A.4: Probing of chemically and spatially resolved atomic profile at buried interfaces using reflectivity beam line at Indus 1.

Understanding of chemically and spatially resolved atomic profile at nano-scaled buried interfaces of thin film and multilayer is fundamentally interesting and also important in many applied fields. Properties of these structures are strongly influenced by presence of small quantity of impurities, layer composition, interfacial microstructure and chemical nature. Progress in understanding and predicting the properties relies on quantitative information about the distribution of these parameters at the atomic scale. Precise measurement of such information at deeply embedded interfaces are very scarce owing to the fact that there are not many techniques available to measure such information wherever layer thicknesses of ~ nm are involved. We have used resonant soft x-ray reflectivity (R-SoXR) measurements at Indus-1 soft x-ray reflectivity beamline to probe low electron density contrast (EDC) and low-Z system. The method has excellent chemical sensitivity to low-Z materials, high contrast variation and high resolution.

Samples are fabricated using ion beam sputtering on Si substrates with varying position of B_4C layer with respect to Si layer (Fig. A.4.1(a)). Measured hard x-ray reflectivity (XRR) profiles of three samples clearly appear very similar (Fig. A.4.1(b)). Thus, XRR is not sensitive to Si/B_4C interface having low EDC, $\Delta p/p=0.05\%$, and to compositional changes in the film if any due to lack of element-specificity. However, R-SoXR near BK-edge of B_4C shows (Fig. A.4.1(c)) different profiles which reveals sensitive to Si/B_4C interface due to contrast variation.

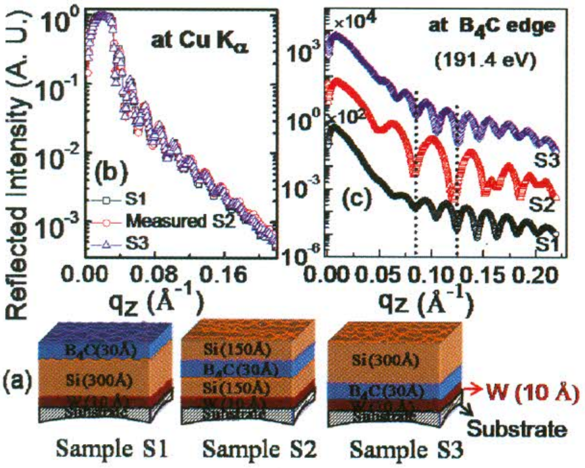

Fig. A.4.1: (a) Schematic of three samples with varying spatial positions of B_4C layer. (b) Measured XRR profile. (c) Measured R-SoXR profiles at a selected energy of 191.4 eV (B K-edge of B_4C).

In B_4C films, there may be a partial decomposition of B_4C to elementary boron and subsequently oxides. The question arises whether there is a chemical change in the B_4C layers, and if so, to quantity it and to determine the elemental distribution from the surface down to a depth of ~30 nm. The variation of R-SoXR near B_2O_3 edge reveals presence of B_2O_3in S1 (Fig. A.4.2). However, no variation of R-SoXR profiles near B K-edge of boron reveals that boron is not present in S1.

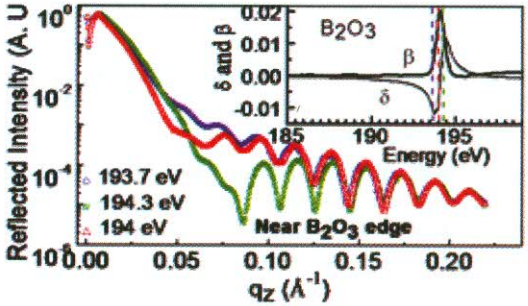

Fig. A.4.2: Measured R-SoXR profiles of sample S1 at selected energies near B K-edge of B_2O_3. Inset shows measured optical constants.

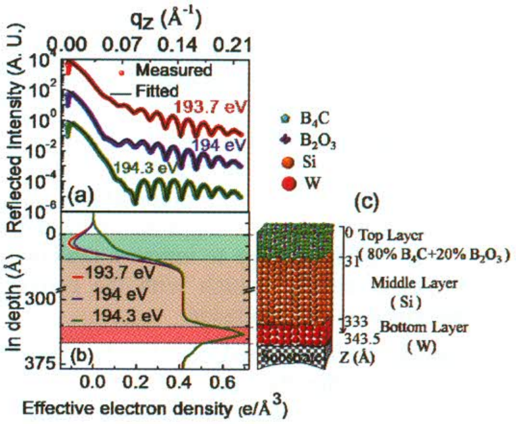

Fig. A.4.3: (a) Measured R-SoXR profiles along with fitted curves of S1near B_2O_3 edge. (b) EDP indicates different layers structure. (c) Schematic illustrates the vertical depth profile of composition.

To quantify atomic percent of B_2O_3 and the spatial distribution in B_4C layer of sample S1, R-SoXR measured data are fitted and electron density profile (EDP) is obtained (Fig. A.4.3). The best-fit data reveals the top B_4C layer is composed of ~80% B_4C and ~20% B_2O_3, The spatial distribution of composition is illustrated in Fig. A.4.3 (c). For more details, please refer to M. Nayak et al., Nature Sci. Rep.5, 8618 (2015).

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