

HRTEM and EELS Analyses of Interfacial Nanostructures in Ti/Si_{1-x}Ge_x/Si(100)

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1. Introduction

With the dimension of electronic devices decreasing, contact resistance at metal/Si interfaces has become one of the most important problems. As one of the solution of this problem, it has been proposed to use Si_{1-x}Ge_x films as intermediate layers between Si substrates and metal films such as Ti[1]. It is known that under annealing, interfacial reactions between Si_{1-x}Ge_x and Ti layers lead to a production of C49-Ti(Si_{1-x}Ge_x)₂ and finally to a transformation into C54-Ti(Si_{1-x}Ge_x)₂ which is a low resistivity phase. For well-defined controlling of the interfacial reactions in the system, it is necessary to characterize nanometer-sized grains localizing at the interfaces. It is generally difficult to identify elemental composition and phases of them only by means of high resolution TEM (HRTEM) and electron diffraction. In the present study, we applied the combination technique of HRTEM and electron energy loss spectroscopy (EELS) to the Ti/Si_{1-x}Ge_x/Si system.

2. Experimental

Si_{1-x}Ge_x layers were epitaxially grown on Si(100) substrates by chemical vapor deposition. We prepared two types of Si_{1-x}Ge_x layers with different Ge fractions, that is, $x=0.14$ and 0.46 . Then Ti films with a thickness of 30 nm were deposited on the Si_{1-x}Ge_x layers by electron-beam evaporation. The interfacial reactions of Ti/Si_{1-x}Ge_x were promoted by annealing at 550 °C for 10 minutes and following rapid thermal annealing (RTA) at 750 °C for 30 seconds, the same as in practical device processes. The cross-sectional specimens of the Ti/Si_{1-x}Ge_x/Si hetero-structures were prepared by ion-milling method with 3 kV Ar⁺ ion. HRTEM observations and EELS measurements of the specimens were performed using a 200 kV TEM (JEM-2010F) equipped with an EELS spectrometer (Gatan DigiPEELS 766). EELS spectra from local areas 5-25 nm in diameter were obtained by using of a spectrometer-entrance aperture with TEM operated in the imaging mode.

3. Results and Discussion

Higher Ge fraction ($x=0.46$) sample

Fig.1(a) shows a cross-sectional TEM image of Ti/Si_{0.54}Ge_{0.46}/Si(100) after 550 °C annealing. Under the poly-crystalline layer with a thickness of about 30 nm, the

islands about 100 nm in size are formed at intervals and it is suggested that the islands trigger discontinuous C54 films after following 750 °C RTA [2,3]. From the HRTEM observation (Fig.1(b)), we clarified that their structure is Ti₆Ge₅-type. From appearance of the Si core-loss edge in EELS spectrum obtained by one of them (Fig.2(a)), it is concluded that the islands contain Si atoms, namely, are Ti₆(Si_{1-x}Ge_x)₅. Although the shape of the Si core-loss edge

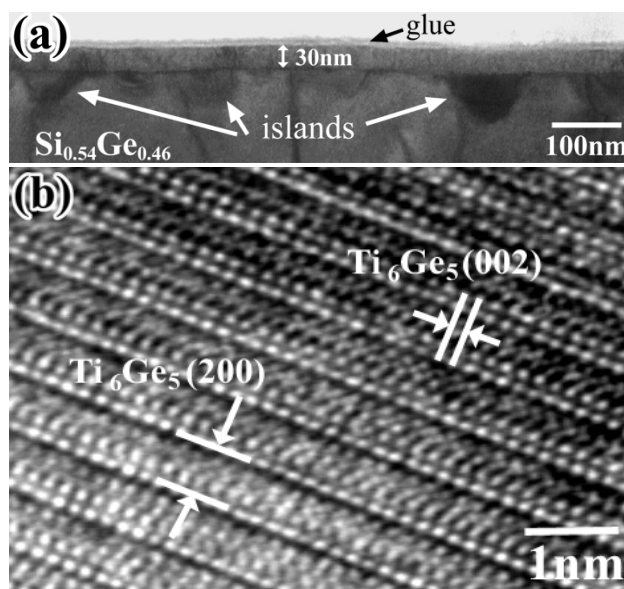


Fig. 1. Cross-sectional TEM images of the higher Ge fraction ($x=0.46$) sample. (a) low magnification image, (b) HRTEM image of an island shown in (a).

is sensitive to the bonding condition of Si atoms, those from the surface layer and the islands are very similar (Fig.2(a)). The result means that the poly-crystalline surface layer is also Ti₆(Si_{1-x}Ge_x)₅. From the results, we can obtain the following reaction model. First, Si and Ge atoms may diffuse into the deposited Ti layer from the Si_{1-x}Ge_x layer and then the Ti₆(Si_{1-x}Ge_x)₅ layer may be produced. Then Ti atoms may diffuse into less strained parts of the Si_{1-x}Ge_x layer and form the Ti₆(Si_{1-x}Ge_x)₅ islands, since the Si_{1-x}Ge_x layer contains compressive strains periodically induced by the lattice misfit between the substrate Si (100) and the Si_{1-x}Ge_x layer.

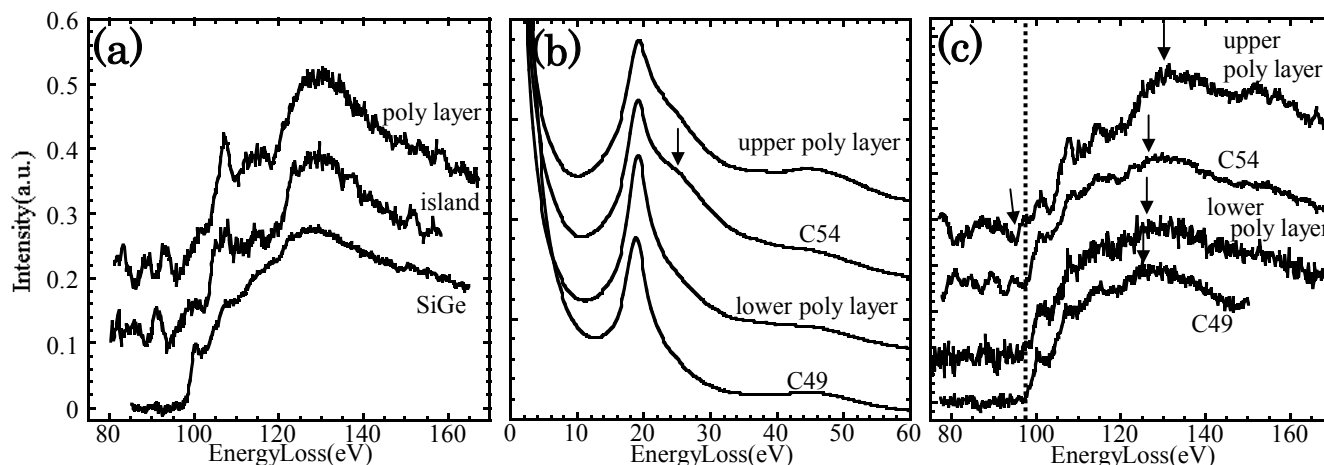


Fig. 2. EELS spectra from samples in which Ge fraction (a) $x=0.46$ and (b)(c) $x=0.14$. By detailed comparison, it is found that the lower poly layer in (c) is C49-Ti(Si_{1-x}Ge_x)₂ and that the poly layer and the island in (a) and the upper poly layer in (c) are the same phase, namely, Ti₆(Si_{1-x}Ge_x)₅.

Lower Ge fraction ($x=0.14$) sample

C54 layer with a thickness of about 50 nm is formed in lower Ge fraction samples ($x=0.14$) after following 750°C RTA (Fig.3(a)). Ti atoms may diffuse into the Si_{1-x}Ge_x layer not locally but uniformly owing to the less strain in lower Ge fraction film. It is also found that two poly-crystalline layers about 5-10nm in thickness exist over the C54 layer shown as black arrows in the figure and they are not detected by X-ray diffraction. In HRTEM image (Fig.3(b)), it is suggested that the lower poly-crystalline layer consists of C49 structure grains. In Figs.2(b) and (c), we show EELS spectra from these layers and a C49 layer in $x=0.14$ sample after 550°C annealing for a reference. By detailed comparison among them, it is concluded that the upper layer is Ti₆(Si_{1-x}Ge_x)₅ the same as the islands mentioned above and that the lower poly-crystalline layer is C49-Ti(Si_{1-x}Ge_x)₂. These layers may have failed to transform into the C54 phase by some surface effects. Since both C49-Ti(Si_{1-x}Ge_x)₂ and Ti₆(Si_{1-x}Ge_x)₅ are the high resistivity phases, they are undesirable for contact devices.

4. Conclusions

In the present study, we analyzed the nano-structures in the annealed Ti/Si_{1-x}Ge_x/Si systems and succeeded in clarifying their existence and structures by HRTEM and EELS. From the results, atomic diffusion and reaction processes in the interfacial reactions were discussed. It is considered that the HRTEM/EELS combined analysis is a very powerful technique to characterize advanced device materials.

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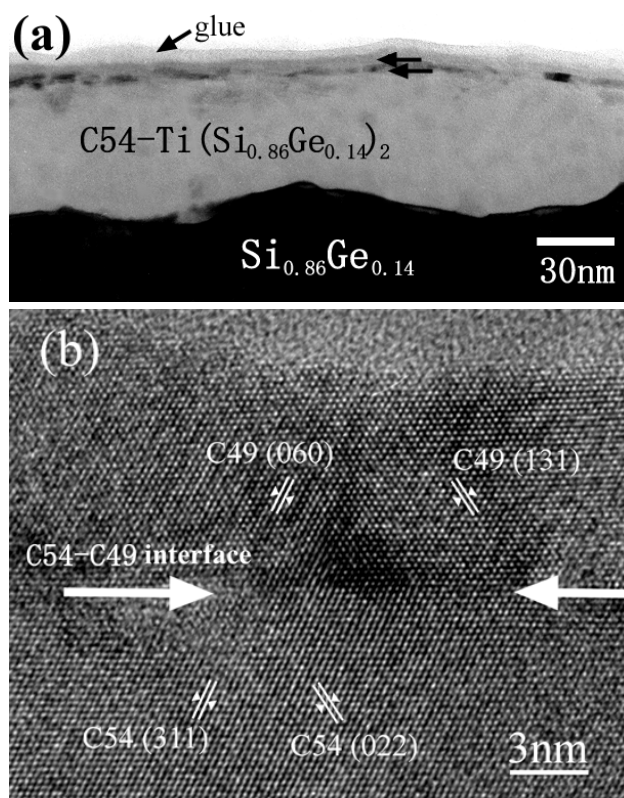


Fig. 3. Cross-sectional TEM images of lower Ge fraction ($x=0.14$) sample. (a) low magnification image, (b) HRTEM image of an interface shown in (a).

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