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Research Article

Two material removal modes in chemical mechanical polishing: Mechanical plowing vs. chemical bonding

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Abstract: With the rapid development of semiconductors, the number of materials needed to be polished sharply increases. The material properties vary significantly, posing challenges to chemical mechanical polishing (CMP). Accordingly, the study aimed to classify the material removal mechanism. Based on the CMP and atomic force microscopy results, the six representative metals can be preliminarily classified into two groups, presumably due to different material removal modes. From the tribology perspective, the first group of Cu, Co, and Ni may mainly rely on the mechanical plowing effect. After adding H_2O_2, corrosion can be first enhanced and then suppressed, affecting the surface mechanical strength. Consequently, the material removal rate (MRR) and the surface roughness increase and decrease. By comparison, the second group of Ta, Ru, and Ti may primarily depend on the chemical bonding effect. Adding H_2O_2 can promote oxidation, increasing interfacial chemical bonds. Therefore, the MRR increases, and the surface roughness decreases and levels off. In addition, CMP can be regulated by tuning the synergistic effect of oxidation, complexation, and dissolution for mechanical plowing, while tuning the synergistic effect of oxidation and ionic strength for chemical bonding. The findings provide mechanistic insight into the material removal mechanism in CMP.

Keywords: chemical mechanical polishing; corrosion wear; material removal mode; mechanical plowing; chemical bonding

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1 Introduction

Chemical mechanical polishing (CMP) has been widely used as a critical manufacturing technology in the semiconductor industry to achieve local and global planarization of wafers [1–5]. With the rapid development of various semiconductor devices, the number of materials needed to be polished significantly increases. Take logic chips for example, with the feature size shrinking to 7 nm and smaller, the number of materials required to be polished reaches more than 10. Moreover, the number of materials needed to be polished simultaneously at the interconnect barrier planarization stage reaches 4–5, such as Cu/Ru/Ta/TaN/low-k [6–8]. Meanwhile, the defects need to be controlled to satisfy the stringent requirement of photolithography. For instance, the dishing should be below 4 nm [9]. However, different materials vary considerably in chemical and mechanical properties, making it difficult to swiftly adapt the CMP performance, especially for a surface containing multiple materials.

To address this problem, researchers investigated the fundamental CMP mechanism and tried to classify the material removal modes in CMP. Take Cu and Ta for examples. For Cu, a representative material in microchips, researchers concluded that Cu could be removed via the mechanical plowing effect [10–16], which is quite similar to the mechanical disruption of the surface passivating film of WO_3 in W CMP [17]. Kawaguchi et al. [14] applied their original CMP simulator based on the tight-binding quantum chemical

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molecular dynamics method to investigate the atomistic Cu CMP mechanism. It was revealed that the O atom from H_2O_2 intruded into the Cu bulk and dissociated the Cu–Cu bonds, leading to the rise of a Cu atom, which was then mechanically sheared by the SiO_2 abrasive grain. Guo et al. [15] employed molecular dynamics simulations with the ReaxFF reactive force field to clarify the atomistic Cu CMP mechanism. It was revealed that Cu atoms were mainly removed through the fracturing of Cu–Cu bonds and Cu–O bonds by extrusion shear action of SiO_2 abrasive particles. In addition, Guo et al. [16] reported that the synergistic activity of H_2O_2 and glycine could weaken the bond energy between Cu atoms, making it easier to remove Cu atoms with SiO_2 abrasive particles mechanically. Li et al. [13] concluded that the synergistic effect of chemical corrosion and abrasive wear led to Cu removal during CMP. The mechanical action was primarily due to the abrasive particles trapped in pad asperities. In comparison, a few researchers reported that the chemical bonding effect could lead to Cu removal. Wen et al. [18] showed that as one of the three removal pathways in Cu CMP, interfacial Cu–O–Si bridge bonds could be formed between Cu–OH and Si–OH, and then Cu–Cu bonds could be dissociated by mechanical sliding.

For Ta, another representative material in microchips, researchers inferred that Ta could be removed with the chemical bonding effect [19–22], which is quite similar to the chemical tooth in glass CMP and the tribochemical removal pathway of silicon [23, 24]. Vijayakumar et al. [19] inferred that chemical bonds might be formed between Ta and SiO_2. The mechanical tearing of Ta–O–Si bonds could result in the removal of Ta_2O_5 during Ta CMP. Li et al. [21] reported that the Ta material removal rate (MRR) increased along with the specific surface area of SiO_2 particles. Additionally, Li et al. [22] found that the Ta MRR was directly correlated with the effective hydroxyl content of SiO_2 particles. The results suggested that chemical bonding between Ta and SiO_2 might be formed to promote Ta removal. By contrast, a few researchers showed that the mechanical plowing effect could affect Ta removal. Sulyma et al. [25] reported that Ta could combine with anions to form a weak surface film, easily mechanically removed by abrasive particles.

There have been many attempts to unravel the removal mechanisms of different materials in CMP based on either macroscopic experimental results or simulations. However, each research primarily focuses on a single material. In some cases, the material removal mechanisms concluded by different researchers are discrepant. As aforementioned, the surface needing to be planarized in microchips usually comprises multiple materials. Therefore, systematic investigation of multiple materials is needed to guide the CMP process effectively. In addition, CMP is a nanoscale material removal process, but microscopic evidence is lacking. In sum, the material removal mechanisms in CMP remain elusive and merit exploration. Accordingly, six representative metallic materials used in semiconductors were examined in this paper. Based on the CMP and atomic force microscopy (AFM) results, two material removal modes were put forward from the tribology perspective. The finding obtained from the study provides a practical guide to optimizing the CMP performance of different materials.

2 Materials and methods

The CMP experiments were conducted on a commercial polisher (Universal-150, Hwatsing Co., Ltd.). The Cu film, Co disk, Ni film, Ta film, Ru film, and Ti disk of 50.8 mm diameter were polished. The slurries consisted of ultrapure water, colloidal SiO_2 (YZ8040, purchased from Shanghai YZ-Lapping Material Co., Ltd.), and chemical reagents. The pH value was adjusted with KOH and HNO_3. For Cu, Co, and Ni, the slurries consisted of water, 1 wt% colloidal silica, 0.1 M glycine, H_2O_2, and at pH 4. For Ta, Ru, and Ti, the slurries contained water, 1 wt% colloidal silica, 25 mM K^+, H_2O_2, and at pH 10. Table 1 lists the CMP process conditions. The weights before and after CMP were measured with a microbalance (MSA225S, Sartorius, 10 μg readability). The MRR was calculated using the weight loss method [26]. The surface morphology and roughness were measured with an optical 3D surface profiler (SuperView W1, Chotest). The scan area was 97.9 μm × 97.9 μm.

AFM (E-Sweep, Hitachi) was used to assess the surface mechanical strength.

Table 1 CMP conditions.

Condition	Value
Calibrated down pressure (psi)	4
Carrier speed (r/min)	100
Platen speed (r/min)	100
Slurry flow rate (mL/min)	100
Time of a single polishing	60 s for Cu, Co, Ni, and Ti 30 s for Ta and Ru due to the limited thickness
Polishing pad	Politex (Dupont)

A diamond probe (NC-LC, Adama) rubbed the sample surface in liquid. For Cu, the solutions consisted of 0.1 mM glycine, H_2O_2, and at pH 4. The experimental conditions were: 1 μN applied load, 2 μm/s relative sliding velocity, 2 μm relative sliding length, and 1 reciprocating sliding cycle. For Ta, the solutions consisted of 25 mM K^+, H_2O_2, and at pH 10. The experimental conditions were: 10 μN applied load, 2 μm/s relative sliding velocity, 1 μm relative sliding length, and 10 reciprocating sliding cycles. Afterward, a silicon nitride probe (MSCT, Bruker) was used to scan the surface topography.

3 Results and discussion

3.1 Effect of corrosion on the CMP results of different metals

The oxidizer H_2O_2 can corrode the metal surface. Figure 1 shows the effect of H_2O_2 on the CMP results of different metals. Based on the changing trends of the MRR and the surface roughness S_a along with the H_2O_2 concentration, the six metals can be roughly classified into two groups. For the first group of Cu, Co, and Ni, the MRR and the surface roughness S_a first increase and then decrease. In comparison, for the second group of Ta, Ru, and Ti, the MRR gradually increases, and the surface roughness S_a first decreases and then levels off. The typical surface morphologies

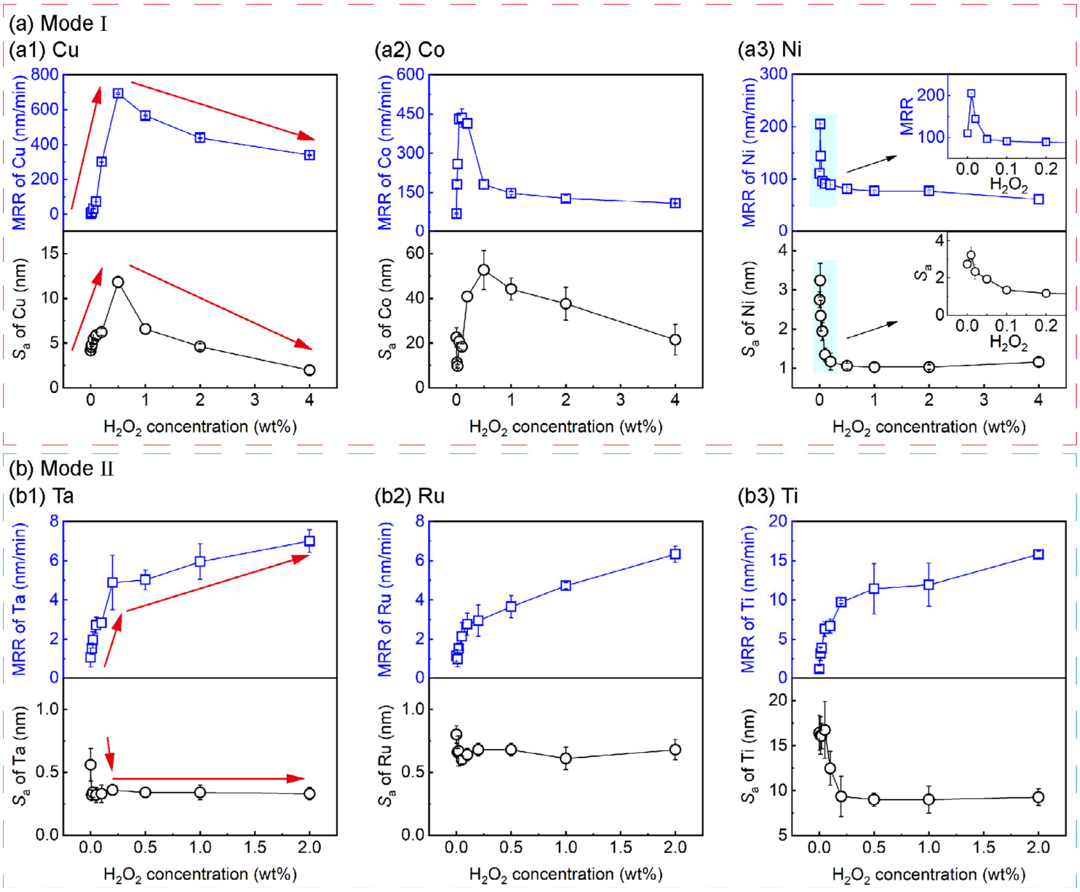

Fig. 1 Effect of the H_2O_2 concentration on the CMP results of different metals. (a1) Cu, (a2) Co, and (a3) Ni. (b1) Ta, (b2) Ru, and (b3) Ti.

after polishing are displayed in Figs. S1–S6 in the Electronic Supplementary Material. The different CMP results of the two groups might be attributed to different material removal modes. The underlying mechanism will be discussed later.

3.2 Two material removal modes in metal CMP

Considering that Cu and Ta are the most widely used interconnect material and barrier material in microchips, respectively [27, 28], they were selected as the research representatives of each group and mainly studied in Sections 3.2 and 3.3. AFM wear experiments were conducted in liquid with a diamond probe to examine the surface mechanical strength [29]. Figure 2 shows the AFM results. Given that the diamond probe is chemically inert, it is likely that only the mechanical effect needs to be considered when rubbing. According to the Archard model [30], the wear amount negatively correlates with mechanical strength. The lower the mechanical strength is, the higher the wear depth is. With the H_2O_2 concentration increasing, the wear depth of Cu first increases and then decreases, indicating that the mechanical strength of the Cu surface first decreases and then increases. In comparison, the wear depth of Ta first increases and then keeps almost unchanged, suggesting that the mechanical strength of the Ta surface maintains the same level with sufficient H_2O_2.

Essentially, metal CMP is a nanoscale corrosion wear process. In this study, H_2O_2 can corrode the metal surface to form oxides, which can then be worn by SiO_2 particles. There are two typical wear modes in tribology [31]: abrasive wear and adhesive wear. For abrasive wear, the material is removed mainly through the mechanical plowing effect, and the MRR is associated with the mechanical strength of the surface film [32]. For adhesive wear, the material is removed primarily with the adhesion effect, such as chemical bonding, and the MRR is related to the number of adhesive junctions [33].

Based on the above results and literature, two material removal modes in metal CMP are put forward, namely mechanical plowing and chemical bonding, corresponding to the two groups shown in Fig. 1. As shown in Fig. 3(a), for Cu, a representative of the first group, the changing trend of the AFM wear results along with H_2O_2 (Fig. 2(a)) is consistent with that of the CMP MRR results (Fig. 1(a1)), implying that the mechanical strength can affect the material removal. The mechanical plowing effect may primarily cause material removal. Specifically, adding a low concentration of H_2O_2 can enhance corrosion by the synergistic effect of oxidation, complexation, and dissolution in the presence of a complexing agent of glycine. The surface becomes loose and is of low mechanical strength [34, 35]. Consequently, with the

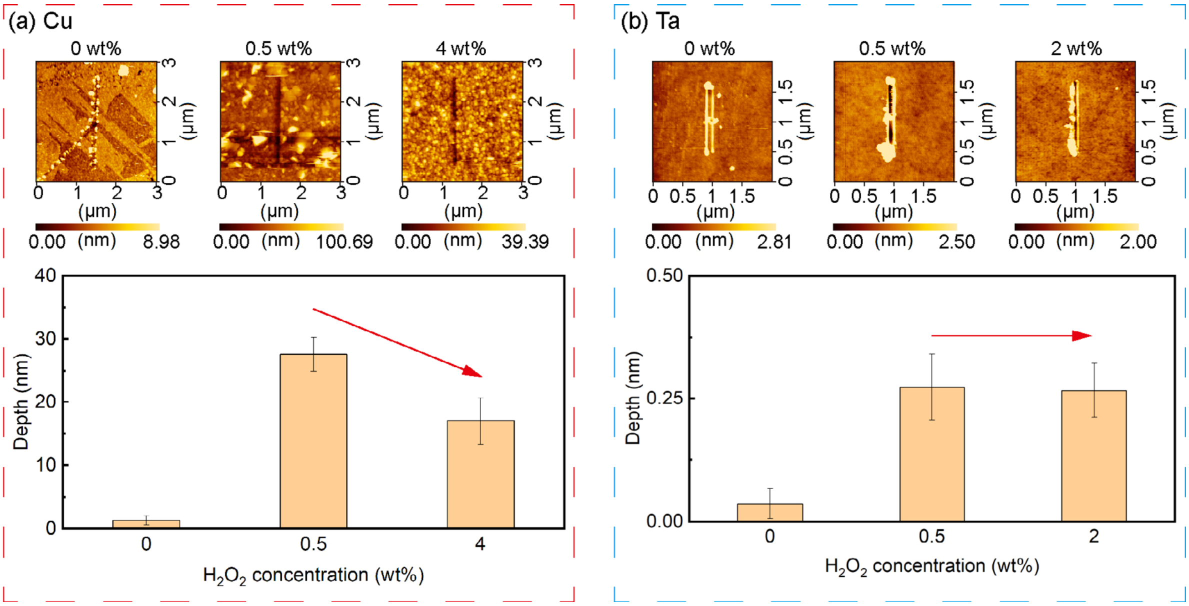

Fig. 2 Effect of the H_2O_2 concentration on the AFM results. (a) Cu. (b) Ta.

mechanical plowing effect of SiO_2 particles, the MRR increases while the surface quality worsens. As the H_2O_2 concentration increases, excessive oxidation can gradually suppress corrosion. The surface grows compact with relatively high mechanical strength [34, 35]. Therefore, the MRR decreases while the surface quality improves.

As shown in Fig. 3(b), for Ta, a representative of the second group, the changing trend of the AFM wear results along with H_2O_2 (Fig. 2(b)) is different from that of the CMP MRR results (Fig. 1(b1)), suggesting that the material removal can be independent of the mechanical strength. The chemical bonding effect may give rise to material removal. More specifically, after adding H_2O_2, oxides are formed on the surface. Generally, the oxide film is of relatively high mechanical strength and is difficult to be removed merely by the mechanical plowing effect [36]. Under mechanical stress, tribochemical reactions between the oxides and SiO_2 particles can occur. As a result, chemical bonds of Ta–O–Si can be formed, and the binding energy of the neighboring Ta–Ta bonds can be reduced [19]. As SiO_2 particles slide, the weakened Ta–Ta bonds can be broken, and the material removal can be realized without introducing lattice dislocations. With the H_2O_2 concentration increasing, oxidation gradually becomes uniform over the whole surface. Meanwhile, the chemical bonding effect can only impact the topmost atomic layer [24], and thereby the surface roughness first decreases and then levels off. In addition, more interfacial chemical bonds are formed, and thus the MRR increases.

In sum, two material removal modes are preliminarily concluded in metal CMP. From the microscopic point of view, the generation reason of the two material removal modes may be depended on the difficulty degree of forming new chemical bonds between the surface oxides and SiO_2 particles and breaking the metallic bonds weakened by corrosion. If breaking the weakened metallic bonds is more accessible, the material removal mode can be mechanical plowing, like Cu. Otherwise, the material removal mode can be chemical bonding, like Ta. More investigation is ongoing to reveal the detailed material removal mechanisms further.

3.3 Controlling CMP performance based on the two material removal modes

Different CMP controlling strategies are needed for the two material removal modes. For the mode relying on the mechanical plowing effect, the CMP performance can be controlled by adjusting the surface mechanical strength via tuning the synergistic effect of oxidation, complexation, and dissolution [34]. For the mode depending on the chemical bonding effect, the CMP performance can be modulated by adjusting the bond number via tuning the synergistic effect of oxidation and ionic strength [37].

Cu and Ta were used for demonstration. Cu and Ta will be polished simultaneously during the interconnect

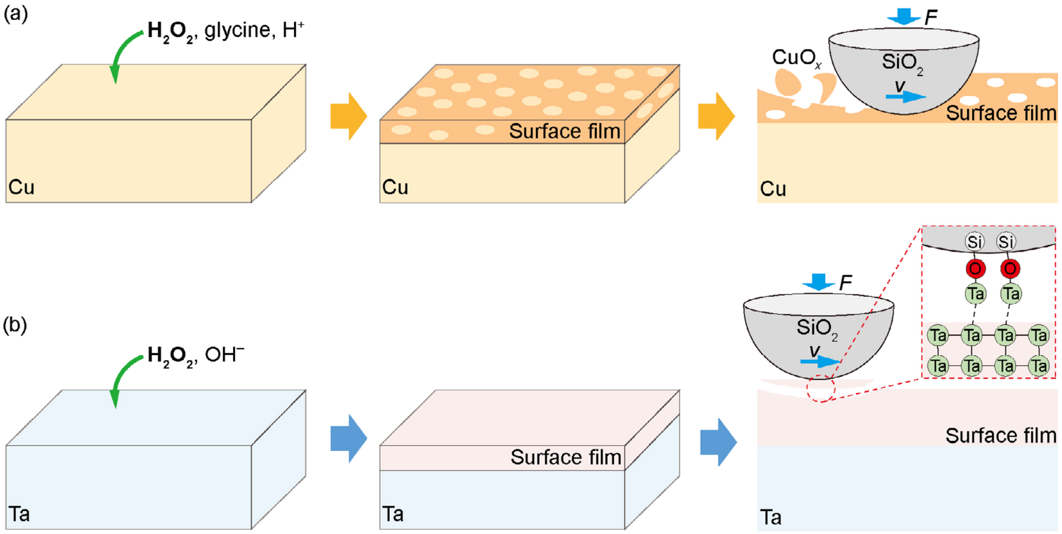

Fig. 3 Two material removal modes. (a) Mechanical plowing. (b) Chemical bonding.

barrier planarization stage, as shown in the inset in Fig. 4(a). Ideally, an MRR selectivity of about 1 between Cu and Ta and excellent surface quality are required to achieve planarization. As shown in Fig. 4(a), with the increase in the K^+ concentration, the Cu MRR slightly changes, while the Ta MRR significantly increases. The increment in the Ta MRR can be explained as follows. Adding K^+ can neutralize the surface potentials of Ta oxides and SiO_2 particles. According to the DLVO theory [38], tribochemical reactions between Ta oxides and SiO_2 can be enhanced, increasing the number of chemical bonds. Then, after adding the complexing agent L-arginine and the corrosion inhibitor benzotriazole (BTA), the Cu MRR varies greatly, while the Ta MRR slightly changes. The variation of the Cu MRR can be attributed to the change in corrosion and the resultant mechanical strength. After optimization, an MRR selectivity of about 1 and ultra-smooth surfaces of Cu and Ta (Fig. 4(b)) are achieved. The results in Fig. 4 confirm the two material removal modes to some extent in turn.

This study preliminarily classifies two material removal modes in metal CMP from the tribology perspective: mechanical plowing and chemical bonding. In addition, feasible measures for controlling the CMP performance are presented and confirmed for the two material removal modes. Furthermore, the material removal modes can also be applied to other materials, such as sapphire, GaN, SiC, diamond, and metal alloys. The findings obtained from this study may open new avenues to swiftly optimize the CMP performance, especially for a surface containing multiple materials.

4 Conclusions

This work polished and characterized six representative metals used in semiconductors. Two material removal modes were proposed based on the experimental results and the tribology principles. The conclusions can be drawn as follows:

1) The six metals can be classified into two groups based on the CMP performance. As the H_2O_2 concentration increases, for the first group of Cu, Co, and Ni, the MRR and the surface roughness increase and then decrease. In contrast, for the second group of Ta, Ru, and Ti, the MRR gradually increases, and the surface roughness first decreases and then levels off.

2) Two material removal modes were put forward from the tribology perspective: mechanical plowing and chemical bonding. For Cu, it may be primarily worn by the mechanical plowing effect. Adding H_2O_2 can enhance and then suppress corrosion, influencing the surface mechanical strength. Therefore, the MRR and the surface roughness increase and decrease. By

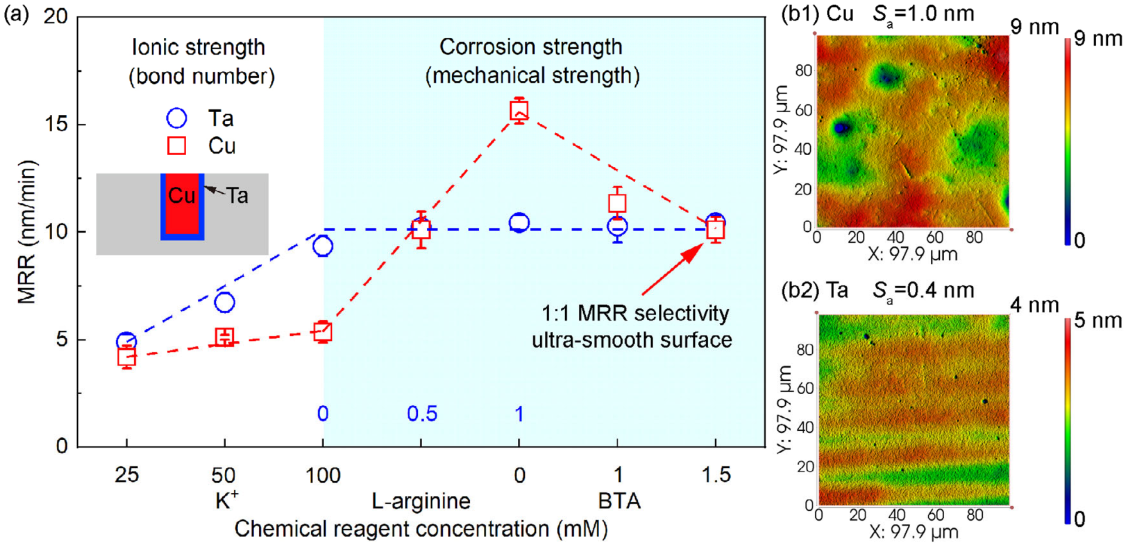

Fig. 4 Controlling Cu and Ta CMP performance based on the two material removal modes. (a) The MRR results. The basic slurry consisted of water, 2 wt% colloidal silica, 1 wt% H_2O_2, 25 mM K^+, and at pH 10. (b) The surface morphologies of (b1) Cu and (b2) Ta after polishing with the optimized slurry containing additional 75 mM K^+, 1 mM L-arginine, and 1.5 mM BTA based on the basic slurry.

contrast, Ta may be primarily removed by the chemical bonding effect. The addition of H_2O_2 can result in the enhancement of oxidation, increasing interfacial chemical bonds. Hence, the MRR increases, and the surface roughness decreases and reaches a stable value.

3) Different CMP controlling strategies were proposed and confirmed with Cu and Ta. The CMP performance can be controlled by adjusting the synergistic effect of oxidation, complexation, and dissolution for mechanical plowing while tuning the synergistic effect of oxidation and ionic strength for chemical bonding.

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Declaration of competing interest

The authors have no competing interests to declare that are relevant to the content of this article. The author Linmao QIAN is the Editorial Board Member of this journal.

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