Determination of Interstitial Oxygen Concentration in Heavily Doped Silicon by Combination of Neutron Irradiation and FTIR

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Abstract We used neutron irradiation and FTIR method to determine the oxygen concentration of heavily doped silicon. The relationship between irradiation dose and resistivity was established and the effect of A-center on the determination of oxygen concentration was analyzed. The neutron irradiation combined with FTIR provides a handy and practical method for the determination of oxygen concentration in heavily doped silicon.

PACC: 3320E, 6180H

1 Introduction

Fourier Transform Infrared Spectroscopy (FTIR) is a routine method to determine interstitial oxygen concentration $[O_i]$ in silicon. In heavily doped substrates, particularly with the dopant concentration $> 5 * 10^{18}\,cm^{-3}$, the quantitative measurement of $[O_i]$ by this method is impossible due to strong absorption of free carriers[1]. Recently, Tsuya et al.[2] reported the infrared measurement of $[O_i]$ in heavily-doped silicon after electron irradiation. Upon electron irradiation, the free carriers of high concentration are trapped by irradiation induced complex defects and resistivity of silicon crystal increased so that FTIR measurement of $[O_i]$ in heavily doped silicon becomes possible. Besides electron irradiation, other irradiations such as gamma rays or neutrons can also be utilized. However, owing to the limitation of beam fluence of the electron accelerator, the accumulation of electron irradiation is so long that electron irradiation is hampered in practical use. Considering the Neutron Transmutation Doping (NTD) technique has been widely used in semiconductor material technology, it may be anticipated that the combination of neutron irradiation and infrared optical measurement would provide a handy and practical method for determination of $[O_i]$ in heavily doped silicon. In this paper, we shall describe the infrared optical measurement of $[O_i]$ in heavily doped silicon wafers which have been changed to high resistivity ones by the neutron irradiation of high energy and

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fluence.

2 Experiment

The samples used in this experiment were heavily and lightly boron doped P-type silicon wafers with typical resistivity of 0.015 and 10Ωcm, respectively, cut from ⟨100⟩CZ crystal of 76mm in diameter. According to its fluence of neutron irradiation, the classification of the samples and the experimental condition of neutron irradiation are listed in table 1.

Table 1 Experimental conditions of neutron irradiation

class of irradiation	doping level of samples	neutron fluence(n/cm^2)	sample temp. (℃)
LLI	P+,P	7×10^{16}	40
MLI	P+	6.4×10^{17}	40
HLI	P+	3.3×10^{18}	40

LLI, MLI, HLI: low, medium, high level irradiation, respectively

The fast neutron irradiations with $E>2$MeV were carried out at swimming pool light water research reactor. Absorption spectra were measured at room temperature by a Nicolet 170X FTIR spectrometer with a resolution of $1cm^{-1}$. A calibration factor of $3.14\times10^{17}cm^{-2}$(GB1557-89 Standard of China) was used and multiple reflections were taken into account.

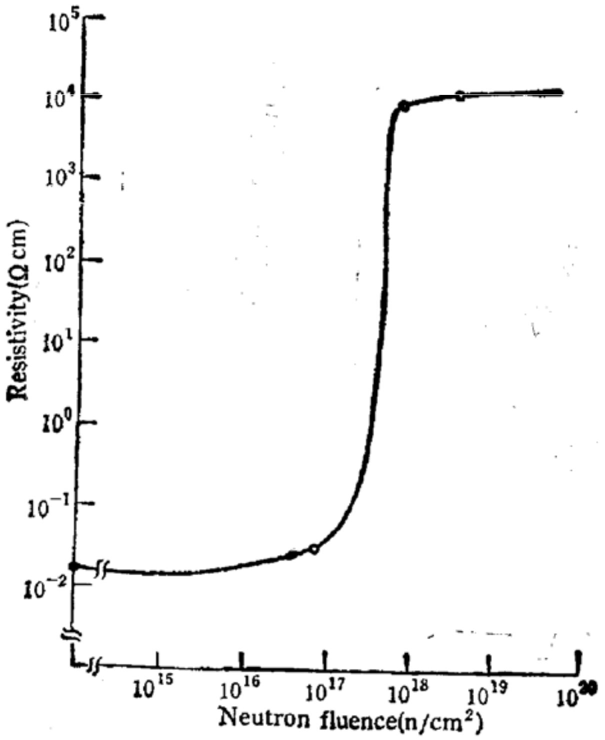


Fig. 1 Resistivity variation of heavily doped silicon after irradiation with various fast neutron fluences

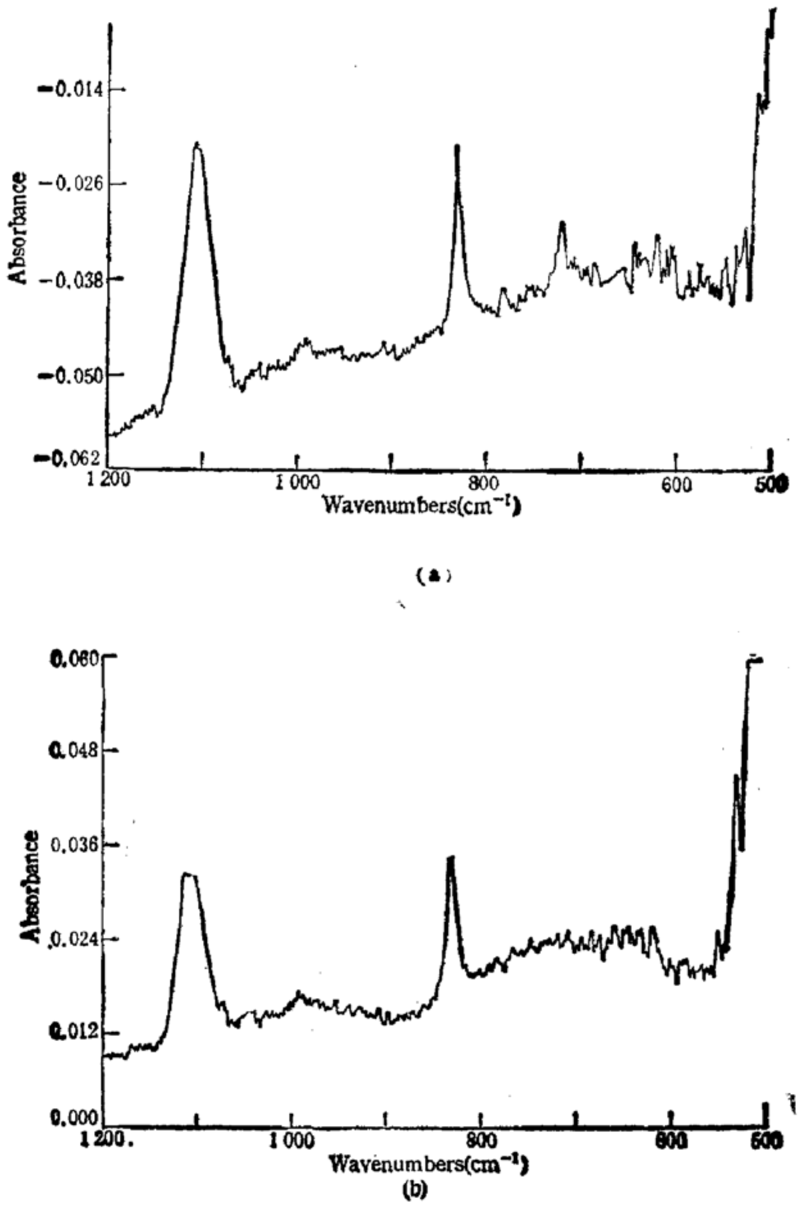
3 Results and Discussion

3.1 Variation of resistivity after irradiation

Before and after irradiation, Hall coefficient and conductivity of sample were measured using Van der pauaw technique. As shown in Fig.1, when the neutron fluence is more than $7.5\times10^{17}cm^{-2}$, the heavily doped silicon has been changed into high resistivity one and $6\times10^{17}cm^{-2}$ is the threshold fluence.

3.2 Infrared measurement of $[O_i]$

Fig.2(a),(b) shows the absorption spectrum of heavily doped Si after HLI and MLI, respectively. After irradiation, the effect of free carrier absorption was eliminated, and the absorption peak at $1106cm^{-1}$ and $830cm^{-1}$ due to interstitial oxygen and A center (Oxygen-Vacancy pair), respectively, appears clearly. From Fig. 2(a),(b),



(b)

Fig. 2 Infrared absorption spectrum of P-type heavily doped Si
(a) after MLI (b) after HLI

the $[O_i]$ of P-type heavily B-doped silicon after HLI and MLI was calculated to be $4.33\times10^{17}cm^{-3}$ and $4.52\times10^{17}cm^{-3}$, respectively.

3.3 Effect of A-center on precise measurement of $[O_i]$

As mentioned above, after the neutron irradiation of fluence more than 6×10^{17}

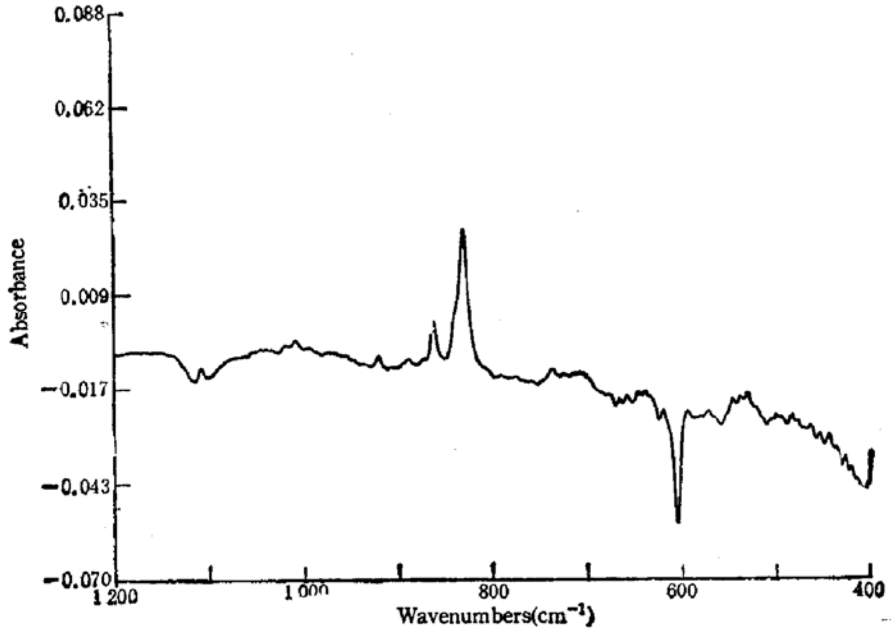


Fig. 3 Subtracted absorption spectrum of a pre-irradiated lightly doped Si from that of a post-irradiated one after 10 min annealing.

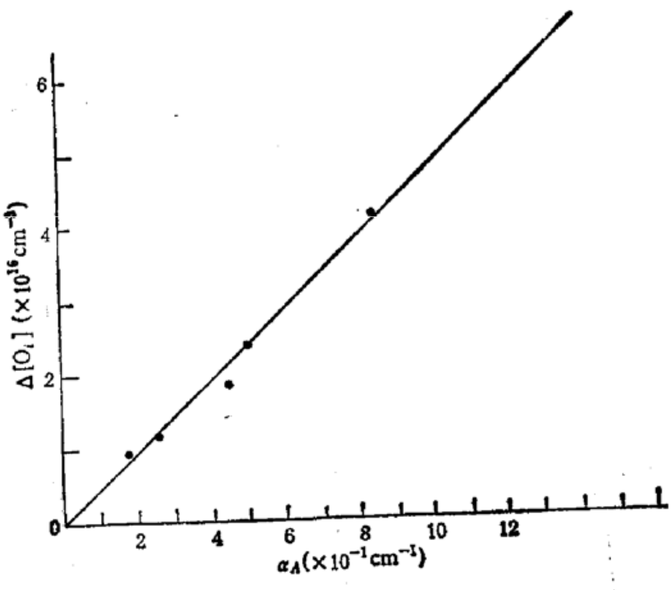


Fig. 4 Correlation of the $[O_i]$ decrease with the absorption coefficient of A centers observed in lightly doped Si

n/cm^2, a sharp absorption peak attributed to the main irradiation induced defect A-center (VO) at $830cm^{-1}$ appears as shown in Fig. 2. Consequently, the absorption peak at 1106 cm^{-1}decreases to some extent. Therefore, it is important to take into account the effect of A center and evaluate the $[O_i]$ precisely. Since the dissociation of A center has begun at temperatures higher than 350℃. The variation of the A center can be controlled by furnace annealing. After the irradiation of 7×10^{16} neutrons/cm^2, the lightly doped Si wafers were annealed at 400℃ for different periods from 10 to 45 minutes. Fig.3 shows the resultant absorption spectrum obtained by subtracting the spectrum of a pre-irradiated specimen from that of a post-irradiated ones after 10 min annealing. The distinct decrease of $[O_i]$ as well as the appearance of A centers is observed and the absorption peak at $862cm^{-1}$ is attributed to multivacancy.[3] It also shows the sharp decrease of the carbon absorption peak at 605 cm^{-1} due to the generation of defects after irradiation.

By subtracting the spectrum of a pre-irradiation specimens from the spectra of samples for different annealing periods separately, the correlation of the $[O_i]$ decrease ($\Delta[O_i]$) with the absorption coefficient of A center (α_A) were established (as shown in Fig.4) and can be expressed as follows:

$$\Delta[O_i] = k\alpha_A$$

where $k = 0.47\times10^{17}/cm^2$.

From the results described above, the interstitial oxygen concentration of heavily doped Si wafers in our experiments were evaluated. For the MLI silicon wafer, yielding

$$\alpha_A = 1.02cm^{-1} \quad \Delta[O_i] = 4.8\times10^{16}/cm^3$$

$$[O_i] = 4.52\times10^{17} + 0.48\times10^{17} = 5.00\times10^{17}/cm^3$$

For the HLI silicon wafer, yielding

$$\alpha_A = 1.14cm^{-1}, \Delta[O_i] = 5.4\times10^{16}/cm^3$$

$$[O_i] = 4.33\times10^{17} + 0.54\times10^{17} = 4.87\times10^{17}/cm^3$$

After correction, the $[O_i]$ of heavily B-doped Si wafer was determined to be $4.94\times10^{17}/cm^3$. The error of measurement of $[O_i]$ from the neutron irradiations of different fluences was $< 3\%$. The formation of A centers in heavily doped Si led to the slight decrease of $[O_i]$ by about 10—11%.

4 Conclusions

In this investigation, it shows that the resistivity of heavily doped silicon was increased from 10^{-2} to $> 10^3\Omega cm$ after fast neutron irradiation with fluence of more than $6\times10^{17}/cm^2$ and the effect of free carrier absorption was eliminated when the infrared absorption measurement was carried out. Taking into account the slight decrease of $[O_i]$ obtained from IR absorption spectrum due to the formation of A center, the interstitial oxygen concentration in heavily doped Si can be successfully determined by FTIR with a precision within 3%.

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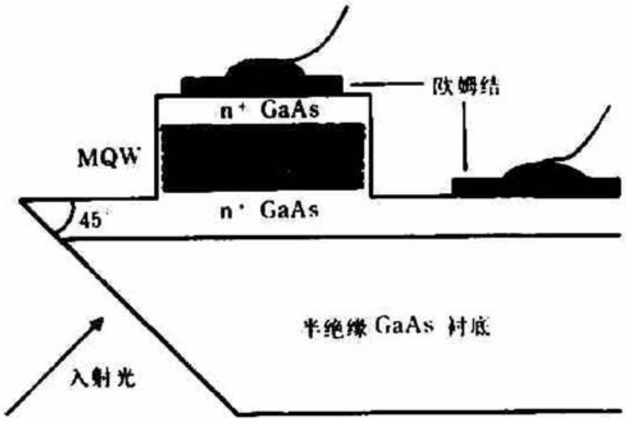

图 1 多量子阱红外探测器器件结构

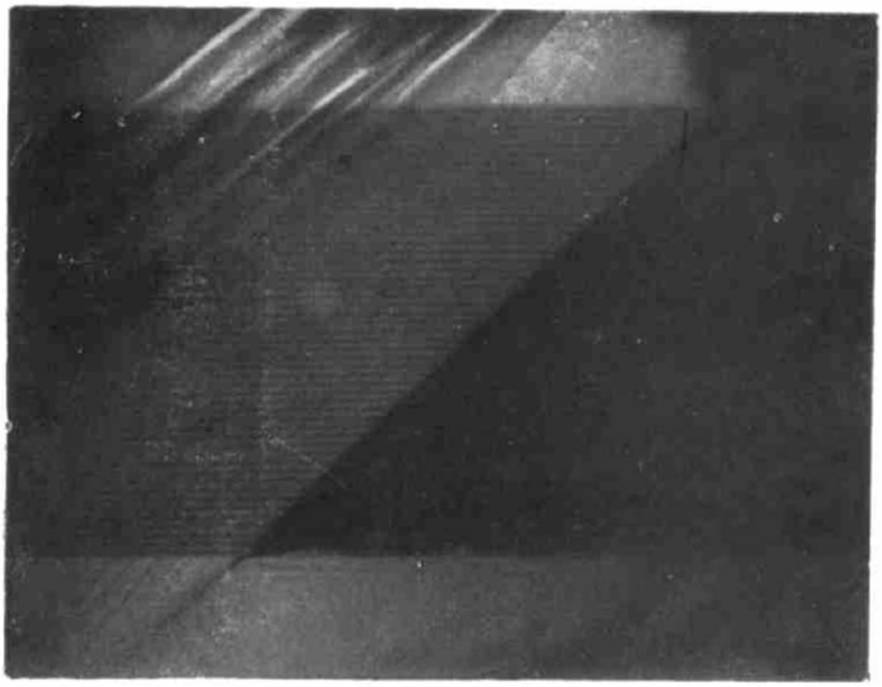

图 2 腐蚀台面边缘处断面的 REM 像