Low-voltage scanning transmission electron microscopy of core-shell nanowires.

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A conventional scanning electron microscope (SEM) can be converted into a hybrid low-voltage scanning transmission electron microscope (LVSTEM) operating at 30 kV, using a dedicated specimen holder. The images recorded collecting electrons diffracted at high angles β (20°<β<60°) [1] show dark field (DF) features, where the main contrast mechanism is the mass-thickness. In this approach, the probe size is larger than the atomic column and therefore channeling effects should be less crucial than in high-voltage (200-300 keV) high-angular annular dark field imaging (HAADF).

LVSTEM has been applied to the analysis carbon nanotubes or materials that can be damaged by high-energy electrons . LVSTEM can also be exploited as a fast screening tool, capable of nanometric spatial resolution, for core-shell nanowires (c-s NWs) analysis. NWs are generally produced in large numbers per growth and therefore it is extremely important to find a versatile technique that allows a reliable and fast analysis of their intimate structure, as chemical interfaces or nature of the catalyst particle, with statistical relevance.

To understand if LVSTEM can give quantitative information and the contrast value expected for c-s InGaAs/GaAs NWs, we used as a first test sample three $In_xGa_{1-x}As$ (x=0.05, 0.12, 0.24) quantum wells (QWs) embedded in GaAs, analysed in cross section geometry. The QWs nominal thickness is 5nm.

In figure 1a) a DF image of InGaAs QWs in GaAs matrix is reported; the three brighter lines close to the surface are the QWs, whose atomic number is higher than that of GaAs, thus indicating that the image shows pure DF features. In the inset, the contrast profile measured in the white dotted box, where sample thickness is about 40nm, is reported. The dependence on the In content of the contrast C, calculated from the experimental image as $C(\%) = 100 \times (I_{InGaAst} - I_{GaAs})/(I_{GaAs})$, is shown in figure 1b) (red bold line). The maximum contrast C measured in LVSTEM for the QW with x=24% is about 5.5%, while in the high voltage (200 keV) approach C ranges between 8 and 12% This difference cannot be ascribed to absorption which is found to be negligible in this thickness range, but it is due to the fact that the probe size (5-6 nm) is comparable with the QW thickness. We calculated that for a 6 nm probe size on a 5 nm QW a reduction of 45% in the contrast is expected. The theoretical contrast calculated using the power law $I \sim Z^{1.7}$ [2] (where Z is the mean atomic number) and rescaled to take into account the probe size is reported in figure 2b (black line) and the agreement with experimental contrast is good (the theoretical contrast without probe size effect is also reported in the same graph as a dotted line).

The LVSTEM was then applied to the detection of In-rich core of InGaAs/GaAs c-s NWs[3]. We underline that the core is invisible with conventional SEM imaging techniques. As shown in figure 2a), the presence of an In-rich core is clearly visible in the DF image, and

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it is evidenced by the longitudinal line profile reported in figure 2b). The reduction of the image intensity at 600 nm from the tip is related to a strong variation in Z along the wire, due to the transition between the core region and the pure GaAs portion. The magnified image of this region, indicated by the white dotted box (in the inset), shows a dark line in the InGaAs core parallel to NW growth direction. Interestingly, this feature disappears when the sample holder is tilted by 1°; the sensitivity of the image to sample tilt suggests that channeling plays an important role also in LV approach, even if probe size is larger than atomic columns. More in detail, the dark line inside the core can be associated to channeling effects induced by the lattice strain between the core and the outer shell. The influence of strain in HAADF images in high voltage STEM has been demonstrated [4][5], but this effect in LVSTEM is pointed out here for the first time. The application of LVSTEM to quantitative analysis is limitated both by the channeling effect and the lack of knowledge on c-s NWs internal geometry, however the the core is clearly detectable. Further analysis to verify the possibility to obtain more quantitative information are currently in progress.

In conclusion, we have shown that LVSTEM can be applied to the analysis of c-s NWs and that the contrast can be affected by the probe size and, surprisingly, by channeling.

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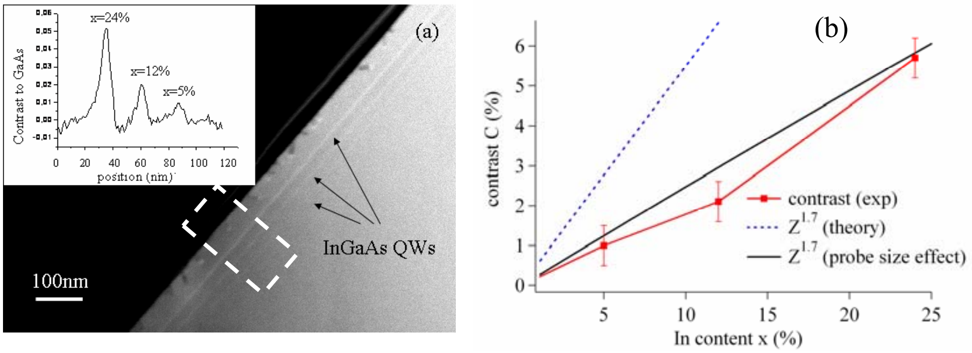

Figure 1. (a) Cross sectional DF image of $In_xGa_{1-x}As$ QWs in GaAs matrix. In the inset, the image contrast collected in the white dotted box is reported (sample thickness 40nm). (b) Comparison between the experimental contrast (C) dependence on In content measured from the DF image (red line), the theoretical C dependence ($I=Z^{1.7}$, blue dotted line), and the expected C dependence taking into account the probe size (6nm).

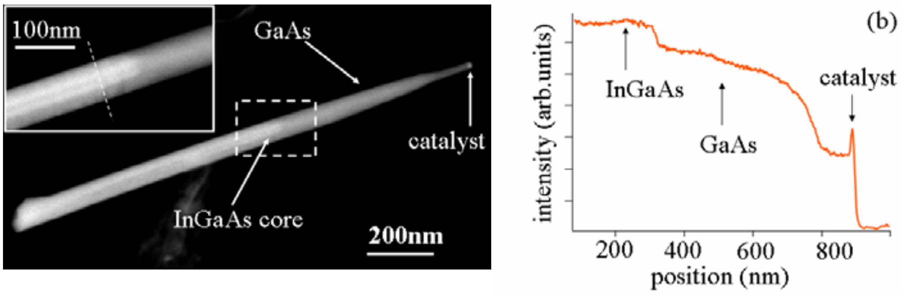

Figure 2. (a) DF image of a representative InGaAs/GaAs c-s NW. In the inset we show a magnified image of the region indicated by the white dotted box. (b) Longitudinal line profile.

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