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X-ray reciprocal space mapping of GaAs/AlAs quantum wires and quantum dots

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Periodic arrays of 150 and 175 nm-wide GaAs–AlAs quantum wires and quantum dots were investigated, fabricated by electron beam lithography, and $SiCl_4/O_2$ reactive ion etching, by means of reciprocal space mapping using triple axis x-ray diffractometry. From the x-ray data the lateral periodicity of wires and dots, and the etch depth are extracted. The reciprocal space maps reveal that after the fabrication process the lattice constant along the growth direction slightly increases for the wires and even more so for the dots.

In the last few years, considerable progress has been made in the field of semiconductor quantum wires and quantum dots.[1–3] Quantum confinement effects have been observed in wires and dots with a size up to about 2000 Å.[1,2] High resolution x-ray diffractometry provides a promising method for structural investigations of periodic arrays of semiconductor surface corrugations,[4–7] quantum wires,[8,9] and quantum boxes.[9,10] It is nondestructive, averaging over a large area of the sample, and requires no sample pretreatment. Moreover, it is mainly sensitive to the crystalline part of wires and dots, which determines the actual size of the quantum confined region.

In this letter we report on reciprocal space maps of the diffraction pattern of reactive ion etched 150 and 175 nm wide GaAs/AlAs periodic quantum wires and quantum dots. A Philips MRD diffractometer with an angular resolution of 12 arcsec was used. Its analyzer crystal, placed in between the sample and the detector (''triple axis diffractometry''—TAD),[11] reduces significantly the extension of the reciprocal space probe. The independent variation of the two diffraction angles ω (between incident x rays and sample surface) and 2θ (between incident and scattered x rays) provides the possibility of reciprocal space mapping.

The lateral macroperiodicity of the wire and dot arrays gives rise to additional intensity maxima (wire satellites W_i and dot satellites D_i) in the diffraction pattern. In principle, the full information about the geometrical shape (height, width, inclination of the sidewalls, period) as well as about the structural quality (strain and crystalline damage) can be obtained from a two-dimensional map of reciprocal space.[7]

The GaAs/AlAs wires and dots were realized by nanostructuring a 30 period AlAs–GaAs multiquantum well (MQW) grown on a 1 μm GaAs buffer. The nominally 8 nm thick GaAs wells are separated by nominally 12 nm AlAs barriers resulting in a total thickness of 600 nm. The MQW was capped by a 20 nm GaAs layer. Beneath the GaAs buffer 25 periods of a 5 ML/5 ML short period AlAs–GaAs SL with a total thickness of approximately 75 nm was grown on the GaAs substrate with a 80 nm GaAs buffer. The samples investigated were prepared by electron beam lithography (EBL) with a Leica Cambridge EBPG5-HR electron beam pattern generator and subsequent magnetically confined plasma reactive ion etching (MCP-RIE)[12] using $SiCl_4$ with a flow rate of 13.5 sccm and O_2 with 1.5 sccm (which is incorporated to ensure the verticality of the nanostructures) at an operating pressure of 0.5 mTorr. The microwave power was 54 W, the rf power 35 W and the resulting dc bias was −230 V. The etching depth was nominally between 600 and 700 nm. The period was nominally 300 and 350 nm and the width of the wires and dots was half the period length. SEM micrographs of the periodical wire and dot arrays revealed vertical, i.e., [110] oriented, sidewalls, and an etching depth of approximately 760 nm. This implies that the GaAs buffer was partly etched. During the etching process the 12 nm Ti/20 nm Au mask was partly attacked. The consequence was a damage of the uppermost part of the dots and wires.

Figure 1(a) shows a reciprocal space map around the (004) reciprocal lattice point (RELP) of an unstructured (as-grown) GaAs/AlAs-reference sample. ''S'' denotes the GaAs-substrate peak, SL_0 and SL_1 the zero and first-order MQW peak, respectively. ''A'' is a symbol for an artifact, the

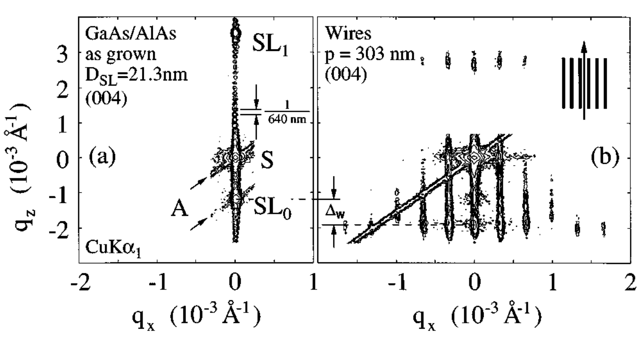

FIG. 1. (a) Reciprocal space map around the (004) RELP of an unstructured control sample. The levels of the isointensity contours are (in units of counts per second): 1.2, 2.4, 3.6, 8, 25, 80, 800, and 18 000. (b) Equivalent map of the quantum wire array with contours at 1.5, 2.4, 3.3, 4.2, 8, 15, 50, 500 and 15 000 cps.

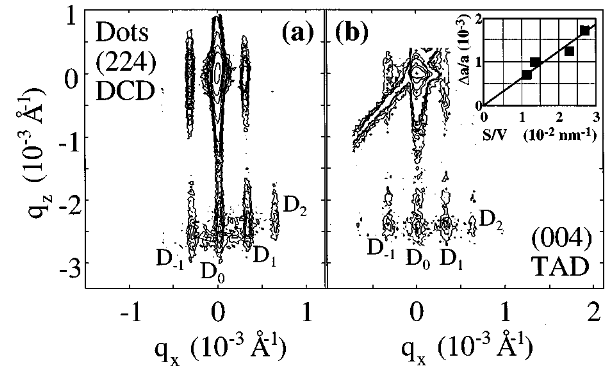

FIG. 2. (a) Reciprocal space map (DCD) around (224) of the dot array with [110] perpendicular to the diffraction plane. D_i denotes the *i*th dot satellite along the q_x direction centered around the SL_0 MQW diffraction peak. Dot satellites around the GaAs substrate/buffer RELP result from partial etching of the buffer. Contours at 4, 7, 11, 15, 22, 64, 256, 1024, and 20 000 cps. (b) Equivalent TAD map around (004). Contours at 1.2, 2.5, 4, 5, 32, 64, 256, 1024, and 20 000 cps. Inset: The average extra strain in growth direction $\Delta a/a$ vs the ratio of surface area and volume S/V of the nanostructures.

analyzer streak. RELPs with high intensity, in the present example the substrate reflection S and the SL_0 peak, are elongated along the Ewald sphere intersecting the growth direction with the Bragg angle Θ_B. Thickness fringes inbetween the MQW peaks SL_0 and SL_1 indicate the good crystalline quality of the system. Their spacing [see Fig. 1(a)] corresponds to the total thickness of the superlattice of approximately 640 nm. The MQW period was determined to be 21.3 nm from a dynamical simulation of a ω–2θ scan over 4° exhibiting satellite of the orders SL_{-7} to SL_{+7} which also gave the widths of the GaAs (8.2 nm) and the AlAs (13.1 nm) layers. In Fig. 1(b), the diffraction pattern of the periodic wire array is shown. Wire satellites accompanying the SL_0 peak and the first-order MQW peak SL_1 are observed. The wire period determined from the spacing of the satellites along the q_x direction is 303 nm. The inset in Fig. 1 defines the diffraction geometry, the arrow is the normal to the diffraction plane, which is defined by the incident and diffracted (004) x-ray wave vectors.

In Fig. 2 the maps for the periodic dot array are shown. The sample was oriented with the [110] direction perpendicular to the diffraction plane (q_x direction coincides with $[1\bar{1}0]$). Two maps around the RELPs (224) and (004) are shown in Figs. 2(a) and 2(b), respectively. Clearly, dot satellites are observed both around the SL_0 satellite RELP and the GaAs buffer peak (D_i denotes their respective order). The latter indicates a corrugation (partial etching) of the GaAs buffer as expected. The half-width of these fringes is much larger than that of the actual GaAs/AlAs dot fringes. Thus the total etch depth extends through the entire MQW structure, which has a total thickness of about 639 nm (30×21.3 nm), and some 100–200 nm into the GaAs buffer. The isointensity contours of the W_i satellites along the q_z direction exhibit thickness fringes which correspond to a thickness of just about 370 nm, i.e., less than the 639 nm. These fringes which are observable in Fig. 2 as well, can only come from the rather perfectly crystalline parts of the sample.

Due to the small overall intensity, the map around the (224) RELP was measured by double crystal diffractometry (DCD) using a diaphragm of 0.1 mm width in front of the detector. The dot period determined from the D_i satellite spacing is 310 nm which coincides quite well with the nominal lateral periodicity of 300 nm.

An important feature is the observation of the shifts of both the zero order wire and dot peak with respect to the reference SL_0 peak along the [001] growth direction. These shifts toward the center (000) of reciprocal space can only result from a larger mean lattice constant a_n^{MQW} in the wires and dots along [001] direction compared to that in the unpatterned GaAs/AlAs MQW reference sample. An anticipated elastic relaxation in strained MQW layers after fabrication, the distortion would be orthorhombic in the case of [110] oriented wires on a [001] substrate, would have the opposite effect:[13] In a pseudomorphic layer structure grown on a GaAs (a_{GaAs}=5.6537 Å) substrate, the GaAs layers of the sample are not strained at all and the AlAs (a_{AlAs}=5.6629 Å) layers are subjected to a small biaxial compression because of the lattice mismatch of 0.162%. When wires or dots are fabricated by deep etching, this strain is expected to be redistributed among the layers of the etched part of the sample, leading to a reduction of the biaxial compressive strain in the AlAs layers and the occurrence of a biaxial tensile strain in the GaAs quantum wells. A reduction of a_n^{MQW} within the dots would be the consequence of elastic relaxation. However, from Figs. 1 and 2 follows, that on the contrary, the nanostructured MQW is even more strained in the growth direction (q_z direction). The shift of the zero order wire satellite with respect to the SL_0 peak of the unstructured MQW Δ_W [see Fig. 1(b)] is about 136 arcsec, the shift of the zero order dot satellite D_0 [see Fig. 2(b)] is even larger: Δ_D=230 arcsec. This corresponds to an enlargement of the average MQW-lattice constant a_n^{MQW} from 5.663 Å in the reference sample to 5.669 Å in the wires (the extra average strain in the growth direction $\Delta a/a$ equals 1.1×10^{-3}), and to 5.673 Å in the dots ($\Delta a/a=1.8\times10^{-3}$). The origin of this *increase* of a_n^{MQW} does not follow from the x-ray analysis. However, in SEM investigations on similar dots with even thicker AlAs barriers (approximately 70 nm), which have been exposed to air for about 30 min after etching, a *visible* oxidation of the AlAs layers was observed.[14] Therefore it is most probable that the oxidized AlAs layers on the sidewalls splay the quantum wires and dots mainly along the MQW growth direction. In any case, the dots are affected stronger than the wires because they can expand or contract in all three dimensions of space whereas the wires are fixed along the direction of the corrugations by the buffer. Furthermore, the exposed surface area is larger for the dots than for the wires. This idea is confirmed by the inset of Fig. 2(b), where the extra average strain in growth direction $\Delta a/a$ of all investigated wire and dot samples is plotted against the ratio of surface area and volume S/V of the nanostructures (the line is a guide to the eye). The dots are of cylindrical shape (radius r, height h), so the surface area of their sidewalls is $2\pi rh$ and their volume is $r^2\pi h$ leading to $S/V=2/r$. The wires are approximated by rectangular blocks (width w, length l, height h), which have a sidewall surface area of $S=lh+wh\approx lh$ since $w\approx150$–175 nm is much smaller than the length $l \approx 100$ μm. Consequently S/V equals $1/w$ for the wires. From the inset, it can be seen that the extra average strain $\Delta a/a$ is proportional to S/V in the first approximation. In a previous photoreflectance work of deep etched GaAs/AlGaAs quantum dots, evidence was seen of an increasing strain in the quantum wells along the growth axis with decreasing dot size,[3] consistent with the present observation of an enlarged a_n^{MQW}.

In conclusion, x-ray reciprocal space mapping is particularly useful for the characterization of periodical one- and zero-dimensional systems. The period and the etching depth can be deduced. Furthermore it could be clearly demonstrated that with this method small changes of the strain status in multilayer samples caused by the nanofabrication process itself can be detected. Moreover, information on the crystalline quality of the fabricated lateral structures was obtained.

Note added in proof. Recently, Vaclav Holy has shown that the in-plane lattice constant of periodic arrays of quantum wires or dots is not necessarily determined by the position of the diffraction satellites in asymmetric reciprocal space maps [like (224)].

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