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D10.5

Demonstration of relocating nanoobjects based on in-situ deposited fluorescence

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Contents

Executive Summary

In this deliverable report we describe a multistep workflow for re-locating pre-selected nano-objects based on i) the design and arrangement of markers, ii) their creation on a sample surface and iii) the nano-transfer to an X-ray beamline based on the X-ray fluorescence signal of the deposited markers. The workflow relies on a "marker design on demand" principle developed within the Joint Research Action 5 "Advanced Nano-Object Transfer and Positioning" and also includes hierarchical marker arrangement concepts. A software protocol has been created and optimized in Matlab and Python that assists in the position coordinate transformation from a nano-instrument at a nano-science centre to a focused X-ray beamline at an Analytic Large Scale Facility (ALSF). With the SEM or AFM stage coordinates for each marker and nano-object serving as input, the software tool calculates the new motor positions for the X-ray experiment. With this workflow comprising the marker design, the software protocol and the hardware implementation we provide a versatile solution for various nano-transfer demands not only for ex-situ but more importantly for in-situ or operando studies where the transfer and re-location of small objects is challenging. The workflow proposed here has been successfully tested during several experiments using different nano-science centres and ALSF infrastructures within the participating institutions of the NFFA consortium, and as such proofs the intra- and inter- institutional capabilities of the nano-transfer workflow developed within JRA5. At the same time it shows the benefit of the mutual interactions between the NFFA partners and the overall success in spreading the technical achievements across European institutions. As such, the JRA5 nano-transfer workflow may serve as a seed to establish standard procedures for re-locating of small objects facilitating a multi-analysis of the regions of interest permitting to bridge different techniques and providing a novel level of insight.

1. Concept

The workflow developed within the joint research action JRA5 "Nano-Object Transfer and Positioning" is modular in nature and as such easily adoptable to further specific needs. It provides a platform permitting a routine nano-transfer between nanoscience instruments and focused X-ray beamlines based on the X-ray fluorescence of deposited markers. The workflow covers the design of the marker size and their lateral arrangement including hierarchical principles, the marker creation on the sample surface, the software protocol assisting in the sample stage coordinate transformation between the respective instruments, as well as the hardware implementation. Several aspects of this workflow have already been described in more detail in the following deliverable reports: D10.1 "Software routine for coordinate definition and relocation of single nano-objects"; D10.2 "Implementation of transfer and positioning system at nanolabs and ALSFs"; D10.4 "Methods for deposition of hierarchical markers"; and D7.14 "Test of re-alignment of nanostructures in synchrotron experiment by in-situ marker deposition", a report on the cross-JRA cooperation between JRA2 - Research on High Precision Manufacturing and JRA5.

2. Design Specification

Here we describe the specifications established within the JRA5 "Advanced Nano-Object Transfer and Positioning" for each step of the workflow to ensure a successful nano-transfer.

2.1 Marker Design Including Hierarchical Principles

Figure 1 shows a scheme of the "Marker Design on Demand" proposed within the JRA5 to ensure an efficient nano-transfer based on a hierarchical marker arrangement. Indeed, Figure 1 serves as logo for the new "Nano-Object Transfer & Positioning" offer within the category "Lithography & Patterning" of Installation 1 of the transnational access program open to NFFA users. It documents the approach to apply several levels of hierarchies in the marker system to facilitate a simple and fast nano-transfer exploiting different prominent positions on a sample (like edges, defects etc. that can be easily located and identified) and different detection schemes (secondary and backscatter electrons in the SEM, optical microscope for zero hierarchy level marker and X-ray fluorescence for all higher hierarchy level markers). A more detailed description on how a hierarchical marker concept is implemented in the workflow for the nano-transfer can be found in the JRA5 deliverable report D10.4 "Methods for deposition of hierarchical markers".

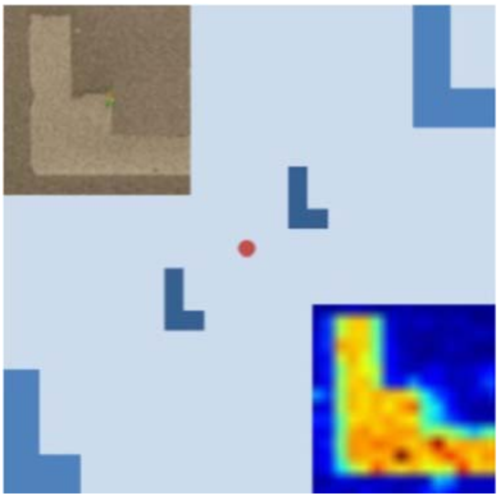

Figure 1: Logo of the JRA5 offer "Nano-Object Transfer & Positioning" of the transnational NFFA user program, indicating several hierarchical levels and the SEM and X-ray florescence scanning images taken from the same marker at DESY NanoLab[1] in Hamburg and at the X-ray beamline ID01 at ESRF in Grenoble (T F. Keller, M. Abuin, A Stierle, DESY), taken from the deliverable report D10.4.

2.2 Marker Creation by the IBID/EBID Process

A successful workflow for a nano-transfer needs optimized markers placed at well-defined sample positions. The sample edges may serve as the 0th level hierarchy. Intermediate, 1st hierarchical level markers are needed to bridge larger distances on millimetre sized samples that guide towards the regions of interest. 2nd hierarchy level markers must be located in the vicinity of the nano-objects.

A versatile tool to create the markers in such well-defined positions is the electron- (EBID) or ion- (IBID) induced deposition of a precursor gas in an SEM or FIB-SEM. EBID or IBID permits to mark regions of interest after the pre-selection process based on the microscopic view onto distinct nano-objects. As such, the most useful, promising and meaningful nano-objects can be selected well in advance of the X-ray beamtime. The marker optimization in the framework of the JRA5 and the development of this workflow is described in the deliverable report D10.4 “Methods for deposition of hierarchical markers”. In cooperation between JRA2 and JRA5 a series of thickness calibrations were performed in order to create markers with a desired thickness. The results are described in detail within the deliverable report D7.14 “Test of re-alignment of nanostructures in synchrotron experiment by in-situ marker deposition”, in the framework of the cross-JRA cooperation between JRA2 - Research on High Precision Manufacturing and JRA5.

Figure 2 shows AFM height images of a series of markers written in the SEM that were used to calibrate the two EBID process parameters i) acceleration voltage of the electrons and ii) the spot-size, i.e. the aperture defining beam-size of the electron beam. This calibration procedure shows how an optimal marker thickness can be obtained by calibrating EBID/ IBID processing parameters.

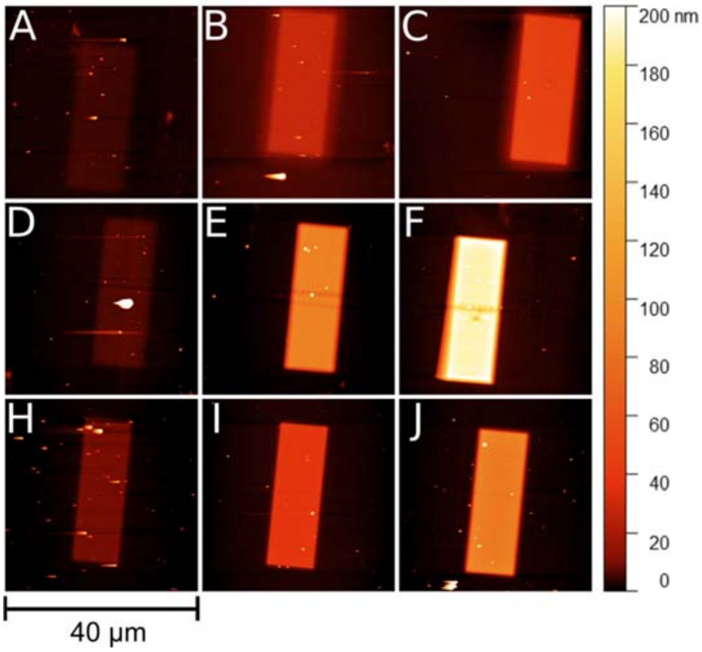

Figure 2: AFM height measurements of markers created by EBID in a SEM with varying the processing parameters spot size and acceleration voltage. a-c) deposited at 20 kV, d-f) at 5 kV, h-j) written at 10 kV. Spot-sizes in the left, center and right column are 7, 6 and 5, respectively (C. Seitz, T. F. Keller, DESY), taken from the deliverable report D7.14.

The bar on the left hand side of Figure 2 shows the colour code indicating that the thickness of all tested EBID markers deposited with all applied settings is lying in the range of 2-200 nm. Based on height profiles across each of the AFM topographic image in Figure 2, the resulting thickness values were obtained and are now being used for calibration of the EBID process. Using this thickness calibration for the acceleration voltage and the spot size, well defined and optimized X-ray fluorescent markers can be created with nanometre precision by in-situ deposition using the EBID process.

2.3 Software Protocol Assisting the Nano-Transfer

Figure 3 shows the graphical user interface (GUI) of the Advanced Nano-Object Transfer and Positioning protocol in the Matlab implementation, see, e.g., the JRA5 deliverable report D10.1.

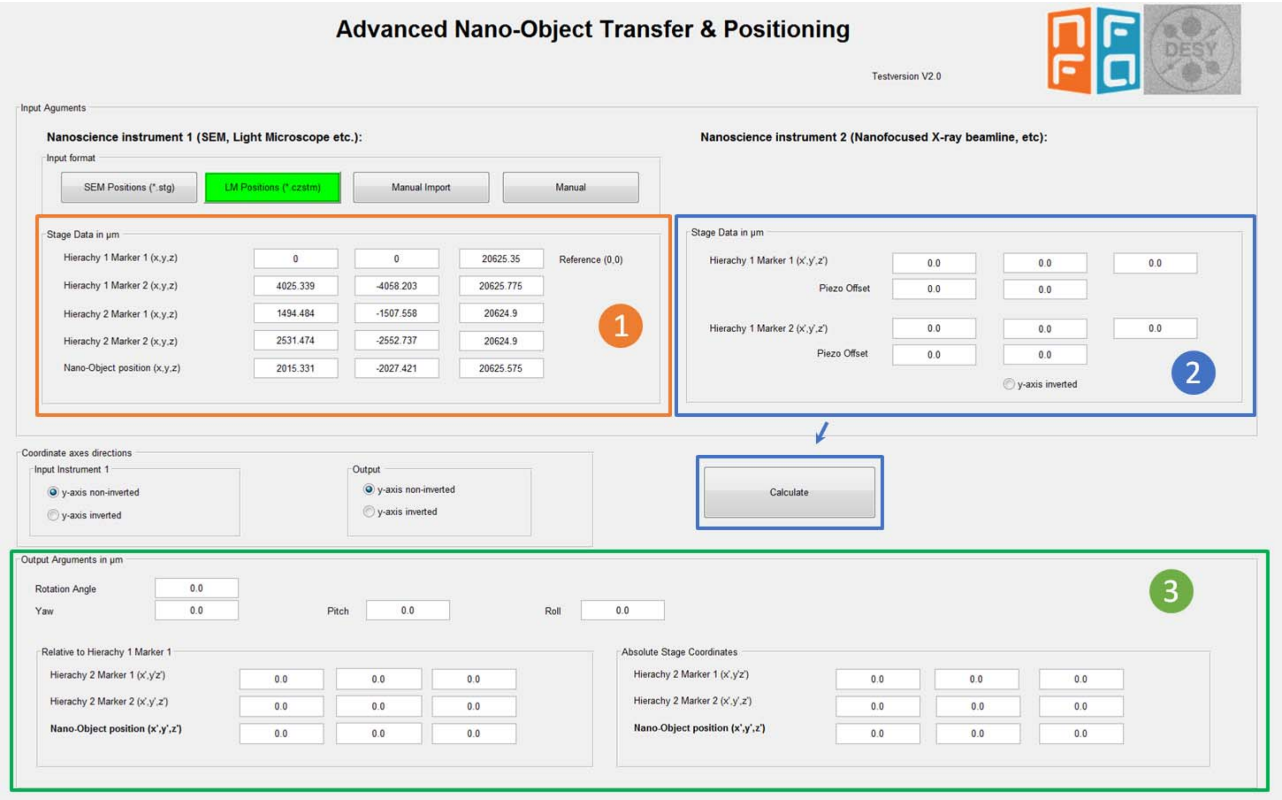

Figure 3: Screenshot of the MATLAB scripts in 3 steps that users have to follow in this version of the software (A. Böhme, M. Abuin, T.F. Keller, A. Stierle). Taken from JRA5 deliverable report D10.1.

As indicated in the screen shot shown in Figure 3, the GUI is divided into 3 sections: the section for the input coordinates obtained with the nano-instrument at the nano-science centre, by e.g., using an SEM, optical light microscope or AFM, marked with “1”; the input section with the new motor position coordinates on the instrument or beamline, where the region of interest shall be re-located, marked with “2”; and the output of the coordinate transform calculation with the resulting coordinates of the region of interest, marked by “3”. The output furthermore comprises coordinate-transformed mutual relative distances and absolute values for all saved positions (markers and nano-objects), which can be exported in a tabulated ASCII format.

3. Results

3.1 Nano-Transfer Based on in-Situ Deposited Fluorescence

To demonstrate the re-location of nano-objects based on in-situ deposited fluorescence, we are describing here the successful nano-transfer achieved during an X-ray beamtime from DESY NanoLab to the coherence beamline P10 at PETRA III, DESY. The aim was to gain insight into the structure-functionality relations of nano-photonics by analysing femtosecond laser printed arrays of crystalline single Ge and SiGe nanoparticles with relevance due to their potential as a substitution for plasmonic metal-based metamaterials. The crystallinity and strain information was probed by coherent X-ray diffraction with the aim to apply the Bragg coherent x-ray diffractive imaging technique[2] at beamline P10 at PETRA III at DESY.

3.2 Nano-Transfer - Localization at Nanoscience Center

Figure 4 shows an SEM overview with 2 level hierarchy markers, i.e., two 0th level hierarchy Pt based IBID markers at the bottom of the SEM image, and a 1st level hierarchy EBID marker in close vicinity to the sample area containing the arrays with Ge and GeSi single nanoparticles. The 0th level hierarchy Pt based IBID markers may be visualized with the optical beamline microscope available at the P10 beamline, as marked inside the SEM image. The final step of the nano-transfer relies on the X-ray fluorescence of the Pt based markers in order to ensure any offset between the optical microscope and the lateral X-ray beam position on the sample surface.

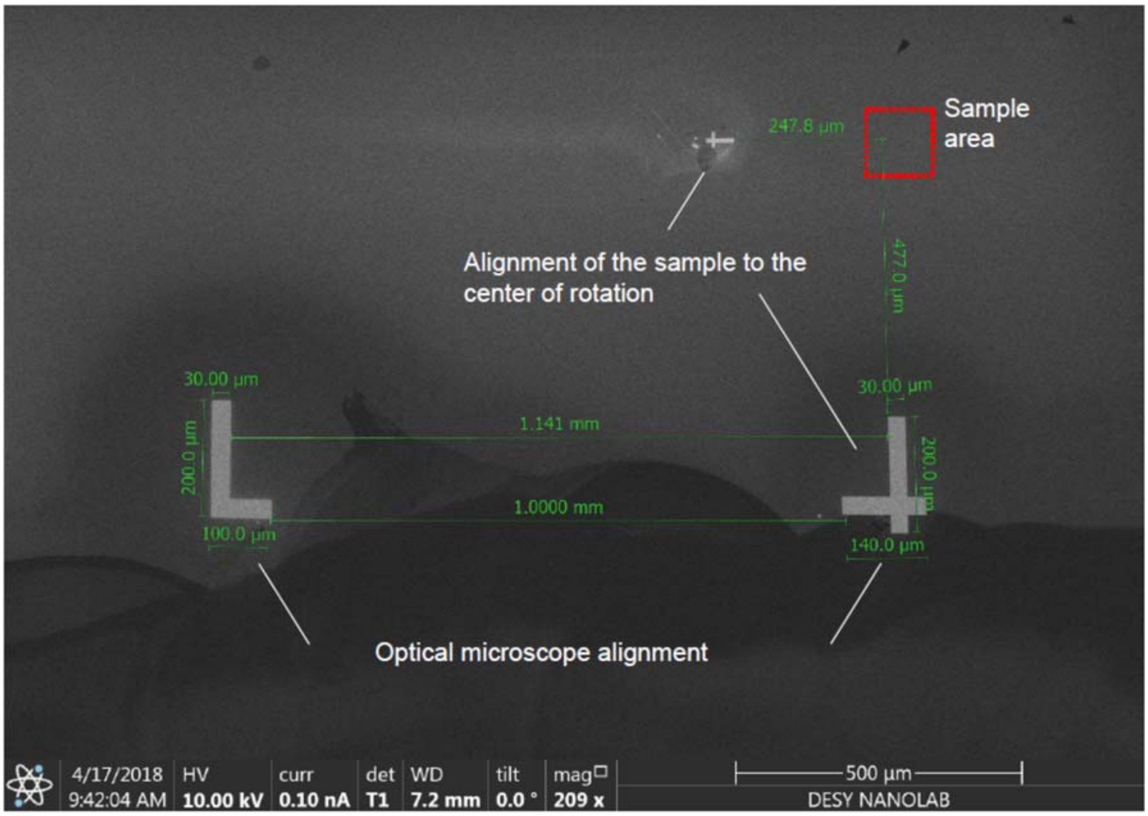

Figure 4: SEM overview image[1] with three Pt based EBID/IBID markers showing two hierarchical levels with two big markers on the sample edge on the bottom, and a higher hierarchical level marker in the upper half of the image in the vicinity of the region of interest containing the Ge nanoparticle nano-array. (S. Kulkarni, D. Dzhigaev, T. F. Keller).

3.3 Nano-Transfer – Re-Localization at the coherence beamline P10 at PETRA III

The nano-transfer of the Ge and GeSi nanoparticle arrays at the coherence beamline P10 at PETRA III, DESY, relies on a two-level hierarchy. First, the 0th level hierarchy Pt based IBID markers are located based on the coarse optical microscope image (not shown). Second, the same 0th level hierarchy IBID markers are re-located with the Pt L_3 X-ray fluorescence using the focused X-ray beam, see, e.g., the X-ray scanning image of the Pt marker region in Figure 5. In the next step, the relative lateral positions (i.e., obtained from SEM stage coordinates) are considered and used along with the "Nano-Object Transfer and Positioning" protocol or by manual calculation of the motor positions at the beamline P10 to re-locate the smaller and thinner 1st level hierarchy EBID marker in the vicinity of the nano-particle arrays.

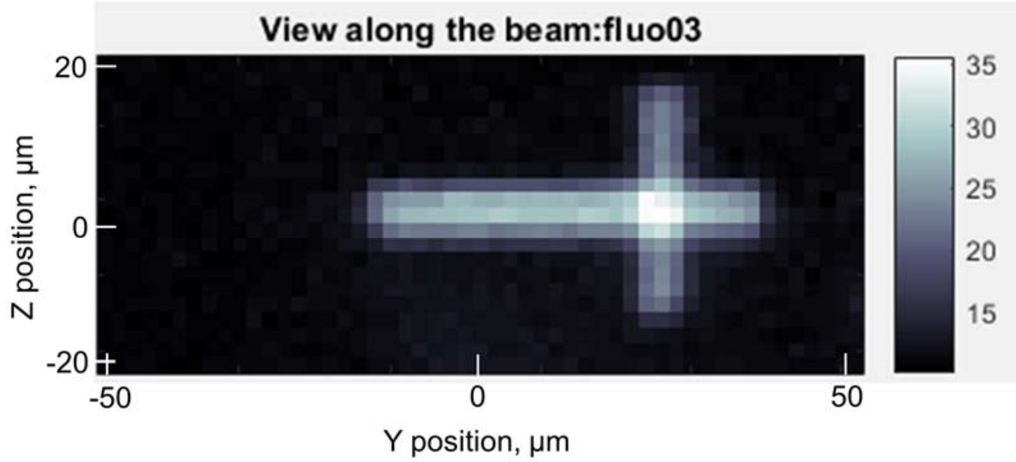

Figure 5: Mapping of the Pt X-ray fluorescence signal from the smallest marker deposited close to the sample area. (D. Dzhigaev, T. F. Keller et al.).

Once the 1st level hierarchy EBID marker in Figure 5 is re-located, the nominal motor positions of the region of interest containing the Ge and GeSi nanoparticle arrays can be calculated and the motor positions changed accordingly. Figure 6a shows the optical bright-field image of a nanoparticle area, whereas Figure 6b is a sample scanning image using the Ge X-ray fluorescence.

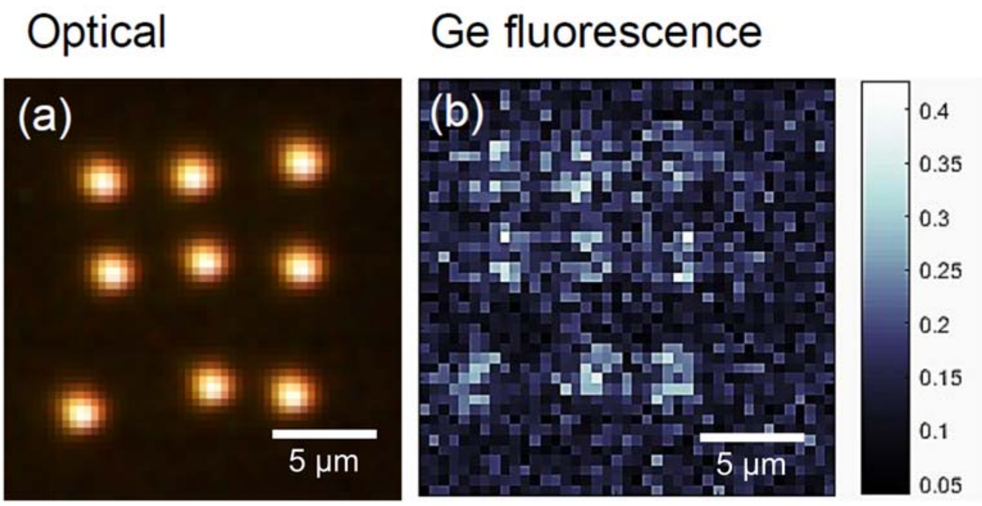

Figure 6: Mapping of the nanoparticles. (a) Optical bright-field image of an array of Ge nanoparticles. (b) The same group as visible by mapping the sample to obtain a Ge-X-ray fluorescence scanning image at beamline P10 at PETRA III, DESY, (D. Dzhigaev, T. F. Keller et al.).

Figure 6b clearly demonstrates that the re-location of the Ge nanoparticle array from DESY NanoLab to the coherence X-ray beamline P10 at DESY was successfully achieved using in-situ deposited fluorescence markers. Indeed, the nanoparticle array was recovered in a reasonable time not wasting the valuable and limited X-ray beamtime.

4. Conclusions and perspectives

This deliverable report documents the workflow for relocating nanoobjects based on in-situ deposited fluorescence. It relies on i) the pre-selection of the region of interest containing the nanoobjects inside an SEM and the subsequent deposition of hierarchical fluorescence markers by the EBID/IBID process. The marker design and arrangement thereby largely relies on a “marker design on demand” facilitating an easy to handle, time-saving nano-transfer that is facilitated by a software protocol transforming the stage coordinate positions in the SEM to the motor positions at the X-ray beamline.

As an example of a successfully achieved nano-transfer, where the region of interest was re-located in a reasonable amount of time, we are describing here an experiment at beamline P10 at PETRA III, DESY. The required task for the Nano-Object Transfer and Positioning was to ensure the re-location of a Ge or GeSi array of single nanoparticles that should be analysed with a focused X-ray beam. For the re-localization routine, the optical visibility of zero level hierarchy markers in the optical beamline light microscope have been considered, whereas the first level hierarchy markers as well as the pre-selected region of interest containing the Ge / GeSi nano-array have been re-located using the Pt and Ge X-ray fluorescence, respectively.

Overall, this deliverable report shows that the workflow for a nano-transfer based on in-situ deposition of hierarchical fluorescence markers as developed within the JRA5 Advanced Nano-Object Transfer and Positioning is efficient and reliable. In the future, it will be offered and transferred to X-ray beamlines, which currently implement a focusing option and have documented their interest in exploiting this nano-transfer workflow. Furthermore, the workflow is already offered to external users and can be requested not only via the transnational access of the NFFA user program within the category “Lithography & Patterning” of Installation 1 of the transnational access program, but also by regular users of PETRA III, or on a collaborative basis also from users of ESRF or SOLEIL.

References

[1] Stierle, A., et al. "DESY NanoLab." Journal of large-scale research facilities JLSRF 2 (2016): 76.

[2] Dzhigaev, D., et al. "Bragg coherent x-ray diffractive imaging of a single indium phosphide nanowire." Journal of Optics 18.6 (2016): 064007.