Effects of Carbon Pre-Germanidation Implantation on the Thermal Stability of NiGe and Dopant Segregation on Both n- and p-Type Ge Substrate

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In this work, the effects of carbon pre-germanidation implantation on the thermal stability of NiGe and dopant segregation on both n-type and p-type Ge substrate were investigated systematically. As-prepared NiGe films with carbon pre-germanidation implantation to different doses were characterized by means of sheet resistance measurement, X-ray diffraction (XRD), scanning electron microscopy (SEM), cross-sectional transmission electron microscope (X-TEM) and secondary ion mass spectroscopy (SIMS). The presence of carbon is proved to improve the thermal stability of NiGe formed on both n- and p-type Ge significantly, as well as to lead to dopant segregation (DS) of P and B at the NiGe/Ge interface. The homogeneous distribution of C within NiGe films and stuffing of C atoms at the NiGe/Ge interface is responsible for the enhanced thermal stability of NiGe and DS of P and B during germanidation process.

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As the continuous downscaling of metal-oxide-semiconductor field-effect-transistors (MOSFETs) to 10 nm and beyond nodes, germanium (Ge) material, owing to its high carrier mobility for both electrons and holes, has resurfaced as a promising candidate channel material to achieve enhanced device performance for future high speed complementary metal-oxide-semiconductor (CMOS) technology.1–5 As the continuous downscaling of metal-oxide-semiconductor field-effect-transistors (MOSFETs) to 10 nm and beyond nodes, germanium (Ge) material, owing to its high carrier mobility for both electrons and holes, has resurfaced as a promising candidate channel material to achieve enhanced device performance for future high speed complementary metal-oxide-semiconductor (CMOS) technology.1–5

Similar to Si-based MOSFETs technology in which nickel monosilicide (NiSi) is commonly employed for source/drain (S/D) contact metallization, nickel monogermande (NiGe), has also been chosen as the most promising contact material for Ge devices due to its low resistivity, low activation temperature, and the feasibility for self-aligned-germanidation process.6–10 However, the development of Ge-based CMOS technology is still confronting with enormous difficulties, such as the poor thermal stability of NiGe films, as well as large contact resistance especially for n-type substrates due to low activation concentration.11 Because of the low melting point of Ni and Ge are pretty fast which, in turn, lead to poor thermal stability of NiGe films at relatively lower annealing temperature compared to its NiSi counterpart.9,13–15

The agglomeration of NiGe films inevitably gives rise to increased sheet resistance and leakage current, and this hinders its widespread application in advanced Ge MOSFETs. In order to solve this problem, many methods such as alloying Ni with Pt, Zr, Ta and Pd,16–19 inserting an ultrathin Ti, Yb, Pb or Pt interlayer between Ni and Ge before germanidation,20–23 adding a capping layer24 and forming epitaxial NiGe on tensile-strained Ge-on-Si substrate,25 have been extensively reported to improve the thermal stability of NiGe films. The incorporation of carbon, in previous work, has been proved to be an effective way to suppress the agglomeration of NiSi.26–32 and NiSiGe,33 as well as to improve the thermal stability of NiGe films formed on undoped Ge substrate.34 For NiGe films formed on n- and p-type Ge substrate, how the incorporation of carbon influences the thermal stability of NiGe films and the dopant segregation at the NiGe/Ge interface is, however, rarely touched. Especially, despite excellent performance for Ge p-MOSFETs has been achieved,35 the fabrication of high performance Ge n-MOSFETs is still struggling because of high diffusivity and low activation concentration of n-type dopants such as P in Ge.36

It is well known that carbon plays a very important role in forming n-type ultra-shallow junction, as well as to enhance the active doping concentration in that it can prevent the rapid diffusion of P by forming PV- clusters.37 Besides, the incorporation of carbon can also effectively suppress the diffusion of B by forming carbon-interstitials (C-I) pairs.38 Therefore, it is of great importance and significance to perform a systematic investigation to elucidate the effects of carbon on stabilizing NiGe films and on the dopant segregation of P and B at the NiGe/Ge interface in this work.

Experimental

The process flow for the experiment is summarized in Fig. 1a. Approximately 1.5-μm-thick Ge layers were epitaxially grown on 8-inch p-type Si(100) substrate by Reduced Pressure Chemical Vapor Deposition (RPCVD). Note that the top 1.0-μm-thick Ge layer is free of defects as shown in Fig. 1b. After Ge pre-amorphization implantation (PAI) with dose to 6 \times 10^{15} cm^{-2} at 20 keV, phosphorus (P) or boron (B) ions to a dose of 3 \times 10^{15} cm^{-2} was implanted into Ge substrate at 6 keV and 2.5 keV, respectively. Prior to activation annealing, a 200-Å-thick SiO2 was deposited on wafers to prevent the out-diffusion of dopants by plasma enhanced chemical vapor deposition (PECVD). Rapid thermal annealing (RTA) was then employed at 600 °C/60 s in N2 ambient to activate dopants as well as to repair the implantation induced damage. After removing the PECVD oxide in dilute HF solution (1:10), carbon (C) ions with doses ranging from 0 to 6 \times 10^{15} cm^{-2} were implanted into n- and p-type Ge substrate at an energy of 2 keV. According to the SRIM simulation,39 C should peak at ~6 nm below the Ge surface. After cleaning Ge wafer in 1:100 diluted HF solution for 60 s to remove native oxide, the wafers were loaded into a sputtering chamber for the deposition of 10-nm-thick Ni films. Afterwards, the wafers were sliced into small pieces about 2 cm x 2 cm in size. Subsequently, the sample pieces were annealed isochronally for 30 s in a rapid thermal process ( RTP) chamber in N2 from 350 °C to 700 °C. Any unreacted Ni was selectively etched in 1:10 diluted HCl solution at 55 °C. The as-prepared NiGe samples were characterized by four-point probe measurement for sheet resistance (Rsh), X-ray diffraction (XRD) for phase identification, scanning electron microscopy (SEM) for both surface and interface morphology, transmission electron microscopy (TEM) for microscopic observations, and secondary ion mass spectroscopy (SIMS) for the distributions of carbon, phosphorus and boron in NiGe films.

Results and Discussion

For NiGe films with different C doses formed on both n- and p-type Ge substrate, the variation of sheet resistance (Rsh) with germanidation...
Epitaxial growth of Ge on silicon substrate
Ge pre-amorphization implantation
Phosphorus/Boron implantation
20-nm SiO₂ deposited on Ge wafers
RTA at 600 °C and then remove SiO₂
Carbon implantation into Ge with various doses at 2 keV
Native oxide removal (HF:H₂O = 1:100)
10-nm Ni film deposition
RTP (350 °C to 700 °C for 30 s)
Unreacted Ni removal (HCl:H₂O = 1:10)

Figure 1. (a) Process flow for the experiment and (b) transmission electron microscopy image of Ge epitaxial layer on Si substrate.

The temperature is shown in Fig. 2, respectively. As seen, with and without C, the sheet resistance of NiGe films formed on n- and p-type substrate shows similar tendency as the increase of germanidation temperature. For NiGe films without (w/o) C as references, the sheet resistance starts to increase at 300 °C for n-type and 450 °C for p-type substrate at the onset of agglomeration of NiGe films. At germanidation temperature ≥ 550 °C, a sharp increase in Rₕₙ is evident indicating the integrity breakup of NiGe films and this will be verified by top-view and cross-sectional SEM images later. In stark contrast, for NiGe films with C formed on both n- and p-type substrate, the temperature at which Rₕₙ starts to increase is shifted from 500 °C and 450 °C for references to higher temperature e.g. 600 °C, indicating significant improvement of thermal stability for NiGe films by the incorporation of carbon. However, as the increase of carbon doses from 0 to 6 × 10¹⁵ cm⁻², the sheet resistance continuously increases, manifesting that the thermal stability of NiGe films is improved at the expense of increased sheet resistance. Compared to references without C, the larger sheet resistances for NiGe films with C may be attributed to more grain boundary scattering as well as the change of both preferred orientations and grain size of NiGe by the incorporation of carbon within NiGe films. Therefore, a tradeoff between the increase in sheet resistance and improvement of thermal stability should be made for the practical application of NiGe films in Ge-based devices.

For NiGe films formed on both n- and p-type Ge substrate at 600 °C to different C doses, the XRD diffractograms are shown in Fig. 3a and 3b respectively. For phase identification, conspicuous difference can be observed for NiGe films with and without C as the increase of C doses. For NiGe films formed on n-type substrate, some peak like NiGe(301) is only detected for the reference without C whereas some peak like NiGe(112) only occurs at the presence of C. With regard to NiGe films formed on p-type substrate, the NiGe(301) peak is only seen at the presence of C. As a result, it can be concluded that the incorporation of carbon within NiGe films indeed results in the change of the preferred crystalline orientation of NiGe grains. Some certain crystalline orientations like NiGe(112) and NiGe(301) for n- and p-type Ge substrate respectively are favored at the presence of carbon which may withstand high germanidation temperature. If compared to Ni-Si system, NiGe₂, the counterpart of NiSi₂ emerging at high silicidation temperature, is, however, always absent no matter the germanidation temperature. The fact that the phase transformation NiGe⁺Ge → NiGe₂ is not thermodynamically favorable should be ascribed to.⁴⁰

In Fig. 4 and 5, the top-view and cross-sectional SEM images of NiGe films with different C doses on n-type Ge substrate are shown respectively. As seen, the NiGe films for the reference without C is uniform and continuous until 500 °C as shown in Fig. 4a and 5a. However, severe agglomeration takes place at 550 °C as demonstrated in Fig. 4b, where a large dark patch representing exposed Ge substrate dispersed in NiGe films. This agglomeration results in the drastic increase in sheet resistance shown in Fig. 2a. In sharp contrast, even after germanidation temperature at 600 °C, the NiGe films with C to various doses display continuous and uniform surface morphology as well as pretty smooth NiGe/Ge interface in Fig. 4c–4g and

Figure 2. Variation of sheet resistance of NiGe films with germanidation temperature to different C doses on (a) n-type and (b) p-type Ge substrate.

Figure 3. XRD diffractograms of NiGe films formed on (a) n- and (b) p-type substrate at 600 °C with different C doses.
Fig. 5c–5g, respectively. This is also well consistent with the low sheet resistance in Fig. 2. It is reported that the incorporation of carbon can improve the morphology and phase stability of NiSi effectively. It can be speculated that the enhancement of thermal stability for NiGe is also ascribed to the presence of carbon in NiGe films. The grain boundary and interface energies would be modified by carbon atoms segregated at NiGe grain boundaries and at the NiGe/Ge interface which stabilizes NiGe films. From the view of kinetics, the improved atomic diffusion along grain boundaries and the NiGe/Ge interface due to the presence of C, giving rise to delayed agglomeration similar to NiSi case. Furthermore, as the increase of C dose, it is clearly shown that the NiGe/Ge interface is becoming smooth and this can be explained by increasing segregated carbon atoms at the NiGe/Ge interface which leads to decreasing interfacial energy thus stabilize the NiGe/Ge interface even more effectively.

The top-view and cross-sectional images of NiGe films formed on p-type substrate with different C doses are demonstrated in Fig. 6 and Fig. 7 respectively. Compared to their counterparts formed on n-type substrate, the NiGe films without C is uniform and continuous until 450 °C shown in Fig. 6a and Fig. 7a and severe agglomeration occurs at 500 °C as displayed in Fig. 6b and Fig. 7b which is also in good agreement with the drastic increase in sheet resistance shown in Fig. 2b. Similarly, the NiGe films formed on p-type substrate with different C doses also exhibit continuous and uniform surface morphology as well as smooth NiGe/Ge interface at high germanization temperature e.g. 600 °C, as depicted in Fig. 6c–6g and Fig. 7c–7g, respectively. The cause for the improved thermal stability by the incorporation of carbon is provided explicitly in previous section. It is worth noting that not like the NiGe films formed on n-type substrate, the NiGe grains are becoming bigger as the increase of C dose on p-type substrate. This could be interpreted as what follows. The incorporation of carbon can lead to the decrease of boron-interstitials (B-I) pairs by forming carbon-interstitials (C-I) pairs and the released B atoms, in turn, result in reduced nucleation density of NiGe which facilitates the 3D growth of NiGe grains. As the increase of C dose, the nucleation density of NiGe is becoming lower due to more released B atoms. As a result, the NiGe grains grow faster and the grain size is bigger for NiGe with larger C dose. However, for NiGe films formed on n-type Ge substrate, C atoms react with PV clusters to form stable carbon-vacancy-phosphorus (C-V-P) clusters in Ge. Consequently, no available released P atoms can give rise to reduced nucleation density of NiGe and this is responsible for small size of NiGe grains on n-type Ge substrate. On the other hand, it was reported that the introduction of P in Ge didn’t essentially alter the lattice structure of Ge whereas the B incorporation can induce large strain due to its small atom size than Ge. The growth of NiGe grains during germanization could be facilitated by the strain due to the incorporation of B. As a result, compared to NiGe films formed on n-type Ge
substrate, NiGe grains grow faster and become bigger on p-type Ge substrate.

The cross-sectional TEM images of NiGe films without C (500 °C on n-type Ge, 450 °C on p-type Ge) and with C to $2 \times 10^{15}$ cm$^{-2}$ (600 °C on both n- and p-type) are shown in Fig. 8 and Fig. 9, respectively. As seen, the thickness of NiGe films formed on both n- and p-type Ge are approximately 23 nm. In Fig. 8a and Fig. 9a for the references without C, the NiGe films formed on both n- and p-type Ge substrate with C also coincide well with the NiGe/Ge interface is pretty rough, indicating that the grooving tendency for NiGe films without C indeed occurs. However, for NiGe films with C shown in Fig. 8b and Fig. 9b, the surface morphology and the NiGe/Ge interface is obviously more smooth and flat. In the enlarged TEM images of NiGe films, compared to the rough NiGe/Ge interface for the reference without C, the NiGe films with C show well-defined NiGe/Ge interface with highly textured structure, demonstrating the formation of atomically flat NiGe/Ge interface without obvious defects. The uniform NiGe films as well as flat NiGe/Ge interface evidenced by TEM on both n- and p-type Ge substrate with C also coincides well with SEM images and the explanation is explicitly provided in previous SEM section.

For NiGe films with and without C formed on n- and p-type Ge at 500 °C, the secondary ion mass spectroscopy (SIMS) depth profiles for different elements are displayed in Fig. 10a and 10b, respectively. As seen, the flat plateaus of Ni and Ge are revealed clearly indicating the formation of uniform NiGe films formed on both n- and p-type Ge at 500 °C. For the NiGe film with C formed on n-type Ge in Fig. 10a, a plateau of C within NiGe film as well as a small shoulder at the NiGe/Ge interface is evident, and this manifests the homogeneous distribution of C within NiGe and the segregation of C at the NiGe/Ge interface. Due to the low solubility of C in NiGe, most C atoms should stuff at NiGe grain boundaries and at the NiGe/Ge interface. The segregation of C at grain boundaries and at NiGe/Ge interface is believed to play an important role in improving the thermal stability. Interestingly, an obvious peak of phosphorus or called P dopant segregation for the NiGe film with C is observed whereas the peak doesn’t appear for the reference without C, indicating that the suppression of P diffusion by the incorporation of C. It is reported that the phosphorus diffuses in Ge dominantly via vacancy-phosphorus pairs and the diffusion of vacancy-phosphorus pairs can be retarded in the presence of carbon. As a result, released P atoms during the reaction of Ni+Ge $\rightarrow$ NiGe partly volatilize to the circumstance and the rest have to segregate at the NiGe/Ge interface. Without C, the diffusion of P is not retarded and this leads to the deeper distribution (longer tail) of P as shown in Fig. 10a. Similarly to n-type counterpart, C also distributes homogenously within the NiGe film and a small shoulder occurs at the NiGe/Ge interface on p-type Ge. It is worth noting that with C, an obvious B peak at the NiGe/Ge interface can be seen indicating the significant B dopant segregation at the interface. However, for NiGe film without C as reference, no B peak at the interface appears at all. It has been reported that the diffusion of B is effectively suppressed by the incorporation of C in Si, it is speculated that the diffusion of B in Ge is also suppressed in a similar way by the competition between B and C atoms for the capture of Ge self-interstitials. Due to the formation of carbon interstitials (C-I) pairs similar to the C in n-type Ge, without C/500 °C

n-type, without C/500 °C

n-type, with C/600 °C

p-type, without C/450 °C

p-type, with C/600 °C

p-type, without C/450 °C

p-type, with C/600 °C

n-type, without C/500 °C

n-type, with C/600 °C

(a) n-type, without C/500 °C

(b) n-type, with C/600 °C

Cross-sectional TEM images of NiGe films formed on n-type Ge: (a) at 500 °C without C, and (b) at 600 °C with $2 \times 10^{15}$ cm$^{-2}$ C.

Cross-sectional TEM images of NiGe films formed on p-type Ge: (a) at 450 °C without C, and (b) at 600 °C with $2 \times 10^{15}$ cm$^{-2}$ C.

SIMS depth profiles of elements (a) for NiGe film formed on n-type Ge with $4 \times 10^{15}$ cm$^{-2}$ C and (b) for NiGe film formed on p-type Ge with $2 \times 10^{15}$ cm$^{-2}$ C at 500 °C. For comparison, the SIMS profiles of elements for NiGe films formed on both n- and p-type Ge without C as references are also provided.

![Cross-sectional TEM images of NiGe films formed on n-type Ge](image1)

![Cross-sectional TEM images of NiGe films formed on p-type Ge](image2)

![SIMS depth profiles of elements](image3)
SiGe case, the effective suppression of B diffusion is attributed to the reduction of available interstitials which facilitates the diffusion of B. Therefore, suppressed diffusion leads to the segregation of B at the NiGe/Ge interface in the presence of C. It should be mentioned that the incorporation of C not only improves the thermal stability of NiGe films, but also leads to significant dopant segregation of P and B at the NiGe/Ge interface. This dopant segregation is believed to help in reducing the contact resistivity between NiGe and both n- and p-type Ge substrate, especially for p-type Ge because of enhanced B DS.

Conclusions

In summary, the effects of carbon pre-germanization implantation on the thermal stability of NiGe films and dopant segregation on both n-type and p-type Ge substrate were investigated systematically. It is found that the incorporation of C not only enhances the thermal stability and morphology of NiGe films formed on both n- and p-type Ge substrate by approximately 100 °C, but also suppresses the diffusion of P and B which results in dopant segregation of P and B at the NiGe/Ge interface during germanization process. The uniform distribution of C within NiGe films and the segregation of C at the NiGe/Ge interface should be responsible for the improved thermal stability and suppression for P and B diffusion. These findings in this work make the approach using carbon pre-germanization implantation a viable method in fabricating high performance Ge-based devices in the future.

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