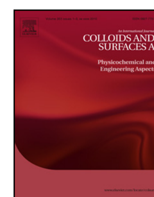
Colloids and Surfaces A: Physicochem. Eng. Aspects 436 (2013) 87–96

Contents lists available at ScienceDirect

Colloids and Surfaces A: Physicochemical and Engineering Aspects

journal homepage: www.elsevier.com/locate/colsurfa



Shear thickening and defect formation of fumed silica CMP slurries

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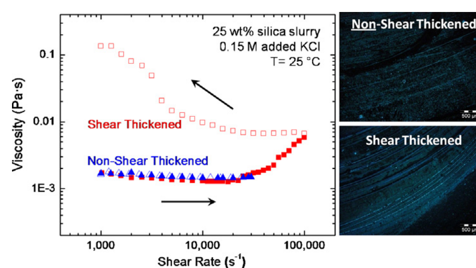
HIGHLIGHTS

- A novel technique that measures rheological behavior while polishing a semiconductor wafer was developed.
- Surface scratch frequency increased when thickening of the slurry was observed.
- The examined scratches are believed to be the result of shear-induced agglomerates.
- These agglomerates are small in number and are found exclusively in the shear thickened sample.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 10 April 2013
Received in revised form 25 May 2013
Accepted 5 June 2013
Available online 15 June 2013

Keywords:
Chemical mechanical polishing
Fumed silica
Rheology
Shear thickening
High shear
CMP defects

ABSTRACT

During the chemical mechanical polishing (CMP) process, it is believed that shear thickening of the slurry, caused by particle agglomeration, has the potential to result in a significant increase in particle-induced surface defects (i.e. scratches, gouges, pits, etc.). In this study, we have developed a methodology for the synchronized measurement of rheological behavior while polishing a semiconductor wafer, the first of its kind (a technique termed rheo-polishing). We investigate the shear thickening of a 25 wt% fumed silica slurry with 0.15 M added KCl and its impact on polishing performance and subsequent surface damage. The thickened slurry displays a ~5-fold increase in viscosity with increasing shear rate. As the shear rate is reduced back to zero, the slurry continues to thicken showing a final viscosity that is ~100× greater than the initial viscosity. Optical microscopy and non-contact profilometry were then utilized to directly link slurry thickening behavior to more severe surface scratching of "polished" TEOS wafers. The thickened slurry generated up to 7× more surface scratches than a non-thickened slurry. Both slurry thickening and surface scratching were associated with a dramatic increase in the population of "large" particles (≥300 nm) which were undetectable in the non-thickened slurry. These "large" and potentially scratch-generating particles are believed to instigate measurable surface damage.

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1. Introduction

Chemical mechanical polishing (CMP) has developed into the primary technique of the semiconductor processing industry for the local and global planarization of integrated circuits [1]. The removal of material is achieved through a synergistic combination of chemical and mechanical attributes of the slurry (hence the name of "chemical mechanical polishing") and the interplay at the polishing pad-wafer interface [2–5]. Recently, great emphasis has been placed on reducing the size of microelectronic devices in order to produce faster and more powerful microprocessors [6]. As a result, the semiconductor industry must constantly improve the performance of the CMP process.

Today's advanced silicon chips contain over one billion transistors in one square centimeter of surface area [5,7]. Interconnecting

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http://dx.doi.org/10.1016/j.colsurfa.2013.06.003

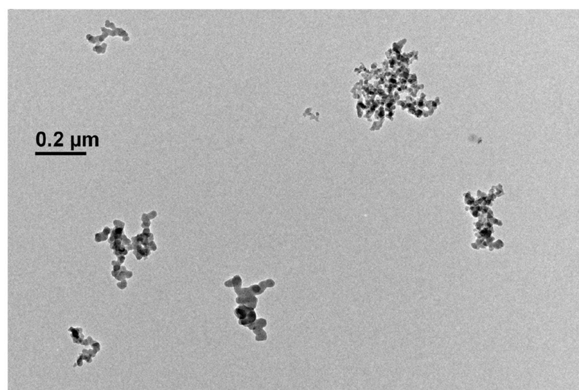

Fig. 1. Transmission electron microscopy image of fumed silica (provided by Cabot Microelectronics Corporation).

such a dense population of electrical elements requires multiple layers of wiring (over eight metal layers can be found in the most recent generation of logic devices) [5]. Each level of wiring involves a minimum of two CMP steps (one metallic CMP step and one dielectric CMP step). As the semiconductor industry moves towards smaller feature sizes and to more layers of wiring (thus, more CMP steps), CMP surface defects such as scratches, gouges, pits, and corrosion need to be reduced. Even mild surface defects on a microelectronic device can greatly diminish device performance [8], while more catastrophic defects, like the severing of a wire line or the fracture of a dielectric layer, can lead to complete device failure [7]. CMP-induced defects have been linked to the existence of small populations of large particles (typically > 500 nm) within the slurry [9,6,10]. These large particles can be impurities from slurry production or a consequence of the aggressive polishing environment.

During the high speed CMP process, the slurry experiences shear rates in excess of 1,000,000 s^{-1} [11]. Recent high shear rheological studies have shown that silica CMP slurries will display shear thickening behavior under process relevant shear rates ($\geq$10,000 s^{-1}) [11–15]. Under this intense shearing, individual slurry particles are driven together to form large agglomerates, which trigger a spike in the slurry's viscosity (termed shear thickening [16,17]). Even though CMP-induced defects have been definitively connected to the presence of large particles within the slurry, there has yet to be a direct correlation between slurry shear thickening and imperfections on a wafer surface.

Here, a method to simultaneously monitor rheological behavior while polishing a semiconductor wafer is presented (deemed *in situ* rheo-polishing). The objective of this manuscript is to correlate changes in slurry viscosity, specifically shear thickening, with the formation of surface scratches during polishing. The ensuing surface defects are then correlated with changes in the slurries' particle size distribution.

2. Experimental

For this study, 25 wt% fumed silica slurries (d = 160±11 nm, determined via dynamic light scattering) with and without the addition of salt (0.15 M KCl) were employed. The fumed silica aggregates (Fig. 1) are made by fusing small (5–50 nm in diameter [18]), primary particles together under a high temperature flame (~1700 °C). The as received fumed silica aggregates are suspended in water and then electrostatically stabilized at pH 11 (well above silica's isoelectric point of pH 2) through the addition of KOH. All slurry material was provided by Cabot Microelectronics Corporation (Aurora, IL) and was a simplified version of the commercial product; consisting of fumed silica, water, and KOH. Concentrated

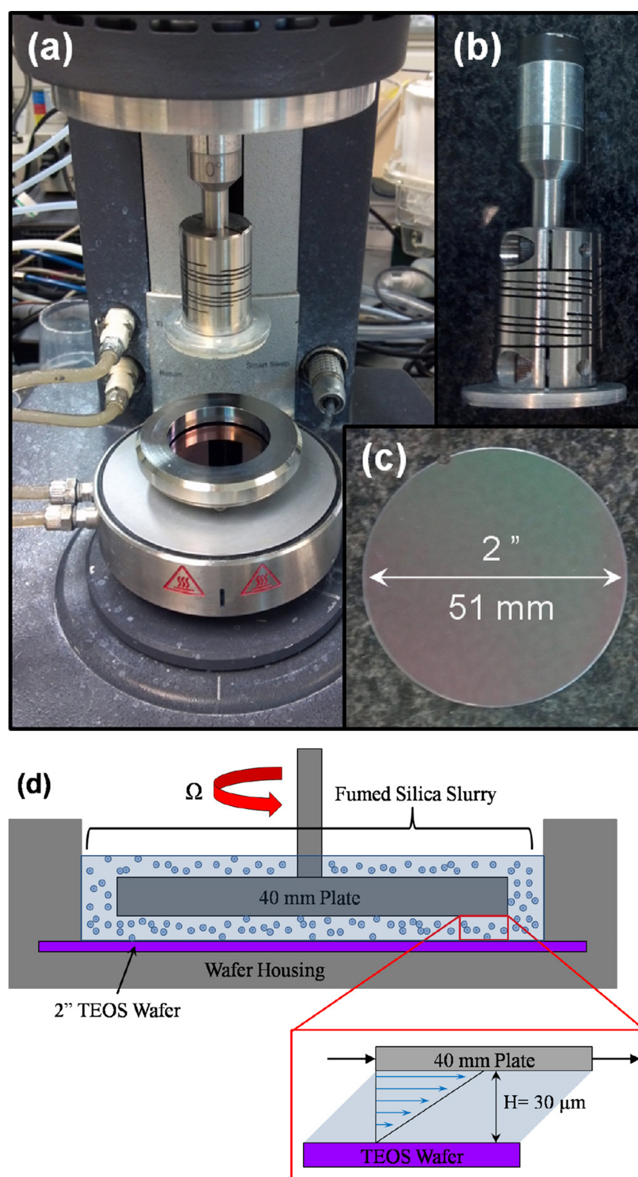

Fig. 2. Rheo-polishing setup with TA Instruments disposable (a) Peltier plate fixture and (b) 40 mm upper plate and (c) 2 in. TEOS wafer, which is inserted into the disposable plate fixture to be "polished." (d) Schematic of the rheo-polishing setup showing the cross-sectional view of the rheometer plate and TEOS wafer covered with fumed silica CMP slurry.

slurries (32 wt%; pH = 11) were diluted to a target solids fraction of 25% (final pH = 10.8) using ultrapure deionized (DI) water and a 0.6 M KCl solution (99% purity; Fisher Scientific, Pittsburgh, PA). The final KCl concentration (c_{KCl}) for the salt adjusted slurry was 0.15 M. After dilution, slurries were stored under ambient conditions for 24 h before commencing rheological tests. Slurry particle concentration (25 wt%) and ionic strength (0.15 M) remained high in order to induce thickening behavior under measurable shear rates (10,000–100,000 s^{-1}; please see our previous publications for further details [14,15]).

In situ rheo-polishing measurements were conducted using TA Instruments' AR-G2 rheometer (New Castle, DE) with a parallel-plate geometry. The top, rotating plate is constructed of disposable aluminum (40 mm in diameter). TA's disposable plate fixture was used as the bottom, stationary plate (Fig. 2a–c). Inserted into the fixture housing (for "polishing"), was a 2 in. (51 mm) diameter silicon dioxide blanket wafer, where the silicon dioxide was produced

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