HRTEM- Sample Preparation for Compressed Rolled- up- Nanomembranes

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Heterostructural semiconductor superlattices have attracted overwhelming consideration over the last years due to their electronic, optical or opto-electronic properties. Recently, an approach to realize radial hybrid superlattices (RSL) by the release and roll-up of strained nanomembranes has been established [1]. However, for certain device applications, a planar structure of such a hybrid superlattice would be more suitable. A planar hybrid superlattice (MeSSL) can be realized by simply pressing a RSL. This superlattice consists of two symmetrical inverted multilayer stacks with a mirror plane in the middle, leading to a phase flip of 180° (Figure 2) [2]. Figure 1 illustrates this process schematically.

In order to investigate the quality of the layer and interface inside the MeSSLs, the Transmission Electron Microscopy (TEM) is used. The aim of this work is to uncover the most suitable TEM preparation method .

At first, cross-sections of the samples were prepared with a Focus Ion Beam (FIB/ ZEISS NVision- 30kV Ga-ions). This preparation method leads to a wide electron transparent lamella with constant thickness. However, the disadvantages of the FIB technique lie in the depth of the Ga- ion implantation as well as amorphization of large parts of the lamella.

As a second TEM preparation method, the conventional ion beam etching technique was applied. For a cross-section, two sample pieces were glued face to face and ground down to a thickness of approximately 10µm using a modified Tripod. Subsequently, the samples were Ar- ion- etched using a RES101 (LEICA) with small angles and low energy.

Both samples were characterized for electron transparency, amorphization and suitability for HRTEM with a TEM TECNAI T20 (200kV/ LaB6) and a TITAN (80…300kV/Cs corrected).

Figure 2 shows a TEM bright field image of a FIB-prepared hybrid superlattice cross section fabricated by pressing a 3nm InGaAs/ 3nm GaAs/ 6nmCr RSL. The material contrast differs between the polycrystalline metal (dark) and the single-crystalline semiconductor (bright). The mirror plane is distinguishable as a double metal layer leading to two inverted multilayer stacks. An excellent quality of the FIB prepared lamella for TEM overview imaging is evident.

Figure 3 displays a HRTEM bright field image of a pressed MeSSL in the region of the AlAs sacrificial layer. An amorphous cover layer is clearly seen. Only a few regions are useful for HRTEM. In order to optimize the quality of the FIB lamella, a further preparation step by Ar-ion etching followed. The FIB lamella was mounted in a special holder of a RES101- ion etching system (LEICA). The lamella was etched with 2kV, 1,2mA, etching angle of 4° without sample movement for 5min per side.

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Figure 4 reveals that, after this final ion etching stage, the amorphous layers were removed. Unfortunately the lamella exhibits numerous distracting bend lines. However, in contrast to the lamella without additional Ar-etching, more satisfying transparent regions for HRTEM investigations were found.

The best result was produced by conventional preparation of an AlGaAs/ GaAs superlattice. Large transparent regions generate the best assumption for HRTEM (Figure 5). But, this method is not applicable in the case of nanomembranes. The FIB targeted preparation technique- possibly with subsequently Ar- ion etching- is the only possibility to prepare a suitable TEM sample because rigorous control over the preparation position is needed.

1. C. Deneke et al, Phys. Stat. Sol. © 5 (9), 2704 (2008)
2. T. Zander et al., Appl. Phys. Lett.94,053102 (2009)

Figure1. Formation of RSL by rolling up and MeSSL by subsequent pressing

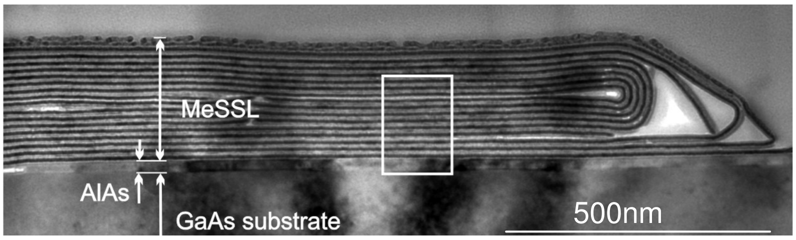

Figure 2.TEM Bright Field image : FIB prepared cross- section of a MeSSL

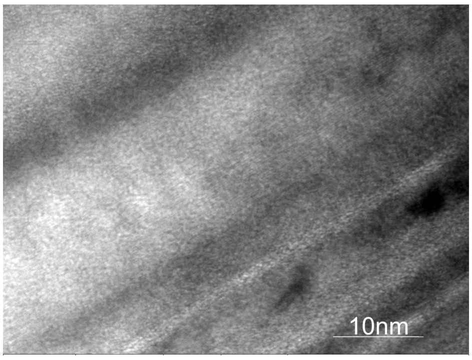

Figure 3. HRTEM: MeSSL- FIB-lamella

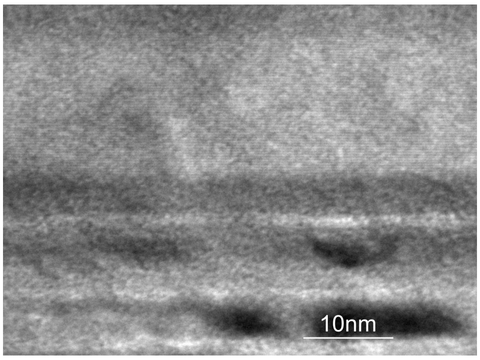

Figure 4. HRTEM: MeSSL- FIB-lamella + Ar- ion etching

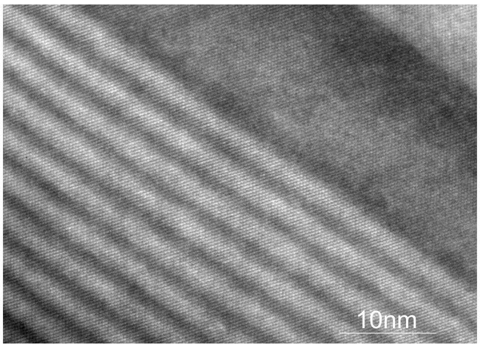

Figure 5. HRTEM: AlGaAs/ GaAs superlattice- conventional Ar- ion etching

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