



Characterization of phosphate electrolytes for use in Cu electrochemical mechanical planarization

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ABSTRACT

Electrochemical mechanical planarization (ECMP) is a potential replacement or complement to conventional chemical mechanical planarization (CMP) techniques. ECMP can operate at very low downforces (<1.0 psi), potentially without slurry particles or oxidizers, and can also be tailored to achieve specific dissolution rates via applied potential. Through the modification of a phosphate-based electrolyte by the use of benzotriazole (BTA), a possible ECMP electrolyte was developed. Utilizing a rotating disk electrode (RDE), a broad range of electrolyte characteristics were screened. The most important parameters investigated were pH, salt concentration, and BTA concentration. The optimal electrolyte composition was found to be around 1 M potassium phosphate with a pH value of 2. Removal rates varied from 750 to 2500 nm min⁻¹ within an operating potential window from 0.5 to 1.0 V vs. Ag/AgCl reference electrode.

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1. Introduction

Chemical mechanical planarization (CMP) has been employed for leveling copper damascene structures during the fabrication of integrated circuits (ICs); however, CMP currently contributes significantly to dishing and surface defects, and could jeopardize the integrity of fragile low-k dielectrics [1]. There is also a strong demand to meet increasing industrial standards, which include achieving higher planarization efficacy, causing less defects, and attaining higher throughputs at lower downforces [2,3]. This is challenging due to the reduction of feature sizes with increasing packing densities and the growing number of interconnect layers present in ICs [2]. Electrochemical mechanical planarization (ECMP) could be used as a replacement or complement to current CMP technologies to potentially address some of these issues; however its potential advantages have not yet been unambiguously demonstrated [4–8].

ECMP utilizes an external circuit to oxidize copper (Cu) and therefore can operate at low downforces [3,4,9] and may not require the use of strong oxidizers, complexing agents, and/or slurry parti-

cles which are known to make CMP waste slurry treatment costly. During CMP, slurry particles have a tendency to form aggregates that can be embedded in the wafer surface and also cause surface scratching. These aggregates can additionally become trapped in the polishing pad, in turn leading to premature degradation of pad life. Because ECMP can possibly eliminate the use of slurry particles, it is estimated that with the reduction in pad replacement, consumable costs could be reduced by up to 30% [3,9].

Recent ECMP studies performed by Goonetilleke et al. [10] and Hong et al. [11] utilize tailored CMP slurries for use during ECMP, however these electrolytes might not fully exploit the electrochemical aspects of ECMP due to the use of peroxides. It has been suggested by Andricacos et al. [12] that phosphoric acid, a common electrochemical polishing (ECP) electrolyte, could potentially be used as an effective electrolyte in ECMP as well. They have also shown promising ECMP results utilizing 1 hydroxyethane-1, 1 diphosphonic acid (HEDP), which is thought to exhibit similar electrochemical properties according to potentiodynamic curves of copper, to that of phosphoric acid [12]. Though studies have numerically shown that planarization using ECP alone is not feasible for small aspect ratio features [13]; utilizing inhibitors in an ECP electrolyte could allow for ECMP to achieve the superior level of planarization needed for future nodes.

Benzotriazole (C₆H₄N₃H, BTA), is widely used as a corrosion inhibitor to prevent dishing and corrosion in Cu CMP [14–18]. Inhibitors are also known to increase planarization [11], therefore

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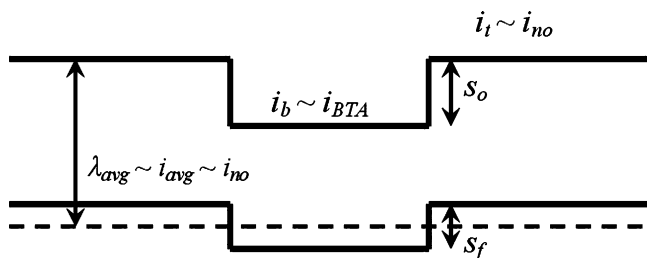


Fig. 1. Schematic diagram showing an idealized topography with an initial feature with step height s_0 . After ECMP has removed λ_{avg} of Cu film, the final step height is assumed to be s_f .

using inhibitors in electrolytes similar to those used during ECP, could produce effective electrolytes for ECMP. Currently there are few or no studies showing the effects of BTA in a phosphate-based electrolyte.

To quantify planarization capability of electrolytes, we use a planarization factor, ε , defined on a feature scale as (Fig. 1):

$$\varepsilon = \frac{s_0 - s_f}{\lambda_{\text{avg}}} \quad (1)$$

where λ_{avg} is the average metal thickness removed (typically estimated by Faraday's law) s_0 is the initial step height before planarization and s_f is the final step height of the feature after planarization [13]. The step heights may be expected to vary with position and feature size on a wafer and therefore, s_0 and s_f , can be thought of as a mean step height. As ε increases, feature scale planarization increases. However, because Fig. 1 illustrates a dramatic simplification of a typical wafer's topography, acceptable planarization values for ε are not definite and would depend on multiple factors such as the step-height reduction that can be achieved by the preceding plating step. We attempt to predict planarization capabilities of ECMP electrolytes using electrochemical experiments on both a custom built bench top ECMP tool and a RDE.

In this study, electrochemical techniques were utilized to investigate a feasible range of applied potentials, the inhibition ability of BTA on a Cu surface in a phosphate system, the predicted planarization efficacy and removal control. Based on the electrochemical results, optimal electrolyte chemistry and electrode potential operating window are predicted. Suggested operating conditions and electrolytes have been tested on blanket wafer fragments using a custom built ECMP tool, and results are shown.

2. Experimental

All rotating disk electrode (RDE) electrochemical experiments were performed at room temperature with a standard three electrode setup using a Cu working electrode (WE) (area = 0.196 cm², purity = 99.9%), Pt wire counter (CE), and a saturated Ag/AgCl reference electrode (REF). The working electrode was hand polished with 600 grit sandpaper and thoroughly rinsed with de-ionized (DI) water before every experiment. Experiments were carried out using a μ AUTOLAB Type III potentiostat and a variable speed four contact rotator (Pine Instrument Co. AFMSRX). Potentiodynamic experiments were scanned from -0.5 to 3.0 V vs. Ag/AgCl reference at a rate of 5 mV s^{-1} .

The electrolyte was prepared using ortho-phosphoric acid (85%, Fisher), potassium phosphate (monobasic KH_2PO_4 /dibasic K_2HPO_4 , Fisher), benzotriazole (Fisher), and de-ionized water. All solutions were prepared as 1 M potassium phosphate where a ratio of $\text{KH}_2\text{PO}_4/\text{K}_2\text{HPO}_4$ was used to reach a pH value as close to the target value as possible; then if necessary phosphoric acid was used to decrease the pH of the solution to the desired value. The follow-

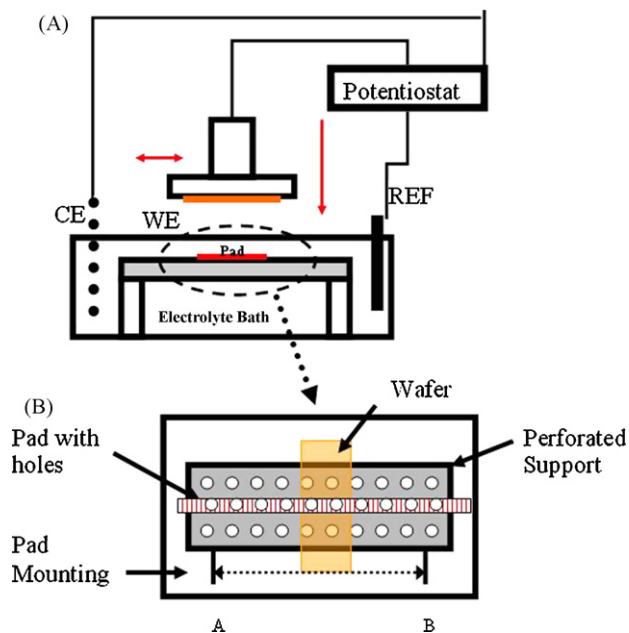


Fig. 2. (A) ECMP tool schematic (side view). The WE is a wafer fragment mounted on a platen that moves linearly in the two-dimensions parallel to the pad. This platen is lowered onto a platform that has a pad mounted on a perforated support. To complete the circuit a Cu CE is placed within the bath, as well as a Ag/AgCl RE. (B) Pad design (top view). Holes are punched out of the pad to match the pattern of holes on the porous support. The wafer sample is positioned perpendicular to the pad such that the pad contacts $\sim 1/4$ of the wafer's area.

ing parameters were examined in the ranges given: pH from 0 to 10, inhibitor concentration from 0 to 0.1 M, and salt concentration from 0.1 to 1.6 M. To help observe changes in surface finish, average surface roughness and step-height analysis results will be obtained with an Alpha-Step IQ KLA Tencor Profilometer.

After characterizing the electrolyte using various electrochemical techniques on a RDE setup, studies were performed utilizing a custom made ECMP tool (Fig. 2A). This tool was designed to accommodate either 2 in. wafers or wafer fragments. Wafer samples were plated using a plating bath consisting of 240 mM cupric sulfate (CuSO_4), 1.8 M sulfuric acid (H_2SO_4), 5 ppm of bis 3-sulfopropyl-disulfide (SPS), 300 ppm of polyethylene glycol (PEG) and 50 ppm of chloride ions (Cl^-) in the form of hydrochloric acid (HCl). Samples were typically plated at a rate of 10 mA cm^{-2} for approximately 20 min. Following plating, sample roughness and thickness were recorded to monitor changes caused by polishing.

The ECMP tool operates in a 2D linear motion, where the wafer sample is mounted on a platform, which is lowered onto a polishing pad that is secured to a perforated support (Fig. 2B). The wafer platform was lowered to either a position in contact with the pad surface (contact mode) or ~ 5 mm above the pad surface (non-contact mode). In order to perform control experiments, these two modes of operation for the ECMP tool are necessary. Holes were punched out of the pad in the same pattern as the support, which accounted for approximately 50% of the pad's area. Perforation in the support and pad allows electrolyte and ionic current to flow freely to the wafer surface. The pad is secured on the support using adhesive. The whole support (including pad) is then lowered and stabilized in the electrolyte bath. To make electrical contact on the front of the wafer, two strips of Cu foil are used. One side of each of these strips is folded over opposite edges of the wafer sample. The other sides of the foil strips are folded together and fastened with a metal clip to make contact to the power source. The Cu foil strips and the sample are then secured with Kapton tape to protect the

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