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In situ scanning x-ray diffraction reveals strain variations in electrochemically grown nanowires

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Abstract

Templated electrochemical growth in nanoporous alumina can be used to fabricate nanowires with applications in magnetic storage devices, hydrogen sensors, and electrocatalysis. It is known that nanowires, grown in such templates, are strained. The strain in nanoscale materials can influence their performance in applications such as catalysts and electronic devices. However, it is not well established how the nanoporous template affects the lattice strain in the nanowires and how this develops during the growth process due to the lack of non-destructive *in situ* studies with spatial resolution. We have measured the strain and grain size of palladium nanowires in nanoporous templates during the growth process. For this, we performed *in situ* scanning x-ray diffraction with a submicron focused x-ray beam. We found a tensile strain in the nanowires and that it is more pronounced along the growth direction than in the confined direction of the templates. The tensile strain measured *in situ* is higher than previous *ex situ* reports, possibly due to hydrogen absorption during the growth. With the spatial information made possible with the focused synchrotron x-ray beam, we could observe local variations in the strain as a function of height. A region of local strain variation is found near the bottom of the nanowires where growth is initiated in branches at the pore bottoms. Knowledge of how nanoporous templates influences the strain of the nanowires may allow for atomic scale tailoring of the catalytic activity of such nanowires or minimizing strain to optimize electronic device performance.

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(Some figures may appear in color only in the online journal)

1. Introduction

Nanoscale structures are traditionally defined using lithographic techniques such as UV lithography [1], electron beam lithography [2], and nano-imprint lithography [3]. A cost-effective alternative to these traditional lithography methods could be to use nanoporous alumina as templates for ordered arrays of nanodots or nanowires [4, 5]. When aluminum is anodized under certain conditions, the oxide film becomes porous with self-organizing nanopores in a hexagonal pattern [6]. Growth of metal nanowires inside such templates can be performed using electrodeposition, where metal ions from an electrolyte are reduced at the bottom of the pores resulting in the growth of metal nanostructures that adapt the shape of the pores. This has been used to fabricate nanowires of, for example, Ni [7] and Co [8, 9] for magnetic memories and superconducting Sn nanowires [10]. Pd nanowires have been fabricated and used in applications such as hydrogen sensing [11] and electrocatalysis [12–14].

Nanowires electrodeposited in nanoporous templates grow via a nucleation and growth mechanism. The nucleation rate is governed by the applied cathodic potential, which is the driving force for reducing the Pd ions. The kinetics are governed mostly by mass transport via diffusion and for even slight overpotentials, the reaction is mass-transfer limited [15]. Reports of both 2D plane growth [16] and 3D volume growth [17] of Pd crystallites in nanoporous alumina templates exist in literature. The applied potential can alter the crystallographic growth direction [9, 18], exposing different facets to the alumina template walls. This could, in turn, alter the surface energy and the surface stress exerted on the nanowires.

Strain in nanosized-systems influences the catalytic activity both in gas-phase heterogeneous catalysis and electrocatalysis, where tensile and compressive strain have been shown to increase the catalytic activity of different material systems [19–25]. Strain also influence many other physical properties of materials. The bandgap of epitaxially grown semiconductor films can be tuned by growth induced strain due to lattice mismatch [26]. Strain in thin-film semiconductors can also increase the photoluminescence yield due to a modified band structure [27]. In thermoelectric materials, the performance can be increased by strain present in the material [28]. In magnetic metal–organic frameworks, ferroelectric and magnetic order parameters can couple with elastic strain [29]. In MoS₂ nanoribbons, the magnetic moment can be increased and tuned by applied strain [30], and the magnetic anisotropy of nanowires can be tuned with strain [31]. It is known that the confinement of the nanoporous template affects the grain size

[32], strain [33], crystallographic structure [34], and crystallographic orientation [9, 32]. These crystallographic parameters can affect many physical properties of nanostructures, impacting the performance of devices or catalysts. Exactly how the confinement of the nanoporous template affects the growth, strain, and grain size of such metal nanowires is not very well understood. Often metal nanowires grown in nanoporous templates are characterized using transmission electron microscopy (TEM) after the dissolution of the nanoporous template [13, 35–42]. Altering the surrounding of the nanowires, in this way, may influence the strain state of the nanowires. Due to the lack of non-destructive and *in situ* studies, there is little knowledge on how growth in the nanoporous templates influences the strain of the nanowires, which in turn influences their physical properties.

We have previously studied the strain in Pd nanowires deposited into nanoporous alumina templates, *ex situ*, using synchrotron x-ray diffraction [43]. We have also studied strain of Sn nanowires, *in situ*, during growth using synchrotron scanning x-ray diffraction with a 5 μm beam [44]. In this paper, we employ the high flux and penetrative nature of hard x-rays focused to a sub-micron beam (500 nm) at a modern synchrotron source to follow the growth of Pd nanowires, using scanning x-ray diffraction. The smaller beam size and thinner sample geometry should result in a spatial resolution ten times higher than our previous study. This allows us to study the crystal structure, *in situ*, during the growth of the nanowires while they are still inside of the nanoporous template. The low cross-section of water in the hard x-ray regime allows for the measurement to be performed in the electrolyte during the growth. Our study reveals a tensile strain that is more pronounced along the growth direction. The strain along the nanowires is also shown to change during the growth process, with local variations in strain in the nanowires close to the position of branches at the bottom of the nanoporous template.

2. Experimental

2.1. Sample preparation

A hat-shaped sample was machined from a rod of pure polycrystalline aluminum (99.999%). The sample surface was successively ground using 4000-grit (EU grade) sandpaper, followed by successive polishing down to a 1 μm diamond particle suspension (Struers), achieving a mirror-finish. A diamond wire saw was used to cut out a 300 μm wide strip

Figure 1. (a) Artistic illustration of sample geometry together with a graphical illustration displaying the focused x-ray beam through the nanoporous film indicating the need for such a sample geometry. (b) Schematic illustration of electrochemical cell dedicated for *in situ* synchrotron experiments.

on the top of the top-hat-shaped sample. Before anodization, the sample was cleaned by sonication in acetone and ultra-pure water. An electrically isolating polymer (nail-varnish) was used to cover the sample and only expose the polished surface on the top of the strip, as shown in figure 1(a). The thin lamella was fabricated to fit within the focus of the x-ray beam, as illustrated in the inset in figure 1(a). The sample was then anodized at 0 °C in 0.3 M sulfuric acid at 25 V using a two-step anodization process. The first anodization step was for 10 h, afterwards the sample was submerged in a mixture of chromic acid and phosphoric acid (0.185 M CrO_3 and 0.5 M H_3PO_4) overnight to dissolve the oxide grown during the first anodization step. The second anodization step was 3 h long at 25 V followed by electrochemical barrier layer thinning, where the potential was linearly decreased from 25 V to 1 V for 30 min. This process results in a thinner barrier layer, allowing electron tunneling through the oxide layer between the electrolyte and the aluminum substrate. The barrier layer thinning process also results in branching of the pore bottoms.

2.2. Electrodeposition of Pd nanowires

Electrodeposition was performed using a neutral electrolyte containing $\text{Pd}(\text{NH}_3)_4\text{Cl}_2$ and NH_4Cl , which is a well-established electrolyte for Pd electroplating [45]. A neutral electrolyte is used to avoid dissolution of the porous alumina template during deposition [46]. The exact electrodeposition procedure is described elsewhere [43]. The electrodeposition was performed in a PEEK electrochemical cell dedicated for *in situ* synchrotron experiments, as shown in figure 1(b). A graphite counter electrode was used, and the electrolyte was continuously pumped to replenish the electro-active Pd species.

2.3. In situ scanning x-ray diffraction experiment

The *in situ* scanning x-ray diffraction experiment was performed at the ‘*In situ* and Nano Diffraction’ beamline, P23, at PETRA III, DESY in Hamburg, Germany. A sub-micron focused beam with a vertical spot size of ~ 500 nm at the sample position was used. The x-ray energy was 18.275 keV. The electrochemical cell was mounted on a (5 + 2) circle HUBER diffractometer equipped with a Eulerian cradle, as shown in the supplementary information (figure S1 (available online at stacks.iop.org/JPD/54/235301/mmedia)). An X-Spectrum Lambda 750K detector with a pixel size of $55 \mu\text{m}$ and active area of 512×1528 pixels was used at a sample-detector distance of 360 mm, resulting in an angular resolution of 0.01° . The diffractometer was calibrated using an in-house LaB_6 standard. The sub-micron focused x-ray beam impinged on the sample in a transmission geometry, where the beam was parallel to the sample surface. To gain structural information along the nanowires, during the growth, the sample and the electrochemical cell were scanned through the beam along the growth direction of the nanowires, as shown in the schematics of figures 2(a) and (b). The intensity of the direct beam together with the palladium diffraction signal as a function of height, while the sample was scanned through the beam, can be found in the supplementary information (figure S2(a)). The diffraction signal was recorded in and out of the sample surface plane. Detector images measured in-and out-of-plane, before and at the end of the deposition, are shown in figure 2(c), illustrating the diffraction powder rings that were measured on the area detector.

2.4. Data analysis

1D diffraction patterns were generated from the 2D detector images by assigning each pixel a δ and γ value as described

Figure 2. (a) Illustration of the sample before the deposition of Pd. (b) Illustration of the sample at the end of the deposition. (c) Detector images at sample height = 1 μm before and at the end of the deposition of Pd nanowires. (d) Lorentzian fitting of diffraction data in- and out-of-plane.

by Schlepütz *et al* [47], then 2θ for each pixel was calculated as $2\theta = \arccos(\cos[\gamma_{\text{pixel}}] \times \cos[\delta_{\text{pixel}}])$. The Pd(111) and (200) peaks were fitted with a Lorentzian line profile, as shown in figure 2(d). The size-contribution to the peak line broadening is Lorentzian [48]. Since the instrumental resolution and bandwidth of modern synchrotrons are negligible compared to the size broadening from nanometer-sized crystallites, which is the case for nanowires grown in nanoporous alumina, a pure Lorentzian profile was used to fit the peaks. The peak position and FWHM were used to calculate the lattice strain and average grain size along and across the nanowires. The horizontal size of the x-ray beam was much larger than the diameter of the nanowires of 25 nm. Therefore no spatial information could be obtained perpendicular to the nanowire growth direction. The beam intersection across the lamella contains approximately 4000 nanopores. The resulting diffraction pattern is hence an average of many nanowires.

Lattice strain for a specific set of atomic planes was calculated as shown in the equation:

$$\varepsilon_{\text{hkl}} = (d_{\text{hkl}} - d_{\text{hkl}}^0) / d_{\text{hkl}}^0, \quad (1)$$

where d_{hkl} is the lattice plane spacing for a certain set of planes with miller indices hkl and d_{hkl}^0 is here taken as the equivalent lattice plane spacing in bulk Pd with the lattice parameter of

3.98 Å. The grain size was calculated from the line broadening using Scherrer's formula. Diffraction in the sample surface plane, where the Q vector is perpendicular to the growth direction, gives information about lattice strain and grain size perpendicular to the growth direction. Diffraction measured out-of-plane gives information about lattice strain and grain size along the Q vector, which is oriented with an angular offset from the surface normal equal to the Bragg angle θ . We do not measure strain and grain size exactly along the growth direction of the nanowires but with an offset of 8.5° for the Pd(111) out-of-plane reflection and an offset of 10° for the Pd(200) out-of-plane reflection.

3. Results

To study the strain state of the nanowires *in situ* and with spatial resolution, scanning x-ray diffraction was used, which resulted in a 3D data set of spatial, reciprocal and time coordinates. From the scanning diffraction dataset, we can visualize the growth of the nanowires over time by considering the Pd(111) Bragg reflection as a function of height at different time stamps, as shown in figure 3. Figure 3(a) shows the out-of-plane Pd(111) Bragg reflection as a function of height (vertical direction) for several time steps, and (b) similarly shows

Figure 3. (a) The evolution of the Pd(111) out-of-plane diffraction as a function of sample height at several times during the electrodeposition of Pd. (b) Pd(111) in-plane diffraction as a function of height at several times during electrodeposition of Pd.

the in-plane Pd(111) Bragg reflection. The in-and out-of-plane diffraction, at the sample height of 1 μm , is shown as a function of time in the supplementary information (figure S2(b)). During the first 25 min of the growth, the signal was too weak to analyze. Therefore that data were excluded from the analysis. As seen, the growth of the nanowires is initiated at the bottom of the nanopores at height 0. Over time, the Pd(111) intensity profile grows taller indicating growth of crystalline Pd up through the pores. At all times, the intensity of the diffraction signal is the strongest at the bottom of the pores and there is a gradient in diffraction intensity as a function of height. This could be explained by an uneven height distribution of the nanowires during the growth, with some nanowires being taller than others.

Microscopy techniques, such as SEM, have been widely used to study the morphology of nanowires deposited into nanoporous alumina templates [39–42]. Even if destructive preparation is required, such as focused ion beam milling, it is a very useful method for visualization of the nanowires inside of the template. With electron microscopy, we have observed branches at the bottom of Pd nanowires grown in nanoporous templates in a previous study [43]. The barrier layer thinning step in the fabrication of the nanoporous alumina template results in branching of the pore bottoms. This results in branching at the bottom of the nanowires grown in the templates, which is well documented in the literature [35, 49, 50]. An SEM image of the branched pore bottoms can be found in the supplementary information (figure S3). It has been reported for Sn nanowires that growth through the branched pore bottoms affects the growth mode of the nanowires and results in a variation in grain size, strain and mosaicity [44].

To calculate the lattice strain of the nanowires as a function of height, the peak position was extracted by fitting the

Brag peaks with a Lorentzian line profile as seen in figure 2(d). The lattice strain in-and out-of-plane (along and across the nanowires) was calculated from the position of the Pd(111) reflection since it is the most intense reflection. The lattice parameter of bulk Pd, $a = 3.89 \text{ \AA}$, was used as a reference to calculate the lattice strain, ϵ , as shown in equation (1). An illustration of the orientation of the lattice planes probed in the nanowires during out-of-plane and in-plane diffraction is shown in figure 4(a). During out-of-plane diffraction, the Q vector is nearly aligned with the growth direction and hence lattice strain is measured along the growth direction. During in-plane diffraction, the Q vector is oriented perpendicular to the growth direction. Therefore the strain measured in-plane is oriented perpendicular to the growth direction, which is the confined direction of the template. Figure 4(b) shows the local average strain of the Pd nanowires along the out-of-plane direction (along the nanowires) as a function of height and time. Figure 4(c) shows the local average strain of the Pd nanowires along the in-plane direction (across the nanowires) as a function of height and time.

The strain measured in-and out-of-plane shows clear variations along the wires and it also varies at the different stages of the growth. The strain measured out-of-plane, as shown in figure 4(b), is tensile in the nanowires compared to that of bulk Pd. Just above the bottom of the pores, marked by a dotted black line in the figure, a region of less strain can be seen, which is present at all three stages of the growth measured. Above this region, there is a tensile strain in the nanowires that is increasing as the nanowires grow. Also, in the in-plane direction there is a variation in the strain along the nanowires, as seen in figure 4(c). The lattice strain across the nanowires is also tensile but to a lesser degree than the strain measured out-of-plane. Above the bottom of the pores, there is a region

Figure 4. Spatially resolved structural information obtained from *in situ* XRD measurements at three different deposition times. (a) Illustration of the lattice strain directions measured in the nanowires. (b) Variation in out-of-plane lattice strain along the nanowires. (c) Variation in in-plane strain along the nanowires. (d) Illustration of grain size for different directions measured in the nanowires. (e) Grain size along the nanowires measured out-of-plane. (f) Grain size along the nanowires measured in-plane.

of more tensile strain. The strain along the growth direction is more tensile than across the nanowires, which is the confined direction. The lattice is hence more expanded in the direction which is not confined by the nanoporous template. This illustrates an effect on the crystal structure imposed by the nanoconfinement of the templates. A point to notice here is that the trends for the strain in-and out-of-plane are each other's opposite. When there is higher strain in-plane, there is less strain out-of-plane and vice versa. When comparing the strain at 0.5 μm , just above the branched pore bottoms, marked by a dotted black line in the figure, there is a variation in the strain as time proceeds during the growth process. This indicates that lattice strain can develop and decrease in regions of the nanowires that are already grown. Little growth occurs at the height of 0.5 μm during the times shown, as most pores are already filled to this point. Inside the pores, the growth takes place at the top of the nanowires, where Pd ions are electrochemically reduced from the electrolyte and incorporated in

the lattice. Yet, there is still strain developing close to the bottom of the pores during the growth process.

The grain size of the nanowires was calculated from the line broadening using Scherrer's formula. Figure 4(d) shows a schematic illustration of the grain sizes in the nanowires probed during in- and out-of-plane diffraction, calculated from the line broadening. Figure 4(e) shows the local average size of the crystalline grains along the out-of-plane direction (along the nanowires) as a function of height at various times. Similarly, figure 4(f) shows the local average size of the crystalline grains along the in-plane direction (across the nanowires), also as a function of height. The average size of the grains both in- and out-of-plane is around 25 nm, matching the diameter of the nanoporous template of 25 nm. As time proceeds during the deposition and more growth occur, an increase in the grain size is observed along the wires. It can also be seen that the grains are slightly smaller at the bottom of the pores, where the nanowires grow up through the branched pore bottoms.

The branched pore bottoms have a smaller pore diameter, so the crystallite size is restricted and is expected to be smaller, as observed.

4. Discussion

The results presented in this paper were made possible by the recent technical advances at synchrotron facilities with brighter sources and access to intense nano-focused beams in the tender to hard x-ray regime, which opens for new kinds of experiments [51, 52]. A large benefit of x-rays compared to electron-based techniques, such as TEM and SEM, is the penetrative nature of x-rays, which allows for studies to be performed on thicker embedded structures as well as *in situ* and *operando* conditions. With x-rays, it is possible to probe and measure inside batteries [53], embedded transistors [54], and during semiconductor nanoscale growth [55]. With new nano-focused x-ray beamlines, it will be possible to push the spatial resolution down to tens of nanometers and extract chemical information using fluorescence [56] or x-ray absorption mapping. Spatially resolved structural information, as demonstrated here, is accessible with scanning x-ray diffraction. A further strength of these recent advances in the brightness of synchrotron sources is the possibility to reduce the exposure time, allowing us to push the temporal resolution of the experiments. In summary, the penetrative nature of nano-focused beams allows experiments to be performed *in situ* with increased spatial and temporal resolution, where both chemical and structural information can be extracted.

We have shown that we can follow the process of electrochemical crystal growth in nanoconfined templates, *in situ*. With a sub-micron beam, we obtain spatial information about the grain size and lattice strain across and along the growth direction of the nanowires. We have shown that the crystal growth is initiated at the bottom of the pores and the nanowires grow up through the template as a function of deposition time. The grain size measured is around 25 nm, which is the same as the pore diameter, thus indicating that the crystallite size is limited by the nanoconfinement inside the template. With the spatial resolution achieved via scanning diffraction, we could observe that the grain size is smaller near the bottom of the pores, indicating that the grains are smaller when growing through the branches. The scanning diffraction technique allows for strain variations along the nanowires to be visualized at different deposition times. Above the bottom branched part of the pores, there is an evident variation in the strain showing less tensile strain out-of-plane and more tensile strain in-plane. This indicates an effect of the nanoconfined template on the strain state of the nanowires. The strain here also varies as the nanowires grow taller over time. Therefore, it is shown that stress can propagate through the crystalline nanowire as growth proceeds. Measurements performed *ex situ* would not be able to capture this phenomenon of strain development during the growth process. Post characterization using other techniques, such as TEM, is very common after the dissolution of the template. But this might reveal a different story about

the strain state of the nanowires if the stress caused by the nanoconfinement is released upon dissolution of the template.

Our finding of a tensile lattice strain of the Pd crystallites in the nanowires is similar to reports from Shin *et al*, who reported, experimentally, a close-to linear relationship between the lattice parameter along the growth direction and the inverse of Sn nanowire radius [33]. This was shown to be true not only for single-crystalline but also for polycrystalline wires [57]. A nano-sized template gives rise to a larger lattice parameter in the growth direction compared to the bulk material, hence more tensile strain. In later studies, Shin *et al* attribute this to a compressive growth stress acting perpendicular to the growth direction in the confined direction of the template [58]. This causes tensile strain in the growth direction and a relative compressive strain in the confined direction due to the Poisson effect. This prediction is in line with our previous study of Pd nanowires, where we measure the strain both along and perpendicular to the growth direction [43] while Shin's claim of a compressive growth stress is based on a theoretical approach. In our present study, we do not only support Shin's model, but we also show that there are variations in the tensile strain along the nanowires.

Both our previous *ex situ* Pd nanowire study [43] and our *in situ* study of Sn deposition [44] showed a difference in the strain between the growth direction and in-plane direction of $\sim 0.5\%$. However, the magnitude of strain was different where we found a strain of $\sim 1.2\%$ for the Sn nanowires and $\sim 0.3\%$ in the case of Pd. This indicates that different effects play a role in the strain state of the nanowires that are both material-dependent and independent. Pd has a surface free energy almost three times as large as Sn [59], leading to a greater driving force to minimize the surface of the Pd nanowires compared to the case of Sn. However, the relative in-plane compression was comparable between the two cases suggesting that the compressive stress exerted by the nanoconfined template is less material dependent.

A new observation, not present in our previous *ex situ* study, is that the lattice parameters are larger when measured *in situ*. This difference in the observed lattice parameters could be due to lattice expansion from hydrogen absorption during the electrodeposition. Such an effect would not be detected in our *ex situ* measurements due to the desorption of hydrogen in the absence of a high external hydrogen partial-pressure. It is known that during electrodeposition, the potential is high enough to electrochemically evolve hydrogen at the metal electrolyte interface [60]. Therefore an increased hydrogen partial pressure is expected. Pd has a large capacity to store hydrogen [61], which could explain the larger tensile strain observed during this *in situ* study. Hydrogen absorption could also explain the observed strain evolution in the nanowires during the deposition.

5. Conclusions

Understanding how growth in nanoconfined templates influences strain, grain size, and other crystallographic parameters is of importance for templated nanostructures to be

successfully used in future devices. We have demonstrated that we could follow the growth process of the nanowires and extract information about the grain size and strain along the nanowires during the growth process. We found that there is a tensile strain in the nanowires and that it is more pronounced along the growth direction than in the confined direction of the templates. It was also found that the lattice is more expanded during the *in situ* measurement compared to our previous *ex situ* study. This is attributed to the absorption of electrochemically evolved hydrogen in the Pd lattice. With the spatial information made possible with the focused x-ray beam we could observe local variations in strain as a function of height. A region of local strain variation is found near the bottom of the nanowires where growth is initiated in branches at the pore bottoms. This indicates that the branched pore bottoms give rise to local strain in the nanowires. The knowledge of how nanoporous templates influences the strain of the grown nanowires may allow for the atomic scale tailoring of such nanowires. This knowledge of strain induced by growth in nanoporous templates can also be expanded beyond the scope of noble metal catalysts. Strain engineering is also used for magnetic materials [62], solar cells [63], and energy storage [64].

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