Ice and Abrasive Particles: An Alternative CMP Polishing Technique

by

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To Mommy, Daddy, and Ana, for all your help and support.

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ABSTRACT

An experimental study was carried out to determine the feasibility and performance of a CMP polishing pad made from a frozen solution of distilled water, alcohol, and 10% concentration by volume of aluminum oxide abrasive particles. Initial experiments were conducted to characterize the process and the solution. In the final experiments, the solution was frozen in a container with liquid nitrogen. Pieces of aluminum-covered silicon wafers were polished with this frozen pad on a pin-on-disk setup.

The results from the experiments showed that liquid nitrogen is the best method for freezing the solution. The final experiments showed that the material removal rate for this polishing pad fluctuates between 6.2 and 26.9 nm/min with the applied pressure fluctuating between 0.7 and 0.9 psi. Microscope images of the surface of the polished wafer showed that the surface had scratches in the 2 micron range, slightly above the current industry results. The images also showed that contaminant particles caused scratches in the 10 micron range.

Thesis Supervisor: Professor Nam P. Suh

Title: Ralph E & Eloise F Cross Professor and Mechanical Engineering Department Head

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Chapter 1:Introduction

In CMP, Chemical Mechanical Planarization, a surface which initially was not planar is made smooth and planar through a combination of chemical and mechanical forces.[1] The surface to be polished, the pad, and the slurry containing the abrasive particles (generally SiO_2 or Al_sO_3) are the three main components of this process. The CMP process is very important for semiconductor manufacturers not only because it achieves planarization of nonplanarized surfaces, but more importantly, because it is the only process that can achieve global planarization, essential for the production of multilevel interconnections.[2]

Currently, the CMP process uses three-body abrasion to polish semi-conductor wafers. Abrasive slurry is inserted between the wafer and a soft pad to obtain the polishing action. Several problems arise when using this soft pad for polishing wafers. For instance, the current polishing pads must be broken in before using them, besides having to charge, dress, and clean them before and after each use.

To increase the material removal rate, a hard, flat abrasive pad can be used in direct contact with the wafer to polish it. The problem with this approach is that, in general, a hard, flat abrasive pad is not only difficult to manufacture, but also it would require frequent replacement due to wear. Furthermore, with a harder pad, besides increasing the risk of surface damage, a lower step height ratio (SHR) is achieved, which means that there is good planarization but poor uniformity. If a higher polish pressure is used to increase the material removal rate, the SHR is also increased which means that less planarization is achieved.

Research was conducted to find a hard, flat abrasive pad that is easy to manufacture, provides a good surface quality, and achieves a good material removal rate (MRR). It was proposed that slurry consisting of water and abrasive particles be frozen in a container and the resulting frozen pad be used to polish wafers. It was discovered that, due to the high wetting angle of the abrasive particles, water was not enough to wet these particles. Therefore, alcohol was mixed with the water and abrasive particles to correct this problem. To achieve a flat surface, a mold with a lid was machined to be used to freeze the abrasive solution. The lid provides a way to ensure the flatness of the top surface to be used for polishing.

Two main experiments were conducted. The objective of the first experiment was to determine the effect of both the abrasive particles and the alcohol on the freezing of water, as well as to determine whether it is best to freeze the solution with a freezer or with liquid nitrogen. In this set of experiments, the solution was frozen with either a freezer or liquid nitrogen to determine the best cooling method for this polishing pad. First, distilled water was frozen alone, as a control. Then alcohol was mixed with water and frozen. Finally, a solution of water and abrasive particles, at various concentrations by volume, was prepared and frozen.

The objective of the second experiment was to determine the effectiveness of the frozen polishing pad on polishing pieces of aluminum coated silicon wafers. The effectiveness of the pad was characterized by measuring the material removal rate (MRR) and noting the surface quality of the wafer.

Finally, a curve detailing the cooling of the container and the solution was characterized. This curve was generated by measuring the temperature changes in the container as well as the solution over a period of time. This curve is useful to gain further understanding of the freezing process as well as determining what changes can be made to the freezing process to make this process more effective.

The objective of this research is to develop a revolutionary polishing pad that is easy to manufacture, use, and replace while providing a good MRR and a good surface quality of the polished wafer. To achieve this objective it is essential that the freezing process as well as the effect of the components of the solution on the freezing process be understood.

This first chapter provides a general overview of the project. The motivation behind the research is described briefly. Furthermore, a brief description of the main experiments conducted is presented.The second chapter provides some more detailed background information on the existing CMP polishing technology. This chapter provides an appreciation of the importance of finding a new polishing pad. The third chapter consists of the theoretical information behind the experiments. Furthermore, any equations necessary to understand the experiments are presented in this section. This information is essential in understanding the results obtained from the experiments.The fourth chapter contains the experimental setup for the different experiments. It includes details about the different experiments and the equipment used in each. The fifth chapter

contains the results from the two different experiments and the cooling characterizations as well as a discussion of the results obtained from these experiments.Finally, the seventh chapter contains the conclusions drawn from the results obtained as well as future work that should be done to explore further avenues in this area.

Chapter 2: Background Information

This chapter contains detailed background information about the CMP process, some properties of water under freezing conditions, some properties of aluminum oxide particles, and finally, some properties of alcohol. The information contained in this chapter is essential to understand the implications of the results obtained in the experiments, discussed in Chapter Six.

Section 2.1: The CMP Process

CMP (Chemical Mechanical Planarization) is a process by which a surface is smoothed and made planar with the aid of chemical and mechanical interactions with an abrasive slurry.[1] The current process has three main components: the surface to be polished, the pad, and the slurry which contains abrasive particles. The pad is the key element transferring mechanical forces to the surface to be polished, while the abrasive slurry is the key element for the chemical and mechanical effects.[2] The slurry helps to achieve high polishing rates and planarity, via chemical interactions, while the abrasive particles in the slurry (commonly SiO_2 or Al_sO_3) transfer the mechanical energy between the pad and the surface to be polished.[3]

A more elemental version of the CMP process was used for thousands of years to polish optical surfaces. Contemporary development of CMP is due to the interest of the semiconductor industry in this type of process.[4] CMP is important to the semiconductor industry, not only because it achieves planarization of nonplanarized surfaces, but also because it is the only process that can achieve global planarization, which is essential to building multilevel interconnections; the ability to do global planarization is the most important advantage of this process.[4] Currently, the CMP process is used to planarize dielectrics and metal films which are used in the fabrication of silicon integrated circuits (SiIC). Usually, the material removal rate (MRR) is less than $0.5\mu m$, while the remaining thickness should be within 0.01 and $0.05\mu m$ of the target.[5] Since the CMP process seems to be such a great match for the semiconductor industry, it would be no surprise that sometime in the future, IC chips will be fabricated widespread using the CMP process.

The current CMP process uses a three-body abrasion method. This consists of moving the sample to be polished against a pad and passing slurry between the surface of the sample and the

pad to remove material in order to achieve good planarization results. The abrasive particles in the slurry provide the mechanical wear necessary to break particles off of surface of the sample.[6] The combination of the chemical and the mechanical parts in the process assures a good result. Cleaning the sample is the last step in the CMP process.

Three degrees of planarity exist: surface smoothing, local planarity, and global planarity. Surface smoothing consists of smoothing the corners and filling the high aspect ratio holes. Local planarity refers to surfaces that are locally flat, while the surface height may vary across the die. Conversely, global planarity refers to a flat surface across the entire sample.[7]

Several advantages to using CMP in the production of IC chips exist. As mentioned above, CMP is the only process which can currently achieve global planarization required to build multilevel interconnections. Furthermore, surfaces from a wide range of materials can be planarized (Al, Cu, Ti, TiN, W and alloys; insulators such as: SiO_2, doped SiO_2 glasses, Si_3N_4 and polymers; and finally, polysilicon), while the reliability, speed, and yield (lower defect density) are all increased. At the same time, the topography is reduced to allow for tighter design rules and additional interconnection levels. CMP is also used as an alternative process for patterning metal used instead of dry etching, which eliminates the need to properly dispose of the hazardous gases.[8]

On the other hand, the CMP process also has some disadvantages. For example, three-body abrasion can introduce possible new defects, such as undesirable scratches, stress cracking, delamination, and corrosion caused by the chemical interaction between the surface and the slurry chemicals.[9] Furthermore, the polishing pad must be broken in before use, and must be charged, dressed, and cleaned before and after each use. It is necessary to condition the pad regularly to avoid pad glazing, a phenomenon where the surface of the polishing pad deforms and wears, resulting in non-uniform MRR when polishing the wafer.[10]

To increase the MRR, two-body abrasion can be performed by using a hard, flat abrasive pad in direct contact with the wafer for polishing. The problem with this approach, besides frequent replacement and manufacturing difficulty, is that, a lower step height ratio (SHR: the variation present in the contour of the surface) occurs with a harder pad, resulting in good planarization

but very poor uniformity. If a higher polishing pressure is used to increase the MRR, less planarization is achieved due to the increase in the SHR.[11]

Section 2.2: Properties of Water Under Freezing Conditions

Since the project involves the creation of ice pads, an understanding of the freezing process of water is essential. After water supercools, it begins freezing on the walls of the container or on suspended particles. Two interesting things happen once the first ice crystal has been formed. First, the ice crystal grows dendritically, releasing a large amount of latent heat. Second, as water freezes, the volume increases by about 10%, exerting pressure on the remaining fluid around it. Furthermore, if the container is uniformly cooled, the temperature decrease is much greater in the center of the container compared to other regions of the container.[12] On the other hand, when the container is cooled unilaterally (only one side of the container is cooled maintaining the remaining sides at the same temperature), the fluid cools from the cooled surface on, and ice "grows" from that surface on.

When solid particles are mixed in the water, they may either be pushed away by the front of freezing water (the interphase between the liquid and frozen sections of the water, that advances as the ice grows from the cooling source) or they can be encapsulated into the network of crystallized water. Which situation occurs is dependent on whether the critical velocity, which is dependent on the temperature gradient between the source of cooling and the water, is above or below a certain critical value. When the velocity at which the front of freezing water propagates is above 3.3μm/s, the position of the particles does not change as they are incorporated into the network of crystallized water. On the other hand, when the velocity of the freezing front is below 3.3μm/s, the particles are swept along by the front and accumulate ahead of it. Therefore, a larger temperature gradient will result in a larger critical velocity, and a smaller temperature gradient will result in a smaller critical velocity. For this reason, if the liquid solution can be frozen by using a method that creates a large temperature gradient, such as the use of liquid nitrogen, the particles in the solution will frozen without a change in their spatial location.[13]

Section 2.3: Properties of Aluminum Oxide Particles

Aluminum oxide is a ceramic material that consists of a hexagonal close-packed array of anions. Aluminum oxide has a surface energy per unit area equal to 1.2 J/m^2. Since it controls the kinetics involved in sintering, surface energy is an important aspect in this process. Similarly, surface tension seems to be the driving force behind sintering. Surface tension is also closely related to the phenomenon of wetting angle or angle of contact between the solid and liquid phases. Because this angle of contact seems to be large in the case of aluminum oxide, it does not bond readily with water. To reduce the surface tension between the aluminum oxide and water is essential in achieving wettability of the aluminum oxide. [14]

Section 2.4: Properties of Alcohol

Alcohols, organic derivatives of water, are among the most organically polar groups (groups that are charged).[15] Their hydroxyl group is very polar and can undergo hydrogen bonding with water molecules, which makes them very soluble in water. Isopropyl alcohol (2-propanol isopropyl alcohol) is an inexpensive alcohol known as rubbing alcohol. Water and alcohol molecules are similar in the sense that they contain the strongly polarized hydroxyl groups that can form hydrogen bonds with other molecules. Some of the properties of isopropyl alcohol include: boiling point (82°C) and density (0.79g/mL).[16]

Because of this strong tendency to undergo hydrogen bonding, when water and alcohol are mixed together some of their properties change. The property that is of most interest for this research is the change in density of water that occurs when alcohol is mixed with water. This change in density is due to the destabilization of the molecule in which the alcohol promotes a more tightly folded arrangement, thus decreasing the density of the solution.[17]

Alcohol also has an effect on the alumina abrasive particles. In their natural unmixed state, the alumina particles have a high wetting angle. This high wetting angle is responsible for its low solubility in water. Thus, simply mixing water and alumina particles will result in a two phase solution that is not well integrated. However, alcohol decreases the wetting angle of the alumina particles, making it more soluble in substances such as water.

Chapter 3: Experimental Setup

Three sets of experiments were conducted. The first set of experiments determined the best way to freeze the slurry. In the second set of experiments, wafers were polished to determine how effective the pad of frozen slurry is for polishing wafers. The cooling experiments were conducted to determine a cooling curve for both the container and the slurry. The experimental setup can be divided into three categories: setup for experiment one, setup for experiment two, and setup for the cooling experiments.

Section 3.1: Setup for The Freezing Experiments

This section includes a description of the Freezer Experiment along with any materials needed to carry out this experiment. The Freezer Experiment can be divided into two groups of sub-experiments. In the first group of sub-experiments, Experiment 1A, the solution was frozen with a freezer. The aim of Experiment 1A was to determine the best way constitution of the solution to be frozen. In the second group of experiments, Experiment 1B, the solution was frozen only using liquid nitrogen. The aim of Experiment 1B was to determine the effects of the different components of the solution on the resulting polishing pad. The combination of these two experiments aims to determine whether it is best to freeze the solution in a freezer or with liquid nitrogen.

Section 3.1.1: Experiment 1A: Freezer Experiment

This section describes the details of the experiments carried out to determine whether the mixture of water and alumina would be good enough to create a polishing pad or if another component would have to be added to the solution to lower the wetting angle of the alumina (such as isopropyl alcohol). Furthermore, the results of these experiments can be used to compare freezing the solution with a freezer with freezing it with liquid nitrogen.

The setup for Experiment 1A consisted of a freezer, an ice cream maker, and a container with a removable polished aluminum baseplate constructed to hold the solution for freezing, shown in Fig. 3.1. By using a removable baseplate, the surface of the ice pad that is in contact

with the surface of the aluminum baseplate can be viewed easily by simply removing this baseplate.

For Experiment #1 (Control), 1 cup and 1 oz. distilled water (equivalent to 1 inch thickness of water in the container) at room temperature was poured into the container. The container with the water was set on the top shelf of the freezer.

For Experiment #2, 1 cup and 1 oz. distilled water at room temperature with 1 tsp. Micropolish II 0.3 (m Alpha Alumina) abrasive particles were combined. This solution was poured into the container, and the container was placed on the top shelf of the freezer.

Figure 3.1: *Container with a Removable Baseplate*

For Experiment #3, the solution was made into a slush with an ice cream maker. The ice cream maker was left in the freezer overnight. A solution consisting of 1 cup and 1 oz. distilled water and 1 tsp. abrasive particles was poured into the ice cream maker. The purpose of making a solution into a slush was to try to create a better distribution solution that could be poured into the container and frozen on the top shelf of the freezer.

Finally, for the last experiment for this group, Experiment #4, a mixture of 1 cup isopropyl alcohol, 4 cups water, and 2 tsp./cup-of-water abrasives was combined and 1 cup and 1 oz. of this

solution was poured into the container. The container was then placed on the top shelf of the freezer.

Section 3.1.2: Experiment 1B: Liquid Nitrogen Experiment

This section includes the details of the Liquid Nitrogen Experiment. This experiment aimed not only to determine how well freezing the solution with liquid nitrogen would work, but also to determine what the effect of the different components of the solution have on the resultant ice pad. This information can be very helpful in understanding the interactions between the three components.

The setup for Experiment 1B consisted of the same container with the removable baseplate, with cooling coils added to the baseplate to hold the liquid nitrogen, as shown in Fig. 3.2, and liquid nitrogen to freeze the solution. For Experiment #1 in this group, 2 cups and 2 oz. distilled water were combined with 1/8 cup alcohol and 4 tsp. abrasive particles. Of this solution, 1 cup and 1 oz. was poured into the container. Liquid nitrogen was allowed to flow through the copper cooling coils to freeze the solution from the baseplate up.

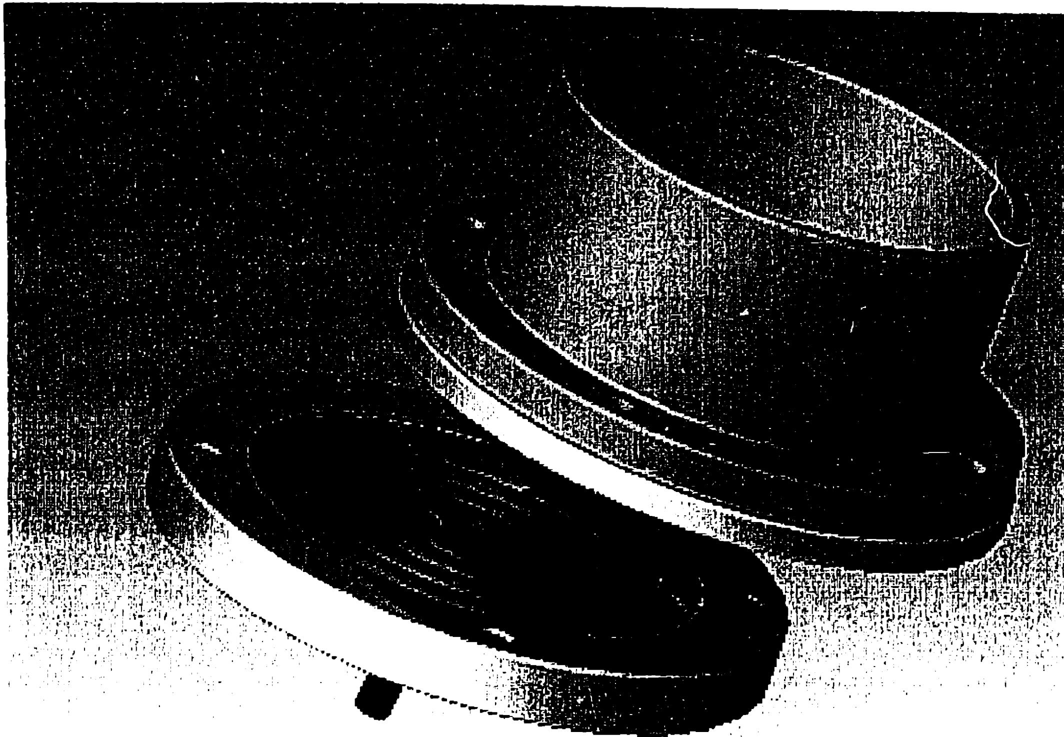


Figure 3.2: *Container with the Cooling Coils Added to the Baseplate*

For Experiment #2, only distilled water was used. Distilled water was poured into the container, and liquid nitrogen was used to freeze the distilled water.

For Experiment #3, 2 cups distilled water and 1/8 cup alcohol were combined and 1/4 cup of this solution was poured into the container. The solution was cooled with the liquid nitrogen.

For Experiment #4, 1 cup distilled water and 2 tsp. abrasive particles were combined and poured into the container. The solution was cooled with liquid nitrogen once again.

Section 3.2: Setup for the Polishing Experiments

This section describes the Polishing Experiments. The aim of this set of experiments was to determine whether the polishing pad obtained from the frozen solution of water, abrasive particles, and alcohol, gives good results. The important measurements in this set of experiments are the MRR (see EQ 4.3-4.5 in Chapter 4) along with the error in its calculation (see EQ 4.7 in Chapter 4) and the surface finish of each piece of wafer (some representative images are included in Chapter 5). The pressure applied to the pieces of wafer was also calculated (EQ 4.6 in Chapter 4) to determine how constant this applied pressure was.

The setup for this experiment consisted of the holder used in the previous experiments with the cooling coils used for the liquid nitrogen, liquid nitrogen to freeze the solution, a plastic lid to make the top surface flat, a pin-on-disk setup to polish the wafers, a scale to weigh the wafers before and after polishing them, a 499.97 gram weight mounted on the pin-on-disk setup to create the pressure for polishing, alcohol and soft tissue to clean the wafers prior to weighing them, and a pair of tweezers to handle the wafers.

For this second experiment, a series of steps were carried out to get accurate results from the polishing of the wafers.These wafers were 4 inch diameter wafers with a 10 nm layer of aluminum and a 500μm +/- 10μm silicon base layer. First, a scale was calibrated and set to zero. Each of three whole wafers was weighed 3 times, recording the weight up to μg. The average weight of the three readings was taken. The wafers were then broken into similar pieces (obtaining 5 samples from the 3 whole wafers, and each piece was also weighed three times, where up to

μg were recorded for each measurement, and the average of all three measurements was taken to determine the average weight.

A batch solution of 10% concentration by volume was prepared by mixing 269.5 grams of abrasive particles, 300 mL of alcohol, and 330 mL of distilled water, adding up to a total volume of 700 mL (refer to EQ 4.1 and 4.2 in Chapter 4 for the calculation of the concentration by volume). This solution was frozen using liquid nitrogen. Each piece of wafer was polished for 3 minutes on the pin-on-disk setup. The resulting polished pieces were weighed 3 times each (up to 6 digits were recorded) and an average new weight was calculated.

Section 3.3: Setup for the Cooling Experiments

This section describes the details of the Cooling Experiments. These experiments were conducted to determine how the container and the slurry freeze as they are cooled with liquid nitrogen.

The setup for this experiment consisted of the holder from the two other experiments with the cooling coils, liquid nitrogen for cooling, and a digital temperature reader. A solution of 10% concentration by volume of abrasive particles was prepared by mixing 12 mL abrasive particles, with 50 mL distilled water, and 58 mL isopropyl alcohol. To characterize the cooling curve for the slurry, the solution was poured into the holder where it was cooled down with liquid nitrogen for 480 seconds, at which time, the liquid nitrogen was turned off (the tip of the temperature reader was inserted in the middle of the solution). The temperature of the frozen solution was monitored for another 1365 seconds to get an idea of the behavior of the frozen pad as it warmed up. Similarly, to characterize the cooling curve for the container, the container was cooled down with the liquid nitrogen for 540 seconds, while the tip of the temperature reader was held against the surface of the baseplate. The cooling curve was determined from this information.

Chapter 4: Technical Background

This chapter describes the formulae needed to calculate the percentage by volume of abrasive particles in the solution and the corresponding mass that is added to the solution. Furthermore, it includes the formulae on how to calculate the area of the piece of wafer, the thickness removed, the MRR and the error associated with this calculation as well as the pressure applied to the wafers upon polishing.

Section 4.1: Abrasive Concentration

This section details how to calculate the abrasive concentration in the solution. This calculation is very important for the polishing experiments because a constant concentration of 10% percent by volume needs to be maintained.

To determine the percentage by volume of the abrasive particles in the solution, the following calculations must be done. The volume of abrasive particles in cm^3 (or mL) is:

$$V = \frac{x}{100} \times T \qquad (4.1)$$

where,

T = total volume of solution in mL (alcohol, abrasive particles, water)

x = percentage of the solution that the abrasive particles should occupy (in volume)

V = volume of abrasive particles in cm^3 = mL

Since the abrasive particles don't come compacted, but loosely packaged in a container is it necessary to convert this volume into weight and use this weight to measure the amount of particles that is added to the solution. To convert the volume into a weight:

$$M = V \times 3.85 \frac{g}{cm^3} \qquad (4.2)$$

where,

M = weight in grams (g) of the abrasive particles

Density of alumina ($\rho_{alumina}$) = 3.85 g/cm^3

Section 4.2: Material Removal Rate and Pressure Calculations

This section describes how to calculate the area of the piece of wafer and the thickness removed from the wafer through polishing. These two calculations lead to the calculation of the MRR, which is an important measurement in this project, since it allows a means of comparison with other material removal processes. How to calculate the error in this calculation is also discussed in this section. Finally, the formula for the calculation of the pressure applied to the wafer is also discussed. For a detailed derivation of these formulae, please refer to Appendix A.

Two of the most important measurements needed to determine whether this polishing pad is effective or not are material removal rate (MRR) and the pressure exerted on the pad and wafer system (P). To determine the MRR, the area of the piece of wafer must be calculated:

$$A_p = \frac{M_p}{(\rho_{al} \times t_{al}) + (\rho_{si} \times t_{si})} \times 10^6 \qquad (4.3)$$

where,

A_p = surface area of the wafer in millimeters [mm]

M_p = mass of the wafer in grams [g]

ρ_{al} = density of aluminum in Mg/m^3 (use ρ_{al} = 2.7 Mg/m^3)

t_{al} = thickness of the aluminum layer of the wafer in micrometers [µm]

ρ_{si} = density of silicon in Mg/m^3 (use ρ_{si} = 3.2 Mg/m^3)

t_{si} = thickness of the silicon layer of the wafer in micrometers [µm]

Besides the area of the piece of wafer, the thickness of the wafer removed through polishing must be calculated by measuring the change in mass of the piece of wafer and using the area of the piece of wafer calculated in the above equation.

$$\Delta t = \frac{\Delta M}{\rho_{al} \times A_p \times 10^{-6}} \times 10^{9} \qquad (4.4)$$

where,

Δt = change in thickness [nm]

ΔM = change in mass of the piece of wafer [g]

Finally, the MRR can be calculated by using the change in thickness obtained from the above equation.

$$MRR = \frac{\Delta t}{time} \qquad (4.5)$$

where,

MRR = material removal rate in nanometers per min [nm/min]

time = amount of time the wafer was polished in minutes [min]

The pressure that was applied to the wafer and polishing pad can be calculated by using the following formula:

$$P = \frac{\frac{M}{1000} \times g \times (1.450377 \times 10^{-4})}{A \times 10^{-6}} \qquad (4.6)$$

where,

P = pressure applied to the wafer and polishing pad [psi]

g = gravity ($9.8 m/s^2$)

Section 4.3: Error Analysis

The error involved in the calculation of the MRR can be calculated by using the following:

$$u_{MRR} = \sqrt{\left(\frac{\partial MRR}{\partial \Delta M}\right)^2 \times (u_{\Delta M})^2 + \left(\frac{\partial MRR}{\partial M_p}\right)^2 \times \left(u_{M_p}\right)^2} \qquad (4.7)$$

Chapter 5: Results and Discussion of the Results

Section 5.1: Cooling Experiments

The results for the cooling experiments can be divided into two sections: the cooling of the container and the cooling of the slurry. The data obtained from monitoring the temperature of the container throughout 9 minutes can be seen in Appendix B. The curve resulting from this data is illustrated in Fig. 5.1. As can be seen from the curve, for the first minute the temperature did not change significantly. However, once the initial cooling period passed, the temperature dropped steadily until the last minute and 15 seconds where it seems to have reached steady state. Unfortunately, since the maximum temperature that can be read with the temperature reader is -50 Celsius, no temperatures were recorded past -50 Celsius.

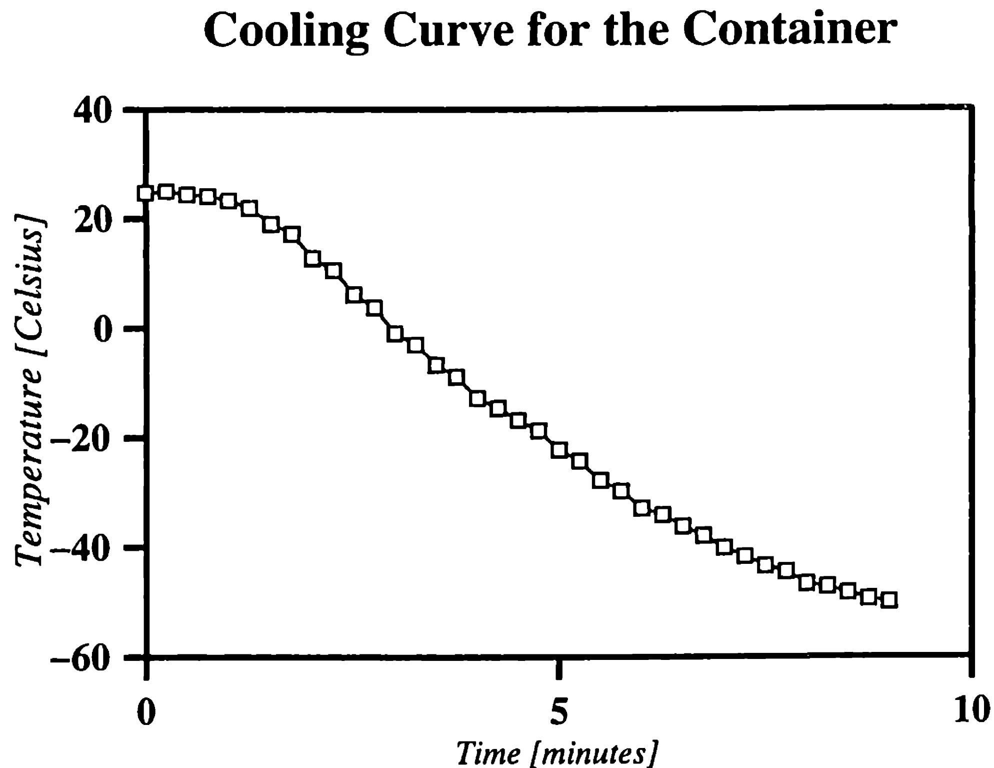


Figure 5.1: *Cooling Curve for the Container*

The data obtained from monitoring the temperature of the slurry throughout 25.5 minutes can be seen in Appendix C. The curve resulting from this data is illustrated in Fig. 5.2. During the first minute and 30 seconds of cooling, the temperature of the slurry remained almost constant, suggesting that the baseplate of the container was cooling down during this time. After the initial minute and a half up to the 8 minute marker, the temperature decreased steadily. After the first 8 minutes, the liquid nitrogen was turned off because the slurry was already completely frozen. After this point, even though the cooling source was turned off, the baseplate continued to cool down the slurry, until it reached a minimum temperature of -42.8 Celsius after 5.25 minutes after turning off the liquid nitrogen. This can be potentially beneficial since the cooling source can be turned off before achieving the desired temperature, thus conserving liquid nitrogen. After this point, the temperature of the slurry increased steadily at a slower rate, as can be seen by studying the curve between time 11.75 and 25 minutes.

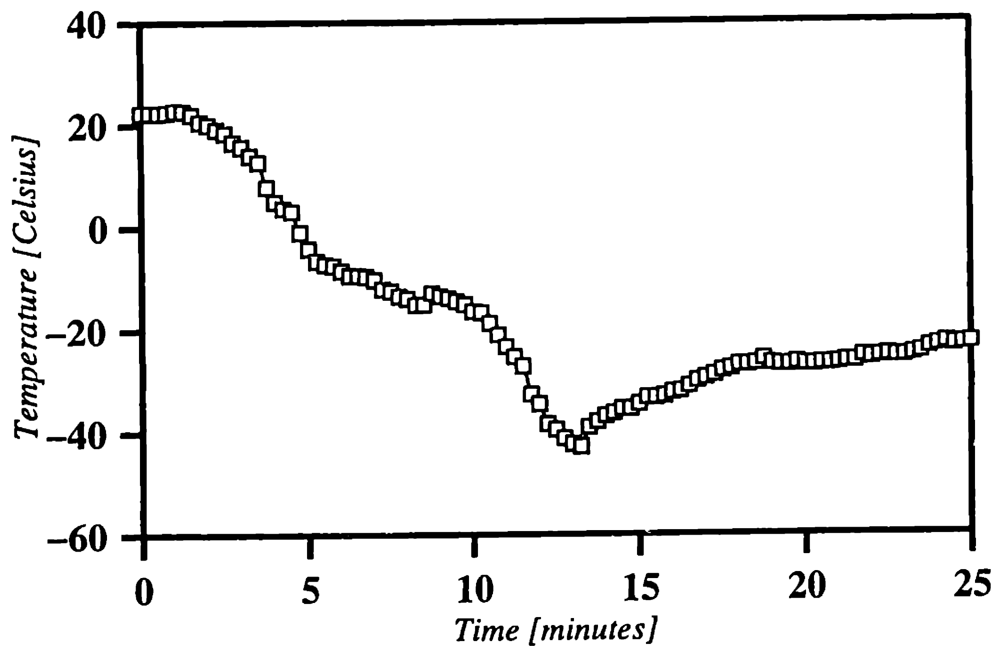


Figure 5.2: *Cooling Curve for the Slurry*

Section 5.2: Freezing Experiments

The results for this set of experiments can be divided into two groups of results. The first group of results corresponds to Experiment 1A, while the second group of results corresponds to Experiment 1B.

Section 5.2.1: Results for Experiment 1A: Freezer Experiment

For Experiment #1 (the control), the bottom surface (the surface in contact with the aluminum baseplate) of the frozen water was very smooth and appeared to be very flat. The top surface was very bumpy and cracked.

For Experiment #2, the resulting ice pad had a thin, smooth, and flat bottom layer of frozen abrasives, as shown in Fig. 5.3. This suggested that the water is not enough to wet the aluminum oxide abrasive particles. There appeared to be a marked difference in concentration between a 1/4 inch ring along the outside edge of the ice pad and the center of the ice pad. This is clear evidence that the abrasive particles were not distributed uniformly throughout the ice pad.

Figure 5.3: *Ice Pad Obtained in Experiment #2 (Distilled Water and Abrasives)*

distilled H_2O and 1 Tsp. Al.O_2 made
(1 cup, 10oz) into watery slush

Figure 5.4: *Ice Pad Obtained in Experiment #3 (Frozen Slush)*

For Experiment #3, the resulting ice pad, as shown in Fig. 5.4, also had a film of abrasive particles on the bottom surface. The top surface was bumpy and had an excess of abrasive particles suggesting once again that the abrasive particles were not distributed uniformly throughout the ice pad. This is partially due to the fact that not even the action of making a slush first to partially already fix the abrasive particles is a solution for the high wetting angle problem exhibited by the abrasive particles.

For Experiment #4, the top surface of the ice pad, shown in Fig. 5.5, appeared to be very smooth. The bottom surface was also very smooth, and the absence of the film of frozen abrasive particles suggests that the alcohol lowered the wetting angle of the abrasive particles, so that now they are being completely wetted. The abrasive particles, the distilled water, and the alcohol seemed to be very well integrated. There was no evident difference in concentration.

From this set of experiments, it was concluded that the addition of alcohol is necessary to ensure that the abrasive particles are wetted. This will in turn ensure that the particles are uniformly distributed in the resulting ice pad.

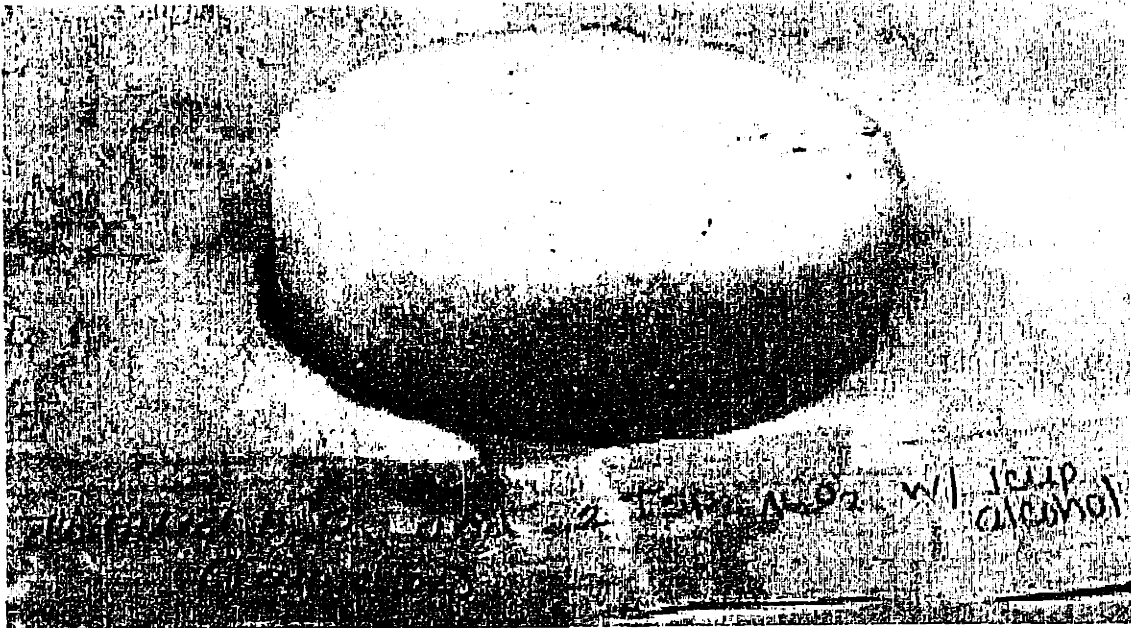


Figure 5.5: *Ice Pad Obtained in Experiment #4 (Addition of Alcohol to the Solution)*

Section 5.2.2: Results for Experiment 1B: Liquid Nitrogen Experiment

For Experiment #1, both the top and bottom surfaces of the ice pad looked very flat. The ice pad, shown in Fig. 5.6, seemed to have a very good distribution of abrasive particles. The good distribution should be true not only on a macroscopic level, but also on a microscopic level. Since the temperature gradient generated by using liquid nitrogen as a cooling agent is large, theoretically, the abrasive particles should be frozen in place, without experiencing any change in spatial location.

For Experiment #2, the resulting ice pad, shown in Fig. 5.7 had many long, deep cracks and was very brittle. Since the ice pad obtained in Experiment #1 did not posses such cracks, both the alcohol and the abrasive particles, must have some effect on cracking and crack propagation. This is evident from Experiments #3 and #4. In Experiment #3 a mixture consisting of only distilled water and alcohol was poured into the container and frozen using liquid nitrogen. The resulting ice pad, shown in Fig. 5.8, only had a couple of long cracks. On the other hand, in Experiment #4, when the solution frozen with liquid

Figure 5.6: *Ice Pad Obtained from Experiment #1 (Solution Frozen with Liquid Nitrogen)*

nitrogen contained only distilled water and abrasive particles, the resulting ice pad had many short cracks. From these experiments, it can be concluded that the abrasive particles act as inclusions to stop the propagation of cracks while the alcohol decreases the amount of cracks. The result is a pad that has no visible cracks.

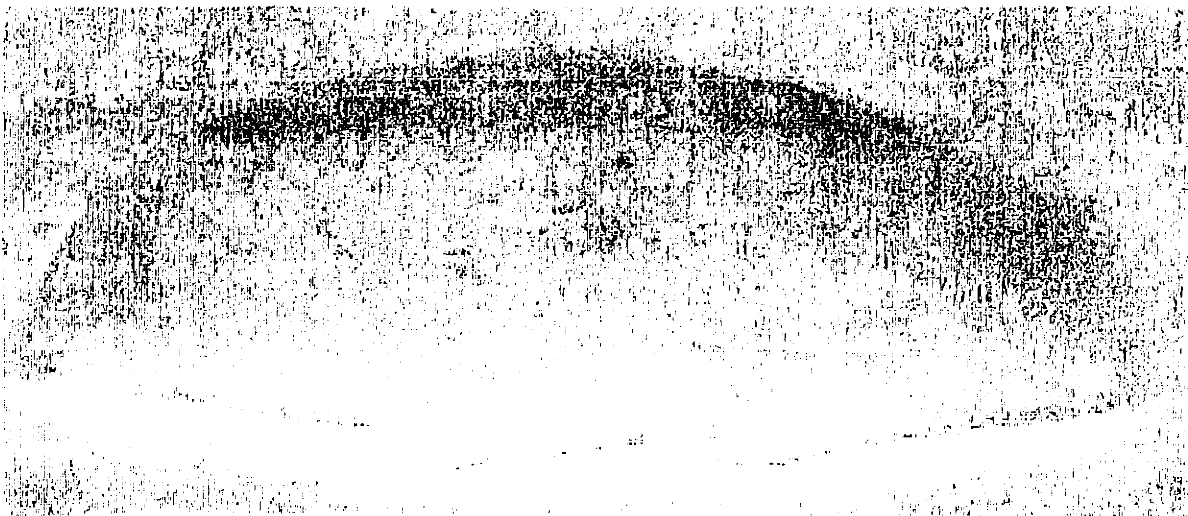


Figure 5.7: *Ice Pad Obtained from Experiment #2 (Frozen Distilled Water)*

Figure 5.8: *Ice Pad Obtained from Experiment #3 (Frozen Water and Alcohol)*

Section 5.3: Polishing Experiments

The most interesting measurement obtained from this experiment is the MRR. For each sample, the material removal rate was calculated along with the resultant pressure applied to the sample. The results from this experiment are shown in Table 1.

Sample #	MRR [nm/min]	Pressure [psi]
1	6.2	0.7
2	10.7	0.8
3	26.9	0.8
4	18.8	0.9
5	8.4	0.8

Table 1: *Resultant MRRs and Pressures for the Polishing Experiments*

It is interesting to note that the MRR varies between 6.2 nm/min and 26.9 nm/min for the same concentration (10% concentration by volume of abrasives) and for a fairly constant pressure. At first, this variation could be thought to come from the uncertainties in the measurements used to calculate the MRR. However, this uncertainty only varies between +/-0.7 and +/-0.9 nm/min. Thus, the uncertainty is not a significant source of variation. However, because this experiment was not conducted under a controlled environment, such as close monitoring of the polishing pad and environment temperatures, control of particles that might be present in a non-cleanroom quality room, and other such factors, it is possible that some of that variation is due to the effect of one or more uncontrolled variables. Furthermore, the pressure varied between 0.7 and 0.9 psi, which is a factor to consider when comparing the MRR results with those of industry or other projects.

Another interesting result to look at is the surface finish of the samples. For example, the most distinctive scratch on the surface of sample #2 was a long scratch of approximately 2 micrometers width as shown in Fig. 5.10. This result is slightly above the current industry results of 1 micrometer. However, when looking at the surface of sample #4, shown in Fig. 5.11, it can be seen that the worst scratch has a width of 10 micrometers which is rather large. As a matter of fact, a scratch of this magnitude could not have been caused by the abrasive particles. Not even

several agglomerated particles of 0.3 micrometers in diameter could have caused this. And as can be seen in Fig. 5.12, the particles are not agglomerated. It can then be concluded that this scratch must have been caused by some particles present.

Figure 5.9: *Surface Picture of Sample #2, seen through a 1000X magnifying lens.*

Figure 5.10: *Surface Picture of Sample #4, seen through a 1000X magnifying lens.*

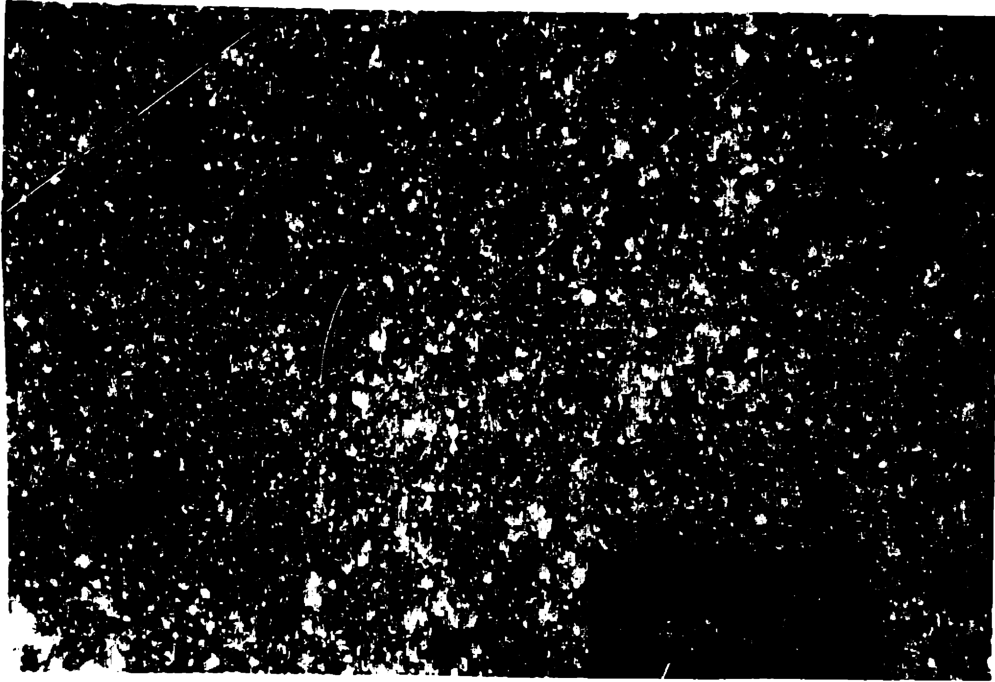


Figure 5.11: *500X Picture Showing the Distribution of Particles in the Solution*

Finally, one important issue raised from this experiment is the actual thickness of the aluminum layer on the wafers. According to the specifications obtained from the wafer supplier, the aluminum layer is supposed to be 1 micrometer in thickness. However, when looking at some of the pictures, such as from sample #2, shown in Fig. 5.13, it can be seen that the process polished through the aluminum layer at some points revealing the silicon layer. It is impossible that the process polished through a 1 micrometer layer when the MRR for this sample was only 10.7 nm/min. Fortunately, this inconsistency in the thickness of the layer of aluminum does not affect the calculations much because the density of aluminum and silicon are quite close (2.7 vs. 3.2 Mg/m^3).

Figure 5.13: *Surface Picture of Sample #2, seen through a 1000X magnifying lens, showing how the entire aluminum layer was polished away, revealing the silicon layer*

Chapter 6: Conclusions and Future Work

Section 6.1: Conclusions

As can be seen from the results discussed in the previous chapter, some very interesting conclusions can be drawn. First, from the cooling curves of the container and the solution, it can be concluded that using liquid nitrogen to perform the freezing is very efficient. All that is required is that the container and solution be cooled down without using a constant feed of liquid nitrogen. It would be enough to cool down the container and the solution prior to polishing and then during the polishing process no liquid nitrogen would have to be fed to the container because the container would still decrease and/or maintain the temperature of the solution. This is also a great advantage because a feedback system is not necessary to monitor liquid nitrogen input and output during the polishing process.

Another conclusion that can be drawn from these results is the efficiency of this method of freezing the solution. According to this theory, because the liquid nitrogen causes a large temperature gradient upon freezing the solution, thus the abrasive particles should not experience a change in their spatial location. According to the microscope image taken of the slurry, the particles are not agglomerated but well distributed. Therefore, since the particles are not accumulated at the top surface, they are not being pushed up by the front of freezing solution, and are thus remaining in more or less a fixed spatial location.

Another factor that contributes to the uniformity in distribution of the abrasive particles within the solution is the addition of alcohol to the water and abrasive particles. The alcohol decreases the wetting angle of the alumina particles and it also creates a hydrogen bond with the water molecules, thus decreasing the density of the solution. The alcohol also inhibits the formation of cracks in the frozen pad, while at the same time the abrasive particles act as inclusions, preventing the propagation of cracks.

Finally, it was discovered that the MRRs for some of the samples, even though lower than desired, still prove potential. One thing to consider is the fact that the pressures under which the wafers were polished were between 0.7 to 0.9 psi, which is considerably less than the amount of pressure that is applied in industry. While the calculations done are not greatly affected by the

inconsistency in aluminum layer thickness, it is important to note that inconsistency and be careful of taking the thickness of the deposited layer as a given fact not to be checked. Furthermore, the surface finish of the samples were generally smooth, although some of the samples had at least one large scratch, which was most probably caused by some loose particles contaminating the environment. Such contamination can be minimized, however, in a clean-room environment.

Section 6.2: Future Work

Like in most cases, just like questions are answered in the process of conducting research, many more questions emerge which need to be explored. Some of the questions that have arisen throughout the course of this research are discussed below.

One aspect that needs to be researched further is the determination of the minimum amount of alcohol that is necessary to wet a certain amount alumina particles. This is a very important parameter to be characterized because it will help conserve alcohol in the long run. Even though alcohol is one of the least expensive components in this project, wasting alcohol is by no means an economically sound choice to make, especially in a production environment.

Another question that arose during the course of this research has to do with the relationship between the concentration of abrasive particles to MRR and surface finish. It would be interesting to see whether the relationship is linear, approaches a steady state, or if it increases up to a saturation point and then deteriorates after a saturation point.

Finally, more polishing experiments should be run with greater pressures. Using greater pressures will make the results easier to compare to the current results in industry.

Chapter 7: Appendices

This chapter contains the appendices with the derivations that are necessary to understand the formulas presented earlier such as the MRR and pressure (Appendix A), calculations. Furthermore, it also includes the raw data obtained from the cooling (Appendix B and C), and polishing (Appendix D) experiments. The final appendix, Appendix E, has the error calculations for the MRR.

Appendix A: Derivation Of MRR and Pressure Calculations

Since the samples used in the polishing experiments were not whole wafers but pieces broken from wafers, the area of each piece of wafer must be calculated by using the mass of the piece, which can be found by weighing the piece of wafer. It is important to note that the wafer consists of two layers of different densities. The bottom layer is made of silicon, while the top layer is made of aluminum. Thus, the total mass per area (mpa) is:

$$mpa = (\rho_{al} \times t_{al}) + (\rho_{si} \times t_{si}) \qquad \text{EQ (A.1)}$$

where ρ_{al} and ρ_{si} are the densities of aluminum and silicon respectively and t_{al} and t_{si} are the thicknesses of the layer of aluminum and silicon respectively.

To find the total area of the piece of wafer (A_p), divide the mass per area by the mass, obtained by weighing the piece of wafer, and multiply this quantity by 10^6 to convert the area to mm^2. Thus, the resulting formula is:

$$A_p = \frac{M_p}{(\rho_{al} \times t_{al}) + (\rho_{si} \times t_{si})} \times 10^6 \qquad \text{EQ (A.2)}$$

where M_p is the mass obtained by weighing the piece of wafer.

The amount of material removed from the thickness of the wafer can be calculated by using the change in mass, obtained by measuring the wafer before and after polishing and calcu-

lating the difference. The product of A_p and the density of the aluminum layer gives the thickness per unit mass that when multiplied by M_p results in the change in thickness, Δt. Thus,

$$\Delta t = \frac{\Delta M}{\rho_{al} \times A_p \times 10^{-6}} \times 10^9 \qquad \text{EQ (A.3)}$$

where the factor 10^{-6} converts the area from [mm^2]to [m^2], while the factor 10^9 converts the change in thickness from [m] to [nm].

Then, the MRR can be calculated by taking the change in thickness and dividing it by the amount of time that it took to remove the thickness. Thus,

$$MRR = \frac{\Delta t}{time} \qquad \text{EQ (A.4)}$$

where the MRR is in [nm/min].

Finally, the pressure applied to the wafer can be found by calculating the force divided by the area through which the force is acting. Thus,

$$P = \frac{Force}{Area} \qquad \text{EQ (A.5)}$$

Knowing that Force = M_p x g (where g is 9.8 kgm/s^2) and that the area is A_p, and adding a factor of 1/1000 to convert M_p from [g] to [kg], the resulting formula is:

$$P = \frac{\frac{M}{1000} \times g \times (1.450377 \times 10^{-4})}{A \times 10^{-6}} \qquad \text{EQ (A.6)}$$

The extra factor of 1.450377 x 10^{-4} is used to convert the pressure from [Pa] to [psi].

Appendix B: Data for the Cooling of the Container

Time [seconds]	Temperature [Celsius]
0	24.7
0.25	25.0
0.5	24.4
0.75	24.1
1	23.3
1.25	21.9
1.5	19.0
1.75	17.2
2	12.8
2.25	10.6
2.5	6.2
2.75	3.8
3	-0.9
3.25	-3.0
3.5	-6.7
3.75	-8.9
4	-12.8
4.25	-14.6
4.5	-16.8
4.75	-18.7
5	-22.2
5.25	-24.2
5.5	-27.8
5.75	-29.7
6	-32.9
6.25	-34.1
6.5	-36.2
6.75	-37.9
7	-40.1
7.25	-41.7
7.5	-43.4
7.75	-44.5
8	-46.7
8.25	-47.2
8.5	-48.3
8.75	-49.4
9	-50.0

Appendix C: Data for the Cooling of the Slurry

Time [seconds]	Temperature [Celsius]
0	22.3
0.25	22.4
0.5	22.3
0.75	22.5
1	22.8
1.25	22.7
1.5	22.0
1.75	20.6
2	20.0
2.25	19.1
2.5	18.4
2.75	16.6
3	15.7
3.25	14.0
3.5	12.8
3.75	7.9
4	5.0
4.25	3.7
4.5	3.1
4.75	-1.0
5	-4.2
5.25	-6.6
5.5	-7.2
5.75	-7.5
6	-8.5
6.25	-9.4
6.5	-9.4
6.75	-9.6
7	-10.2
7.25	-12.0
7.5	-12.4
7.75	-13.4
8	-13.9
8.25	-15.1
8.5	-15.0
8.75	-12.8
9	-13.4
9.25	-13.8
9.5	-14.4
9.75	-15.0
10	-16.4
10.25	-16.6
10.5	-18.7
10.75	-21.0
11	-23.3
11.25	-25.3
11.5	-27.1
11.75	-32.5
12	-34.5
12.25	-38.4
12.5	-39.5
12.75	-41.2
13	-42.3
13.25	-42.8
13.5	-39.0
13.75	-37.9
14	-36.8
14.25	-36.2
14.5	-35.4
14.75	-35.4
15	-34.5
15.25	-33.2
15.5	-33.2
15.75	-32.9
16	-32.2
16.25	-31.9
16.5	-31.0
16.75	-30.0
17	-29.4
17.25	-28.8
17.5	-28.0
17.75	-27.6
18	-26.9
18.25	-26.9
18.5	-26.7

18.75	-25.8
19	-26.7
19.25	-26.9
19.5	-26.9
19.75	-26.5
20	-26.9
20.25	-26.9
20.5	-26.7
20.75	-26.7
21	-26.5
21.25	-26.2
21.5	-26.2
21.75	-25.3
22	-25.6
22.25	-25.3
22.5	-25.1
22.75	-25.3
23	-25.3
23.25	-24.9
23.5	-24.5
23.75	-23.6
24	-23.1
24.25	-22.7
24.5	-23.1
24.75	-22.9
25	-22.7
25.25	-22.9
25.5	-22.9

Appendix D: Results of the Polishing Experiments

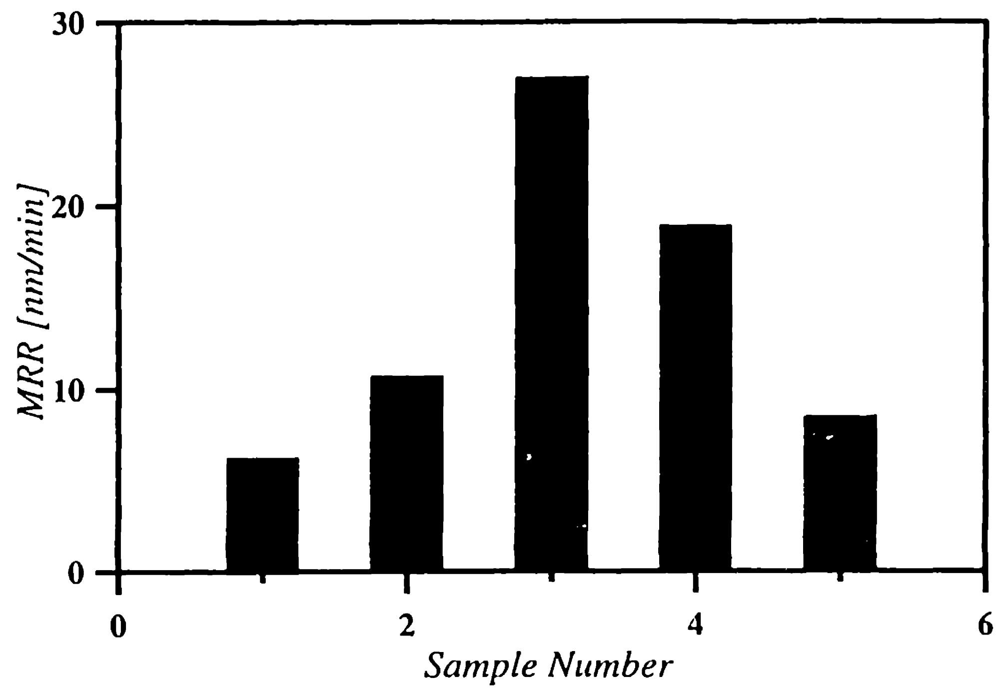
Sample #1		
	mass in grams	error (+/-)
m1	1.130767	0.000001
m2	1.130763	0.000001
m3	1.130749	0.000001
m(avg)	1.130760	
error(avg)	0.000001	
A[mm2]	968.36	
	removed mass	error (+/-)
m1	1.130703	0.000001
m2	1.130714	0.000002
m3	1.130715	0.000001
m(avg)	1.130711	
error(avg)	0.000001	
change in thickness		
	in nm	
change in t	18.741	
MRR [nm/min]	6.24701	
pressure (psi)	0.733858	

Sample #2		
	mass in grams	error (+/-)
m1	1.063641	0.000001
m2	1.063643	0.000001
m3	1.063639	0.000001
m(avg)	1.063641	
error(avg)	0.000001	
A[mm2]	910.89	
	removed mass	error (+/-)
m1	1.063562	0.000001
m2	1.063563	0.000001
m3	1.063562	0.000001
m(avg)	1.063562	
error(avg)	0.000001	
change in thickness		
	in nm	
change in t	31.9862	
MRR [nm/min]	10.6621	
pressure (psi)	0.780166	

Sample #3		
	mass in grams	error (+/-)
m1	1.001480	0.000002
m2	1.001482	0.000002
m3	1.001483	0.000001
m(avg)	1.001482	
error(avg)	0.000002	
A[mm2]	857.65	
	removed mass	error (+/-)
m1	1.001294	0.000002
m2	1.001295	0.000001
m3	1.001295	0.000001
m(avg)	1.001295	
error(avg)	0.000001	
change in thickness		
	in nm	
change in t	80.7544	
MRR [nm/min]	26.9181	
pressure (psi)	0.828589	

Sample #4		
	mass in grams	error (+/-)
m1	0.963639	0.000001
m2	0.963640	0.000002
m3	0.963643	0.000001
m(avg)	0.963641	
error(avg)	0.000001	
A[mm2]	825.25	
	removed mass	error (+/-)
m1	0.963515	0.000001
m2	0.963518	0.000002
m3	0.963511	0.000001
m(avg)	0.963515	
error(avg)	0.000001	
change in thickness		
	in nm	
change in t	56.5487	
MRR [nm/min]	18.8496	
pressure (psi)	0.861127	

Sample #5		
	mass in grams	error (+/-)
m1	1.104011	0.000001
m2	1.104015	0.000001
m3	1.104013	0.000001
m(avg)	1.104013	
error(avg)	0.000001	
A[mm2]	945.46	
	removed mass	error (+/-)
m1	1.103947	0.000001
m2	1.103947	0.000001
m3	1.103951	0.000001
m(avg)	1.103948	
error(avg)	0.000001	
change in thickness		
	in nm	
change in t	25.3323	
MRR [nm/min]	8.44408	
pressure (psi)	0.751637	


Polishing Experiment Results
30
20
10
0
MRR [nm/min]
0
2
4
6
Sample Number

Appendix E: Error Calculations for the MRR

for MRR					
Sample	**Mp**	**delta M**	**error(dM)**	**error(Mp)**	**Error**
1	1.13076	1.130711	524950.11	−524927.37	0.7
2	1.063641	1.063562	558076.07	−558034.62	0.8
3	1.001482	1.001295	592714.19	−592603.51	0.8
4	0.963641	0.963515	615989.35	−615908.8	0.9
5	1.104013	1.103948	537668.12	−537636.46	0.8

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