

Cleaning of silicon surface by surface dielectric barrier discharge

F. Samoila, V. Anita, L. Sirghi

Iasi Plasma Advanced Research Center (IPARC), Faculty of Physics, Alexandru Ioan Cuza University of Iasi, Blvd. Carol I nr. 11, Iasi 700506, Romania

Silicon is the most common material used in microelectronics and micro electromechanic systems (MEMS) devices. In many cases the surfaces of silicon used for these proposes is not clean due to adsorption of airborne and package-released hydrophobic organic contaminants. In previous works we have shown that the negative glow plasma of a glow discharge in air at low pressure is very efficient in removing hydrophobic contaminants from silicon surface. Moreover, adding of water vapour to the working gas produces hydroxyl groups on the oxidized surface of silicon and renders the surface superhydrophilic after the treatment. In the present work we use a surface dielectric barrier discharge (DBD) in air at atmospheric pressure to clean silicon wafers in a Petri dish. The cleaning effectiveness is evaluated by measurements of water contact angle of the silicon surfaces, which showed a drastic decrease from about 80° to less than 40° in about 10 minutes treatment time. Cleaning of the silicon surfaces by the DBD surface discharge plasma has been probed also by increasing capillary force between a silicon AFM tip and cleaned silicon surface.

1. Introduction

Silicon is the most common material used in microelectronics and micro electromechanic systems (MEMS) devices, including the commercial atomic force microscopy (AFM) probes. In many cases the surface of silicon is contaminated due to the adsorption of airborne and package-released hydrophobic organic contaminants [1]. Various cleaning methods are used to remove the organic contaminants molecules or contaminant particles attached to silicon surfaces. These methods can be classified as physical or chemical, wet or dry. In general, the most used method for silicon wafer cleaning is wet chemical cleaning, but, because of the of high cost, problems with waste disposal and safety issues, dry cleaning methods are preferred [2]. The most important of dry cleaning methods are: thermally cleaning, gas phase cleaning, photochemical enhanced cleaning and plasma enhanced cleaning [3]. From all this methods, plasma cleaning is simple, fast and environmentally friendly [4]. In comparison with low-pressure plasma techniques, the atmospheric pressure plasma cleaning represents a more easy and economic procedure. The goal of this work is to study the cleaning effect of surface DBD plasma on silicon surfaces. The effectiveness of plasma cleaning of silicon surfaces is evaluated using two different methods: contact angle measurements and AFM adhesive force measurements

2. Experimental setup

The experimental arrangement used to generate the surface DBD discharge at atmospheric pressure is sketched in Fig. 1. The DBD is generated on the surface of an Al_2O_3 ($k = 9$) plate (area of

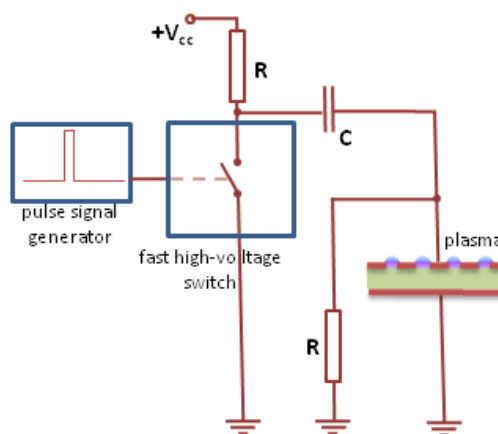


Fig. 1 Sketch of the DBD electric circuit used in surface cleaning

75 × 60 mm² and thickness of 0.5 mm) with aluminium electrodes (0.2 mm in thickness). The grounded electrode (50mm × 50 mm) was bound to one side of the dielectric plate and an aluminium radiator in order to keep the device at the room temperature. The active electrode, on the other side of the alumina plate, had a structure of 12 parallel strips (2 mm × 50 mm with gaps of 2mm) and connected to a high-voltage pulsing generator. The discharge device was placed above a glass Petri dish (60 mm in diameter) to form a miniature reactor for surface treatments. The high-voltage pulses are generated by a fast high-voltage transistor switch made up of a large number of MOSFETs combined in a compact low-inductance bank (HTS 30-06, from Behlke Power Electronics GmbH, Germany). The switch is connected to a *dc* power supply and controlled by a signal generator (Tektronix AFG

3052C) that generated short voltage pulses (5 V in amplitude and 1 μ s in width) with a repetition frequency of 15 kHz. The pulse voltage and intensity were recorded by a digital storage oscilloscope (Tektronix DPO 2024 B).

Typical time variations for voltage, current intensity and instantaneous power during a pulse are presented in Fig.2. Dependence of energy per pulse and average power (at 15 kHz frequency) on d c voltage applied on the fast high-voltage electronic switch is presented in Fig.3. The discharge takes place during the very fast variation of the voltage. As shown in Fig. 2, the voltage changes in a few ns from a positive value (around 400 V) to a large negative value (around -2000V). Then, the voltage decrease to zero in about 20 μ s. Large intensity fluctuations (tenths of Amperes) are noticed during the discharge. Due to stray inductance of the discharge circuit, the discharge current has a large reactive component. This is illustrated by the oscillations of the instantaneous power which shows oscillations with negative values. However, the integral of the instantaneous power during a pulse compute a positive value of the energy dissipated on the surface DBD. Because of the high dielectric constant of the alumina plate and design of the device (small thickness of the dielectric and high ratio of passive over active electrode area), the discharge breaks at relatively low values of *dc* voltage (1000V in our case) [5]. Figure 3 shows the dependence of the discharge dissipated energy and average power on the *dc* voltage used for the high-voltage pulse generation. Thus, at the *dc* voltage and pulsing frequency used for surface treatment (2000 V and 15 kHz, respectively) the average power of the discharge was about 5 W, which assured a surface density of power of 0.2 W/cm².

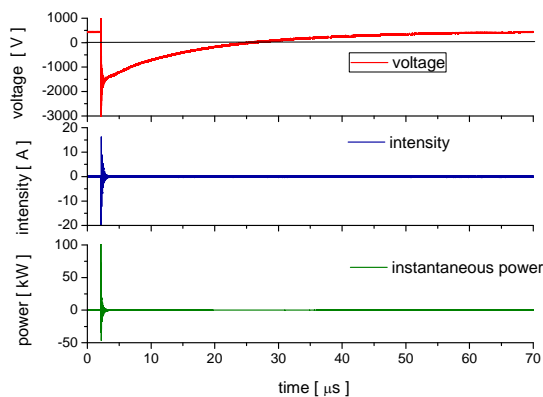


Fig. 2 Time variation of voltage, current intensity and instantaneous power during a surface DBD pulse

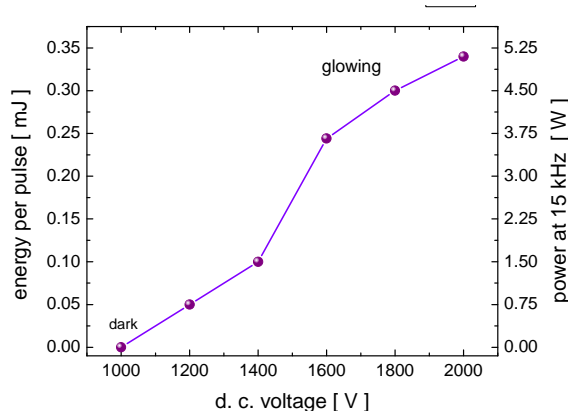


Fig. 3 Dependence of energy per pulse and average power (at 15 kHz) on *d c* voltage applied on the fast high-voltage electronic switch.

3. Results and discussion

Thus, the efficiency of the surface DBD plasma treatments was evaluated by water contact angle measurements. Adsorption of hydrophobic airborne contaminants during long storage time renders the silicon surface less hydrophilic. The very reactive oxygen and hydroxyl species produced in surface DBD plasma react with the adsorbed contaminant molecules and break them to form volatile compounds. Removing of the hydrophobic contaminants on silicon surface restores the good hydrophilicity of silicon surface. Figure 4 shows the variation of water contact angle on silicon surface as function of DBD plasma treatment time. The initial value of water contact angle was around 80°, which is attributed to surface adsorption of hydrophobic organic contaminants. After 10 minute of exposure of silicon wafer to the surface DBD plasma, the water contact angle had a significant decrease to about 40°. This decrease in contact angle value indicates that DBD plasma is an efficient cleaning method. However, differently from the silicon cleaning in low-pressure plasma [1], the water contact angle did not further decrease with the increase of the treatment time. This might be an indication that the treatment in atmospheric plasma DBD does not generate hydroxyl groups on the silicon surface.

Cleaning of silicon surface and change of surface hydrophilicity do have a large impact on the capillary adhesive force measured in atomic force microscopy [1]. It is known that a small capillary water bridge forms instantaneously at a nanoscopic contact formed by AFM tip and sample surfaces in humid air and it generates a strong adhesion force [6].

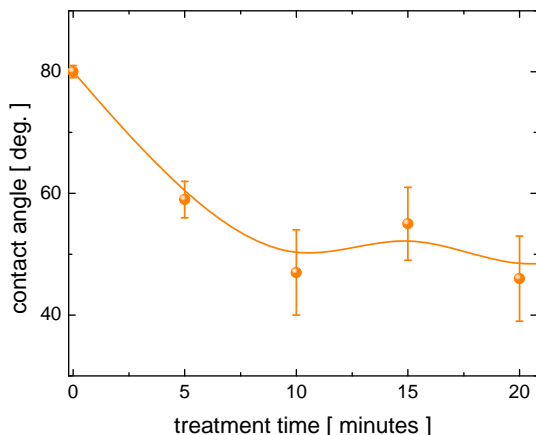


Fig. 4 Variation of silicon contact angle with the DBD plasma treatment time

Figure 5 presents typical force-distance curves measured in ambient air (RH = 30%) with a silicon AFM tip on a silicon (100) surface, before and after 10 minutes of DBD plasma cleaning. Before cleaning the surface the value of adhesive force was around 15 nN and after 10 minutes of DBD plasma treatment the force became 34 nN. Increase of the hydrophilicity of silicon surface led to an increase of the capillary water bridge between AFM tip and silicon surface, which determined a larger adhesive force.

4. Conclusion

Usually, the surface of silicon used in various applications is not clean due to adsorption of airborne and package-released hydrophobic organic contaminants. In the present work we used a surface dielectric barrier discharge (DBD) in air at atmospheric pressure to clean silicon wafers. An efficient pulsed DBD plasma in atmospheric air was obtained at relatively low voltage values by using a DBD device with high dielectric constant and a good design of electrodes and pulsing scheme. The cleaning effectiveness is evaluated by measurements of water contact angle of the silicon surfaces, which showed a drastic decrease from about 80° to less than 40° in about 10 minutes treatment time. Cleaning of the silicon surfaces by the DBD surface discharge plasma has been probed also by increasing capillary force between a silicon AFM tip and cleaned silicon surface

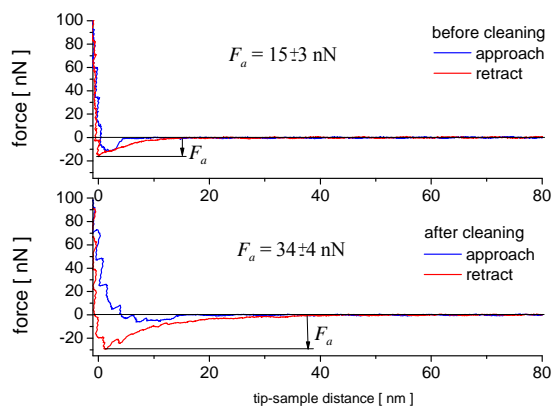


Fig. 5 Force-distance curves acquired with a silicon probe on (100) surface of a silicon wafer before and after DBD plasma cleaning (10 minutes).

5. Acknowledgement

This work was supported by CNCSIS, IDEI Research Program of Romanian Research, Development and Integration National Plan II, Grant no. 267/2011.

References

- [1] L. Sirghi, O. Kylian, D. Gilliland, G. Cecone, and F. Rossi, *J. Phys. Chem.* **B 110** (2006) 25975.
- [2] L. Sirghi, *Rom. J. Phys.* **56** (2011) 144
- [3] A. Thedsakhulwong, W. Thowladda, *J. Metals, Materials and Minerals* **18** (2008) 137.
- [4] A. Pamreddy et al, *Surface & Coatings Technology* 236 (2013).
- [5] V. I. Gibalov, G. J. Pietsch, *Plasma Sources Sci. Technol.* **21** (2012) 024010.
- [6] L. Sirghi, R. Szoszkiewich, E. Riedo, *Langmuir* **22** (2006) 1093.