國立交通大學

電子工程學系 電子工程所碩士班

碩士論文

以低溫微波活化鍺薄膜摻雜之研究

Study on Dopant Activation in Germanium Film by Low Temperature Microwave Annealing

研 究 生：莊尚勳

指導教授：羅正忠 博士

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摘　　要

本論文為利用微波退火製程來活化多晶鍺薄膜中的離子佈植摻雜物，可抑止摻雜物擴散。對於經過離子佈植後產生的非結晶區域，發現薄膜的電阻率高對於微波的吸收率較高。而薄膜的厚度也對於微波的吸收率有影響。我們嘗試以不同的微波強度及時間施加於鍺薄膜，藉由量測其片電阻值進而推算電阻率，觀察其活化的程度。我們發現縮短微波製程的時間可以控制雜質擴散，減少晶圓在高溫的環境。而在晶圓上下加入空白矽晶圓可以降低製程初始的升溫速率，此一作用同樣對於減少雜質擴散及保護晶圓表面有一定的幫助。利用微波退火的低溫活化特性，可防止鍺薄膜中離子佈子摻雜物發生擴散的現象及避免金屬污染，減少製程的步驟。在未來將可應用於元件微縮。

Study on Dopant Activation in Germanium Film by Low Temperature Microwave Annealing

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Abstract:

We study on the microwave annealing process to activate boron and phosphorus in single and poly germanium thin film. The amorphous thin film on the wafer was formed by ion implantation. The higher resistivity of the thin film has the better fraction of microwave absorption. And the film thickness also influences the microwave absorption. The wafers go through different microwave power and process time to observe dopant diffusion. We found to decrease the process time, shorten the wafer stay in the near-maximum temperature of the process, can reduce the dopant diffusion. And the addition of filler wafers above and below the wafer can also suppress the dopant diffusion and prevent the damage on the wafer surface.

誌　謝

經過兩年的時間，很快地要從碩士班畢業了。首先，我要感謝我的指導老師羅正忠教授，帶領我進入了半導體的領域，在知識和做人處世上帶給我很多學習的榜樣。我要感謝國家奈米實驗室的研究員李耀仁博士，給予我研究上相當大的支持與幫助。同時感謝所有在 NDL 曾經幫助過我的工程師及代工小姐。

感謝威良學長帶我找到了碩士班的方向，給予我許多寶貴的建議。感謝智仁學長、翊裳學長幫助我解答了許多實驗上以及生活上的問題。和文新和芳毓同學一起度過在實驗室的日子，是我在這兩年中非常寶貴的回憶。也感謝侑倫學長、博安學長、富國、政昌、國欽、忠慶、建平、舒淳、翼麟和其儒學弟，跟我一起做實驗、量測和討論，一起度過了充實的碩二生活。

最後，我要感謝我的父母莊厚礎先生及嵇美雲女士一路上支持我，讓我可以順利地完成我的學業。

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Chapter 3 Dopant Activation in Germanium by Microwave Annealing

Chapter 1
Introduction

1-1 General Background

Ge has been considered to be a potential material to replace silicon because of its higher mobility for electron and hole. But, the thermal instability of Ge and GeO_x during the process limits the fabrication temperature of Ge-based devices. Recently, high K material and metal gate have become a solution for continuous scaling down. The deposition of high K material can be achieved by atomic layer deposition (ALD) at very low temperature (170~340 ℃). This makes germanium be reconsidered the possibility of replacing Si channel [1-2]. However, the dopant activation is another issue of the fabrication of the Ge-based devices [3-5]. For nanoscale devices, the shallow junction is required to prevent the short channel effect. However, dopant diffusion in Ge film is observed in RTP (rapid thermal processing) method. According to A. Satta *et. al.* [6], the rapid thermal annealing, even at 500℃ for 1 minute, leads to severe implanted dopant diffusion for high dose P in Germanium. However, the lower annealing temperature, the lower activation percentage caused the higher contact resistance and reduced drain current.

1-2 Motivation – Why do we need low temperature dopant activation for germanium-based devices?

Low temperature dopant activation method has been developed by J. H. Park. They have successfully utilized the metal-induced crystallization (MIC) process to activate dopants in amorphous germanium at about 360℃ [7]. However, for n-type doped wafer, only cobalt could get a good result in low resistivity. Other metal impurities work as acceptorlike traps which capture the electron result in increasing resistivity. And low diffusivity in n-type doped germanium is the disadvantage of cobalt MIC. According to Alford *et al.* [8], microwave annealing has been presented as a possible alternative to other rapid thermal process methods in silicon processing. It shows microwave could repair the damage caused by high-dose boron implanted. And the dopant is activated by microwave initiation of solid phase epitaxial regrowth (SPEG) at 500℃.

In this work, we utilized the microwave annealing to activate the dopant in Ge at low temperature. We discuss single and poly crystalline Ge which is the main substrate for both bulk Ge integrated circuits and thin film transistors. In addition, the major effects of microwave process are power, frequency, and load in the chamber, and minors are the temperature. We demonstrate the low-temperature microwave annealing by controlling the power magnitude and loads inside the chamber with using sheet resistance measurement to make a comparison to the conventional RTP method. We found that the microwave could make additional dopant activated at low temperature. The process temperature was controlled under 360℃. By TEM and SIMS analysis, radiation damage caused during implantation process could be repaired by microwave annealing and the severe dopant diffusion could be suppressed due to the low temperature process.

1-3 Organization of the Thesis

In Chapter 2, we first introduced the mechanism of microwave annealing. The theory of microwave annealing has already been studied. The thin film thickness and the conductivity have relation with the microwave absorption. The dopant activation is caused by solid phase epitaxial regrowth. Second, we introduced the microwave system. Several parts containing in the system was showed what its purpose. The filler wafer is for eliminating the plasma.

In Chapter 3, we prepared two kinds of sample for testing the microwave annealing. Poly germanium is deposited by low pressure chemical vapor deposition (LPCVD) on the thermal oxide. It is successfully activated by microwave annealing. Single germanium is deposited by ultra high vacuum chemical vapor deposition (UHVCVD) on the bare silicon wafer. We tried to find how the power and process time influence the dopant activation and diffusion in germanium. And we add the filler wafer for increasing the uniformity of sheet resistance and suppressing the dopant diffusion.

Finally, Chapter 4 gave the conclusion and suggestions of this thesis for the future work.

Chapter 2
The Microwave Annealing Process

2-1 Introduction

According to references [9-10], microwave annealing has already been discussed. It has been utilized on ion-implanted silicon. In this chapter, we introduce the mechanism of microwave annealing and the microwave system.

2-2 Mechanism of Microwave Annealing

Microwave has been reported as a potential method to represent those conventional high temperature methods, such as rapid thermal annealing and flash lamp annealing. According to H. Bosman *et al.* [9], it has been proved that the wafer could absorb the microwave energy and be heated by the skin effect. The skin depth could be calculated from:

$$\delta = \sqrt{\frac{2}{\mu\sigma\omega}} \qquad (1)$$

where δ is the skin depth which is normalized to the free space wavelength $\lambda=2\pi c/\omega$, which c being speed of light in vacuum, ω is the microwave frequency, μ is the permeability of silicon, and σ is the specific electrical conductivity of the material. In (2), the wafer thickness is normalized to the scale length.

$$S = \frac{2}{\sigma Z_0} \qquad (2)$$

where $Z_0=(\mu_0/\varepsilon_0)^{1/2}$ is the free space impedance. We tried to calculate the real values about the microwave power absorption approximately. In (1), the microwave frequency we used is 5.8 GHz and the conductivity of silicon single crystal wafer is about 50 Ω-cm. The relative permeability of silicon is approximate to 1. We can extracted the skin depth of silicon is 4.67μ m. The thickness of the silicon wafer is 150 mm. we can simply calculate the fraction of incident power absorption (A):

$$A = \frac{2\xi}{(\xi + 1)^2} \qquad (3)$$

$$\xi = \frac{L}{S} \qquad (4)$$

We obtained the fraction of incident power absorption of the silicon wafer is 0.033.

According to Zohm *et al.* [10], by discussing the value of σ, we could found the low conductivity thin film has a better absorption of microwave. After ion implantation, the thin film which becomes amorphous with a lot of defects is good for microwave annealing.

Alford *et al.* [8]has already discussed the repairmen of radiation damage by ion implant and activate dopants in silicon by microwave initiation of solid phase epitaxial regrowth (SPEG). So as the same reason, we are trying to use microwave annealing on germanium thin film.

2-3 Introduction of Microwave System

Fig. 2-1 shows the microwave system. The microwave system mainly contains:

(1) Microwave power supply: Supplies high voltage power for the microwave source. Supply includes internal alarms to prevent damage the microwave source, i.e. over volt alarm or over temperature alarm.

(2) Microwave source: Generates the microwave energy required for processing. Internal interlocks prevent overheating, i.e. water flow switch or over temperature sensor.

(3) Isolator: Eliminates excessive microwave energy from the process chamber to prevent damage to the microwave source. Measures all the excess microwave energy and reports to the PLC.

(4) Coupler: A port to measure forward microwave energy going into the process chamber. The measured result is reported to the PLC.

(5) Waveguide: Delivers generated microwave energy into the process

chamber.

(6) Process Chamber: as shown in Fig. 2-2, an octagonal prism shape vessel designed to isolate wafers from the atmosphere while gases and microwave as specified by the recipe are applied to the wafers. The chamber's geometry promoted a uniform microwave energy field.

Fig. 2-3 shows the photo of the loading stage. When the process starts, the loading stage under the process chamber goes up until the chamber sealed. And then, the stage rotates slowly for increase the uniformity of the microwave absorption. After 10 minutes N_2 gas prepurge, the microwave power supplies turns on. As shown in Fig. 2-4, the quartz suspectors above and below the wafer can prevent particles from the environment during process. Because quartz doesn't absorb the microwave energy, they also can decrease the maximum temperature by absorbing heat from wafer. When high microwave power supplies, the tool is capable of generating plasma. Plasma will minimize any unique MW benefit and potential might damage tool. The addition of filler wafers (bare silicon) above and below the process wafer can prevent plasma generation. And we also found some other benefits of filler wafers in Chapter 3.

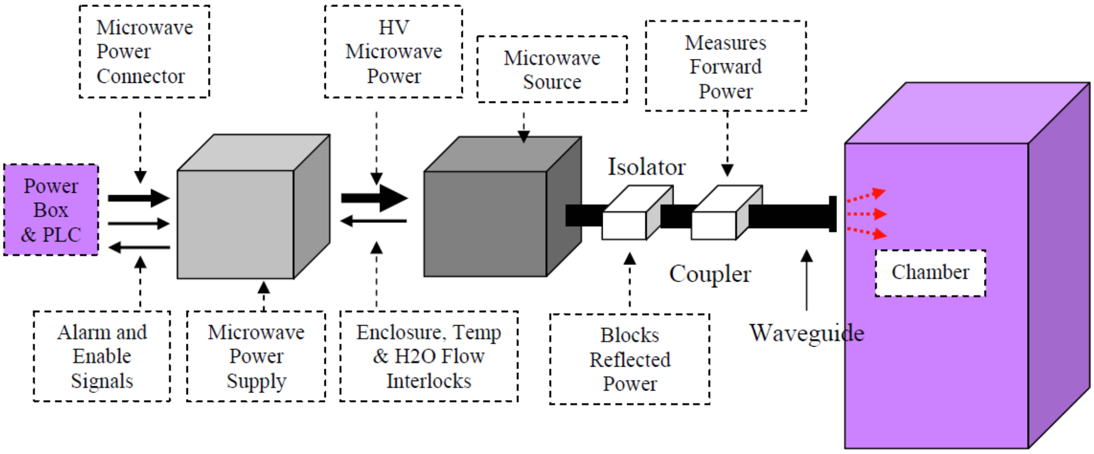


Figure 2-1　Diagram of the microwave system.

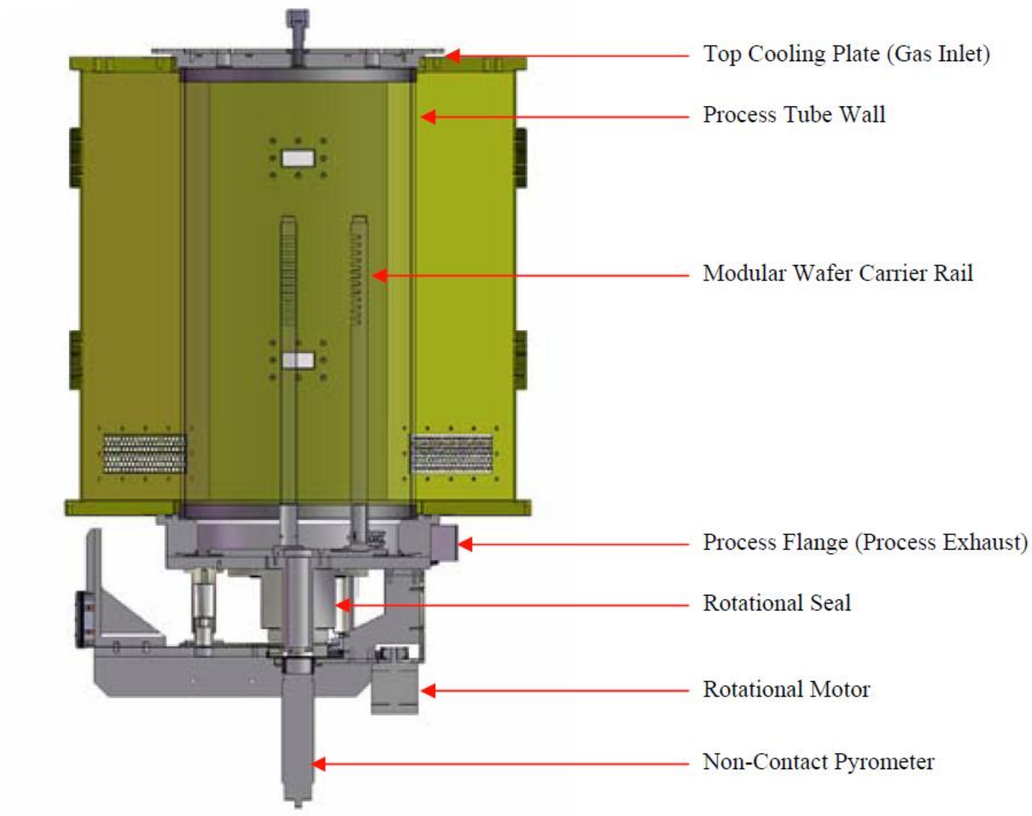


Figure 2-2　Diagram of the process chamber.

Figure 2-3　Photo of the loading stage.

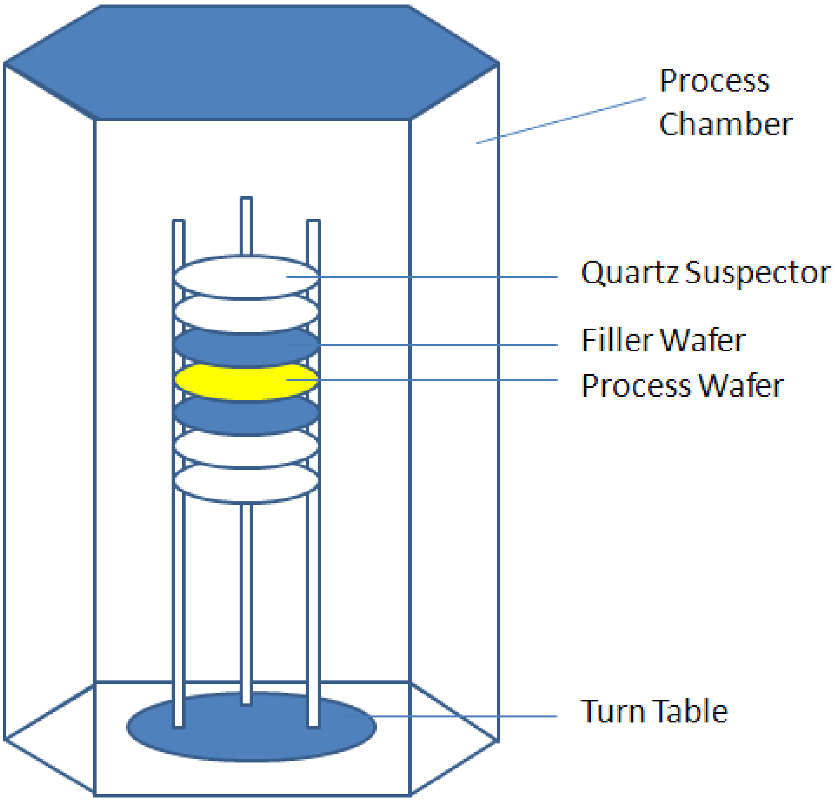


Figure 2-4 Process setting of microwave annealing.

Chapter 3

Dopant Activation in Germanium by Microwave Annealing

3-1 Introduction

In this chapter, we will demonstrate how the microwave annealing process influence the ion implanted germanium with several different conditions, such as the magnitude of microwave power, the amount of the filler wafers and the process time, etc. We found that optimization of the treatment has a tendency of higher power for sufficient dopant activation level and shorter process time for preventing severe dopant diffusion. And the addition of filler wafers above and below the process wafer could improve the uniformity and suppress the dopant diffusion.

3-2 Experimental Procedure

Several Ge films were prepared for dopant activation. A 200 nm Ge epitaxily layers were deposited on a 6-in p-type Si (100) substrate with resistivity 15–25 Ω-cm by UHVCVD. In addition, a 50nm poly-Ge film was deposited by LPCVD on a 500nm thick thermally grown silicon dioxide (SiO_2) film on Si substrate. All samples were implanted at room temperature (RT) with BF_2 (25keV, 1×10^{15} cm^{-2}) and P^{31} (25keV, 1×10^{15} cm^{-2}), respectively. And the wafers which were deposited AlSiCu and tungsten respectively on thermal oxide were also prepared for microwave testing.

The splits by microwave anneal experienced at 5.8 GHz with a power of about 600W~1000W in the chamber from 100 seconds to 600 seconds. Before microwave anneal, a 10 minutes N_2 purge was performed before the microwave was started and the N_2 flow was maintained until the process completed. The process time was measured when the microwave power turned on until the power turned off without regardless of temperature, which is different from that of RTA. In addition, there are three different load configurations for the comparisons of different loading designs, respectively. Configuration (A) is the standard setup without loading wafers for the dopant activation processes, as shown in Fig. 3-1. Then, another adds additional two quartzes and two filler wafers in configuration (B) with fixed space between process and filler wafers, as shown in Fig. 3-2. The other adds two more filler wafers in configuration (C), as shown in Fig. 3-3. The profiles of temperature versus time by microwave were shown in Fig. 3-4, which was detected from the bottom loading wafer in the chamber. From Fig. 3-4, as one added the filler wafers in the microwave chamber, the maximum temperature and temperature increasing slope would be decreased because the filler wafers would share the microwave power in the chamber.

The SIMS, sheet resistance (Rs), the X-ray diffraction (XRD), and cross-sectional transmission electron microscope (TEM) image were measured. Meanwhile, the split, without microwave treatment, activated by rapid thermal anneal (RTA) in a Heat-pulse AG601i RTA system at 360℃, and 550℃.

3-3 Results and Discussions

As Fig. 3-4 shows, we successfully control the total process temperature about 360℃. The temperature goes up very fast at the beginning of the process and saturated at about 360 ℃ for the process time - 10 minutes. This could be used on forming the shallow junction in source/drain engineering.

For poly germanium samples, the sheet resistance measured by 4-point probe, as shown in Fig. 3-5. The result of both microwave annealed and rapid thermal processes are listed. The condition of microwave annealing is 100% power with two quartzes above and below the wafer (Fig 3-1) for 10 minutes. We found it is difficult for phosphorus-implanted sample annealed at low temperature by RTP in poly germanium. However, by microwave annealing, the result of sheet resistance is close to the one by rapid thermal annealing at 450℃. It could tell microwave annealing has the ability of dopant activation in germanium thin film at low temperature. And the wafers deposited on AlSiCu also went through the microwave treatment. And we found that the resistivity remained the same value before. Because the microwave is uniform in process chamber, it did not cause a voltage drop on the metal surface. AlSiCu won't be damaged through microwave annealing.

Next, we discussed the behavior of dopant with different conditions of microwave annealing. In Table 3-1, The sheet resistance extracted from (a) which is 100% power two quartzes below and above the wafer for ten minutes is 167.4Ω /□. As shown in Fig. 3-6, about 20% of the implanted P diffused into the germanium thin film. And we tried to add the filler wafers to suppress the dopant diffusion.

In Table 3-1, we compare the sheet resistances of (b), (c) and (e). The sheet resistance by rapid thermal annealing at 450℃ and 550℃ for one minutes are 235 Ω/□ and 120 Ω/□, respectively. We found that (c) has the lowest value and even lower than RTA 450℃ for one minute. (b) seems has not enough microwave power to activate sufficient dopants. So the higher power is needed. And we tried to adding two more filler wafer as (e) shows. We found that the sheet resistance is larger than 100% power with two quartzes. But the process temperature is as high as 100% power. We thought the decay of microwave through two filler wafers both two sides is more severe than (c). The microwave energy reaching to the process wafer is not enough.

As shown in Fig. 3-7, the diffusion of P of the sample with one cycle of ten minutes is more severe than (d) which annealed for two cycles, each cycle is five minutes. It is the reason why (c) has a lower Rs value. Therefore, we considered the longer time the wafer stay in saturated temperature is the main reason that causes the dopant diffuse. So the next condition, we reduced the process time to suppress the diffusion effect. In 100-second process time, the maximum temperature is lower than ten-minute process. Therefore, we used higher power to activate dopants and keep temperature at about 380℃. We set two powers (200%) fully turned on for 100 seconds. As shown Table 3-1, (g) has a better value than (d) and a better dopant profile. And the peak of the P concentration in sample (g) is as high as Control sample. Only 10.7% of dopants diffused into bulk in sample (g). We could found the filler wafers could truly suppress the dopant diffusion by comparing the dopant profile of (f) and (g). According to the temperature profile of these with and without filler wafers, we could found two differences. One is the reduction of maximum temperature about 10 to 20℃. The other is the ramp rate at the beginning of the process. The filler wafers could get a more smooth temperature profile. According to the experiments above, we found three possible mechanisms which lead to dopant diffusion: (1) the power magnitude (2) the time that the wafer stay at the temperature nearly saturated (3) the ramp rate at the beginning of the process.

In Fig. 3-8(a), the 60 nm amorphous region was created by ion implantation. After microwave process, SPEG makes dopants get into the lattice site and repair the radiation damage by the implant. After RTA at 550℃ for 60 seconds, the surface became very rough because of germanium out-diffusion. The diameter of the measured hole is about 146 nm and the depth is about 30 nm, as shown in Fig. 3-8(b). It will cause a critical issue of devices fabrication. The microwave process has the ability to repair the radiation damage by ion implantation in germanium. It is only about 2-3 nm amorphous region on the surface of germanium with 200% power 100 seconds, as shown in Fig. 3-8(c). Without suspectors, we found pin holes on the surface, as shown in Fig. 3-8(d). We suggested it caused by the high ramp rate of the beginning of process. The longer process time has the worse surface roughness, as shown in Fig. 3-8(e) and (f). And Fig 3-8(f) has bigger holes than Fig 3-8(e) because of the longer time the wafer stayed in saturated temperature.

The substrate loss is one of the issue of the Ge-base CMOS fabrication. The surface roughness was measured by AFM. In Fig. 9-1(a), we considered that these holes which are narrow and deep on the control sample surface are caused by the formation of dislocation as the germanium deposited by UHVCVD. In Fig. 3-9(b), after RTA at 600℃ for 30 seconds, the surface by RTA process became rougher as increasing the process time. However, in Fig. 3-9(c), after 200% 100sec microwave process, the surface of these samples still remained as good as the control sample because of the low temperature microwave process.

3-4 Summary

This work has demonstrated that poly and single germanium thin films can be activated by microwave annealing. By TEM and SIMS analysis, radiation damage caused during implantation process could be repaired by microwave annealing and the severe dopant diffusion could be suppressed due to the lower temperature process as compared with conventional RTA process.

In addition, shorten process time and suspectors are needed for suppressing dopant diffusion. Higher power should be used in short process time for sufficient activation level. And the microwave annealing is compatible with metallic process.

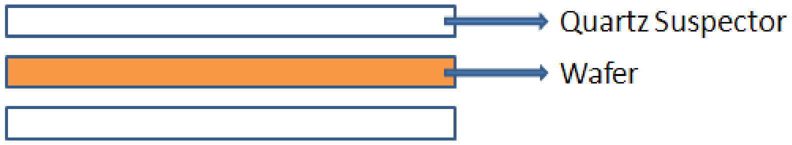


Figure 3-1　Process setting of Configuration (A).

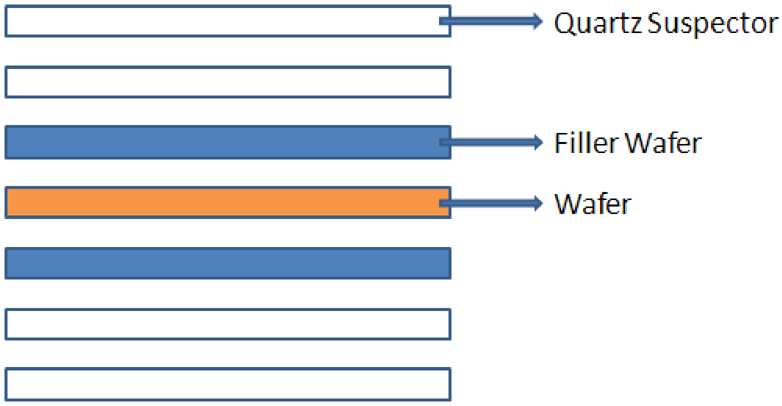


Figure 3-2　Process setting of Configuration (B).

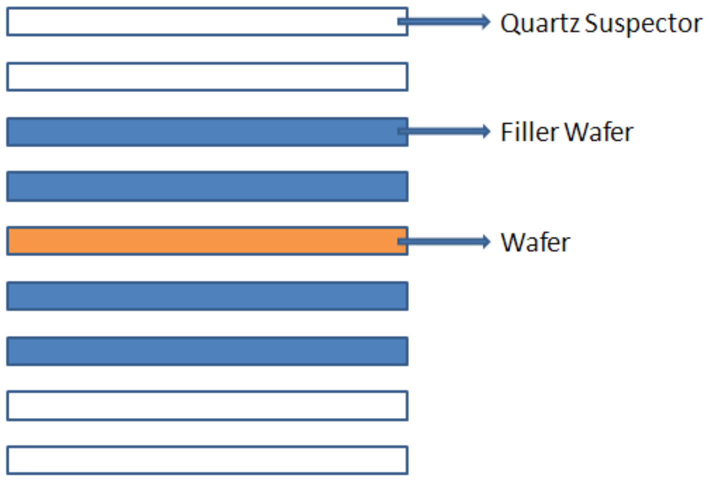


Figure 3-3 Process setting of Configuration (C).

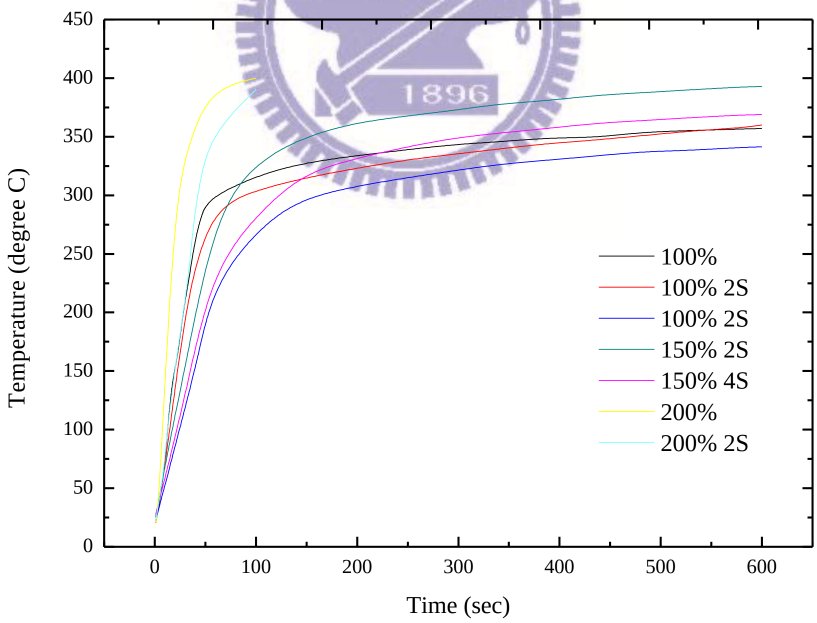
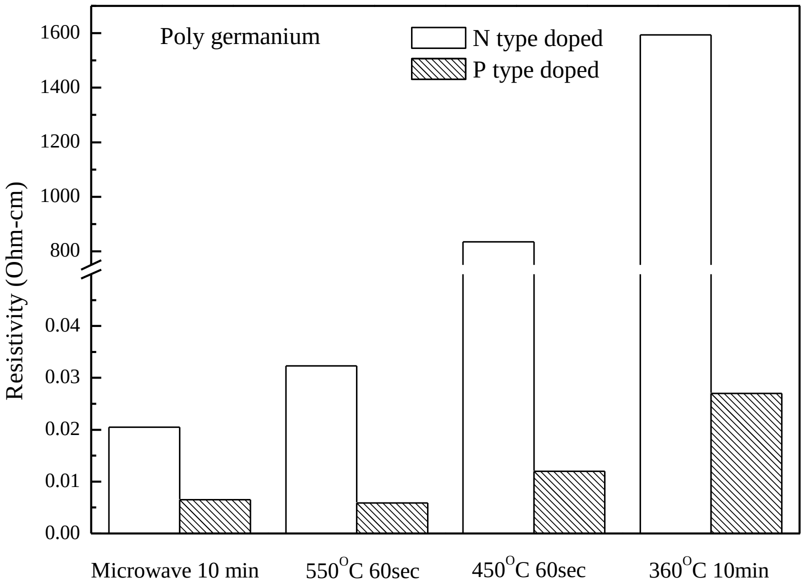
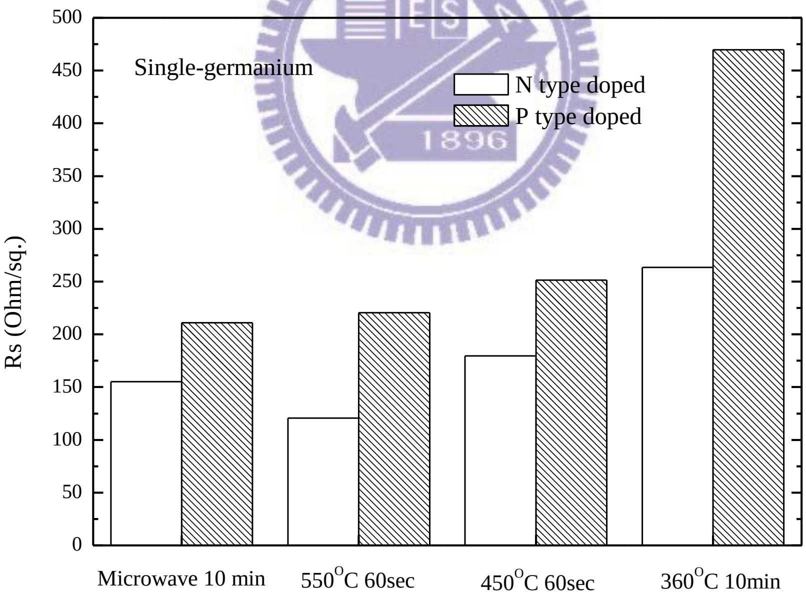


Figure 3-4 Temperature versus process time during microwave annealing in different setting conditions, Q is presented quartz suspector and F is presented filler wafer.



(a)



(b)

Figure 3-5 (a) The resistivity of poly germanium and (b) the sheet resistance of single germanium by microwave annealing and RTA in different process temperature.

	a	b	c	d	e	f	g
Power	100%	100%	150%	150%	150%	200%	200%
Suspector	2	4	4	4	4	4	4
Filler wafer	0	2	2	2	4	0	2
Process time (sec)	600	600	600	300 (two cycles)	600	100	100
Rs (Ω/□)	167.4	316.3	162.3	215.4	276.3	133.2	200.1
Tmax (℃)	360	341.3	392.8	373	368.9	400	390

Table 3-1 Conditions of microwave annealing examined for investigating the dopant diffusion mechanism.

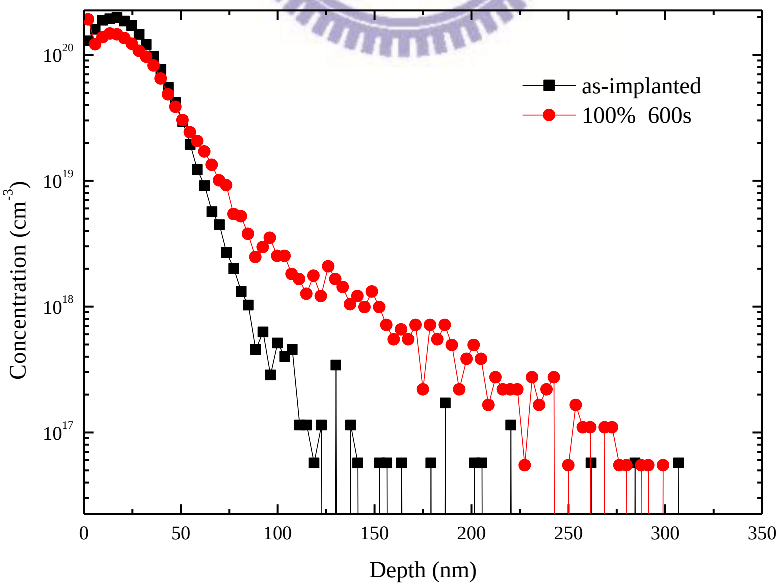


Figure 3-6 Diffusion profiles of implanted P before and after 100%/600s microwave annealing.

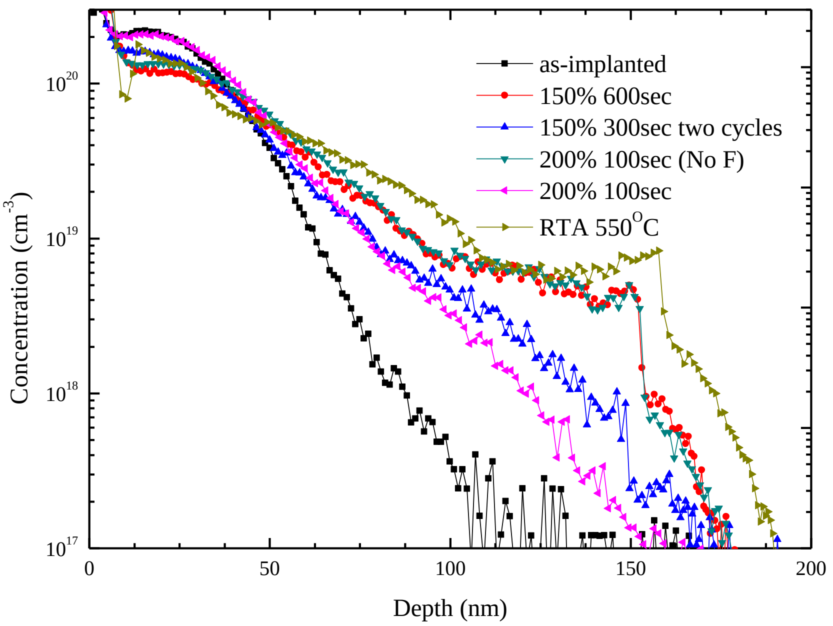
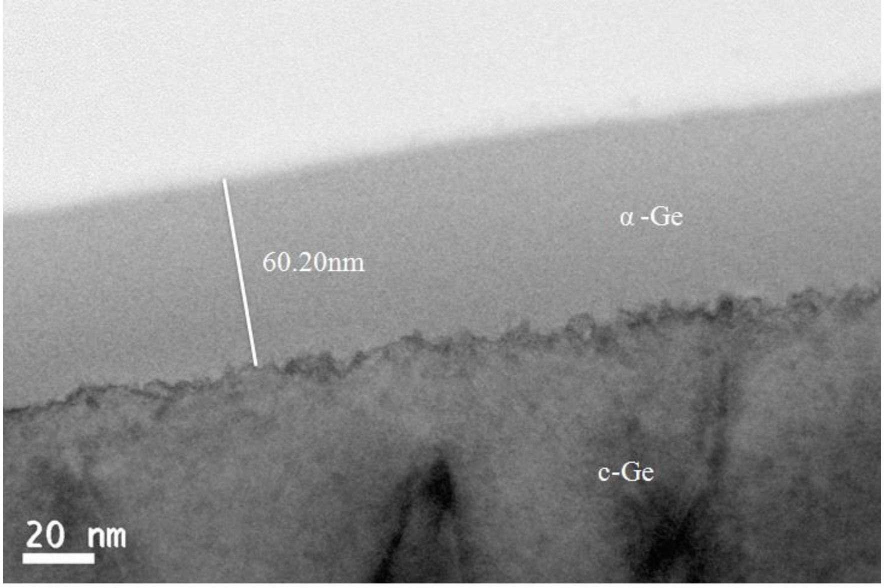
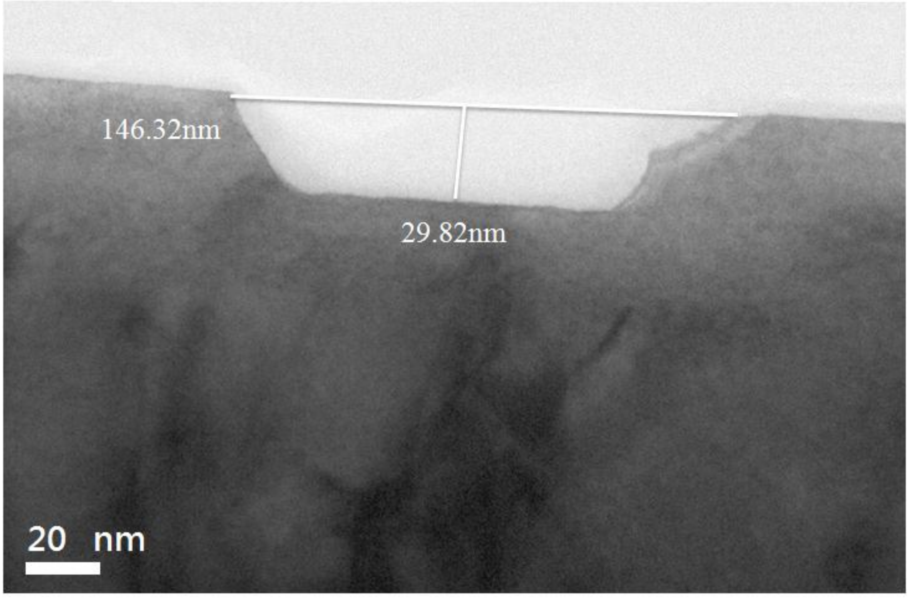


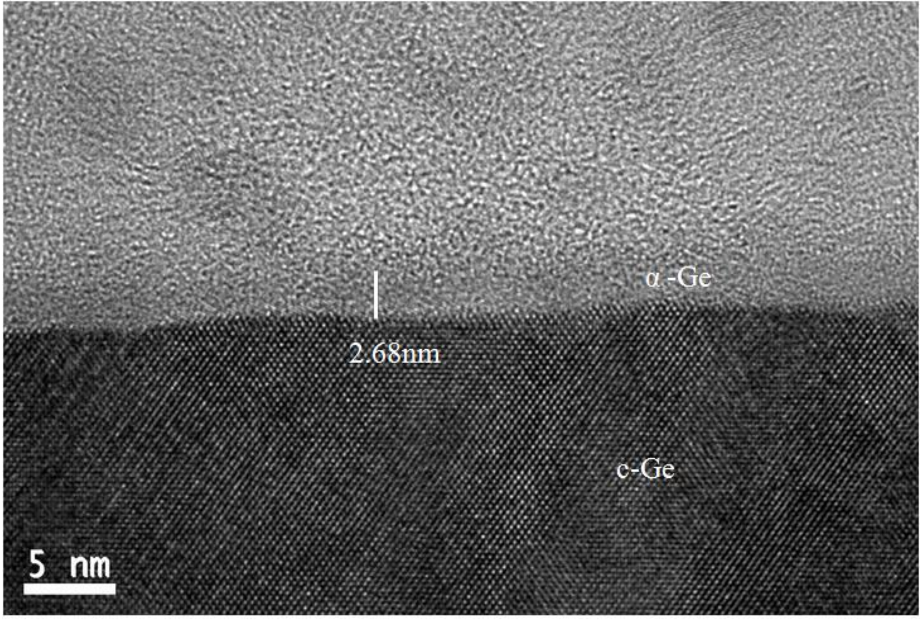
Figure 3-7 (a) Diffusion profiles of implanted P before and after microwave annealing in different conditions and 550℃/60s RTA.



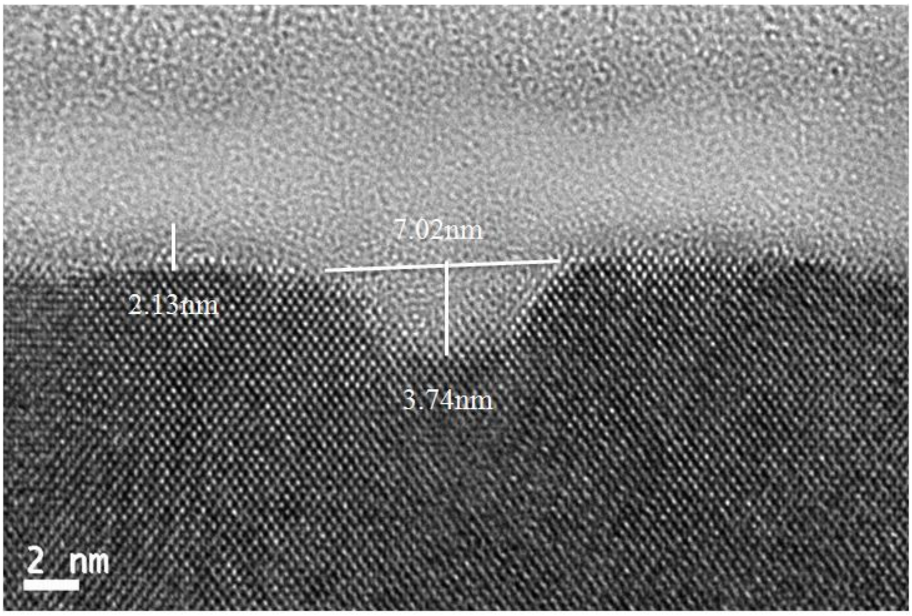
(a)



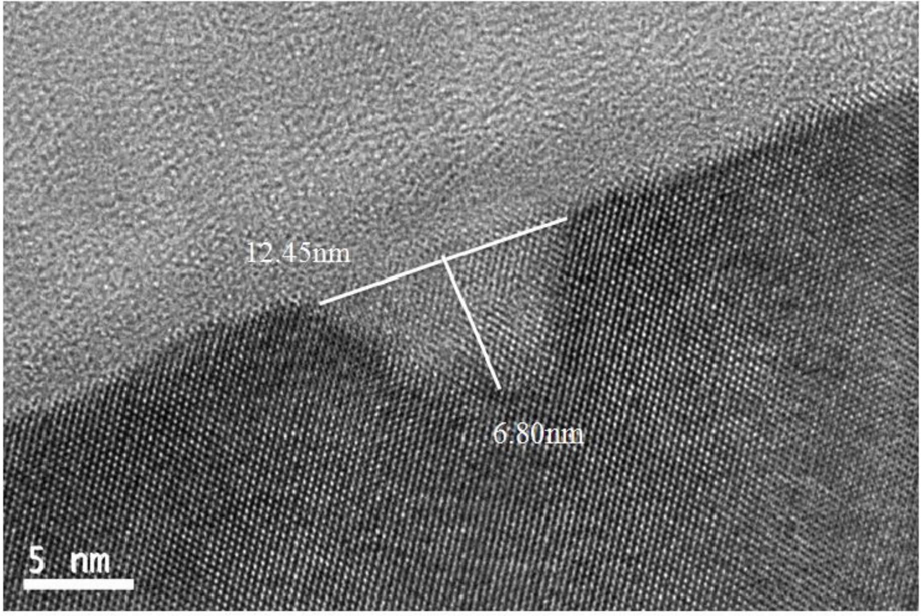
(b)



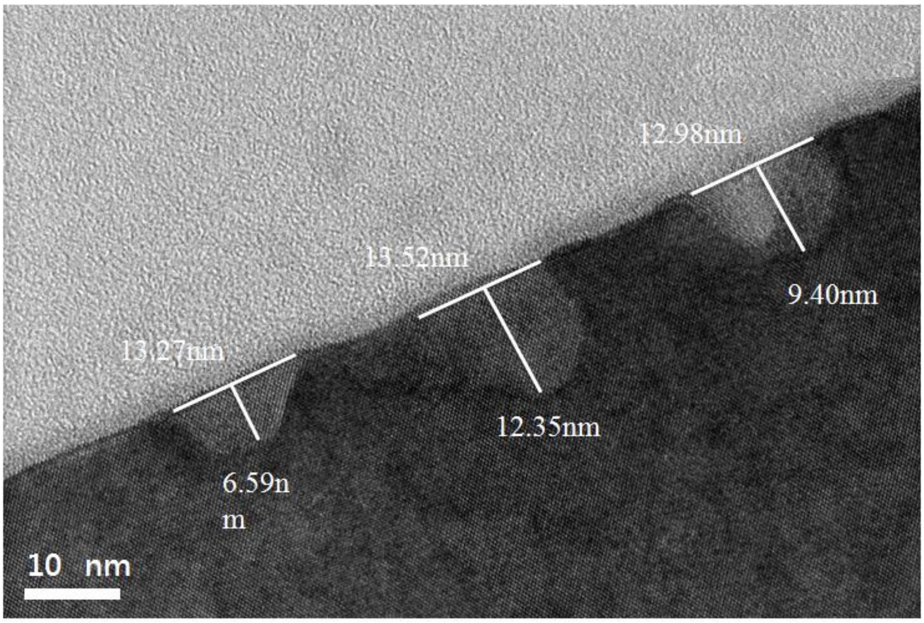
(c)



(d)

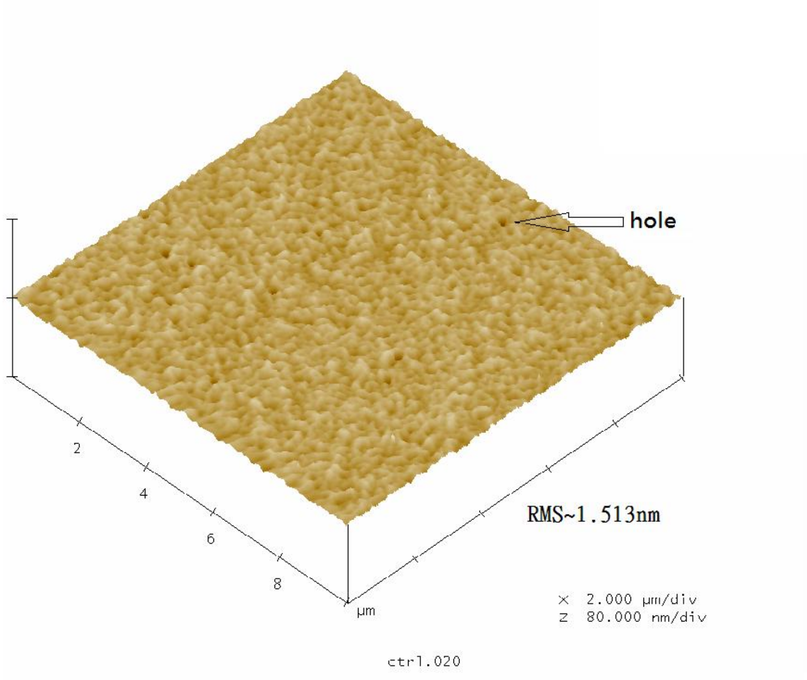


(e)

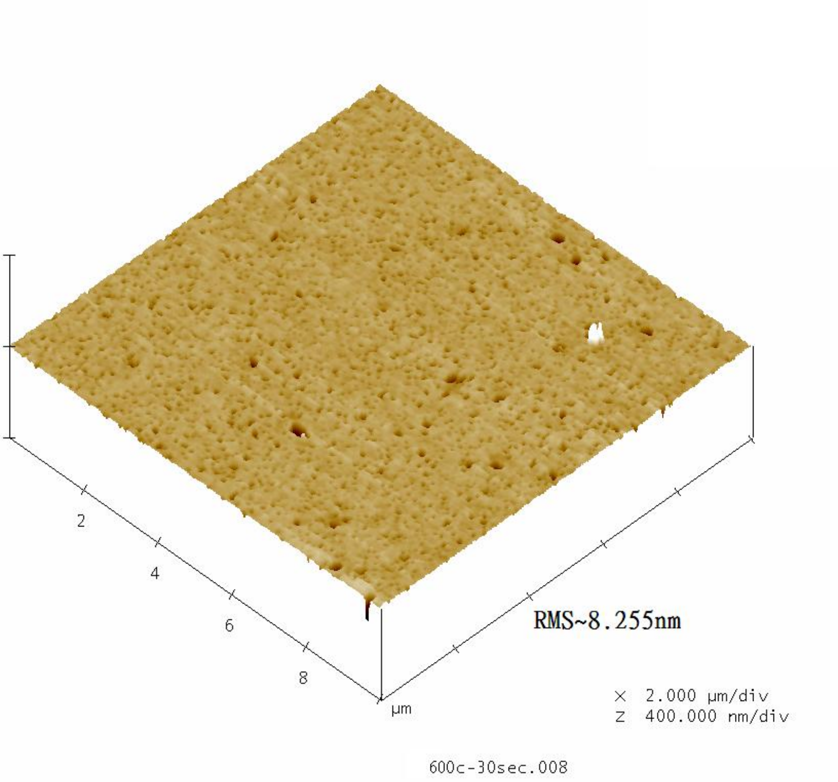


(f)

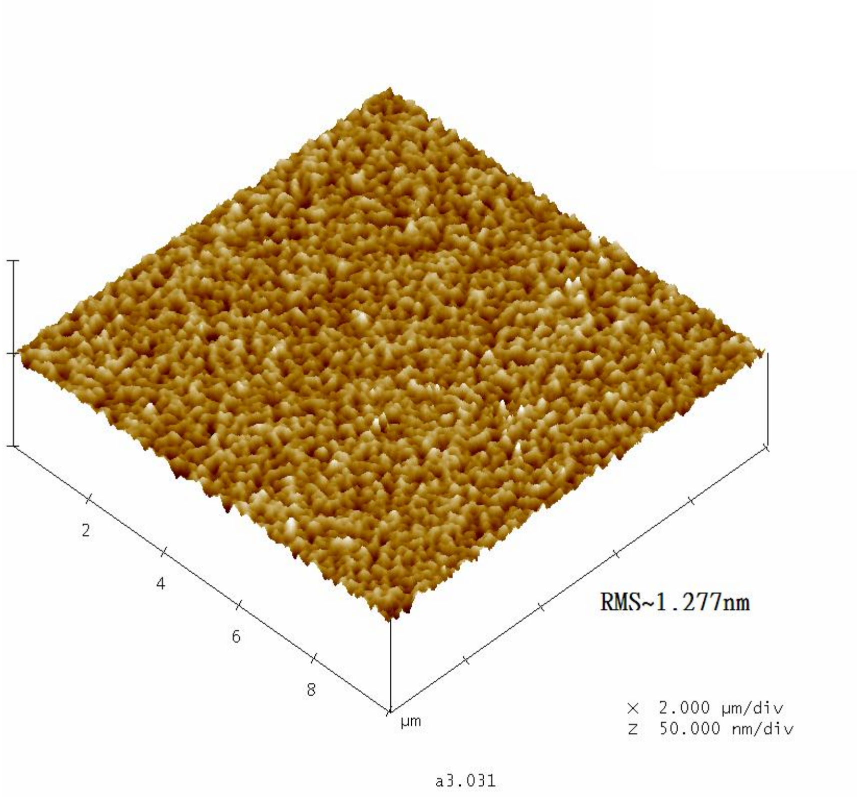
FIG. 3-8 TEM of (a) as-implanted, (b) 550℃/60sec RTA, (c) 200%/100sec with filler wafers, (d) 200%/100sec without filler wafers, (e) 150%/300sec two cycles and (f) 150%/600sec.


hole
2
4
6
8
µm
RMS~1.513nm
× 2.000 µm/div
Z 80.000 nm/div
ctrl.020

(a)


2
4
6
8
µm
RMS~8.255nm
× 2.000 µm/div
Z 400.000 nm/div
600c-30sec.008

(b)



(c)

Fig 3-9 AFM of (a) control sample, (b) 600℃/30sec RTA and (c) 200% 100sec microwave annealing.

Chapter 4
Conclusions and Future Work

4-1 Conclusions

In this thesis, we have introduced the mechanism of microwave annealing and the microwave system. The microwave annealing can successfully used on poly and single germanium thin film. We suggested three possible mechanisms three possible mechanisms which lead to dopant diffusion: (1) the power magnitude (2) the time that the wafer stay at the temperature nearly saturated (3) the ramp rate at the beginning of the process. And we found shorten process time and adding the filler wafers could make less damage on the germanium surface.

4-2 Future work

By investigating the microwave annealing of germanium, we can use it on device fabrication. For germanium substrates, the performance of conventional germanium CMOS devices may be improved by microwave annealing because of the higher dopant activation level.

For thin film transistor fabrication, the poly germanium could be used as active layer. By microwave annealing, the thin film will be less damage at low temperature.

Now, it becomes more popular of 3D-IC integration. A critical aspect currently plaguing the integration of the second (or higher) level devices is the lack of dopant activation techniques at sub-400℃. Microwave annealing has the potential to be a candidate of the annealing methods for 3D-IC fabrication.

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碩士論文題目：

以低溫微波活化鍺薄膜摻雜之研究

Study on Dopant Activation in Germanium Film by Low Temperature Microwave Annealing