Vacuum 88 (2013) 17–22

Contents lists available at SciVerse ScienceDirect

Vacuum

journal homepage: www.elsevier.com/locate/vacuum

Energy and angle dependent photoemission study on Si/Ge multilayers using synchrotron radiation

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ARTICLE INFO

Article history:
Received 26 April 2012
Received in revised form
9 August 2012
Accepted 10 August 2012

Keywords:
Synchrotron
Photoemission spectroscopy
Multilayers
Depth profiling

ABSTRACT

The present report adds to the ongoing research on Si/Ge nanostructures by discussing the results obtained utilizing unique features of Synchrotron radiation (SR). Based on the structural and electronic properties of this system investigated in our earlier work, we have synthesized $[Si(5\ nm)/Ge(5\ nm)]_{\times 10}$ multilayers (MLS) and exposed it to a range of incident photon energies (utilizing synchrotron radiation) and at different incident angles for the photoemission spectroscopic measurements. It is shown how the interface features are enhanced simultaneously suppressing the signals from topmost atomic layers. Also the effect of incident angle variation is discussed, which gives a clear picture of interface electronic properties of Si/Ge system. Significant changes in Si 3p, Ge 4p and s band features are observed in the valence band density of states. The overall results present an interesting observation of thoroughly investigating the basic building bilayer stack of the multilayer structure by combining energy and/or angle variation with low energy sputter depth profiling.

1. Introduction

The photoionization cross sections are dependent on incident photon energy, which makes it useful in studying different phenomena such as Fano resonance and Cooper minima [1]. Unique features of synchrotron radiation add to the analysis of such parameters [2,3]. In the case of semiconductors, photoemission spectroscopy (PES) using synchrotron radiation (SR) is extremely useful in sensing the role of defects, composition, crystallinity and local atomic bonding configuration as well as in probing the quantum size effect [4]. All these effects can be qualitatively/quantitatively examined using suitable combinations of electronic, structural and other properties [5–8]. These nanostructures have unique arrangement of energy levels, which are responsible for modifying the optical absorption and electronic transitions drastically [9,10] as compared to their bulk counterparts. Apart from these, various new effects like band bending, strain/stress, surface and interface effects, bond contractions etc. are observed. This is because the nanomaterials that lie in the domain in between the two extremes of bulk and single molecule exhibit properties that are entirely different from either of them [11–13]. Semiconductor nanostructures, particularly two-dimensional (thin films) are of current research interest owing to their structure dependent optical and electrical characteristics (conductivity, carrier diffusion length, mobility, absorption etc.) that can be custom designed to create new generation of miniature circuits and other electronic/photonic devices [14–29]. In such applications it is necessary to produce very high quality contamination free layers, which can only be done with the use of specific techniques revealing minute information on the electronic and structural properties. An elegant method of identifying the character of certain features in the valence band spectra is to employ resonant photoemission technique. Photoemission resonance is closely related to intra-atomic transitions and thus enhancements of photoemission features of a certain element often occur at an absorption edge for the respective element. Therefore, by using a synchrotron radiation source, the relative change in photoemission cross-section for various electron states can be used to determine the partial Density of States (DOS). A 'resonance' may occur whereby the photoemission intensity for a particular state may be enhanced at certain photon energies and angles [4,30,31]. This is caused when a high binding energy (BE) core level has its electrons excited to states whose energy is just above the Fermi level. The atom can relax by emitting an Auger electron, or a resonant enhancement of a valence band state can occur. Another characteristic for resonant photoemission is the fact that the kinetic energy (KE) of the spectral feature, which is resonantly enhanced, disperses linearly with photon energy. Especially in the case of solid samples, this might be

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http://dx.doi.org/10.1016/j.vacuum.2012.08.005

a very useful additional criterion for establishing whether the intensity increase of a valence band feature is truly resonant or not.

This method can particularly be useful for thin film nanostructures, because information can be obtained from different depths depending on requirement facilitating observations of individual layers in an MLS. This advantage can be a great addition to the ongoing research interest, which is now being shown worldwide in developing thin-film technologies due to the capability of producing high quality layers with reasonable growth rates and unusual properties [32–34]. In this respect, there has been considerable research on Si/Ge system in order to look for new possibilities of fabricating better performance devices such as high speed wireless communication systems and also for finding out the possible ways of making direct bandgap material that can perform better with the advantage of low cost as compared to costlier GaAs technology. Thus, it becomes essential to probe this system layer by layer in detail utilizing advanced characterization tools. Taking into consideration all the above mentioned aspects, we aim to investigate the electronic properties of Si/Ge thin film structures mainly looking into the depth profiling combining destructive (ion sputtering) and non-destructive (varying energy/angle) methods.

2. Experimental details

The multilayer structures (MLS) of [Si (5 nm)/Ge (5 nm)]$_{\times 10}$ with the starting layer of Ge were prepared by electron-beam evaporation technique under UHV conditions on float glass substrates at room temperature (base pressure $\sim 5 \times 10^{-10}$ Torr) [35]. The deposition rate of 0.01 nm/s for both Ge and Si was controlled using quartz crystal thickness monitor. Initial characterization of the samples was done using laboratory x-ray photoelectron spectroscopy and x-ray reflectivity to check the quality and mixing of the layers. Advance variable angle/energy PES measurements were performed on the UGC-DAE CSR PES Beamline installed at INDUS-1, RRCAT, Indore, India [36,37] by either adjusting the incident energy or by rotating the sample with respect to the source or analyzer to adjust incident angle. Energy calibration of the spectrometer (OMICRON EA 125) was done using known Fermi levels of standard samples such as Au, Ag and Pt. The energy resolution was ~0.8 eV. All the samples were properly grounded in order to avoid any charging effect. For depth profiling, low energy Ar^+ ions (~1 keV and 1–2 μA) were used to avoid any surface damage and intermixing during sputtering of the samples. The sputtering was done till we saw the first Si/Ge interface, so that we could simultaneously probe both layers.

Room temperature micro-Raman spectra were obtained on an integrated Raman system [Horiba Jobin Yvon HR800] with an excitation wavelength of 632.2 nm (HeNe, power = 9 mW). The spot size was <2 μm and the wave number accuracy was ±1 cm^{-1}. The micro-structural parameters of the multilayer were measured with grazing incidence x-ray reflectivity (GIXRR) setup of Bruker D8 Discover diffractometer equipped with a sealed Cu tube as the source of x-rays at $\lambda = 0.1542$ nm operated at 40 kV and 40 mA.

3. Results and discussion

3.1. Micro Raman measurements

The Raman spectrum recorded on as-deposited Si/Ge MLS shows some interesting features as shown in Fig. 1. This spectrum can be divided into two main regions: main broad peak corresponding to Ge-TO as a convolution of three signals centered on ~266 cm^{-1}, indicating that the deposited layers are amorphous/nanocrystalline in nature containing mostly the amorphous portion. The peak is shifted by ~4 cm^{-1} as compared to amorphous

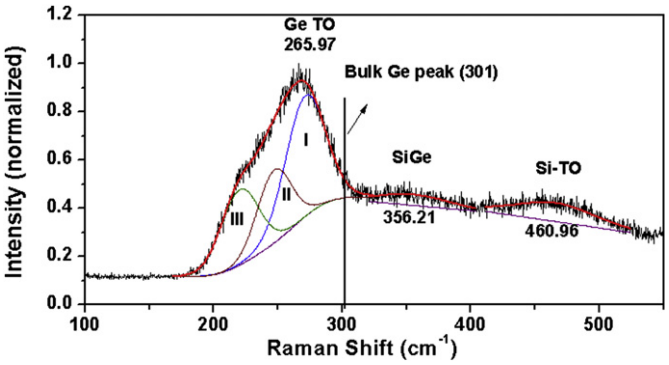

Fig. 1. Micro Raman spectra of as-deposited [Si (5 nm)/Ge (5 nm)]$_{\times 10}$ MLS.

bulk Ge (~270 cm^{-1}) and also from the crystalline bulk peak at ~301 cm^{-1}. The peak is deconvoluted in three peaks as shown in the figure. It is well reported that the shift toward lower value is due to the formation of very small size crystallites (a few nm) [14,15].

Similarly, the spectra also show a broad feature around ~491.5 cm^{-1} amorphous/nanocrystalline Si TO phonon mode. The shift toward lower wave number as compared to bulk in this case also indicates the presence of nano size particles in the deposited MLS. However, the peak position of Si_xGe_{1-x} is also marked in the figure, where one can see that it does not have contribution in the overall spectrum, confirming that there is practically no detectable intermixing between the constituent layers at their interfaces, although more sensitive measurement like x-ray reflectivity revealed some alloy formation at the interfaces.

3.2. Grazing incidence X-ray reflectivity measurements

For the as-deposited MLS, as depicted in Fig. 2, well-defined Bragg's peaks are observed upto the 3rd order, conforming that the Si/Ge interfaces are flat and parallel to the substrate surface. The x-axis of the spectrum is converted from 2θ to the momentum transfer vector Q_z (in order to account for the information in vertical direction in the film) using the relation:

$$Q_z = \frac{4*pi*\sin(\theta)}{\lambda} \quad (1)$$

Where θ is the incident angle and λ is the wavelength of Cu Kα radiation (0.154 nm). The reflectivity patterns are fitted using standard Parratt formalism to compute total multilayer thickness, bilayer periodicity, electron density and the interface quality in

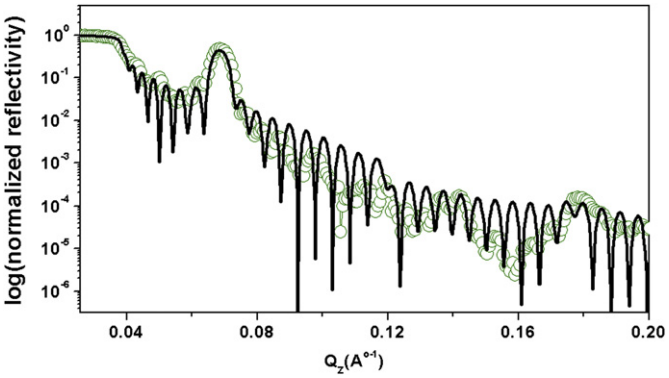

Fig. 2. GIXRR patterns of as-deposited [Si (5 nm)/Ge (5 nm)]$_{\times 10}$ MLS.