

CHAPTER IV

EXPERIMENTS AND ANALYTICAL TECHNIQUES

This chapter describes the experimental condition and the analytical techniques used in this investigation. A mentioned previously, the processing of post-etched residue removal will be studied based on the current tool and process applicable. The cleaning efficiency will be studied by noting the contribution of such process parameters as scrubbing time and solution concentration. The characterization can be broken into in three parts: 1) cleanliness evaluation of the part after cleaning process by using SEM and AFM; 2) cleanliness evaluation by using ion chromatography technique to identify the fluoride ion left on the part after clean; and 3) electrical performance measurement including the defect reduction from the cleaned part.

4.1 Process Description

In this study, we focused on post-etched redeposit removal process. The standard recipe consists of the following steps: 1) scrubbing; 2) rinsing; and 3) dry. In the first step of scrubbing, parts were immersed into the NaOH solution. The redeposit is speculated to have reacted with cleaning solution, and the accompanied scrubbing force helped to lift the residue off from the part. One key element of this scrubbing action is the download force in which it was fixed at 150 grams during this study. The NaOH concentration was controlled during mixing process. Finally, the rinsing step used the DI water to rinse the parts followed by blow drying with nitrogen gas.



(b) Rinsing step



To improve the removal efficiency of post-etched residue, a total of 2 process parameters were studied: scrubbing time and NaOH concentration. The scrubbing force, process step sequence, and DI rinsing time were fixed.

To define the process window, alumina pitting defect presence was used as a gating criterion. As shown in Figure 4.2, when there is an excessive exposure of the parts to the NaOH, the alumina portion of the slider will start to "pit". Although alumina pitting is a cosmetic defect – i.e. do not alter the electrical nor magnetic performance of the device, it was found necessary to limit the size and magnitude of this defect in order to avoid any unanticipated reliability failure in the drive.



Figure 4.2. Example of inspection location for pitting defect and post-etched sidewall cleanliness.

4.2 Tools and equipment

4.2.1 Process equipment

- 4.2.1.1 Photoresist spin coater
- 4.2.1.2 Photoresist laminator
- 4.2.1.3 UV stepper machine
- 4.2.1.4 Developer machine
- 4.2.1.5 Reactive ion etching machine
- 4.2.1.6 Automated photoresist stripping machine
- 4.2.1.7 Automated scrubbing machine

4.2.2 Analytical equipment

- 4.3.2.1 SEM
- 4.3.2.2 AFM
- 4.3.2.3 Auto titrator
- 4.3.2.4 Ion Chromatography

4.3. Experiment Procedure

The process characterization contained 4 steps as following.

4.3.1. Screening Process Recipe

First step of this study was to screen for the condition that is free of cosmetic defects. This condition was used to define the process window. The concentration of cleaning solution, sodium hydroxide, and scrubbing time can all contributed to alumina pitting. Higher concentration and longer scrubbing time created more pitting. Samples were prepared with 3 different NaOH concentration range. Visual inspection under high power microscope was use to quantify the pitting in each concentration as a function of the scrubbing time. SEM was also used to characterize the redeposit removal efficiency. (See Figures 4.3.)



Figure 4.3. A flow diagram of screening process used in screening the redeposit and defect to quantify the impact of each cleaning recipe factors.

4.3.2 Etched sidewall cleanliness comparison between current cleaning recipe and the new recipe.



Figure 4.4. A diagram illustrating how the comparisons was made for each

cleaning condition.

4.3.2.1. Prepared part with the same etching depth and using the same RIE machine followed by resist stripping to remove the photoresist.

4.3.2.2. Cleaned with NaOH scrubbing process. The cleaning process parameter such as scrubbing force, brush position, scrubbing stroke, DI rinsing pressure and nitrogen blow pressure were all set to the same setting. The concentration of NaOH was checked by the titration and controlled at +/- 3 of standard deviation as confidence level.

4.3.2.3. Cleanliness was checked after NaOH cleaning by SEM and AFM. The statistical techniques were used to compare result between the current base recipe and the experimental recipe. The inspection data was categorized as the attribute data so that they can be compared for the effectiveness of two sample groups by using proportion analysis with confidence level at 95% to detect the significance.

4.3.2.4. Ion chromatography was used to identify the fluoride ion left on the part after cleaning. The quantity of each sample is 40 pieces per trial with a total 10 trials.

Following images in figure 4.5 and 4.6 are the cleanliness inspection criteria using SEM and AFM.



(a) No posted-etch residue(redeposition) on etched sidewall



(b) Found post-etched residue (redeposition) on etched sidewall

Figure 4.5. SEM images showing the cleanliness quality from (a) cleaned and (b) uncleaned sidewalls.



(a) No spike (protruded residue) on etched sidewall



(b) Found spike (protruded residue) on etched sidewall

Figure 4.6. AFM images showing the cleanliness quality from (a) cleaned and (b)

uncleaned sidewalls.

4.3.3.1. Quasi-Static Test (QST) was used to check the electrical performance of the read-write head in the slider form. QST yield based on the product specification was used for the comparisons.

4.3.3.2. Dynamic Electrical Ttest (DET) was used to check the electrical performance of the read-write head in the Head Gimble Assembly (HGA) form. The test was performed to simulate the read/write function on the magnetic disk to see the yield and performance of reader and writer.

4.3.3.3. Early Touch Down (ETD) was used as a key monitoring parameter during the DET. ETD is a good indicator which signifies the occurrence of head-disk contact that is normally associated with the presence of the contaminants on the slider body.

4.3.3.4. Touchdown limit failure is a parameter of Drive assembly process to check the read-write operating performance. This failure is similar with the ETD on dynamic electrical test; but this test is performed on a completed HDD.

Figure 4.7 shows the overall experimental diagram. The read-write head performances checking are illustrated in the Figure 4.8. For part cleanliness by SEM and AFM will be tested on the read-write head include the fluoride ion residue by IC. The electrical performances on "reader" and "writer" are checked on read-write head and also in hard disk drive.



Figure 4.7. A diagram showing the experimental flow.



Figure 4.8. A block diagram showing the electrical performance check.

4.4 Analytical Techniques

Note that contaminants associated with the ABS formation process – i.e. photolithography and etching – are very complex and demands a close scrutiny in order to be effectively eliminated. The contaminants commonly induced during the ABS formation process usually have submicron feature size; thus rendering conventional high-power optical microscopy ineffective for its characterization. SEM and AFM are needed in this case in order to perform the surface inspection after the associated cleaning process. However, SEM and AFM have limited throughput and field of view. The challenge then involves making quantitative data analysis from a limited data set. In this case, proper use of statistical analysis tools is necessary.

4.4.1 Scanning Electron Microscopy (SEM)

SEM is an extremely versatile for imaging surface and subsurface microstructure. Phillip XL30S was used to investigate the part cleanliness in this study. SEM was used to scan post-etched sidewall surface cleanliness to compare the residue after cleaning. The samples were prepare by attach on SEM stud and coat the Palladium (Pd) to reduce charging from substrate material before scan. The specimens were loaded into the chamber and used resolution at 5 kV. The images were taken and counted for the number of part that contains the residue for comparisons.



Figure 4.9. Picture of the Phillip XL30S SEM.

4.4.2 Atomic Force Microscopy (AFM)

The AFM consists of a microscale cantilever with a sharp tip (probe) at its end that is used to scan the specimen surface. Dimension 500 (DI 5000) of Veeco was used to investigate the part cleanliness. AFM was used to scan post-etched sidewall surface cleanliness to compare the protruded residue (spike at the etch sidewall).



Figure 4.10. Picture of the Dimension 5000 AFM.

4.4.3 Ion Chromatography (IC)

Dionex DX500 system was used for measurement the fluoride, chloride, nitrate, phosphate, and sulfate on the hard disk drive component. For this study fluoride ion is the focus since post-etched redeposit consisted of fluoride compound.