Student Paper

Plasma Activation of Si surfaces: An Easier and Safer Approach for Microsphere Lithography

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Lithography still remains as the primary enabling technological step in fabricating many kinds of devices even though the minimum size of such devices keeps shrinking down to nanometer scale [1,2]. Today the creation of precisely engineered CMOS devices involves the use of advanced steppers, reflective optics and DUV light sources, which demand large capital investment (~$5B/fab) and processing costs (~$1M/mask). This level of cost is neither sustainable nor practical for low-cost and simpler devices such as bio-chemical sensors with sub 100nm features. These simpler-to-fabricate devices demand a more affordable and less-complex technique to define the minimum size typically between 500 and 50nm, owing to their higher tolerance for low yield. One of such promising alternatives to expensive optical/DUV lithography in this range is the microsphere lithography (ML) that relies on the deposition of a monolayer of tiny ($d \leq 1\mu m$) polymer and/or glass spheres to form a hexagonal-closed-packed (HCP) lattice on a chosen substrate (typically glass or Si). ML is especially useful for building artificial 2D-crystals for magnetic, plasmonic, photonic sensors and growth templates that can be defined with a single lift-off or etching step, as found in many low-cost applications [3,4]. Being a maskless process and requiring no radiation source, ML is probably one of the cheapest and simplest approaches to form nanostructures down to 100nm, as long as the intended pattern is a simple 2D, preferably HCP, lattice.

Three main steps involved in ML are surface treatment, coating, and post additive/subtractive processes (Fig 1). The most critical is the surface treatment step that subsequently allow for self-assembly of the spheres. Existing methods for this step either involve purely chemical *functionalization*, i.e. coating the surface with unique molecules for a highly specialized bonding group attached to the spheres, or use of so-called piranha treatment, i.e. rather strong acids (H_2SO_4/HF) in elevated temperatures ($\geq 60^{\circ}C$), to form hydrophilic Si surfaces by increasing the silanol (Si-O) groups. While the former may require specialized chemistry limited to a unique set of applications, the latter uses copious amount of dangerous acids in hazardous conditions. Hence a safer and less complex scheme would be very desirable. In this paper, we propose a novel method for surface treatment using *plasma activation*, which is relatively simple, cost effective and a comparatively safer method. More specifically, we use O_2 plasma treatment to activate the Silanol groups on the surface of the Si substrate to change its hydrophilicity, and quantify the result of this process by force-spectroscopy and direct assembly techniques. As can be seen from the force-spectroscopy results in Fig. 2, exposure to O_2 plasma leads to activated Si surfaces that matches to the response of the *piranha* treatment. Similar experiments repeated using specialized AFM cantilevers that have carboxyl-terminated SiO_2 spheres as contacts reveal a larger stiction response of the activated surface (Fig.3) that increases with RF power in the plasma chamber. Similar *F-d* curves of plasma treated surfaces are also studied as a function of pressure, O_2 flow-rate, temperature and time (plasma and ambient) to optimize processing and handling conditions for the proposed plasma technique.

The key factor for ML technique to be effective is the creation of large area of monolayers with minimum point and line defects. Inaccurate sphere/solvent ratios tend to form either dense multilayers of spheres on one end or very patchy and highly defective surfaces on the other end. Thus, in this study, we not only report on optimum plasma conditions for effective surface activation but also compare the best deposition conditions (i.e sphere density) and effectiveness of the different deposition methods such as drop coating, spin coating and float transfer on the properly activated surfaces. For instance, simple drop coating of 0.5μm polystyrene spheres (2.5% w/v) dispensed in a 1:1 ethanol solution, volume adjusted to the dimensions of substrate, produced results (see Figs. 4&5) comparable to the spinning technique at 1000 RPM (Fig.6), which implicate, for all intents and purposes, that plasma activation is comparable to other standard approaches for surface treatment for ML. Another unique implication of this work is the fact that we report a direct and comparative analysis of plasma activation process between Si surfaces. Hence the optimized processing conditions reported herein can be used for tuning both the ML and wafer-bonding process commonly used in MEMS, SOI wafer production and 3D integration of ICs.

References

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[4] W.Y. Fu, K.K.Wong and H.W. Choi, *Appl. Phys. Lett.,* vol.95, p.133125. 2009.

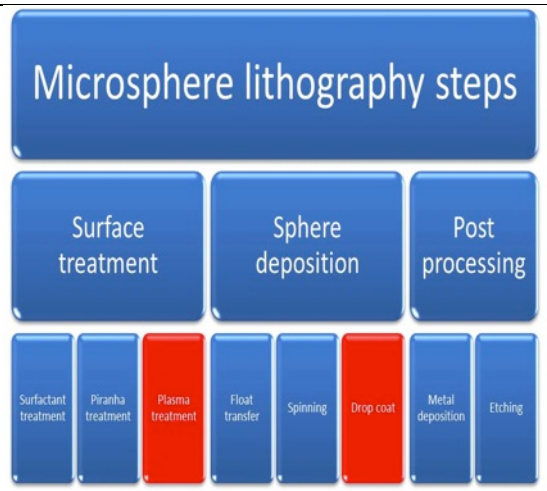

Fig. 1 *Steps involved in ML. Our focus is in this work is highlighted in red boxes*

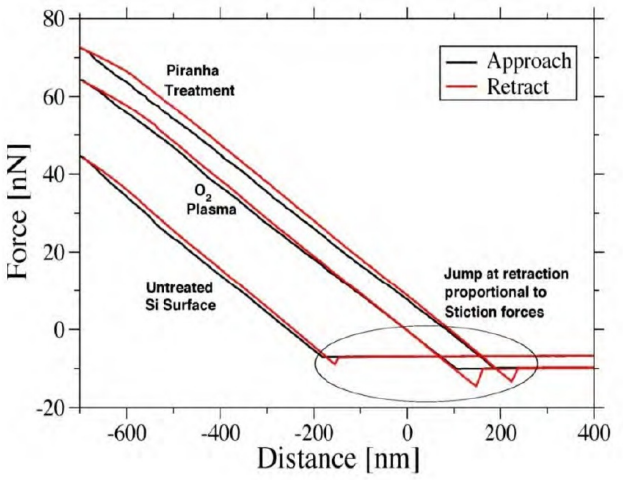

Fig. 2 *Force-Distance curves measured using AFM on surface (plasma & piranha) activated and untreated samples.*

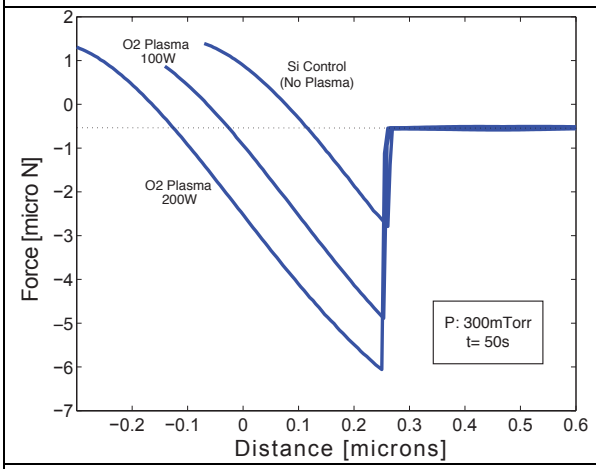

Fig. 3 *Force spectroscopy of unexposed and plasma activated Si surfaces obtained using SiO_2 sphere contacts.*

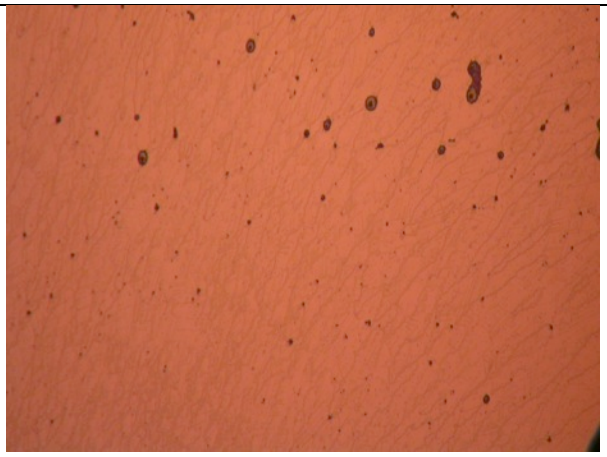

Fig. 4 *Optical image* (×200) *showing a monolayer of drop-coated PS spheres (d=0.5µm, 2.5%w/v) diluted 1:1 in ethanol.*

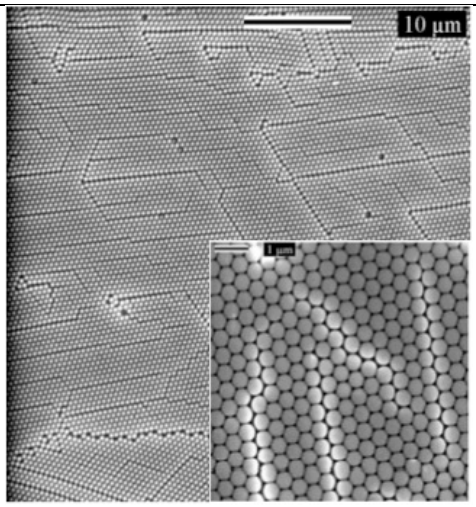

Fig. 5 *SEM image showing monolayer of PS spheres in Fig.4 produced on plasma activated Si via drop coating.*

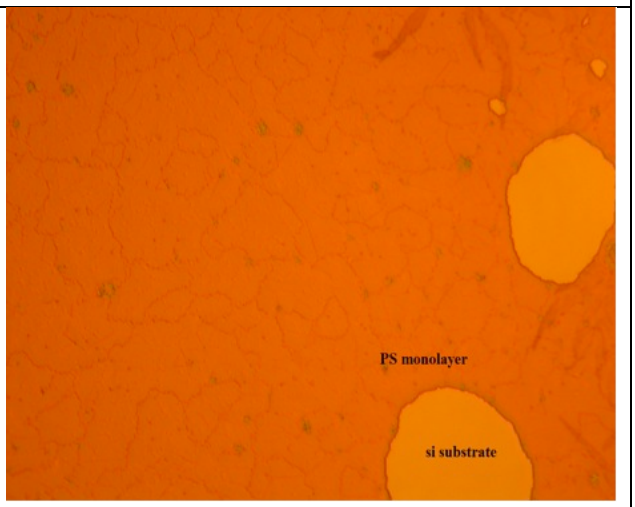

Fig. 6 *Optical Image* (×200) *showing a monolayer of spin-coated PS spheres (d=0.5µm, 4%w/v) diluted*