

Comparative study on multiple transmission–reflection infrared spectroscopy and infrared methods for the measurement of substitutional carbon and interstitial oxygen contents of polycrystalline silicon



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ABSTRACT

Multiple transmission–reflection (MTR) infrared (IR) spectroscopy was established as a new IR method to measure the interstitial oxygen and substitutional carbon contents of polycrystalline silicon wafers at ambient temperature. The superiority of MTR–IR to conventional IR was verified through practical measurements of polycrystalline silicon samples. With single incidence, the following characteristics of the former were superior to those of the latter. (1) The areas of the absorption peaks of interstitial oxygen at 1107 cm^{-1} and substitutional carbon at 605 cm^{-1} can be amplified linearly with transmission times N between 10 and 12, thereby extending the limit of detection of oxygen and carbon to approximately one order of magnitude lower. (2) The strength of the interference fringes of the baseline can be decreased to 5 times that of the single incidence for a 1 mm polycrystalline silicon slice. (3) In contrast to conventional IR method in which data are gathered from one sampling point at each measurement, MTR–IR collects information from many sampling points in a long sample for one-time measurement. In conclusion, MTR–IR is more sensitive, reliable, and reproducible than the conventional IR method in terms of measuring the interstitial oxygen and substitutional carbon contents of polycrystalline silicon.

1. Introduction

The photoelectric conversion efficiency of solar cells must be raised, and their production cost must be reduced to expand solar power generation markets and become accepted by consumers. Solar energy mainly involves monocrystalline and polycrystalline silicon. Solar cells have been developed using monocrystalline silicon, polycrystalline silicon, silicon ribbons, and film materials, such as microcrystalline silicon thin-film, multi-compound thin-film, and dye sensitized thin-film. Monocrystalline silicon should be replaced with polycrystalline silicon because of several factors. 1) With the improvement of the production technology of silicon stick, the length of its middle part with high purity increased, while the one of its two ends with low purity decreased. Correspondingly, the supply of the latter, which can be usually used to make polycrystalline silicon solar cells, is dwindling. 2) polycrystalline silicon square slices, which are more economical than monocrystalline ones, can be obtained directly by casting and directional solidification methods. 3) polycrystalline silicon production is improved

continuously, and a fully automatic casting furnace can be utilized to manufacture silicon ingots that weigh more than 200 kg and have grain sizes reaching the centimeter level. 4) The obtained results of single crystal silicon technology have also been applied to produce polycrystalline silicon cells.

With the development of solar cells, polycrystalline silicon has been used to replace monocrystalline silicon. Measurement methods for the contents of various substances, such as substitutional carbon (Cs) and interstitial oxygen (Oi), of polycrystalline silicon have been explored. Xin Fang and Wenzhong Shen studied the distribution of Cs and Oi concentrations in polycrystalline silicon through an infrared (IR) method and found that oxygen content increases gradually from top to bottom of a polycrystalline silicon ingot; by contrast, carbon content decreases little by little [1]. Lydia and McCormick used the IR method to measure the carbon content of polycrystalline silicon to improve the quality and accuracy of its spectrogram through annealing and low-temperature measurement [2]. Three major national standards of polycrystalline silicon include solar-grade polycrystalline silicon (GB/T

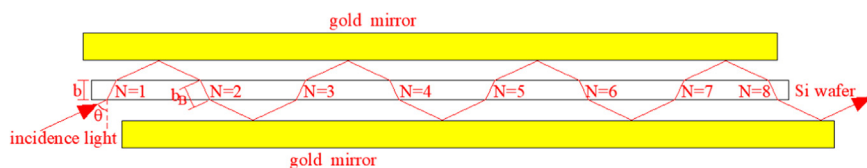
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where the incident light passes through at $\theta = 74^\circ$ (Brewster's angle of silicon = 74°). $N = 8$. The maximum N can reach 12 by extending the length of the gold mirrors and the silicon wafer. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

25074–2010) [3], the Method of Determining Interstitial Oxygen Content in Silicon by Infrared Absorption (GB/T1557–2006) [4], and the Test Method for Substitutional Atomic Carbon Content of Silicon by Infrared Absorption (GB/T 1558–2009) [5]. However, an ordinary IR method cannot be utilized to measure Cs and Oi contents in the grain boundaries of polycrystalline silicon compared with those of monocrystalline silicon, thereby leading to test errors.

Considering that an ordinary IR method inaccurately measures the Oi and Cs contents of polycrystalline silicon, we applied multiple transmission–reflection infrared (MTR–IR) spectroscopy, which was developed by Shoujun Xiao, the author's doctoral tutor, to measure the Cs and Oi contents of polycrystalline silicon, and our results were compared with those of conventional IR method [14].

2. MTR–IR

An MTR–IR equipment was designed and fabricated by Shoujun Xiao and Hongbo Liu [6–14] and used to qualitatively and quantitatively analyze the chemically modified molecular membranes on a silicon surface (Fig. 1). An incident light re-enters a silicon wafer through the reflection of golden mirrors placed on both sides of a silicon slice. Through many times inside a silicon chip, the optical path is increased, and absorbance is amplified. According to the principle of an integral sphere, or phase elimination interference, the transmitted light and the reflected light meet; therefore, a wave crest and a trough are added and offset to eliminate the effect of film interference [14].

3. Experimental part

3.1. Methods and materials

Double-sided polished polycrystalline silicon wafers (resistivities of 10 and 3000 Ω cm for Czochralski [CZ] and float zone [FZ], respectively, 1 mm thick; Shanghai JUN HE Electronic Materials Co., Ltd., China) were cut into rectangular ones (16 mm \times 50 mm) for IR analysis. CZ silicon wafers and FZ were respectively used as a sample and a reference to measure the impurity concentrations of Oi and Cs. The silicon wafers were carefully cleaned with a piranha solution (concentrated H_2SO_4 /30% H_2O_2 , 3:1, V/V), which reacts violently with organic materials, for 4 h to remove organic pollutants, boiled in $\text{NH}_3\cdot\text{H}_2\text{O}/\text{H}_2\text{O}/\text{H}_2\text{O}_2$ mixture (1:1:1, V:V:V) for 30 min, cooled to room temperature, rinsed with water, and stored in water. The silicon samples were immersed in 0.1% HF for 3 min to eliminate the native passivation silicon oxide layer and then dried with a stream of nitrogen before measurement was performed.

3.2. Measurement

The optical setup was designed to adapt to any commercial IR spectrometers. In this study, Bruker Tensor 27 was used. The MTR accessory with a Brewster incident angle of 74° was also utilized. Unless otherwise specified, a Deuterated Triglycine Sulfate (DTGS) detector with 100 scan times at 4 cm^{-1} resolution was used for measurement in a wavenumber range of 400–4000 cm^{-1} .

The silicon sample was inserted between the two gold mirrors. One end of the silicon slice was protruded approximately 5 mm out of the

Fig. 1. Scheme of the MTR setup [6]. After multiple transmissions and reflections on a silicon wafer placed between two gold mirrors, the incident light is guided to the detector. For clarity, the transmitted light is drawn without the reflected light. The red line denotes that the IR light goes through the accessory. b is the thickness of a silicon wafer, and b_B is the thickness of a silicon wafer

incident spot to ensure that the first incident shot was on the silicon surface. The two guiding mirrors could be moved back and forth to obtain the maximal luminous flux in the DTGS detector. The incident angle was controlled at 74° by using a microadjuster with a minimal angle scale of 0.225 [6].

The whole measurement procedure was performed in accordance with ASTM F 1188 and ASTM F 1391 [15,16]. Twelve random samples from different batches were used. Each sample was measured successively four times via MTR and conventional IR by slightly relocating the silicon wafer at each time to measure different sampling points. For each sample, 4 different sampling points were measured with conventional IR, whereas 48 different sampling points were examined through MTR–IR if $N = 12$.

4. Results and discussion

In Fig. 2, the baseline ripple of MTR–IR (0.002) is nearly 5 times smaller than that of the conventional IR method (0.01). The spread of light in polycrystalline silicon is complex because of the inconsistencies of various crystal cells of polycrystalline silicon and the existence of a grain boundaries region. Scattering and refraction are generated in the silicon chip, and numerous small ripples in the baseline appear. Polycrystalline silicon with a thickness of 1 mm does not produce interference fringes and is used here because the thickness of the wafer is considerably greater than the length of interference. However, the structure of polycrystalline silicon is complex. At a thickness of 1 mm, small ripples still appear on the baseline. These small ripples are more noticeable in the conventional IR method than in MTR–IR. The spectral quality of polycrystalline silicon is better in MTR–IR than in the conventional IR method because of the incidence of Brewster's angle (eliminating p polarization interference) and the effect of integrating sphere (reflection and transmission interference cancellation) [14].

The MTR–IR and conventional IR spectra of the same polycrystalline silicon slice are depicted in Fig. 3. The comparison of the sample spectra of the MTR–IR and conventional IR-examined polycrystalline silicon

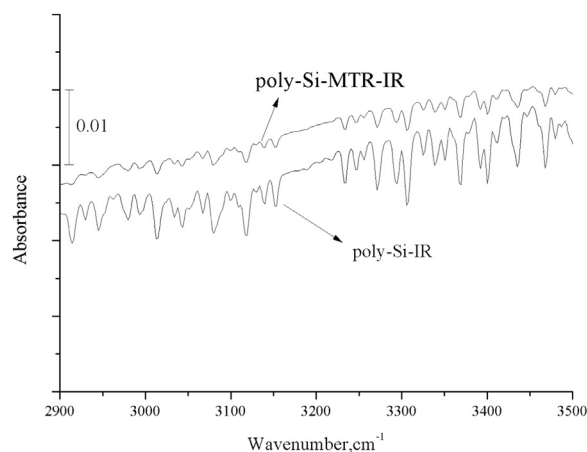


Fig. 2. Comparison of baseline spectra for a polycrystalline silicon sample measured with MTR–IR and conventional IR methods. Sample length = 50 mm, $n = 3.42$, $N = 12$, $b = 1.0$ mm, $b_B = 1.042$ mm, $b_{MTR} = 12.503$ mm, and incidence angle = 74° .

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