Application of TEM for Distinguishing the Primary and Secondary Abrasives of Undiluted CMP Slurry

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***Abstract*-Determination of morphology and size distribution of primary and secondary abrasives of slurry is essential for optimization of CMP performance. The undiluted slurry was characterized in liquid phase using silicon based Nano-pipettes in TEM. To validate utility of Nano-pipettes, comparatively quantitative analysis was performed by cryo-TEM technique.**

I. Introduction

One of the major challenges in scaling down of IC manufacturing process is the increase in interconnect delay time, which limits the performance of devices [1, 2]. Delay time primarily depends on resistance (R) of metal lines and interfaces, and their capacitance (C). For minimizing RC delay time, multilevel metallization is utilized. It assists in reduction of length of metal lines, by dividing the length of interconnect over multiple levels as compared to single level [2]. With reduction of features of semiconductor devices down to nanoscale, it is required to maintain a smooth and planar surface before and after each processing step. Compared to physical processes like reactive ion etching and etch-back method, the chemical mechanical planarization (CMP) is a simple and cost-effective process for achieving local as well as global planarization [1].

CMP requires polishing pad, substrate, slurry containing abrasives, and conditioner for polishing pad. During CMP, material removing rate (MRR) is known to depend on chemical selectivity and physical stability of slurry under different conditions of pH environment, temperature, and pressure [3]. MRR and defects, such as scratches, depend on size, size-distribution, and dispersion or aggregation/agglomeration of micro/nano-particles of abrasives found in CMP slurry. CMP performance can be tuned by adjusting the above these essential parameters of primary and secondary abrasives [4].

In current slurry metrological techniques, it is challenging that the information of size, size distribution and shape are obtained comprehensively by ensemble methods. The slurry samples usually need to be diluted before characterizing the size distribution of abrasives. However, dilution can cause slurry instability [5]. On the other hand, transmission electron microscope (TEM) is capable of determining particle counts and their shapes at nanoscale resolution. However, slurry sample preparation using conventional copper grids leads to particle aggregation/agglomeration. And it becomes very difficult to distinguish primary and secondary abrasives particles. As an alternative, the native state of aqueous samples can be approached by sampling with Holey grid/cryo-TEM (Fig. 1). Nevertheless, cryo-TEM "rely on highly sophisticated and expensive machinery. Thin sectioning of samples in a frozen state is a technical and tedious procedure" [6].

For the purpose of observing the primary and secondary abrasives in undiluted CMP slurry, a silicon-based microchip specimen (Nano-pipette) was designed [7], which can be used to sample and seal the undiluted CMP slurry in the native aqueous state for TEM observations. The Nano-pipette is designed successfully to prevent agglomeration/aggregation of abrasives [8]. In this study, we demonstrate the application of Nano-pipette for distinguishing between primary and secondary abrasives of the undiluted silica slurry, and validate the usefulness of Nano-pipette by comparing the results obtained from copper grid/TEM and cryo-TEM analysis.

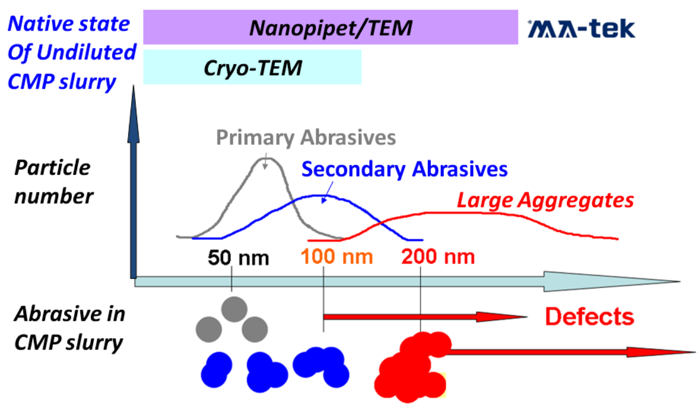

Fig. 1 Distinguishing between the primary and secondary abrasives of undiluted CMP slurry by using Cryo-TEM and nano-pipette in TEM.

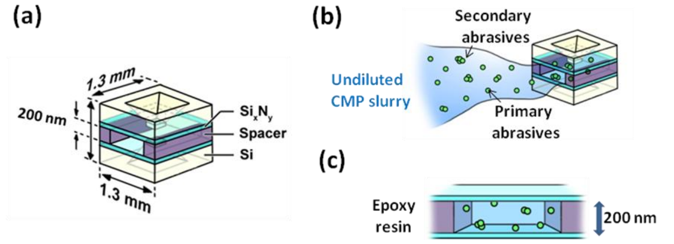

Fig. 2 Geometry of window type Nano-pipette and sampling processes for sample preparation. (a) The schematic diagram of the device, with dimensions and materials as indicated; (b) Sorting of the aqueous of undiluted CMP slurry by the Nano-pipette; (c) Magnified schematic diagram of the chamber showing the sealing of aqueous solution in Nano-pipette by epoxy resin.

II. EXPERIMENT

The undiluted silica CMP slurry samples, provided by Material Analysis Technology Inc., are used to prepare TEM samples with copper grid, Holey-grid, and Nano-pipette, respectively.

Nano-pipette Fabrication

Six-inch width and 625-µm thickness wafers were used to prepare Nano-pipettes. Nano-pipette was created through silicon-based semiconductor manufacture processes, including thin-film growth, lithography and etching process. Two wafers were bonded with a silicon oxide spacer, and the bonded wafer was sliced to individual Nano-pipettes by wet etching processes. An electron transparent window in both sides of wafer is 50 µm × 50 µm with approximate 100-nm thickness silicon nitride film. And a 200-nm height chamber gap was designed by control the thickness the spacer of silicon oxide. The device configuration and its dimensions are shown in Fig. 2a.

Nano-pipette Sampling Processes

One microliter undiluted CMP slurry sample was placed on a glass slide and loaded by suction into the Nano-pipette within 3 minutes, and then was sealed by epoxy resin. This specimen was then mounted onto the TEM holder for subsequent imaging processes. The sample preparation method is shown in Fig. 2b and 2c.

III. RESULTS AND DISCUSSIONS

The abrasives of undiluted silica slurry were observed by TEM. Electron micrographs of the abrasives, which were recorded from randomly selected locations, are shown in Fig. 3. Images of silica slurry samples prepared by drying the slurry onto copper grid show the particle aggregation/agglomeration (Fig. 3a). The abrasives aggregate/agglomerate randomly. As a result, it is difficult to distinguish the primary and secondary ones. On the other hand, the primary and secondary abrasives can be distinguished clearly when TEM micrographs were recorded using Holey-grid and Nano-pipette. Images are shown in Fig. 3b and 3c.

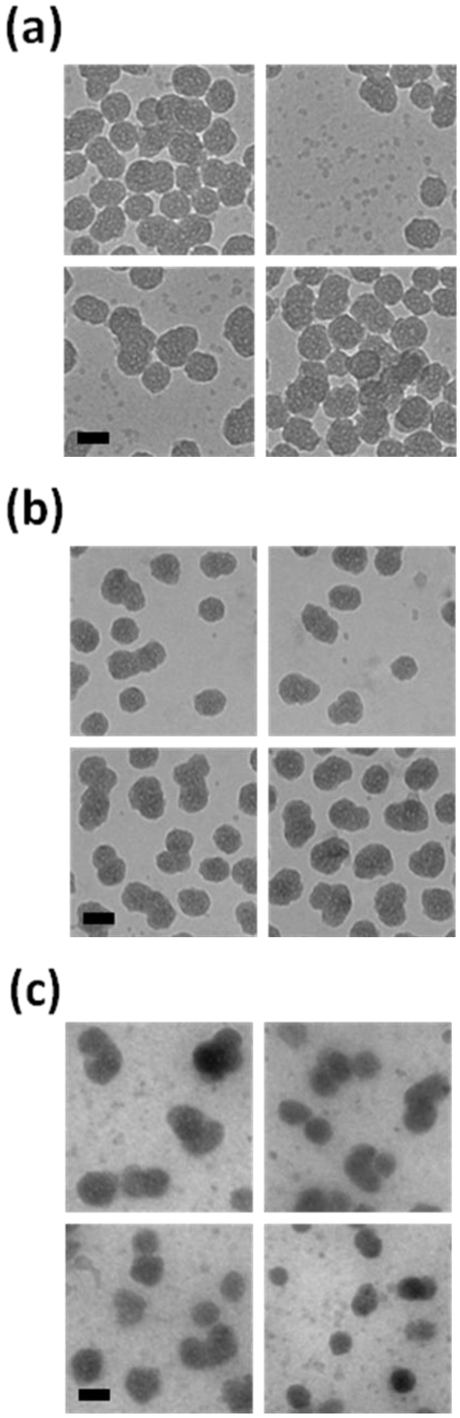

Fig. 3 TEM images of the abrasives of undiluted CMP slurry at four randomly selected locations. Images were recorded for samples (a) dried on copper grid (with SiO_x-film), (b) frozen in Cryo-TEM grid, and (c) in liquid phase in Nano-pipette. Scale bar is 50 nm.

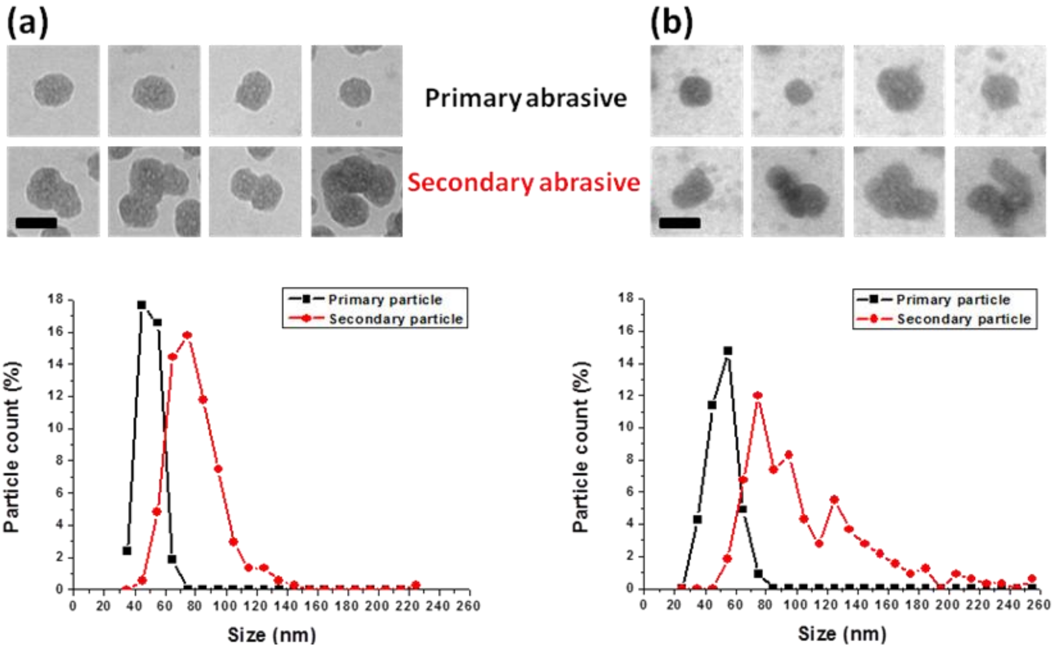

Fig. 4 Abrasive size distribution of primary abrasives (black square) and secondary abrasives (red circle) in undiluted CMP slurry characterized by (a) Cryo-TEM and (b) Nanopipet/TEM. Calculated abrasive number is larger than 300 counts. Scale bar is 50 nm.

The information about size of the primary and secondary abrasives is determined individually. The mean size of abrasive of the silica slurry characterized by Holey-grid and Nano-pipette are 68.4 ± 21.8, and 86.7 ± 41.9 nm, respectively. And the relevant statistical data are presented at Table I. The size distributions of abrasives as observed using Holey gird and Nano-pipette are similar. Fig. 4 shows distribution of percentage of abrasive particle counts with size of abrasive particles.

Table I

STATISTICAL DATA OF PRIMARY AND SECONDARY ABRASIVES IN UNDILUTED CMP SLURRY

Classification	Parameter	Holey grid/ Cryo-TEM	Nanopipet/TEM
Primary abrasives	Number percentage (%)	38.5	36.2
	Mean size (nm)	49.8 ± 6.4	51.4 ± 9.1
Secondary abrasives	Number percentage (%)	61.5	63.8
	Mean size (nm)	80.1 ± 19.9	106.7 ± 39.9
Total Abrasives	Mean size (nm)	68.4 ± 21.8	86.7 ± 41.9

IV. Summary

The undiluted CMP slurry is characterized quantitatively, and size distribution of its primary and secondary abrasives is determined in liquid phase. Determination of the composite size distribution and morphology of abrasives in native phase would be helpful for tuning slurry composition for optimized CMP. Furthermore, the image analyzing software-based analysis of TEM images of abrasives in liquid phase [9] will facilitate correlation of size-shape distribution and agglomeration behavior of abrasives for future CMP studies.

Acknowledgment

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