



Chemical mechanical polishing of steel substrate using colloidal silica-based slurries



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ABSTRACT

AISI 52100 steel has been widely used in the mechanical industry due to its excellent mechanical properties and high availability. In some cases, an ultra-smooth surface of AISI 52100 steel is needed and is even indispensable for the satisfactory performance of devices. In this paper, chemical mechanical polishing technique was employed to prepare the ultra-smooth surface of AISI 52100 steel. Colloidal silica was used as the abrasive. The effects of pH, complexing agent such as glycine, H_2O_2 and benzotriazole (BTA) on the polishing performance were studied. It is revealed that, with the increase of pH, the static etching rate (SER) and the material removal rate (MRR) are both gradually reduced, and the post-CMP surface roughness R_a decreases. This is attributed to the fact that compact and passive iron oxides, especially Fe(III) oxides, gradually form on the top surface. At pH 4.00, in the presence of glycine, and with the increase of the H_2O_2 concentration, the SER is further suppressed, and the surface roughness R_a gradually decreases; the MRR initially dramatically increases due to the fact that, with the addition of small amount of H_2O_2 , the porous iron oxide layer with relatively low mechanical strength can be rapidly formed on the surface. Moreover, glycine intensifies the chemical dissolution by chelating iron ions, especially Fe(II) ions, and thereby the mechanical strength of the oxide layer further weakens. Then, after reaching the peak value, the MRR gradually decreases when the H_2O_2 concentration further increases since the compactness of the oxide layer gradually increases. With the increase of the BTA concentration, the MRR is gradually suppressed and the surface roughness R_a decreases due to the formation of Fe-BTA passivating film on the top surface. Finally, a two-step polishing process was developed. The polishing results show that, within 20 min, a rough surface of AISI 52100 steel with the R_a value of 188 nm can be polished into an ultra-smooth surface with the R_a value of 1.8 nm.

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1. Introduction

Various types of steels, such as AISI 52100 steel, have been widely used in the mechanical industry due to their excellent mechanical properties and high availability [1,2]. Generally, the surface quality of steel has significant influence on the functional properties of the engineering components, such as wear resistance and energy loss caused by friction [2–5]. It was reported that, during the typical pin-on-disk tests, with the AISI 52100 steel disk as one of the friction pair, the coefficient of friction became lower when the roughness of the disk was smaller under the lubricated condition [6], which indicates that the surface roughness is critical to the lubrication state of the precise components. During the last few decades, the requirements for precise

components were dramatically increasing. In some other cases, a nanoscale ultra-smooth steel surface is needed and is even indispensable for the satisfactory performance of devices [7,8]. However, the surface quality obtained by mechanical polishing cannot meet the requirement of nanoscale surface roughness. Therefore, all kinds of finishing techniques have been developed to obtain high-precision surface quality of steel such as AISI 52100 steel [4,7–13]. Mulik et al. [4] used a magnetic abrasive finishing (MAF) technique working together with SiC abrasives to process a planar workpiece of hardened AISI 52100 steel, and a surface roughness value of 51 nm was obtained within 120 s. On the basis of the MAF technique, Mulik et al. [10] developed an ultrasonic assisted magnetic abrasive finishing (UAMAF) technique, which integrated ultrasonic vibration and MAF technique, and the tests on a planar hardened AISI 52100 steel surface showed that the surface roughness value of 22 nm could be obtained within 80 s. However, since the material removal of both MAF and UAMAF techniques mainly relies on the direct mechanical abrasion of the protruding

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regions of the steel surface by the abrasives, it might be difficult to further reduce the surface roughness. In order to further improve the surface quality, chemical action should be introduced. Chi et al. [8] adopted the float polishing technique working together with the polishing fluid to process a planar AISI 52100 steel surface, and the surface roughness value of 5 nm was obtained. Specifically, the polishing fluid was made of diamond abrasives at pH 8.3. As an ultra-precision processing technique, chemical mechanical polishing (CMP) technique has been widely used in semiconductor fabrication. CMP combines the synergistic effect of chemical etching and mechanical abrasion, and can achieve both local and global planarization of the substrate surface [14–17]. Recently, the application of CMP technique has been extended to other areas, such as high value-added and precise instruments. Kao et al. [7] prepared colloidal silica nanoparticles of different sizes, and then studied their efficacy in AISI 52100 steel CMP. The polishing results showed that a super-smooth surface with the surface roughness of 8.4 nm was obtained. Peng [11] carried out CMP experiments on an AISI 52100 steel substrate using alumina-based slurries. The results showed that the polishing performance, including material removal amount and surface roughness R_a , strongly depended on the oxidizer, oxidizer contents and abrasive contents. And by optimizing the slurry formulation, a super-smooth surface with the surface roughness of 7.61 nm was realized. Some other researchers have done excellent work on stainless steel CMP, which can provide a reference for the study of AISI 52100 steel CMP. Yun et al. [12] used CMP process to prepare ultra-smooth 304 and 430 stainless steel substrates for flexible thin-film transistors. The results showed that, by using alumina-based slurries, the surface roughness of about 2.5 nm could be realized. Hu et al. [13] used colloidal silica as the abrasive and investigated the effects of pH and oxidizer on the polishing performance of 304 stainless steel, and it was reported that the combination of oxidizer and strong acidity was the prerequisite for a high material removal rate (MRR), but the microscopic defects of 1–2 μm in size could not be avoided on the polished surface.

However, the fundamental issues of steel CMP, especially the interactions between slurries and substrate surface, have not been well investigated. In this paper, CMP technique was employed to polish AISI 52100 steel. Colloidal silica was used as the abrasive. The effects of pH, complexing agent such as glycine, H_2O_2 and benzotriazole (BTA) on the polishing performance of AISI 52100 steel were first investigated, and then the chemical reactions occurring on the steel surface during the polishing process were analyzed, and the corresponding polishing mechanism was proposed. Finally, a two-step polishing process was developed.

2. Experimental details

Polishing slurries were prepared with de-ionized (DI) water, NexSil™ 85K colloidal silica (purchased from Nyaacol Nano Technologies, Inc., Ashland, MA, USA. Primary particle size is about 50 nm) and reagent grade chemicals such as H_2O_2 , glycine and BTA. pH was adjusted using diluted HNO_3 and KOH. Specifically, in order to ensure that the amount of the added H_2O_2 was accurate enough, 1.0 wt% H_2O_2 stock solution was prepared.

Polishing experiments were carried out on a CETR CP-4 benchtop polisher using the AISI 52100 steel disks (2" diameter and 1 mm thickness) with the following polishing conditions: the down pressure 6.0 psi, the table speed/carrier speed 150 rpm/150 rpm, the offset distance between the table and the carrier 63 mm, the slurry flow rate 100 mL/min and the polishing time 1 min. An IC1010/Suba-IV composite pad with K-type groove (purchased from Dow Electronic Materials) was used as the polishing pad. Before the experiment, a pad break-in conditioning was carried

out with a diamond conditioning disk for 10 min. In between each polishing, a pad ex-situ conditioning was carried out for 10 sweeps. The MRR of AISI 52100 steel was determined by measuring the weight loss with a Sartorius ME36S microbalance (0.001 mg resolution). Each experiment was repeated four times. After polishing, the surface morphology was measured with a Veeco MicroXAM optical interferometer surface mapping microscope, and then the surface roughness was evaluated with the scanning probe image processor software. The corresponding measured surface area was 173 $\mu\text{m} \times 128 \mu\text{m}$. In order to investigate the slurries' chemical aggressiveness, static etching tests were carried out at room temperature by immersing the AISI 52100 steel disks (22 mm diameter and 1 mm thickness) into the tested slurries (without silica particles). The test time was set to 3 min, and the static etching rate (SER) was calculated by measuring the weight loss.

Electrochemical experiments were carried out to describe the electrochemical properties of the AISI 52100 steel surface in various slurries (without silica particles) using a Potentiostat/Galvanostat Model 273A work station (Princeton Applied Research) with a 200 mL three-electrode cell. A platinum electrode was used as the counter electrode, an Ag/AgCl electrode with 3.5 M KCl reference solution was used as the reference electrode, and a cylindrical AISI 52100 steel electrode with 5 mm diameter encased in epoxy resin was used as the working electrode. Prior to each measurement, the working electrode was first mechanically polished with an abrasive paper made of brown fused alumina (P2000), and then was carefully degreased with ethyl alcohol and cleaned with DI water in sequence. In order to prepare the fresh AISI 52100 steel surface, the working electrode was held at -1.2 V (vs. $E_{\text{Ag/AgCl}}$) for 60 s for pre-treatment. Then the potentiodynamic polarization measurement was carried out when the open-circuit potential reached a stable value, where the step height was set to 2 mV and the scan rate was set to 5 mV/s.

The chemical compositions of the AISI 52100 steel surface were characterized by X-ray photoelectron spectroscopy (XPS). For the sample preparation, an AISI 52100 steel coupon of 7.5 mm \times 7.5 mm in size was first pre-polished to obtain a fresh surface, then it was immersed into the designated solution for 10 min, next it was rinsed with DI water and dried with high pressure air jet. Finally, it was inserted into the high vacuum chamber subsequently. The XPS measurements were carried out on an ESCALAB 250 XI (Thermo Scientific Instrument, USA) which utilizes monochromatic aluminum k-alpha X-rays. All spectra were obtained at a 90° photoelectron takeoff angle from the surface. For each sample, the high-resolution spectra, corresponding to Fe, O and N respectively, were obtained using 30.0 eV pass energy. Afterwards, the data analysis was performed with the CasaXPS software.

3. Results and discussion

3.1. Effect of pH on the polishing performance of AISI 52100 steel

AISI 52100 steel has been widely used in the mechanical industry, such as being used as the basic material for bearings [1]. In Chinese standard, AISI 52100 steel corresponds to GCr15 steel. The chemical composition of AISI 52100 steel is listed in Table 1. As can be seen, the main chemical element of AISI 52100 steel is iron, which accounts for more than 95.90 wt% [7,11], which means that the interactions between iron on the AISI 52100 steel surface and slurries have crucial impact on the polishing performance.

pH is critical to metal CMP by influencing the status of the metal surface, i.e. immunity, corrosion or passivation [18,19]. The effect of pH on the polishing performance of AISI 52100 steel is shown in Fig. 1. The slurries were composed of 4.0 wt% colloidal silica and with different pH. It can be seen that, with an increase in pH, the

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