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Micro Silicon Bubble Formation during Argon Micro Sputtering in SEM
SEM中アルゴンマイクロスパッタによるマイクロシリコンバブル形成

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Local sputtering by Argon micro plasma jet performed in Scanning Electron Microscope (SEM) is being developed by our group. Sputtered area can be achieved in micro diameter depending on the hole size of an orifice gas nozzle which is used as anode placed in SEM chamber. However, the formation of silicon bubble and blister around the sputtered area, which can be observed frequently, is one of the imperfections of sputtering process. Therefore, to understand of this phenomenon, silicon bubble formation due to local sputtering by Argon micro plasma jet performed in SEM is studied.

1. Introduction

Micro plasma technologies have rapidly progressed and are widely used in medical, environment and industrial business due to its merit properties such as non thermal plasma, low power consumption, low operating cost and the ability to be operated in the sub atmospheric and atmospheric pressure [1].

Recently, local micro sputtering in SEM chamber by DC Argon (Ar) plasma using small orifice gas nozzle has been developed by our group [2]. The advantages of micro sputtering performing in SEM chamber are useful for the application which requires the micro size operation and low oxidation impurity in work piece.

In this paper, the prominently interesting phenomenon observed in our previous study is presented. Except a micro size sputtered area was achieved, Silicon (Si) bubble surrounded the sputtered area were noticed in some conditions. In this paper, the formation of Si bubble is on focused to understand its phenomenon.

2. Experimental apparatus and procedure

Fig. 1 shows the experimental schematic diagram of micro plasma jet used in this research. Micro sputtering was performed, during SEM-working, by injecting Argon gas through stainless steel orifice gas nozzle anode (OGN) placed on the SEM stage holder in the SEM chamber (Hitachi S-3000N) for DC plasma generation OGN has a hole at the OGN tip size of Ø50 µm, and inner and outer diameters of the nozzle of 1.63 and 1.81 mm, respectively.

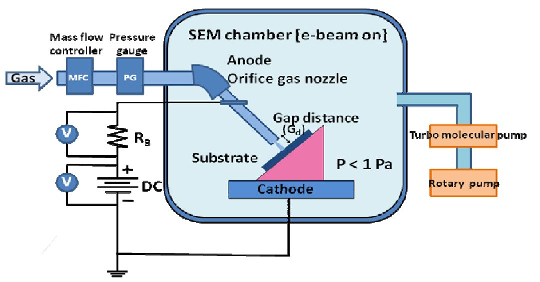

Fig.1. Schematic diagram of micro plasma jet set up in SEM.

A mass flow controller was used to control Ar gas flow rate (GFR) supplied through the OGN at 2.5 - 5 sccm. During SEM-working, gas profile could be observed at the gap distance (G_d), between OGN and the counter electrode (Si substrate), which was adjusted by moving either the OGN or the counter electrode using a micrometer. A turbo molecular pump and a rotary pump were additionally installed on the SEM chamber to evacuated the injected gas and maintain a low vacuum pressure (P < 1 Pa) in the SEM chamber.

DC source having 1 kV maximum voltage was supplied for the discharge through a 10 MΩ ballast resistor (R_B) to limit the current in series. Discharge current (I_d) monitored by the voltage drop across R_B was controlled by applying voltage at 1.5 µA, and discharge voltage (V_d) was calculated from the difference between supplied voltage and voltage drop across R_B. To study the bubble formation, the experimental condition was divided into two parts. The first part was gap distance dependence. GFR was fixed at 2.5 sccm while the G_d was varied ranging from 100, 130 and 170 µm. Another condition was GFR dependence. GFR was varied ranging from 2.5, 3.7 and 5 sccm at 170 µm G_d. In both cases, the experiments were performed in the same discharge time ranging from 1, 10 and15 seconds.

3. Results and discussion

Top view SEM images for sputtered surface on Si substrate after 15 seconds of sputtering in the case of 2.5 sccm GFR at G_d of 170 µm is shown in Fig. 2 (a). The sputtered area surface was very smooth having a small black spot locally surrounding the sputtered area. The black spot shape looked similar to a small hollow dome standing on the surface of Si target as shown in the Fig.2 (c). After confirmation the hardness of the dome-like structure by touching the black spot by the needle, the black bubble was broken but there was no scalp bouncing off to other area. Therefore, the black spot was named bubble due to its shape and soft surface.

From the experimental results, the number of bubbles is depended on the discharge time and reverse proportional to the gap distance as shown in the table I. When the gap distance was increased, the number of bubble was decreased. The bubbles size became small in the case of long gap distance and high GFR, as depicted in the Fig.2 (b) which using 5 sccm GFR at 170 µm G_d. The Bubble diameter noticed in this study size was ranging from less than 1 µm to 26 µm with height ranging from 0 to 3 µm. The largest bubble diameter was found in the case of 2.5 sccm GFR at 170 µm G_d and 3 µm height.

From the previous study [2], Si target could be archived higher sputter yield in the case of low GFR than that of high GFR at the same I_d due to the higher ion energy. In the case of high GFR, there is high probability of collision between sputtered atom and neutral atom causing high rate of sputtered atom backscattering to be redeposited on the target decreasing sputter yield. From the results, the tendency of low density and small bubble size could be observed in the case of high GFR which is consistent with the sputter yield. In another word, the density and size of bubble is direct proportional to the sputtered yield.

Orifice gas profile is an important factor which could be used for explanation the formation of bubble and speculation the sputtered area shape. As shown in Fig. 2 (d), the gas profile has brighter color at the center of gas flow path and near the OGN hole due to high density of Ar gas. A cluster of high gas density flows at the center of gas profile before evacuated by TMP. The shape of sputtered area is consistent with the orifice hole shape which effects on the shape of gas profile as well. From the results, the bubbles were found locally at the edge of sputtered area, this could be due to many parameters such as incident angle of ion bombardment, difference of ion energy and flux, difference of gas and plasma profile at the area which is far from the center of operating point.

At the center of sputtered area, Si target is bombarded by dense incident Ar ion. When Si atom gains sufficient energy, it will be sputtered. In contrast, the bubble is formed due to knock-in of operated gas molecule during bombardment as mentioned in [3]. The

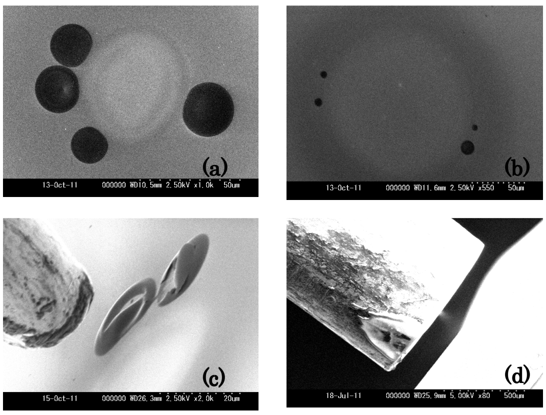

Fig.2 SEM images for (a) sputtered surface on Si substrate by Ar micro plasma using 2.5 sccm GFR, (b) 5 sccm GFR at 170 µm after 15 seconds of sputtering, (c) hardness testing of bubbles, and (d) orifice gas nozzle with injecting Ar gas in SEM chamber.

Table I. Number of bubbles at the various gap distances using 2.5 sccm gas flow rate discharge current of 1.5 µA

Gap distance (µm)	Discharge voltage (volt)	Number of bubbles Discharge time (second) 15	10	1
100	806	Crowded of Si bubbles and flakes	Crowded of Si bubbles	-
130	838	17	11	-
170	878	4	3	-

gas molecule will be accumulated under the surface of Si surface in amorphous Si layer which caused by ion bombardment at the surface of crystalline Si layer [4-5]. If the bubble has a large of internal pressure; the bubble will be deformed and ruptured causing blistering.

The remarkable point is that the bubble size and the thickness of bubbles surface found in our research were much larger and thicker than that of other groups [4-5]. The bubble thickness and diameter size observed in this study was more than 20 nm and 1 µm, respectively. This phenomenon could be come from the fact that micro sputtering in this research was performed in very narrow gap distance and in the normal direction of ion incident angle to the Si target which could have effect on the size and depth of bubble [3]. Moreover, the ion kinetic energy used in our research (≈ 800 eV) was lower than that used in conventional bubble formation [4-5]. As could be seen in the Fig.2, Si bubble color is quite darker than that of other area, it is expected that dark color is due to the secondary electrons cannot overcome work function energy between double walls (bubble and Si surface) to escape from the bubble surface. Therefore, they are trapped inside of the bubbles which make the bubble area dark. However, the outstanding of bubble formation noticed in this research is that they appear locally at the edge of sputtered area.

4. Conclusion

The silicon bubble causing during DC Ar micro sputtering performing in SEM chamber was studied. The diameter of bubble size was ranging from a few µm to 26 µm. The number of bubbles is depended on the discharge time and reverse proportional to the gap distance when gas flow rate and discharge current were fixed.

5. Acknowledgments

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