Analysis of Oxide Thickness Measurement Techniques of SiO_2:
Nanometrics Nanospec Reflectometer and Color Chart

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Abstract

The objectives of this experiment were to analyze two different methods developed for oxide thickness measurement, Nanospec/AFT and a color chart, and gain an experience of working inside a clean room environment. The sample examined was an oxide layer on silicon substrate (SiO_2), half doped with arsenic ion and half undoped. The equipment used in this experiment were Beta Scientific Instrument Co. Nanometrics Nanospec/AFT and a color chart. Factors causing the difference in the thickness through the wafer of SiO_2 on Si sample were discussed. Values for the oxide thickness layer obtained from Nanometrics Nanospec were found to decrease from 2227 to 2221 to 2219 A° then increase to 2237 A° from spot 1 to 4 in the undoped side, respectively. However, the results from spot 5 to 8 in the doped side were 2821, 2791 2777 and 2777 A° respectively, showing an overall decrease from the center to the edge of the wafer. Using the color chart, the thickness of the oxide layer was determined to be 2200 A° for the undoped zone corresponding to the red violet side and 2700 A° for the doped zone corresponding to the gold with slight yellow orange side. Differences in thickness were caused by the difference in the oxidation rate resulted from dopant and thermal flow. The experiment implies that both methods are nondestructive. The color chart method is simple, inexpensive while Nanospec is more accurate. Color chart is subjective and only gives values to two significant figures. Nanospec requires a calibrated standard in order to be quantitatively measure the sample. A typical reflectometer system cannot accurately measure values less than 100 Å.

Introduction

In microfabrication, thermal oxidation is a method used to produce a thin layer of oxide on the surface of a wafer, usually silicone. In this process, an oxidizing agent diffuses into the wafer at high temperature ranging from 600 ^{o}C to 1200 ^{o}C and reacts with it, creating an oxide layer. [1] The oxidation reaction occurs at Si-SiO_2 interface. Silicon atoms are consumed from substrate to form SiO_2. Therefore, the growth of oxide is both out and into substrate. [1]

The oxide layer in a semiconductor device is a very efficient electrically insulating layer on the surface of a material. This oxide layer serves as a dielectric in numerous devices. [2] Moreover, the oxide layer is a favored masking layer in many steps during device fabrication. [1,2] The oxide growth rate and therefore its thickness X_0 can be predicted by Deal-Grove model. At constant temperature, the time t required on bare silicon is given by:

$$t = \frac{X_0^2}{B} + \frac{X_0}{B/A} \tag{1}$$

where B is linear rate constant and B/A is parabolic rate constant. [1,2] The growth rate of oxide depends on several parameters such as substrate orientation, process time and temperature, dopant, gas flow rate and vapor pressure. [1]

Doping is a method to control the electrical properties in semiconductors. Through doping, the crystal composition is thus slightly altered so that it contains either a higher concentration of electrons or holes, which makes the semiconductor n-type or p-type, respectively. The doping of semiconductors can be performed by carrying out the doping after the film deposition by performing the implantation of dopants. [2]

Various techniques have been developed for measuring the oxide thickness, including reflectometry utilized by nanometrics Nanospec, ellipsometry and the use of a color chart. Nanometrics Nanospec uses the optical interference method, a simple and nondestructive way that usually measures thermal oxide thickness from 100 Å to more than 1 μm. [2] The principle of this method is based on characterizing the interference pattern created by light reflected from the air/SiO_2 interface and that from the Si/SiO_2 interface, as illustrated in Figure 1. A difference in optical path occurs between these two rays of light and a phase shift difference results. Therefore, the thickness of any film whose refractive index is known can be found by reflectance as a function of wavelength. [2]

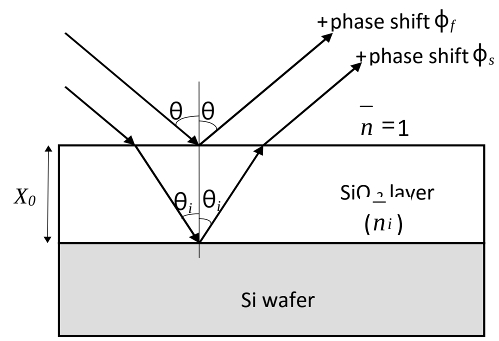

Figure 1. Optical interference method for the measurement of oxide film thickness. [2]

The second technique for oxide film thickness measurements is ellipsometry, the most popular technique used to assess the properties of silicon dioxide films. Ellipsometry provides a non-destructive technique for accurately determining the oxide thickness in the range of 20 Å to 60,000 Å with an accuracy of ±2%. [2] The components of an ellipsometer are light source, polarizer, compensator, analyzer and a detector. An ellipsometer operates by shining polarized monochromatic light onto the wafer surface at an angle. The light is then reflected from both the oxide and the silicon surface. A phase modulation unit, data measurement and processing system work together to measure the difference in polarization, which determines the thickness of the sample. [1,2]

The third method used to measure an oxide film thickness is comparing the film color with a calibrated chart as shown in Figure 2 for SiO_2. This technic is simple but less accurate than the other two. Each oxide thickness has a specific color when it is viewed under white light perpendicular to its surface. The color chart method is based on in the interference of the reflected and the incident waved. Constructive and destructive interference of reflected light waves causes the colorful patterns often observed in thin films. [2]

Film thickness (μm)	Color	Film thickness (μm)	Color
0.05	Tan	0.68	Bluish
0.07	Brown	0.72	Blue green to green
0.10	Dark violet to red violet	0.77	"Yellowish"
0.12	Royal blue	0.80	Orange
0.15	Light blue to metallic blue	0.82	Salmon
0.17	Metallic to very light yellow green	0.85	Dull, light red violet
0.20	Light gold to yellow, metallic	0.86	Violet
0.22	Gold with slight yellow orange	0.87	Blue violet
0.25	Orange to melon	0.89	Blue
0.27	Red violet	0.92	Blue green
0.30	Blue to violet blue	0.95	Dull yellow green
0.31	Blue	0.97	Yellow to "yellowish"
0.32	Blue to blue green	0.99	Orange
0.34	Light green	1.00	Carnation pink
0.35	Green to yellow green	1.02	Violet red
0.36	Yellow green	1.05	Red violet
0.37	Green yellow	1.06	Violet
0.39	Yellow	1.07	Blue violet
0.41	Light orange	1.10	Green
0.42	Carnation pink	1.11	Yellow green
0.44	Violet red	1.12	Green
0.46	Red violet	1.18	Violet
0.47	Violet	1.19	Red violet
0.48	Blue violet	1.21	Violet red
0.49	Blue	1.24	Carnation pink to salmon
0.50	Blue green	1.25	Orange
0.52	Green	1.28	"Yellowish"
0.54	Yellow green	1.32	Sky blue to green blue
0.56	Green yellow	1.40	Orange
0.57	Yellow to "yellowish"	1.45	Violet
0.58	Light orange or yellow to pink borderline	1.46	Blue violet
0.60	Carnation pink	1.50	Blue
0.63	Violet red	1.54	Dull yellow green

Figure 2. SiO_2 oxide film color chart. [2]

The objectives of this experiment were to analyze two different methods developed for oxide thickness measurement, Nanospec/AFT and color chart, and gain an experience of working inside a clean room environment. The sample examined was an oxide layer on silicon substrate (SiO_2), half doped with arsenic ion and half undoped. The equipment used in this experiment were Beta Scientific Instrument Co. Nanometrics Nanospec/AFT and a color chart. Factors causing the difference in the thickness through the wafer of SiO_2 on Si sample were discussed.

Experimental procedure

In this experiment, two techniques were used for oxide thickness measurements: Beta Scientific Instrument Co. Nanometrics Nanospec/AFT and color chart. The sample examined was an oxide layer on silicon substrate (SiO_2), half doped with arsenic ion and half undoped. The experiment was performed at room temperature and a pressure of 1 atm.

The first part of the experiment was to gain familiarity with the safety rules and regulation in clean room. A safety test was taken prior to attending the laboratory. The cleanroom lobby was entered after brushing the shows. In order to protect the environment from contamination generated by users, the following proper attires were worn before entering the cleanroom: cleanroom jumpsuits, shoe covers, gloves, gead gear and mouth cover.

The second part of the experiment was to use Nanometrics Nanospec reflectometer for measuring oxide thickness. After calibration of the pure Si wafer that was already placed on the stage, the film type was selected in order to process a reflective index of 1.45 for SiO_2. For magnification, an objective lens of 10X was chosen. The image was on focus by assuring that the edge of the superimposed polygon was sharp. Then, the sample was placed. Four points were measured at each side of wafer as demonstrated in Figure 3 Calibration and focusing were repeated whenever needed.

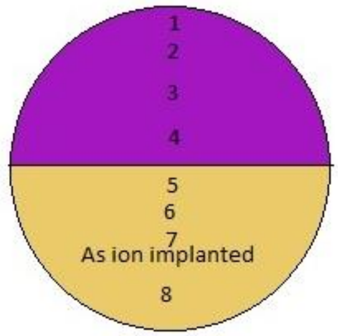

Figure 3. Schematic showing measurement locations for the SiO_2 on Si wafer.

Finally, the wafer sample was visually examined and the colors were matched to the color chart showed in Figure 2 in order to determine the corresponding thickness. It was assured that the line of sight was perpendicular to the wafer's surface.

Results

Using Nanometrics Nanospec reflectometer, measurements were performed at four points on both halves of the silicon wafer, doped and undoped, for a total of eight points. Table 1 summarizes the results for the oxide thickness for SiO_2 on Si wafer collected from Nanometrics Nanospec reflectometer. Based on the data obtained, the undoped zone was found to be thinner than the doped zone. Differences across each side were also observed. Going from spot 1 to 4, values for the oxide thickness layer were found to decrease from 2227 to 2221 to 2219 A° then increase to 2237 A°, respectively. However, the results obtained from spot 5 to 8 were 2821, 2791 2777 and 2777 A° respectively, showing an overall decrease from the center to the edge of the wafer.

Table 1. Oxide thickness for the SiO_2 on Si wafer obtained from Nanometrics Nanospec.

Spot	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Thickness (A°)	2227	2221	2219	2237	2821	2791	2777	2777

Then, the sample under test was visually examined to be gold with slight yellow orange on one side and red violet on the other side. Using the color chart in Figure 2, the thickness of the oxide layer was determined to be 2200 Aº for the undoped zone corresponding to the red violet side and 2760 Aº for the doped zone corresponding to the gold with slight yellow orange side. The resulting colors from the color chart correspond to the maxima amplitude wave in that film thickness.

Discussion

Even though the left and the right side of the wafer were processed under very similar oxidation condition, a large difference in the oxide thickness was observed between the two because the oxidation rate was greatly affected by the arsenic ion implantation. A thicker layer was formed in the side where the arsenic was absorbed due to the increase in the reaction rate. [2,3] More silicon atoms were consumed in the dopant region, forming a thicker oxide layer. [1]

Furthermore, measurements near the edge were expected to be smaller than measurements taken near the center in both sides of the wafer, doped and undoped. Spots near the center of the wafer consumed more silicon atoms than the others due the difference in the thermal flow. An increase in thermal flow increased the oxidation rate, resulting in a thicker layer. During oxidation, the wafer was processed in a horizontal furnace where the top of the tube is slightly hotter compared to the bottom of the tube, which caused a difference in the heat flow across the sample. [3] This difference changes the oxidation rate across the sample, which results in a non-uniform oxide thickness. [2,3]

The values obtained from the color chart are consistent with the values obtained from Nanospec but are less accurate. The color chart method is simple, inexpensive and nondestructive while Nanospec is more accurate and nondestructive. Nevertheless, both methods have some limitations. Determining oxide thickness by comparing a material with a color chart is subjective and it is inaccurate in most cases. In the color chart method, it is assumed that light is normal, no light is adsorbed as it passes through the film and refractive index is independent of the wavelength. Also, light from multiple reflections and polarization effects are ignored. [2,3]

Nanospec requires a calibrated standard in order to be quantitatively measure the sample. [1] A typical reflectometer system cannot accurately measure values less than 100 Å. [2] Therefore, reflectometry measurements are not sensitive to small changes in thin film thickness. The technique also assumes that the refractive index does not vary with thickness. [2]

Conclusion

The objectives of this experiment were to analyze two different methods developed for oxide thickness measurement, Nanospec/AFT and a color chart, and gain an experience of working inside a clean room environment. The sample examined was an oxide layer on silicon substrate (SiO_2), half doped with arsenic ion and half undoped. The equipment used in this experiment were Beta Scientific Instrument Co. Nanometrics Nanospec/AFT and a color chart. Factors causing the difference in the thickness through the wafer of SiO_2 on Si sample were discussed.

Overall, the experiment was successful in showing the accuracy and limitations of the two methods and the effects of dopant and thermal flow on the oxidation rate. Using Nanometrics Nanospec, values for the oxide thickness layer were found to decrease from 2227 to 2221 to 2219 A° then increase to 2237 A° from spot 1 to 4 in the undoped side, respectively. However, the results from spot 5 to 8 in the doped side were 2821, 2791 2777 and 2777 A° respectively, showing an overall decrease from the center to the edge of the wafer. Using the color chart, the thickness of the oxide layer was determined to be 2200 A° for the undoped zone corresponding to the red violet side and 2760 A° for the doped zone corresponding to the gold with slight yellow orange side. Differences in thickness were caused by the difference in the oxidation rate resulted from dopant and thermal flow. Dust on the sample caused some errors in the Nanospec measurement.

The experiment implies that both methods are nondestructive. It also implies that the color chart method is simple, inexpensive while Nanospec is more accurate. [2,3] The color chart method is subjective and inaccurate in most cases. Nanospec requires a calibrated standard in order to be quantitatively measure the sample. A typical reflectometer system cannot accurately measure values less than 100 Å. [2]

References

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