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P-doped Germanium Nanowires with Fano-broadening in Raman Spectrum

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Abstract: The optimized growth conditions for high density germanium (Ge) nanowires and P-doped Ge nanowires on Si (111) substrate were investigated, the phosphorus (P)-doping in Ge nanowires was also characterized. Vapor liquid solid-low pressure chemical vapor deposition (VLS-LPCVD) of Ge nanowires was conducted with different thicknesses of Au film as catalyst, different flow rates of GeH_4 as precursor and PH_3/Ar as co-flow. The morphologies of the Ge nanowires were characterized by scanning electron microscopy (SEM), the P-doping was verified by micro Raman spectroscopy via measuring the P local vibrational peak (342-345 cm^{-1}) and asymmetric broadening of Ge-Ge vibrational peak (about 300 cm^{-1}), respectively. The characterization results show that 1 nm thickness of Au catalyst is the most suitable condition among thicknesses of 0.1, 1, 5, and 10 nm for the growth of high density Ge nanowires at 300 and 350 ℃, and 0.5 sccm is the best flow rate of PH_3/Ar to grow high density and large scale P-doped Ge nanowires among flow rates of 0.5, 1 and 2 sccm. The P impurity can be doped into Ge nanowires effectively during LPCVD process at 350 ℃.

Key words: germanium; nanowires; LPCVD; doping; VLS

1 Introduction

One dimensional semiconducting nanowires/nanorods possess some unique properties different from the corresponding bulk materials, such as low-dimensionality, quantum confinement effects, surface sensitivity and low leakage currents which make them as attractive candidates for building blocks in functional micro/nano systems applied in electronic and optoelectronic fields[1-6]. Germanium (Ge) nanowire, due to its unique characteristics such as low effective mass of electron and hole, along with high carrier mobility (electron mobility: 3 900 $cm^2/(V·s)$, hole mobility: 1 900 $cm^2/(V·s)$), is a superb channel material for semiconductor devices[7]. Besides, Ge is an IV-group element, and has several interesting properties similar to Si that are suitable for nanoelectronic applications in many aspects[8-13]. Therefore, Ge nanowires are being actively investigated for various applications including high-speed field-effect transistors and p-n junction devices[12].

Considerable amounts of studies have been employed on the bulk synthesis of nanowires involving solution, template-assisted, arc discharge, and laser ablation[14,15], *etc*. Also, other methods such as physical vapor deposition (PVD), epitaxial growth from single crystal substrates using supercritical fluid to get nanowire composites and "Pick and Place" have been reported to synthesize high-density arrays of nanowires[16-18]. Generally there are two approaches for synthesis of nanowires: the "top-down" and the "bottom-up" approaches. The "top-down" approach is based on etching technique, such as wet chemical etching or reactive ion etching, etc. In contrast, for the "bottom-up" approach, the nanowires grow from the vapor phase via a catalyst, and liquid phase under high pressure[19,20], *etc*. In most cases, the nanowires grow, through chemical vapor deposition (CVD) method via vapor-liquid-solid (VLS) mechanism[21,22]. In this

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research, Ge nanowires have been synthesized via a VLS growth by low pressure chemical vapor deposition (LPCVD).

Doping impurity can change the Fermi energy and substitute some atoms of semiconductor, and its electrical properties can be changed after doping[23-25]. There are many methods to achieve effective doping such as dual-laser dual-target evaporation source absorption[11], CVD and ion implantation[26], *etc*. Group IV Semiconductor nanowires, such as Si and Ge, can be n-type or p-type with phosphorous (P) or boron (B) doping by exposing them to PH_3 or B_2H_6 during growth. Electronic doping can be achieved by introducing PH_3 or B_2H_6 gases in the nanowires' growth. However, in the case of Ge nanowires, the dominant mechanism for P and B atoms incorporation is through the nanowires' surface via the VLS mechanism. As a doping material, P has its own special advantages compared with the other materials. Liber and co-workers have demonstrated that carrier type (electrons, n-type; holes, p-type) and carrier concentration in single-crystal nanowires can be highly regulated during growth using phosphorus and boron dopants[27].

The wafer preparation accounts a lot in Ge nanowires' growth. Specifically, in case of the nanowires' growth, influences from different orientations of Si substrates have been investigated, and two kinds of substrates of (100) and (111) with H-terminated or SiO_2-terminated have been investigated for the nanowires' growth[28]. The results clearly indicated that high density nanowires can be grown on H-terminated Si (111) substrate compared with other substrates[21,28]. Therefore, Si (111) substrate was chosen for the growth of Ge nanowires in this research. Furthermore, the growth directions of nanowires are mainly <111> and <110> directions, so the vertical-predominated growth of nanowires on wafer surface can be realized on Si (111) substrate[29,30]. Also, HF treatment is essential for the growth of Ge nanowires since the HF solution in the colloid solution etches away residual silicon dioxide that remains on the substrate, resulting in better attachment between nanoparticles and substrate surface[28]. Some reported results also demonstrated that the samples without any native oxide removal or samples left in air for 1-2 h between oxide removal and Au deposition obtain random directions of nanowires' growth[31].

The growth of nanowires often relies on the catalyst preparation process which endows the catalyst a crucial role in VLS growth. Consequently, patterning of catalyst is an emerging challenge for realizing controlled synthesis of low-dimensional materials. In this research, Au was chosen as a catalyst for Ge nanowires' growth since the Au-Ge system exhibits one of the lowest growth temperatures[18]. VLS-LPCVD growth of Ge nanowires by low temperature decomposition of GeH_4 was realized in this research, the undoped and P-doped Ge nanowires on Si (111) substrates have been synthesized using Au as catalyst and GeH_4 as precursor.

2 Experimental

In the previous works, Ge nanowires on Si (100) and Si (111) substrates were prepared by a VLS-CVD method using Au as catalyst and 10% GeH_4/H_2 as precursor, and the results showed that high density Ge nanowires grown vertically on Si (111) when HF treatment of substrates was employed just prior to the CVD process[21,28]. In this research, Ge nanowires with P-doping were synthesized and the effect of doping was characterized by micro Raman spectrum measurement[32]. Low temperature decomposition of GeH_4 for growing Ge nanowires has been performed in this research, and the undoped and P-doped Ge nanowires on Si (111) substrates have been synthesized by LPCVD using Au as catalyst and GeH_4 as precursor. In the synthesis process, Ge nanowires grown from GeH_4 can be illustrated as the following steps: 1. Si substrates with Au catalyst were heated to the desired temperature; 2. Au catalyst activated the thermal decomposition of GeH_4; 3. Au-Ge alloy was formed with the diffusion of Ge atoms for nucleating of Ge nanowires; 4. Au catalyst particles solidified at the tip of Ge nanowires[21].

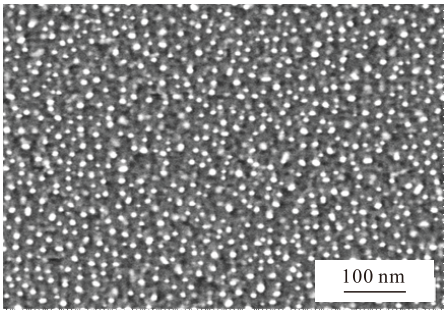

Fig.1 SEM image of surface of Si (111) substrate after Au deposition and HF treatment

In this research, H-terminated Si (111) substrates with a resistivity of 0.2 $\Omega \cdot cm^{-1}$ were used for growing Ge nanowires. Before the Ge nanowires' growth, the residual contamination on the substrates was

removed by a cleaning process that first cleaned with deionized water, acetone, deionized water, H_2SO_4 mixed with H_2O_2 (2:1) step by step, and then treated by HF solution (5%) to obtain H-terminated surface. After these steps, substrates were loaded into electron beam (E-beam) evaporation chamber for deposition of Au catalyst, in this deposition process the current of E-beam evaporation was 45 mA, and the deposition rate was about 0.1 Å/s. For optimization of nanowires' growth conditions, different thicknesses of Au catalysts have been investigated, namely 0.1, 1, 5 and 10 nm. As shown in Fig.1, it is obvious that the Au particles were deposited uniformly on the substrate and the sizes of Au particles are approximately 5-10 nm.

The LPCVD system used for growing Ge nanowires consists of the sample chamber for nanowires' growth, the gas control unit, and the temperature control unit. It is a resistively heating system, and the temperature of the sample holder is considered to be constant at the center of the chamber. In order to grow Ge nanowires, the substrate was heated to 300 or 350 ℃, and then exposed to precursor gases in the LPCVD chamber. In this LPCVD process, growth of Ge nanowires was carried out at 300 ℃ or 350 ℃ with 10% GeH_4/H_2 as precursor under a total pressure of 5 Torr, the typical growth was conducted in a period of 30 min. And for the synthesis of the P-doped (n-type) Ge nanowires, PH_3 diluted in Ar (10%) was used as co-flow during the nanowires' growth.

For the purpose of effective doping of P in Ge nanowires, different flow rates of PH_3 gas at different temperatures were investigated in this process. Morphologies and P-doping of the Ge nanowires were characterized by scanning electron microscopy (SEM) and micro Raman spectroscopy, respectively.

3 Results and discussion

3.1 Synthesis of Ge nanowires on a large scale

As shown in Fig.2, it is obvious that all the substrates were covered by undoped Ge nanowires, some nanowires have tapered morphology for deposition of Ge atoms via CVD mechanism on the sidewalls of nanowires. For the undoped Ge nanowires shown in Fig.2, the typical diameters of nanowires are 15-30 nm, and the lengths of them are 400-1 000 nm. Compared with the nanowires observed in Fig.2, the undoped nanowires shown in Fig.3 are less ordered and in smaller scale. There are kinks and nanowires which are disordered as shown in Fig.3(c), the kinks in larger nanowires occur via multiple twinning events facilitated by the slow growth and anisotropic catalyst/wire interfaces[33]. The flow rates of GeH_4/H_2 (1 sccm) and Ar (1 sccm) are same in the undoped Ge nanowires' growth as shown in Fig.2 and Fig.3. Therefore, 1 nm thickness of Au is a better condition than 5 and 10 nm for synthesis of undoped high density Ge nanowires on Si (111) substrates with the same flow rate of precursor.

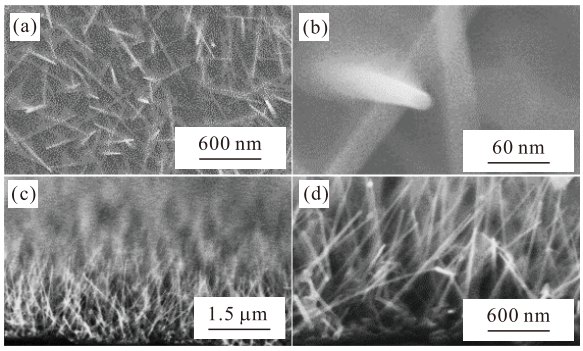

Fig.2 SEM images of Ge nanowires grown on Si (111) substrate (Au: 1 nm; GeH_4/H_2: 1 sccm; Ar: 1 sccm). (a) Top-view of the Ge nanowires; (b) Magnified view of the Ge nanowires; (c, d) Cross-section view of the Ge nanowires

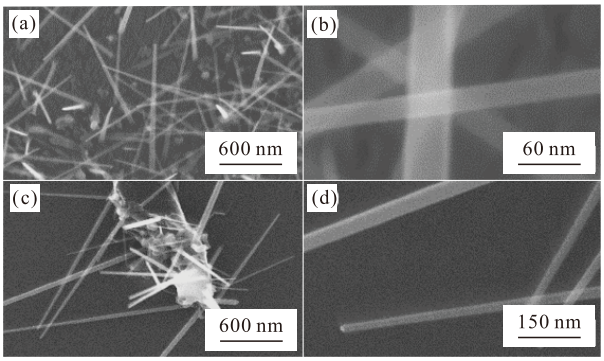

Fig.3 SEM images of Ge nanowires grown on Si (111) substrate with different thicknesses of Au: (a, b) 5 nm; and (c, d) 10 nm under the GeH_4/H_2 1 sccm and Ar 1 sccm flows

From the morphologies of undoped Ge nanowires observed in Fig.4 and Fig.5, it can be concluded that, the Si substrates ((100) or (111)) with 1 nm thickness of Au catalyst (Figs.4 (b, d) and Figs.5 (b, d)) result in higher density and larger scale of Ge nanowires than that of 0.1 nm thickness of Au catalyst (Figs.4 (a, c) and Figs.5 (a, c)). Also, it is apparent that for the substrates with different thicknesses of Au (0.1 or 1 nm), more ordered, higher density and larger scale of undoped Ge nanowires can be grown on Si (111) substrate (Figs.4 (c, d) and Figs.5 (c, d)) than that of Si (100) substrate (Figs.4 (a, b) and Figs.5 (a, b)). This result corresponds to the reported result that the synthesis of undoped

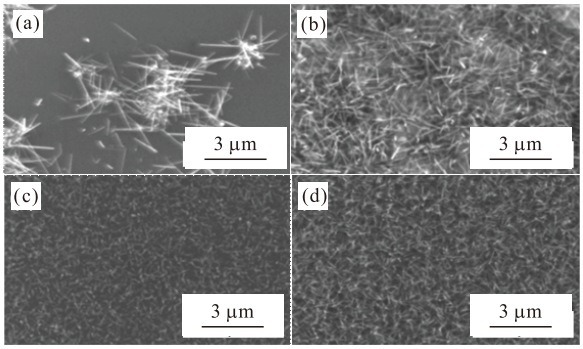

Fig.4 SEM images of Ge nanowires grown under different conditions: (a) Si (100), Au 0.1 nm, GeH_4/H_2 1 sccm, Ar 0.1 sccm; (b) Si (100), Au 1 nm, GeH_4/H_2 1 sccm, Ar 0.1 sccm; (c) Si (111), Au 0.1 nm, GeH_4/H_2 1 sccm, Ar 0.1 sccm; (d) Si (111), Au 1 nm, GeH_4/H_2 1 sccm, Ar 0.1 sccm

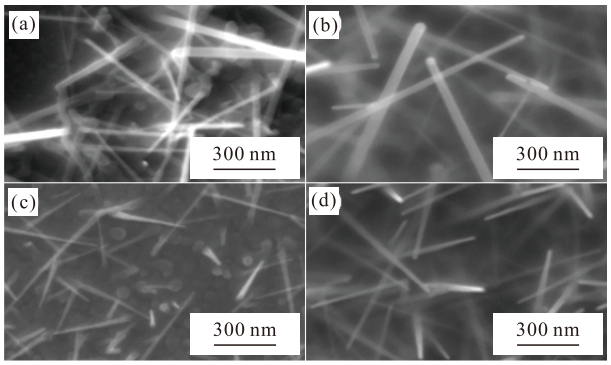

Fig.5 SEM images of Ge nanowires grown under different conditions: (a) Si (100), Au 0.1 nm, GeH_4/H_2 1 sccm, Ar 0.1 sccm; (b) Si (100), Au 1 nm, GeH_4/H_2 1 sccm, Ar 0.1 sccm; (c) Si (111), Au 0.1 nm, GeH_4/H_2 1 sccm, Ar 0.1 sccm; (d) Si (111), Au 1 nm, GeH_4/H_2 1 sccm, Ar 0.1 sccm

Ge nanowires on H-terminated Si (111) substrate can realize higher density Ge nanowires than that of other substrates[28]. The flow rates of precursor have no obvious influence on the undoped Ge nanowires' growth since the morphologies of Ge nanowires shown in Figs.2 (a, b), Fig.4 (d) and Fig.5 (d) with the same Si (111) substrate and Au catalyst (1 nm) are almost the same.

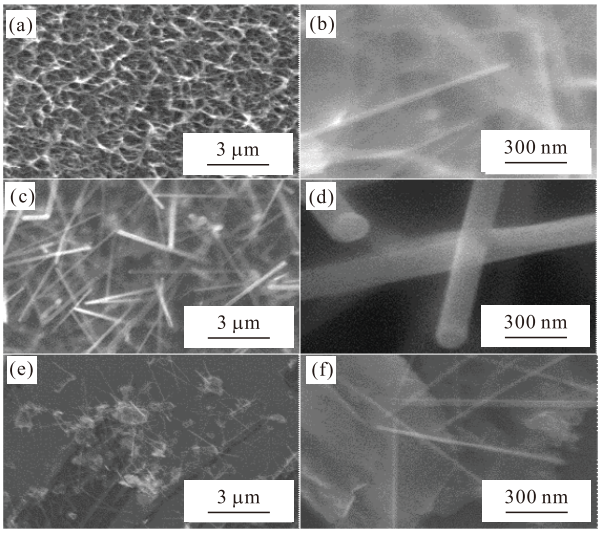

Fig.6 SEM images of Ge nanowires grown on Si (111) substrate under different conditions: (a) Au 1 nm; GeH_4/H_2 1 sccm; PH_3/Ar 1 sccm; (b) Magnified view of (a); (c) Au 5 nm; GeH_4/H_2 1 sccm; PH_3/Ar 1 sccm; (d) Magnified view of (c); (e) Au 10 nm; GeH_4/H_2 1 sccm; PH_3/Ar 1 sccm; (f) Magnified view of (e)

For the P-doped (n-type) Ge nanowires' growth, the PH_3/Ar co-flow was employed in the nanowires' growth process. From the growth results shown in Fig.6, the influence of the thickness of Au on P-doped Ge nanowires' growth is the same as the influence from the thickness of Au on undoped Ge nanowires's growth. The Si substrates with 1 nm thickness of Au catalyst (Figs.6 (a, b)) result in higher density, larger scale and more ordered Ge nanowires than that of 5 and 10 nm thickness of Au (Figs.6 (c, d) and Figs.6 (e, f)). Also, it is apparent that with the thickness of Au increasing, the length and diameter of the nanowires increase.

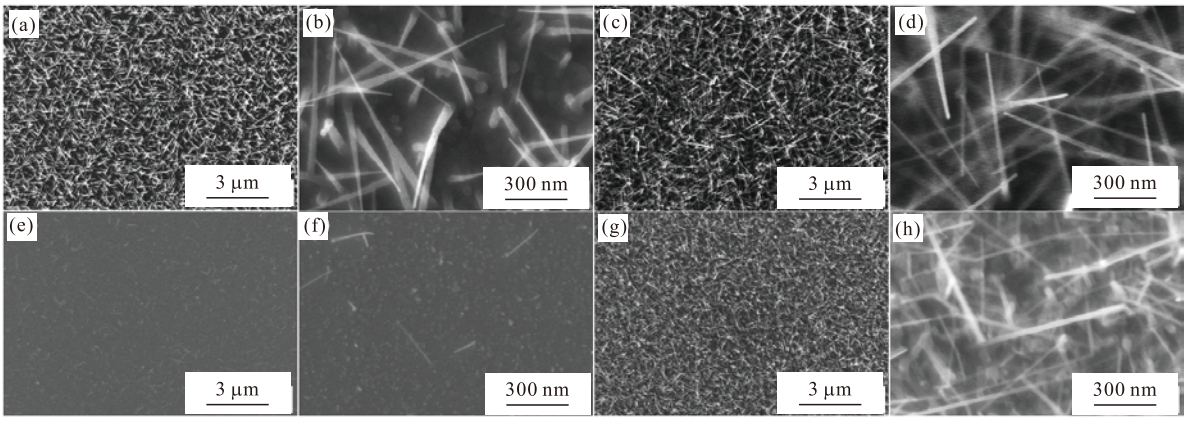

Fig.7 SEM images of Ge nanowires grown on Si (111) substrates with different thicknesses of Au and atmosphere: (a) Au 0.1 nm; GeH_4/H_2 1 sccm; PH_3/Ar 0.5 sccm; (b) Magnified view of (a); (c) Au 1 nm; GeH_4/H_2 1 sccm; PH_3/Ar 0.5 sccm; (d) Magnified view of (c); (e) Au 0.1 nm; GeH_4/H_2 1 sccm; PH_3/Ar 2 sccm; (f) Magnified view of (e); (g) Au 1 nm; GeH_4/H_2 1 sccm; PH_3/Ar 2 sccm; (h) Magnified view of (g)

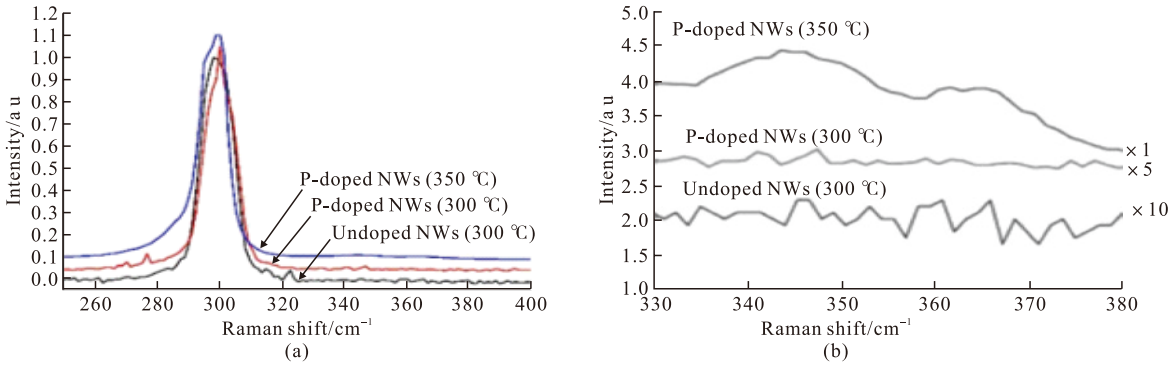

Fig.8 (a) Raman spectra of undoped and doped Ge nanowires (Fano broadening); (b) Raman spectra of undoped and doped Ge nanowires (P local vibrational peak)

From the growth results shown in Fig.7, it is obvious that 0.5 sccm (Figs.7 (a, b)) is a better flow rate of PH_3/Ar than 2 sccm (Figs.7 (e, f)), and 1 nm thickness of Au catalyst (Figs.7 (c, d)) is a better condition than 0.1 nm thickness of Au (Figs.7 (a, b)) to synthesize high density and large scale P-doped Ge nanowires. By comparing the morphologies observed in Fig.6 (a) and Fig.7 (a), it can be found that 0.5 sccm flow rate is more suitable than 1 sccm PH_3/Ar to realize P-doped Ge nanowires' growth. The flow rates of precursors have obvious influence on the doped Ge nanowires' growth since the morphologies of nanowires shown in Figs.7 (c, d) are more ordered, more uniform and denser than that of nanowires shown in Figs.6 (a, b) and Figs.7 (g, h) with the same Si (111) substrate and Au catalyst (1 nm).

Therefore, it can be concluded that 1 nm thickness of Au, 0.5 sccm flow rate of PH_3/Ar are the best condition for the growth of high density and large scale P-doped Ge nanowires among these different conditions.

3.2 Raman spectra of the Ge nanowires

From previous researches, we know that intra- and inter-valence-band transitions provide the continuum responsible for the Fano asymmetry of Ge due to the phonon confinement effect, also, the Raman spectra of Ge nanostructures show downshift and asymmetric broadening[35-37].

In this research, the Raman spectra of undoped and doped nanowires showed that P atoms were doped into the core of Ge nanowires and electrically activated in the Ge nanowires synthesized at 350 ℃. Two evidences can clearly demonstrate this conclusion. Firstly, the peak at about 300 cm^{-1} (the Ge-Ge vibrational peak) has been broadened due to the P-doping in nanowire (350 ℃, GeH_4:PH_3 = 10:2), which can be observed in Fig.8 (a). Secondly, for the P local vibrational peak at 342-345 cm^{-1}, the sample (350 ℃, GeH_4:PH_3 = 10:2) has this clear peak as shown in Fig.8(b).

For other samples synthesized at 300 ℃ with different thicknesses of Au and different flow rates of PH_3/Ar (0.5 and 1 sccm), Raman spectroscopy was also used to characterize their P-doping, however there is no clear P local vibrational peak existing, only clear Ge-Ge vibrational peak can be observed. So doping was not realized in these samples, and 300 ℃ is not a suitable temperature for growing P-doped Ge nanowires during VLS-LPCVD process.

4 Conclusions

The 1 nm thickness of Au is a better condition for the growth of high density Ge nanowires on Si (111) substrates with large scale than other thicknesses of 0.1, 5 and 10 nm. The Si substrates with other thicknesses of Au catalyst can obtain the growth of nanowires on limited scale. And 0.5 sccm is the best flow rate of PH_3/Ar to grow high density P-doped Ge nanowires on a large scale among the flow rates of 0.5, 1 and 2 sccm.

At 350 ℃ during the VLS-LPCVD process, P impurity can be doped into Ge nanowires, and these n-type Ge nanowires can be realized. From the results of Raman spectroscopic characterizations, it has been shown that P atoms were doped into the core of Ge nanowires.

Acknowledgements

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