. The phase of the real space reconstruction provides an image of lattice displacement field, from which the strain and the crystal defects can de deduced. The analysis is in progress, but will take time due to the large amount of data and the poor automation of the "reconstruction" process. In cases when the real space reconstruction fails, a reciprocal space analysis can already provide qualitative information on the microstructure of the crystal, before and after indentation.

We provide below an example of data with its real space reconstruction, for a crystal which has been loaded by a flat punch. The force-displacement curve suggests a reversible (elastic) load (Figure 3), but the diffraction data proves that the load induced stacking faults in the {111} planes (Figure 4) that were not detected by the nanoindenter. This example demonstrates the high sensitivity of CXD to crystal defects, to a level that even top-of-the-art mechanical apparatus cannot challenge.



Figure 3: Reversible indentation of gold nanocrystal with a flat punch. The load-depth curve does not display any discontinuity (a sign of dislocation avalanches) and suggests a reversible mechanical behaviour. The SEM images of the crystal before (left) and after (right) indentation are identical.



Figure 4: Projection of the CXD patterns along the 111 growth axis, before and after flat-punch loading, and the corresponding real-space reconstructions (x-y cuts and x-z cuts). The scale in the real space images is in nm. The colour encodes the phase, whose jumps reveal the presence of crystal defects. The phase origin is irrelevant.

While the analysis is still in progress, we can already draw the following general conclusions:

- We established a reliable procedure to investigate the effect of mechanical loading on the microstructure of nanocrystals.
- The method is high throughput, because both techniques (CDI and nanoindentation) are high throughput and both located in Grenoble.

• The large number of crystals measured in this run will provide interesting mechanical information, but probably deserve more measurements in order to improve the statistical validity of study.

APPENDIX

Full list of the nanocrystals measured by CXD during this experiment, with their corresponding mechanical tests.

| CRYSTAL | | SIZE | Nano-indentation | | CYD scans | | | | | | |
|--------------------------|--|--|---|---|--|---|---|---|--|--|--|
| (SEM&AFM) | | | first pop-in | | CAD scans | | | | | | |
| | COVETAL | Lx | Ly (mm) | Lz | Indontor | Maximal force | Force at | Length of | Displacement before | scan before | scan after |
| SAMPLE | | (nm) 702 | (nm) | (nm) 520 | Flat Punch | (mN) | (mN) | (nm) | (nm) | (πie "45nm") 23 | (file "45nm_2") |
| | 1 4 | 753 | 758 | 330 | Cube Comer | 0.4 | | reve | ersible | 29 | 75 |
| | 1.5 | 744 | 697 | 364 | Cube Comer | 0.03 | | reve | ersible | 31 | 80 |
| | 1,6 | 711 | 720 | | | | | | | 44 | |
| | 1,7 | | | 460 | | | | | | 46 | |
| | 2,3 | | | | Flat Punch | 0.91 | 0.91 | 300.05 | 31.46 | 18 | 72 |
| | 2,4 | 669 | 651 | 620 | | | | | | 37 & 38 | 83 |
| | 2,7 | | | 316 | | | | | | 48 | |
| | 3,2 | 353 | 367 | 205 | Flat Punch | 0.1 | | reversible | | 18 | 97 |
| | 3,0 | | | | | | | | | 53 | |
| | ,0 / 3 | 730 | 7/8 | 111 | Cube Comer | 0 0091 | 0 0001 | Λ | 5.6 | 25 | 80 |
| F | 4.4 | 758 | 748 | 266 | cube conter | 0.0051 | 0.0051 | - | 5.0 | 23 | 05 |
| 2 nm | 4,7 | | | 330 | | | | | | 57 | |
| | 5,2 | 763 | 790 | 348 | Cube Comer | 0.0093 | 0.0093 | 8.6 | 5 | 20 | 87 |
| 4 | | | | | | | | | | 21 | |
| ш | 5,4 | 758 | 734 | 330 | Cube Comer | 0.0078 | 0.0078 | 6.52 | 4.33 | 35 | 85 |
| 2 | 5,6 | 700 | 701 | | | 0.0157 | | 17.01 | | 59 | |
| Ξ | 6,5 | 790 | /81 | 270 | Cube Comer | 0.0157 | 0.0157 | 17.21 | 6.92 | 33 | 92 |
| A S | 6.8 | | | | | | | | | 02 65 | |
| 0, | 7.4 | 702 | 702 | 385 | | | | | | 40 & 41 | 94 |
| | 7,7 | | | 263 | | | | | | 67 | 5. |
| | 8,4 | 697 | 683 | 390 | | | | | | 69 | |
| | 8,8 | | | | | | | | | 71 | |
| | 9,7 | | | 248 | | | | | | 78 | |
| | 10,2 | 744 | 720 | 400 | | | | | | 82 | |
| | 10,4 | 749 | /39 | 365 | | | | | | 80 | |
| | 10,0 | 762 | 725 | | | | | | | 84 73 | |
| | 12.6 | 702 | 125 | | | | | | | 75 | |
| | 13,2 | 772 | 776 | 353 | | | | | | 90 | |
| | | CRY | STAL S | IZE | | N | ano-inder | ntation | | CYD | scans |
| (SEM&A | | | • • • • • | | | first pop-in | | | | | |
| | | (SE | M&AF | | | Maximalfores | Farra at | nrst | pop-in Diambacamant hafara | com h oforo | scans |
| SAMPLE | CRYSTAL | (SE Lx (nm) | M&AF Ly (nm) | Lz (nm) | Indenter | Maximal force (mN) | Force at (mN) | Length of (nm) | pop-in Displacement before (nm) | scan before (file "60nm") | scan after (file "60nm 2") |
| SAMPLE | CRYSTAL | (SE Lx (nm) 1070 | M&AF Ly (nm) 1100 | Lz (nm) 320 | Indenter | Maximal force (mN) | Force at (mN) | Length of (nm) | pop-in Displacement before (nm) | scan before (file "60nm") 26 | scan after (file "60nm_2") 54 |
| SAMPLE | CRYSTAL 7,2 8,2 | (SE Lx (nm) 1070 880 | M&AF Ly (nm) 1100 950 | Lz (nm) 320 527 | Indenter Flat Punch | Maximal force (mN) 0.1 | Force at (mN) | Length of (nm) | pop-in Displacement before (nm) ersible | scan before (file "60nm") 26 29 | scan after (file "60nm_2") 54 23 |
| SAMPLE | CRYSTAL 7,2 8,2 2,3 | (SE Lx (nm) 1070 880 900 | M&AF Ly (nm) 1100 950 940 | Lz (nm) 320 527 603 | Indenter Flat Punch | Maximal force (mN) 0.1 | Force at (mN) | Length of (nm) | pop-in Displacement before (nm) ersible | scan before (file "60nm") 26 29 38 | scan after (file "60nm_2") 54 23 |
| SAMPLE | CRYSTAL 7,2 8,2 2,3 7,3 | (SE Lx (nm) 1070 880 900 970 | M&AF Ly (nm) 1100 950 940 970 | Lz (nm) 320 527 603 416 | Indenter Flat Punch Cube Comer | Maximal force (mN) 0.1 0.005 | Force at (mN) 0.005 | Inst Length of (nm) reve | pop-in Displacement before (nm) ersible 3.07 | scan before (file "60nm") 26 29 38 35 35 | scan after (file "60nm_2") 54 23 29 |
| SAMPLE | CRYSTAL 7,2 8,2 2,3 7,3 8,3 | (SE Lx (nm) 1070 880 900 970 860 860 | M&AF Ly (nm) 1100 950 940 970 1040 | Lz (nm) 320 527 603 416 450 | Indenter Flat Punch Cube Comer Cube Comer | Maximal force (mN) 0.1 0.005 | Force at (mN) 0.005 | 1.89 | pop-in Displacement before (nm) ersible 3.07 | scan before (file "60nm") 26 29 38 35 33 33 | scan after (file "60nm_2") 54 23 29 |
| SAMPLE | CRYSTAL 7,2 8,2 2,3 7,3 8,3 10,3 11,2 | (SE Lx (nm) 1070 880 900 970 860 860 860 | M&AF Ly (nm) 1100 950 940 970 1040 865 860 | Lz (nm) 320 527 603 416 450 550 500 | Indenter Flat Punch Cube Comer Cube Comer Cube Comer | Maximal force (mN) 0.1 0.005 0.0056 | Force at (mN) 0.005 0.0056 | 1.89 3.95 | pop-in Displacement before (nm) ersible 3.07 3.259 | scan before (file "60nm") 26 29 38 35 33 31 | scan after (file "60nm_2") 54 23 29 26 80 |
| SAMPLE | CRYSTAL 7,2 8,2 2,3 7,3 8,3 10,3 11,3 7,4 | (SE Lx (nm) 1070 880 900 970 860 860 860 860 950 | M&AF Ly (nm) 1100 950 940 970 1040 865 860 1060 | Lz (nm) 320 527 603 416 450 550 590 390 | Indenter Flat Punch Cube Comer Cube Comer Cube Comer | Maximal force (mN) 0.1 0.005 0.0056 | Force at (mN) 0.005 0.0056 | 1.89 3.95 | pop-in Displacement before (nm) ersible 3.07 3.259 | scan before (file "60nm") 26 29 38 35 33 31 43 | scan after (file "60nm_2") 54 23 29 26 89 62 |
| SAMPLE | CRYSTAL 7,2 8,2 2,3 7,3 8,3 10,3 11,3 7,4 10,4 | (SE Lx (nm) 1070 880 970 860 860 860 860 950 1049 | M&AF Ly (nm) 1100 950 940 970 1040 865 860 1060 1065 | Lz (nm) 320 527 603 416 450 550 590 390 330 | Indenter Flat Punch Cube Comer Cube Comer Cube Comer Cube Comer | Maximal force (mN) 0.1 0.005 0.0056 0.0074 | Force at (mN) 0.005 0.0056 0.0074 | 1.89 3.95 4.868 | pop-in Displacement before (nm) ersible 3.07 3.259 4.113 | scan before (file "60nm") 26 29 38 35 33 31 43 46 | scan after (file "60nm_2") 54 23 29 26 89 62 32 |
| SAMPLE | CRYSTAL 7,2 8,2 2,3 7,3 8,3 10,3 11,3 7,4 10,4 3,5 | (SE Lx (nm) 1070 880 900 970 860 860 860 950 1049 1170 | Ly (nm) 1100 950 940 970 1040 865 860 1060 1065 1150 | Lz (nm) 320 527 603 416 450 550 590 390 330 267 | Indenter Flat Punch Cube Comer Cube Comer Cube Comer Flat Punch | Maximal force (mN) 0.1 0.005 0.0056 0.0074 0.46 | Force at (mN) 0.005 0.0056 0.0074 0.46 | 1.89 3.95 4.868 73.46 | pop-in Displacement before (nm) ersible 3.07 3.259 4.113 11.19 | scan before (file "60nm") 26 29 38 35 33 31 43 46 53 | scan after (file "60nm_2") 54 23 29 26 89 62 32 |
| SAMPLE | CRYSTAL 7,2 8,2 2,3 7,3 8,3 10,3 11,3 7,4 10,4 3,5 4,5 | (SE Lx (nm) 1070 880 970 860 860 860 860 950 1049 1170 1050 | Ly (nm) 1100 950 940 970 1040 865 860 1065 1150 1000 | Lz (nm) 320 527 603 416 450 550 590 330 267 355 | Indenter Flat Punch Cube Comer Cube Comer Cube Comer Flat Punch Flat Punch | Maximal force (mN) 0.1 0.005 0.0056 0.0074 0.46 0.04 | Force at (mN) 0.005 0.0056 0.0074 0.46 0.04 | 1.89 3.95 4.868 73.46 1.35 | pop-in Displacement before (nm) ersible 3.07 3.259 4.113 11.19 9.8 | scan before (file "60nm") 26 29 38 35 33 31 43 46 53 | scan after (file "60nm_2") 54 23 29 26 89 62 32 52 |
| SAMPLE | CRYSTAL 7,2 8,2 2,3 7,3 8,3 10,3 11,3 7,4 10,4 3,5 4,5 7,5 | (SE (nm) 1070 880 900 970 860 860 860 950 1049 1170 1050 1000 | Ly (nm) 1100 950 940 970 1040 865 860 1065 1150 1000 820 | Lz (nm) 320 527 603 416 450 550 590 390 330 267 355 480 | Indenter Flat Punch Cube Comer Cube Comer Cube Comer Flat Punch Flat Punch Cube Comer | Maximal force (mN) 0.1 0.005 0.0056 0.0074 0.46 0.04 0.094 | Force at (mN) 0.005 0.0056 0.0074 0.46 0.04 0.0094 | 1.89 3.95 4.868 73.46 1.35 8.73 | pop-in Displacement before (nm) ersible 3.07 3.259 4.113 11.19 9.8 4.88 | scan before (file "60nm") 26 29 38 35 33 31 43 46 53 51 | scan after (file "60nm_2") 54 23 29 26 89 62 32 52 38 |
| SAMPLE | CRYSTAL 7,2 8,2 2,3 7,3 8,3 10,3 11,3 7,4 10,4 3,5 4,5 7,5 10,5 | (SE Lx (nm) 1070 880 900 860 860 860 950 1049 1170 1050 1000 1210 | W&AF Ly (nm) 1100 950 940 970 1040 865 860 1060 1065 1150 1000 820 1300 | Lz (nm) 320 527 603 416 450 590 390 330 267 355 480 230 | Indenter Flat Punch Cube Comer Cube Comer Cube Comer Flat Punch Flat Punch Cube Comer Cube Comer | Maximal force (mN) 0.1 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 | Force at (mN) 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 | Length of (nm) 1.89 3.95 4.868 73.46 1.35 8.73 1.5 | pop-in Displacement before (nm) ersible 3.07 3.259 4.113 11.19 9.8 4.88 5.3 | scan before (file "60nm") 26 29 38 35 33 31 43 46 53 51 49 | scan after (file "60nm_2") 54 23 29 26 89 62 32 52 38 35 |
| SAMPLE , E | CRYSTAL 7,2 8,2 2,3 7,3 8,3 10,3 11,3 7,4 10,4 3,5 4,5 7,5 10,5 4,6 5,6 | (SE Lx (nm) 1070 880 900 860 860 860 950 1049 1170 1050 1000 1210 950 | M&AF Ly (nm) 1100 950 940 970 1040 865 860 1060 1065 1150 1000 820 1300 1090 | Lz (nm) 320 527 603 416 450 590 390 330 267 355 480 230 | Indenter Flat Punch Cube Comer Cube Comer Cube Comer Flat Punch Flat Punch Cube Comer Cube Comer | Maximal force (mN) 0.1 0.005 0.0056 0.0074 0.46 0.04 0.004 0.0094 0.006 | Force at (mN) 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 | Length of (nm) reve 1.89 3.95 4.868 73.46 1.35 8.73 1.5 2.65 | pop-in Displacement before (nm) ersible 3.07 3.259 4.113 11.19 9.8 4.88 5.3 6.65 | scan before (file "60nm") 26 29 38 35 33 31 43 46 53 51 49 55 57 | scan after (file "60nm_2") 54 23 29 26 89 62 32 52 38 35 57 42 |
| SAMPLE "EU OG | CRYSTAL 7,2 8,2 2,3 7,3 8,3 10,3 11,3 7,4 10,4 3,5 4,5 7,5 10,5 4,6 5,6 8,6 | (SE (nm) 1070 880 970 860 860 860 950 1049 1170 1050 1000 1210 950 1000 820 | M&AF Ly (nm) 11000 950 940 970 1040 865 860 1060 1065 1150 1000 820 1300 1090 850 975 | Lz (nm) 320 527 603 416 450 590 390 330 267 355 480 230 478 490 | Indenter Flat Punch Cube Comer Cube Comer Cube Comer Flat Punch Flat Punch Cube Comer Cube Comer Cube Comer | Maximal force (mN) 0.1 0.005 0.0056 0.0074 0.46 0.04 0.004 0.0094 0.006 0.008 0.008 | Force at (mN) 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 0.008 0.008 | Arrist Length of (nm) reve 1.89 3.95 4.868 73.46 1.35 8.73 1.5 2.65 5.49 | pop-in Displacement before (nm) ersible 3.07 3.259 4.113 11.19 9.8 4.88 5.3 6.65 5.24 | scan before (file "60nm") 26 29 38 35 33 31 43 46 53 51 49 55 57 60 | scan after (file "60nm_2") 54 23 29 26 89 62 32 52 38 35 57 42 |
| SAMPLE "WU 09" | CRYSTAL 7,2 8,2 2,3 7,3 8,3 10,3 11,3 7,4 10,4 3,5 4,5 7,5 10,5 4,6 5,6 8,6 1,7 | (SE (nm) 1070 880 970 860 860 860 950 1049 1170 1050 1000 1210 950 1000 820 1070 | M&AF Ly (nm) 1100 950 940 970 1040 865 860 1065 1150 1000 820 1300 1090 875 975 1150 | Imiliar Lz (nm) 320 527 603 416 450 550 390 330 267 355 480 230 478 490 281 | Indenter Flat Punch Cube Comer Cube Comer Cube Comer Flat Punch Flat Punch Cube Comer Cube Comer Cube Comer | Maximal force (mN) 0.1 0.005 0.0056 0.0074 0.46 0.04 0.004 0.0094 0.006 0.008 0.008 | Force at (mN) 0.005 0.0056 0.0074 0.46 0.04 0.004 0.0094 0.006 0.008 0.008 | Length of (nm) 1.89 3.95 4.868 73.46 1.35 8.73 1.5 2.65 5.49 | pop-in Displacement before (nm) ersible 3.07 3.259 4.113 11.19 9.8 4.88 5.3 6.65 5.24 | scan before (file "60nm") 26 29 38 35 33 31 43 46 53 51 49 55 57 60 73 | scan after (file "60nm_2") 54 23 29 26 89 62 32 52 38 35 57 42 |
| SAMPLE " WU 09 " H | CRYSTAL 7,2 8,2 2,3 7,3 8,3 10,3 11,3 7,4 10,4 3,5 4,5 7,5 10,5 4,6 5,6 8,6 1,7 4,7 | (SE (nm) 1070 880 970 860 860 860 950 1049 1170 1050 1000 1210 950 1000 820 1070 850 | M&AF Ly (nm) 1100 950 940 970 1040 865 860 1065 1150 1000 820 1300 1090 855 975 1160 840 | Iz (nm) 320 527 603 416 450 550 390 330 267 355 480 230 478 490 281 588 | Indenter Flat Punch Cube Comer Cube Comer Cube Comer Flat Punch Flat Punch Cube Comer Cube Comer Cube Comer | Maximal force (mN) 0.1 0.005 0.0056 0.0074 0.46 0.04 0.004 0.0094 0.006 0.008 0.008 | Force at (mN) 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 0.008 0.008 | Length of (nm) 1.89 3.95 4.868 73.46 1.35 8.73 1.5 2.65 5.49 | pop-in Displacement before (nm) ersible 3.07 3.259 4.113 11.19 9.8 4.88 5.3 6.65 5.24 | scan before (file "60nm") 26 29 38 35 33 31 43 46 53 51 49 55 57 60 73 68 | scan after (file "60nm_2") 54 23 29 26 89 62 32 52 38 35 57 42 |
| SAMPLE BLE "60 nm" | CRYSTAL 7,2 8,2 2,3 7,3 8,3 10,3 11,3 7,4 10,4 3,5 4,5 7,5 10,5 4,6 5,6 8,6 1,7 4,7 5,7 | (SE (nm) 1070 880 970 860 860 860 950 1049 1170 1050 1000 820 1070 820 1070 850 1040 | M&AF Ly (nm) 1100 950 940 970 1040 865 860 1065 1150 1000 820 1300 1090 855 1160 840 1170 | Iz (nm) 320 527 603 416 450 550 390 330 267 355 480 230 478 490 281 588 315 | Indenter Flat Punch Cube Comer Cube Comer Cube Comer Flat Punch Flat Punch Cube Comer Cube Comer Cube Comer | Maximal force (mN) 0.1 0.005 0.0056 0.0074 0.46 0.04 0.004 0.0094 0.006 0.008 0.008 | Force at (mN) 0.005 0.0056 0.0074 0.46 0.04 0.004 0.0094 0.006 0.008 0.008 | Length of (nm) 1.89 3.95 4.868 73.46 1.35 8.73 1.5 2.65 5.49 | pop-in Displacement before (nm) ersible 3.07 3.259 4.113 11.19 9.8 4.88 5.3 6.65 5.24 | scan before (file "60nm") 26 29 38 35 33 31 43 46 53 51 49 55 57 60 73 68 65 | scan after (file "60nm_2") 54 23 29 26 89 62 32 52 38 35 57 42 75 |
| SAMPLE "MDLE "MDLE "MDLE | CRYSTAL 7,2 8,2 2,3 7,3 8,3 10,3 11,3 7,4 10,4 3,5 4,5 7,5 10,5 4,6 5,6 8,6 1,7 4,7 5,7 6,7 | (SE (nm) 1070 880 970 860 860 860 950 1049 1170 1050 1000 1210 950 1000 820 1070 850 1040 860 | M&AF Ly (nm) 1100 950 940 970 1040 865 860 1065 1150 1060 820 1300 975 1160 840 1170 795 | Im Im Lz (nm) 320 527 603 416 450 550 593 300 330 267 355 480 230 478 490 281 588 315 593 593 | Indenter Flat Punch Cube Comer Cube Comer Cube Comer Flat Punch Flat Punch Cube Comer Cube Comer Cube Comer | Maximal force (mN) 0.1 0.005 0.0056 0.0074 0.46 0.04 0.004 0.0094 0.006 0.008 0.008 | Force at (mN) 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 0.008 0.008 | 4.868 73.46 1.35 8.73 1.5 2.65 5.49 | pop-in Displacement before (nm) ersible 3.07 3.259 4.113 11.19 9.8 4.88 5.3 6.65 5.24 | scan before (file "60nm") 26 29 38 35 33 31 43 46 53 51 49 55 57 60 73 68 65 63 | scan after (file "60nm_2") 54 23 29 26 89 62 32 52 38 35 57 42 75 |
| SAMPLE "60 nm" | CRYSTAL 7,2 8,2 2,3 7,3 8,3 10,3 11,3 7,4 10,4 3,5 4,5 7,5 10,5 4,6 5,6 8,6 1,7 4,7 5,7 6,7 5,8 | (SE (nm) 1070 880 970 860 860 860 950 1049 1170 1050 1000 1210 950 1000 820 1070 850 1040 860 960 | M&AF Ly (nm) 1100 950 940 970 1040 865 860 1065 1150 1060 820 1300 1090 850 975 1160 840 1170 795 1000 | Iz (nm) 320 527 603 416 450 550 390 330 267 355 480 230 478 490 281 588 315 593 431 | Indenter Flat Punch Cube Comer Cube Comer Cube Comer Flat Punch Flat Punch Cube Comer Cube Comer Cube Comer | Maximal force (mN) 0.1 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 0.008 0.008 | Force at (mN) 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 0.008 0.008 | Length of (nm) 1.89 3.95 4.868 73.46 1.35 8.73 1.5 2.65 5.49 | pop-in Displacement before (nm) ersible 3.07 3.259 4.113 11.19 9.8 4.88 5.3 6.65 5.24 | scan before (file "60nm") 26 29 38 35 33 31 43 46 53 51 49 55 57 60 73 68 65 63 75 | scan after (file "60nm_2") 54 23 29 26 89 62 32 52 38 35 57 42 75 |
| SAMPLE "60 nm" | CRYSTAL 7,2 8,2 2,3 7,3 8,3 10,3 11,3 7,4 10,4 3,5 4,5 7,5 10,5 4,6 5,6 8,6 1,7 4,7 5,7 6,7 5,8 11,8 | (SE (nm) 1070 880 970 860 860 860 950 1049 1170 1050 1000 820 1070 820 1040 850 1040 860 960 960 | M&AF Ly (nm) 1100 950 940 970 1040 865 860 1065 1150 1000 820 1300 1090 850 975 1160 795 1000 1300 1170 795 1000 300 | Iz (nm) 320 527 603 416 450 550 390 330 267 355 480 230 478 490 281 588 315 593 431 265 | Indenter Flat Punch Cube Comer Cube Comer Cube Comer Flat Punch Flat Punch Cube Comer Cube Comer Cube Comer | Maximal force (mN) 0.1 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 0.008 0.008 | Force at (mN) 0.005 0.0056 0.0074 0.46 0.04 0.004 0.0094 0.006 0.008 0.008 | 4.868 73.46 1.35 8.73 1.5 2.65 5.49 | pop-in Displacement before (nm) ersible 3.07 3.259 4.113 11.19 9.8 4.88 5.3 6.65 5.24 | scan before (file "60nm") 26 29 38 35 33 31 43 46 53 51 49 55 57 60 73 68 65 63 75 | scan after (file "60nm_2") 54 23 29 26 89 62 32 52 38 35 57 42 75 92 |
| SAMPLE "60 nm" | CRYSTAL 7,2 8,2 2,3 7,3 8,3 10,3 11,3 7,4 10,4 3,5 4,5 7,5 10,5 4,6 5,6 8,6 1,7 4,7 5,7 6,7 5,8 11,8 4,9 6,10 | (SE (nm) 1070 880 970 860 860 860 950 1049 1170 1050 1000 820 1070 820 1070 820 1040 850 1040 860 990 990 | M&AF Ly (nm) 950 940 970 1040 865 860 1065 1050 1000 820 1300 975 1160 840 1170 795 1000 820 1300 840 1170 795 0000 820 | Iz (nm) 320 527 603 416 450 550 390 330 267 355 480 230 478 490 281 588 315 593 431 265 480 | Indenter Flat Punch Cube Comer Cube Comer Cube Comer Flat Punch Flat Punch Cube Comer Cube Comer Cube Comer | Maximal force (mN) 0.1 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 0.008 0.008 | Force at (mN) 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 0.008 0.008 | Inst Length of (nm) reve 1.89 3.95 4.868 73.46 1.35 8.73 1.5 2.65 5.49 | pop-in Displacement before (nm) ersible 3.07 3.259 4.113 11.19 9.8 4.88 5.3 6.65 5.24 | scan before (file "60nm") 26 29 38 35 33 31 43 46 53 51 49 55 57 60 73 68 65 63 75 77 | scan after (file "60nm_2") 54 23 29 26 89 62 32 52 38 35 57 42 75 92 |
| SAMPLE "60 nm" | CRYSTAL 7,2 8,2 2,3 7,3 8,3 10,3 11,3 7,4 10,4 3,5 4,5 7,5 10,5 4,6 5,6 8,6 1,7 4,7 5,7 6,7 5,8 11,8 4,9 6,10 8,10 | (SE (nm) 1070 880 900 970 860 860 860 950 1049 1170 1050 1000 820 1070 820 1070 850 1040 850 1040 860 990 920 813 880 | M&AF Ly (nm) 1100 950 940 970 1040 865 860 1065 1000 820 1300 1090 870 975 1160 840 1170 795 1000 820 930 820 | Iz (nm) 320 527 603 416 450 550 390 330 267 355 480 230 478 490 281 588 315 593 431 265 485 612 420 | Indenter Flat Punch Cube Comer Cube Comer Cube Comer Flat Punch Flat Punch Cube Comer Cube Comer Cube Comer | Maximal force (mN) 0.1 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 0.008 0.008 0.008 | Force at (mN) 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 0.008 0.008 | Length of (nm) 1.89 3.95 4.868 73.46 1.35 8.73 1.5 2.65 5.49 | pop-in Displacement before (nm) ersible 3.07 3.259 4.113 11.19 9.8 4.88 5.3 6.65 5.24 | scan before (file "60nm") 26 29 38 35 33 31 43 46 53 51 49 55 57 60 73 68 65 63 75 77 77 79 | scan after (file "60nm_2") 54 23 29 26 89 62 32 52 38 35 57 42 75 92 85 46 |
| SAMPLE "60 nm" | CRYSTAL 7,2 8,2 2,3 7,3 8,3 10,3 11,3 7,4 10,4 3,5 4,5 7,5 10,5 4,6 5,6 8,6 1,7 4,7 5,7 6,7 5,8 11,8 4,9 6,10 8,10 10,10 | (SE (nm) 10700 880 970 860 860 860 950 1049 1170 1050 1000 820 1070 820 1070 850 1040 850 1040 860 990 920 813 980 920 813 920 | M&AF Ly (nm) 950 940 970 1040 865 860 1065 1050 1000 820 1300 1090 850 975 1160 840 1170 795 1000 820 930 880 930 | Iz (nm) 320 527 603 416 450 550 390 330 267 355 480 230 478 490 281 588 315 593 431 265 485 612 435 | Indenter Flat Punch Cube Comer Cube Comer Cube Comer Flat Punch Flat Punch Cube Comer Cube Comer Cube Comer Cube Comer | Maximal force (mN) 0.1 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 0.008 0.008 0.008 | Force at (mN) 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 0.008 0.008 | Length of (nm) 1.89 3.95 4.868 73.46 1.35 8.73 1.5 2.65 5.49 | pop-in Displacement before (nm) ersible 3.07 3.259 4.113 11.19 9.8 4.88 5.3 6.65 5.24 ersible | scan before (file "60nm") 26 29 38 35 33 31 43 46 53 51 49 55 57 60 73 68 65 63 75 77 77 79 | scan after (file "60nm_2") 54 23 29 26 89 62 32 52 38 35 57 42 75 92 85 46 81 |
| SAMPLE "60 nm" | CRYSTAL 7,2 8,2 2,3 7,3 8,3 10,3 11,3 7,4 10,4 3,5 4,5 7,5 10,5 4,6 5,6 8,6 1,7 4,7 5,7 6,7 5,8 11,8 4,9 6,10 8,10 10,10 1,11 | (SE (nm) 10700 880 970 860 860 860 950 1049 1170 1050 1000 820 1070 820 1070 820 1040 850 1040 850 1040 850 1040 813 990 920 813 980 1020 | M&AF Ly (nm) 950 940 970 1040 865 860 1065 1050 1000 820 1300 1090 850 975 1160 840 1170 795 1000 820 930 820 930 820 | Iz (nm) 320 527 603 416 450 550 390 330 267 355 480 230 478 490 281 588 315 593 431 265 485 612 435 350 | Indenter Flat Punch Cube Comer Cube Comer Cube Comer Flat Punch Flat Punch Cube Comer Cube Comer Cube Comer Cube Comer | Maximal force (mN) 0.1 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 0.008 0.008 0.008 | Force at (mN) 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 0.008 0.008 | Length of (nm) 1.89 3.95 4.868 73.46 1.35 8.73 1.5 2.65 5.49 | pop-in Displacement before (nm) ersible 3.07 3.259 4.113 11.19 9.8 4.88 5.3 6.65 5.24 ersible | scan before (file "60nm") 26 29 38 35 33 31 43 46 53 51 49 55 57 60 73 68 65 63 75 77 77 79 | scan after (file "60nm_2") 54 23 29 26 89 62 32 52 38 35 57 42 75 92 85 46 81 77 |
| SAMPLE "60 nm" | CRYSTAL 7,2 8,2 2,3 7,3 8,3 10,3 11,3 7,4 10,4 3,5 4,5 7,5 10,5 4,6 5,6 8,6 1,7 4,7 5,7 6,7 5,8 11,8 11,8 4,9 6,10 8,10 10,10 1,11 3,11 | (SE (nm) 10700 880 970 860 860 860 950 1049 1170 1050 1000 820 1070 820 1070 820 1070 820 1040 850 1040 850 1040 813 990 920 813 980 1020 | M&AF Ly (nm) 1100 950 940 970 1040 865 860 1065 1000 820 1300 1090 850 975 1160 840 1170 795 1000 820 930 820 930 820 | Iz (nm) 320 527 603 416 450 550 390 330 267 355 480 230 478 490 281 588 315 593 431 265 485 612 435 350 | Indenter Flat Punch Cube Comer Cube Comer Cube Comer Flat Punch Flat Punch Cube Comer Cube Comer Cube Comer Cube Comer | Maximal force (mN) 0.1 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 0.008 0.008 0.008 | Force at (mN) 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 0.008 0.008 | Length of (nm) 1.89 3.95 4.868 73.46 1.35 8.73 1.5 2.65 5.49 | pop-in Displacement before (nm) a.07 3.259 4.113 11.19 9.8 4.88 5.3 6.65 5.24 ersible | scan before (file "60nm") 26 29 38 35 33 31 43 46 53 51 49 55 57 60 73 68 65 63 75 77 77 79 | scan after (file "60nm_2") 54 23 29 26 89 62 32 52 38 35 57 42 75 92 85 46 81 77 87 |
| SAMPLE "60 nm" | CRYSTAL 7,2 8,2 2,3 7,3 8,3 10,3 11,3 7,4 10,4 3,5 4,5 7,5 10,5 4,6 5,6 8,6 1,7 4,7 5,7 6,7 5,8 11,8 4,9 6,10 8,10 10,10 1,11 3,11 7,11 | (SE (nm) 1070 880 900 970 860 860 860 950 1049 1170 1050 1000 820 1070 820 1070 820 1070 820 1040 850 1040 860 990 920 813 980 1020 1200 | M&AF Ly (nm) 1100 950 940 970 1040 865 860 1065 1000 820 1300 1090 870 975 1160 840 1170 795 1000 820 930 820 930 820 930 820 930 | Iz (nm) 320 527 603 416 450 550 390 330 267 355 480 230 478 490 281 588 315 593 431 265 485 612 435 350 607 | Indenter Flat Punch Cube Comer Cube Comer Flat Punch Flat Punch Cube Comer Cube Comer Cube Comer Cube Comer | Maximal force (mN) 0.1 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 0.008 0.008 0.008 | Force at (mN) 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 0.008 0.008 | Length of (nm) 1.89 3.95 4.868 73.46 1.35 8.73 1.5 2.65 5.49 | pop-in Displacement before (nm) ersible 3.07 3.259 4.113 11.19 9.8 4.88 5.3 6.65 5.24 ersible | scan before (file "60nm") 26 29 38 35 33 31 43 46 53 51 49 55 57 60 73 68 65 63 75 77 77 79 81 | scan after (file "60nm_2") 54 23 29 26 89 62 32 52 38 35 57 42 75 92 85 46 81 77 87 99+ |
| SAMPLE "60 nm" | CRYSTAL 7,2 8,2 2,3 7,3 8,3 10,3 11,3 7,4 10,4 3,5 4,5 7,5 10,5 4,6 5,6 8,6 1,7 4,7 5,7 6,7 5,8 11,8 4,9 6,10 8,10 10,10 1,11 3,11 7,11 5,12 | (SE (nm) 1070 880 900 970 860 860 860 950 1049 1170 1050 1000 820 1070 820 1070 820 1070 820 1040 820 1040 820 1040 820 990 920 813 980 920 813 980 | M&AF Ly (nm) 1100 950 940 970 1040 865 860 1060 1055 1000 820 1300 1090 850 975 1000 820 1300 1300 820 930 820 930 820 1000 | Im Im Lz (nm) 320 527 603 416 450 550 590 390 330 267 355 480 230 478 490 281 588 315 593 431 265 485 612 420 435 350 607 216 | Indenter Flat Punch Cube Comer Cube Comer Cube Comer Flat Punch Flat Punch Cube Comer Cube Comer Cube Comer Cube Comer | Maximal force (mN) 0.1 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 0.008 0.008 0.008 | Force at (mN) 0.005 0.0056 0.0074 0.46 0.04 0.0094 0.006 0.008 0.008 | Length of (nm) 1.89 3.95 4.868 73.46 1.35 8.73 1.5 2.65 5.49 | pop-in Displacement before (nm) ersible 3.07 3.259 4.113 11.19 9.8 4.88 5.3 6.65 5.24 ersible | scan before (file "60nm") 26 29 38 35 33 31 43 46 53 51 49 55 57 60 73 68 65 63 75 77 77 79 81 | scan after (file "60nm_2") 54 23 29 26 89 62 32 52 38 35 57 42 75 92 85 46 81 77 87 99+ 69 |