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Surface activation for low temperature wafer fusion bonding by radicals produced in an oxygen discharge

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1. Introduction

Wafer bonding is ubiquitous in the semiconductor industry, whether it is applied to the manufacture of substrates for CMOS microelectronics, heterogeneous devices such as high brightness LEDs, or MEMS. High temperature fusion bonding, which involves treatment at temperatures in excess of 700 °C, is well established for the manufacture of SOI substrates. However, where the wafers are of different materials or where at least one of the wafers contains other materials, such as metallization or patterned areas of different doping, exposure to such a large temperature cycle effectively destroys the devices.

Much work has therefore been focused on the development of low temperature bonding techniques for silicon wafers, where the term 'low temperature' means below 400 °C—the upper limit on the temperature to which an aluminium metallization track on a silicon wafer can be taken without significant degradation of the film and the substrate interface. A more ambitious goal is to achieve full bond strength between silicon wafers below 200 °C, which allows moderately temperature sensitive materials such as polymers, piezoelectric, and magnetic materials to pre-exist

ABSTRACT

A new method of exposing silicon/semiconductor wafers to a mixture of radicals is described, in which these species are generated in an oxygen-rich gas discharge confined between a concentric pair of annular mesh electrodes surrounding the wafers. This approach allows the wafer surfaces to be treated without damage from the energetic ions, strong electric fields, and high UV fluxes associated with direct treatment by exposure to gas discharge plasmas. The process is compared with direct oxygen plasma activation for its latitude with respect to treatment duration, effect on wafer surface roughness and bond strength. Wider process latitude and reduced surface roughening are obtained for treatment by radicals compared with direct plasma exposure. Comparative analysis of treated and untreated silicon surfaces by X-ray photoelectron spectroscopy indicates that traces of fluorine present on the wafer surface before treatment are removed with great efficiency by the process.

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on the wafers. Yet another goal is to obtain good bonds without exceeding room temperature much. This allows bio-materials and other highly temperature sensitive materials to be used, and minimizes the thermal expansion mismatch between wafers of different materials. There are many examples [1–4] of devices that would benefit from the improvement of interlayer-free, low temperature silicon-to-silicon wafer bonding.

It has been shown [5] that increased bond strength (to about half the theoretical maximum bond energy density) between two oxidized silicon wafers is obtainable after their surfaces have been exposed to an oxygen plasma for 5 min prior to bonding, and after a post-bonding heat treatment at 200 °C for 2 h. Farrens et al. [6] showed that brief exposure to an oxygen plasma enables a variety of materials including homogeneous pairs of sapphire, silicon dioxide, silicon nitride, and gallium arsenide to be bonded together. High strength bonds between oxidized silicon wafers were achieved with a thermal cycle of less than 300 °C.

Tagaki et al. [7] reported full-strength bonding between silicon wafers without the need for any heating at all using an Ar atom beam sputter etching process followed by bonding in situ under vacuum. The study does not investigate the possible effects of damage from the high-energy Ar atoms, but since the process is known to etch silicon at a rate of approximately 4 nm/min, it is reasonable to expect that the process does result in some degree of damage to the surface.

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Fig. 1. Cross-sectional view of the annular radical generator. The wafer platens and vacuum chamber are also shown. The chamber volume is approximately 22 L.

The system and method described in this paper is an attempt to avoid the shortcomings of the bonding techniques used to date.

1.1. Design and operation of the radical activation system using a gas discharge

Three principles govern the design of this radical generation system:

- 1. To protect the surfaces from strong electric fields, UV fluxes, and ion bombardment damage, the discharge that generates the radicals must be remote from the wafer surfaces.
- To avoid the need for complex RF power supplies, the radical generator must operate under low voltage DC or 50–60 Hz AC excitation.
- 3. To ensure good treatment uniformity, the transport of the critical activating species to the wafer surfaces must occur sufficiently rapidly for the wafer activation to be rate-limited by the surface process rather than the concentration of species in the gas above the surface.

The design of the system [8] is shown in Fig. 1. The gas discharge is enclosed within an annular volume bounded by fine steel mesh walls at internal and external diameters of approximately 160 and 200 mm respectively. This is the radical generator. The mesh walls consist of perforated 0.1 mm thick stainless steel sheet containing a hexagonal array of 0.25 mm diameter circular holes creating a 22% open area. These electrodes are held at ground potential. A third mesh electrode of an intermediate diameter carries the electrical excitation, and the discharge is struck between this and the meshes to either side. Top and bottom, the radical generator is bounded by machinable glass–ceramic rings.

Thus, the first of the design principles is met: the radical generator ring lies beyond the outer diameter of the wafers, and the grounded meshes serve to create a field-free space around the wafers. Within the discharge region, the electric field is parallel to the plane of the annulus and therefore also to the wafer surfaces. This, and the field-free space surrounding the wafers, ensures that any ions carried past the mesh by their own momentum do not strike the wafer surfaces without first having undergone a collision in the gas. Also the pressure is sufficiently high to ensure that any ions propelled past the mesh are rapidly thermalized. The flux of UV photons created within the discharge is heavily attenuated as seen from the wafers; firstly by the small open area of the mesh, and secondly by the low angle of incidence with the wafer surfaces.

The second and third design principles are simultaneously met by choosing a combination of gas pressure and electrode spacing that allows the discharge to sustain itself at less than 300 V DC or AC, and using a pressure that allows the radicals to be transported to the centre of the wafer within a few seconds by diffusion, rather than viscous flow. For the purposes of estimating the rate of transport of species by diffusion, we can make some calculations based on ozone, the most massive radical the system is likely to produce. In focusing on ozone, we do not mean to suggest that we have identified this species as wholly or partly responsible for the activation effect: it has been chosen because it sets a lower bound on the rate at which species can be expected to diffuse within the vacuum chamber. According to Massman [9], the diffusion coefficient of ozone has never been measured in any gas, but his review of molecular diffusivities cites a number of models used to estimate its value in oxygen. He concludes that the best figure to use is $0.145 \text{ cm}^2 \text{ s}^{-1}$ at standard temperature and pressure. The diffusion coefficient is related to temperature and pressure by the expression

$$D_{(T,p)} = D_{(T_0, p_0)} \cdot \left(\frac{p_0}{p}\right) \cdot \left(\frac{T}{T_0}\right)^{1.8}$$
(1)

At a pressure of 600 mTorr and a temperature of 300 K, this gives *D* for ozone in oxygen in the radical generator a value of $172 \text{ cm}^2 \text{ s}^{-1}$. The mean diffusion distance *x* after a time *t* is given by

$$\mathbf{x} \approx (Dt)^{1/2} \tag{2}$$

For a treatment time of 1 min, *x* is approximately 1 m. The distance between the inner diameter of the radical generator and the centre of the wafer is about 80 mm. It can therefore be assumed that after 1 min of operation, ozone that has emerged from the inner mesh of the generator will be evenly dispersed throughout the volume between the wafers. Ozone is the most massive radical likely to be generated in the system, and since diffusion coefficient is approximately inversely proportional to the square root of the mass of the diffusing species [10], all the other radicals will be at least as well dispersed as the ozone. For example, according to Shibata et al. [11], the diffusion coefficient for oxygen atoms in oxygen gas at 300 K and a pressure of 500 mTorr is 336 cm² s⁻¹, which equates to 280 cm² s⁻¹ at 600 mTorr.

Although ozone is generally regarded as an unstable molecule, at room temperature and low concentration, its rate of spontaneous decomposition into oxygen is very low. The dominant process responsible for the decomposition of ozone is chemical interaction with surfaces [12]. By this analysis, ozone can be assumed to be present at the wafer surface during the activation process, as can any lighter species whose lifetime exceeds a few tens of seconds.

Oxygen discharges have been extensively studied, and some of this work has included theoretical prediction and practical measurement of the concentrations of those radicals that are produced in the most significant quantities [13–15]. One species that has attracted much interest is the singlet delta g (SDO) metastable oxygen molecule. This is sufficiently long-lived and energetic to be an aggressive reagent on nearby surfaces. Other radicals include monatomic oxygen, which may also be in excited states. The process we have used employs a brief pump down to a relatively high base pressure, and no dwell time before the oxygen flow is introduced. This ensures that traces of nitrogen from the air, plus water vapour desorbed from surfaces in the chamber, will also contribute to the radicals produced in the discharge.

The electron density within the discharge region (i.e. in the space between the mesh electrodes) was measured using a hairpin probe [16], and found to be approximately 2.5×10^{16} m⁻³. In the space between the wafers, the figure was below the detection limit of the method (5×10^{13} m⁻³).

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