Nano-Scaled Aperiodic Multilayer Systems for X-ray Optics: Quantitative Layer Thickness Determination by HAADF-STEM

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Quantitative high-resolution characterisations by transmission electron microscopy methods are essential for the assessment of the layer growth, of the coating control, and of the reflectivity properties of multilayer systems for X-ray optics [1,2]. Recent developments focus on state-of-the-art thin film X-ray optical multilayer coatings for current and next-generation synchrotron light sources. Aperiodic W-B_4C and W-C gradient or multistripe multilayer systems are of interest as filters with broad-bandwidth characteristics. The relevant structure parameters of such multilayer systems, such as local layer periodicity, layer thickness, and layer orientation, can be determined quantitatively and with high precision from investigations of cross-section specimens [1,2].

High-angle annular dark field Z-contrast STEM (HAADF-STEM) imaging of cross-section specimens allows to locally determine the thickness of the single layers, in addition to analyzing the bilayer thickness. As an example for quantitative layer thickness determination, **Fig. 1.a** shows a cross-section HAADF-STEM micrograph of a cross-section specimen of an aperiodic W-B_4C multistripe multilayer system. The investigations were performed using a TECNAI F 30 transmission electron microscope at 300kV. The experimental image contrast was quantitatively evaluated by applying a geometric phase method (GPA) [1] and by image intensity profiling. By applying these methods precisions of thickness determinations can be obtained that reach the sub-nanometer range. **Fig. 1.b** represents the distribution of bilayer thickness as obtained from evaluating the digital HAADF-STEM micrograph by applying the GPA method. **Fig.1.c** shows the corresponding result of an intensity profile analysis of a HRTEM micrograph. The quantitative values obtained by applying the two methods turn out to be in excellent agreement with each other and with the nominal deposition data. Analyses of periodic multilayer systems by TEM and by X-ray reflectivity measurements allow estimating the influence of interface roughness and electron probe size.

1. D. Häußler, E. Spiecker, S. Yang, W. Jäger, M. Störmer, R. Bormann, and G. Zwicker, phys. stat. sol. (a) 2299 (2005) p. 202.
2. D. Häußler, E. Spiecker, W. Jäger, M. Störmer, R. Bormann, C. Michaelsen, J. Wiesmann, G. Zwicker, R. Benbalagh, J.-M. André, and P. Jonnard, Microelectronic Engineering 84 (2007) p. 454.
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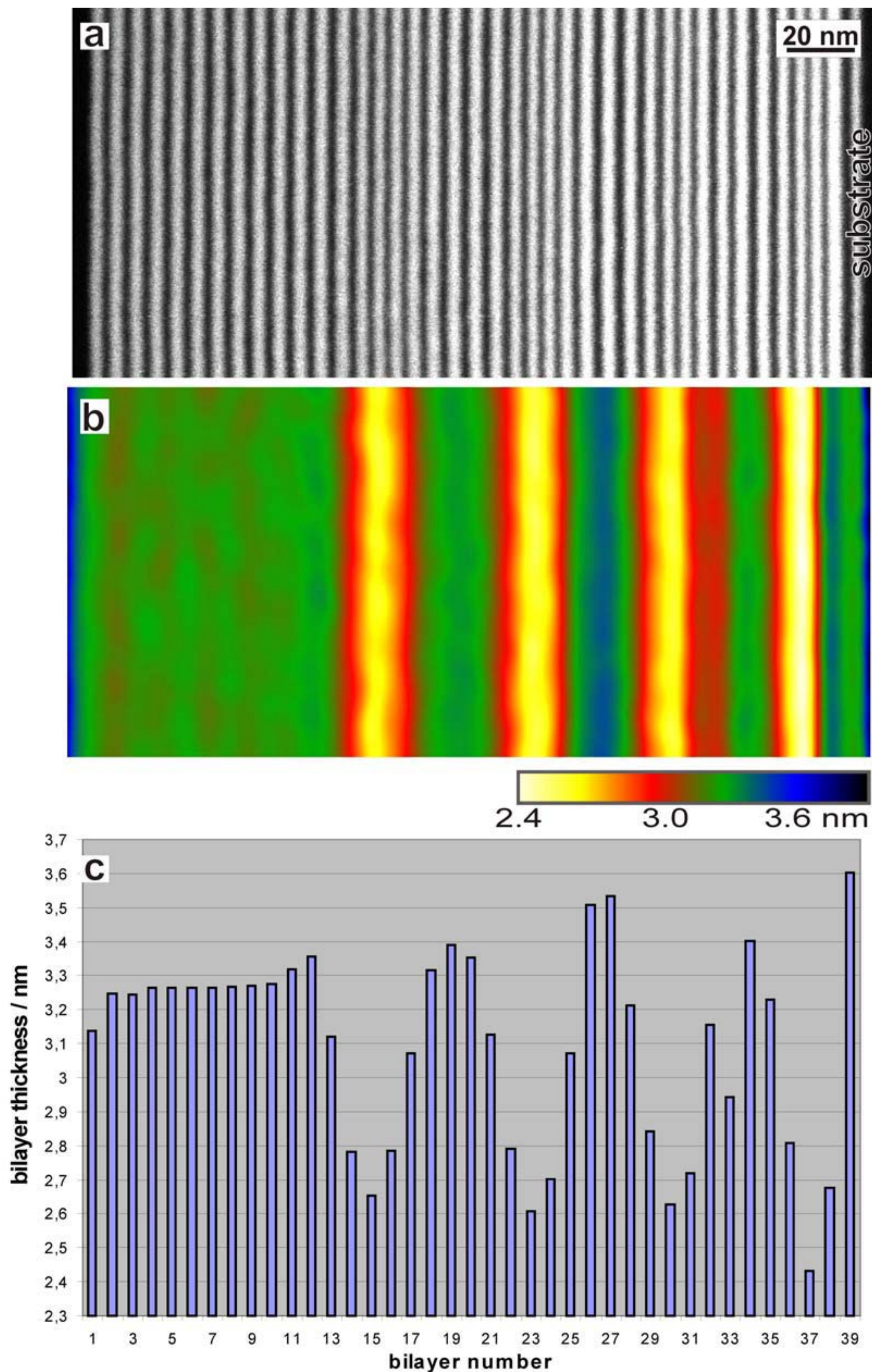

Figure 1. Layer thickness distribution for an aperiodic W/B_4C multilayer system on Si (100). **a**: HAADF-STEM cross-section micrograph. **b**: Colour-coded distribution of bilayer thicknesses as obtained from GPA. **c**: Bilayer thickness distribution (represented by column heights) as obtained from a line scan evaluation of a HRTEM micrograph (not shown) for the identical area of the specimen.

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