STUDY AND ANALYSIS OF CMMRF FOR NANO-FINISHING OF ALUMINIUM ALLOY

A Dissertation submitted
in partial fulfillment of the requirements
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in

Production Engineering

by

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CERTIFICATE

I hereby declare that the thesis entitled "**Study and analysis of CMMRF for Nano-Finishing of Aluminum alloy.**" is an authentic record of my work carried out as requirements for the award of the degree of **Master of Engineering** in **Production Engineering** at **Thapar University, Patiala** under the supervision of **Dr. Ajay Batish**, Professor, Department, Thapar University, Patiala and **Mr. Prabhat Ranjan**, Scientific officer, BARC, Mumbai, during July, 2014 to July, 2015. No part of the matter embodied in this report has been submitted to any other university or institute for the award of any degree.

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It is certified that the above statement made by the student is correct to the best of my/our knowledge and belief.

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ii | P a g e

This thesis is dedicated to my parents.

For their endless love, support and encouragement.

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iv | P a g e

Abstract

Aluminum alloys are widely used in optics as metallic mirrors, micro-electronics at fabrication level and micro-mechanical assemblies like high speed rotating systems towards improvement of friction factor & fatigue failure of rotating elements. In general, the ductile materials like Aluminum & Copper are being machined by Diamond turn machining (DTM) process to get nonmetric surface finish. However it is very difficult to achieve the required surface finishing (surface finish in order of nanometer or sub-nano meter) by conventional random finishing processes like lapping. With newly developed CMMRF (Chemo mechanical magneto rheological finishing) process, experimental trials have been carried out on Al-alloy by altering mechanical properties with the help of chemical reaction (varying type of chemical, type of abrasives and pH value) on the superficial layer of the material. On basis of these experimental trials, 85% improvement has been obtained. A full experimental design has been performed to optimize the process with varying few parameters like, concentration of chemicals, concentration of abrasives, concentration of carbonyl iron particles and working gap. Effect of different parameters has been studied. Average surface finish of less than 5 nm has been obtained, best surface finish of order 0.8 nm has been reported. Hence, the CMMRF process has capability to finish in sub nanometer level/ scale.

Key words: Aluminum; Mirror; AA-7075-T6; Surface finish; Polishing; CMP; MRF; CMMRF.

Table of Contents

List of Figures

List of Tables

Acronyms

% C.V.	Percentage coefficient of variation
2FI	Two factor interaction
AFPs	Advance finishing processes
ANOVA	Analysis of Variance
CCD	Central composite design
CIP	Carbonyl iron powder
CMP	Chemo mechanical polishing/ planarization
CMMRF	Chemo mechanical Magnetorheological Finishing
df	Degree of freedom
DI water	Deionized water
DOE	Design of experiments
EEM	Elastic emission machining
MAF	Magnetic Abrasive Finishing
MR	Magnetorheological
MRAFF	Magnetorheological abrasive flow finishing
MRR	Material removal rate, nm/min.
PRESS	Predicted residual sum of squares
Ra	Center line average roughness value, μm
RMS	Root mean square
RSM	Response surface methodology
UV	Ultraviolet
HVAC	Heating, Ventilation and Air Conditioning
DTM	Diamond turning machine
RSP	Rapid Solidification Processing
MGDA	Methylglycindiacetic Acid
MRF	Magnetorheological Finishing
AFM	Abrasive Flow Machining
3D	Three Dimensional
2D	Two Dimensional
EDM	Electric Discharge Machining

PH	Potential of Hydrogen
TFT	Thin-film Transistor
ER	Electrorheological fluid
NC	Numeric Controller
VLSI	Very Large Scale Integration
XPS	X-ray Photoelectron Spectroscopy
EIS	Electrochemical Impedance Spectroscopy
SEM	Scanning Electron Microscope
ATV	All-Terrain Vehicle
CNC	Computer Numerical Control
ECM	Electro Chemical Machining
EDG	Electro Chemical Grinding
CCI	Coherence Correlation Interferometry
BARC	Bhabha Atomic Research Centre
PED	Precision Engineering Division
Sa	Average Roughness of 3D profile

Chapter 1

Introduction

1.1 Introduction

From last decade the innovation in the field of micro/nanofabrication has given us the opportunity to work in six order of magnitude dimensional range of nm-mm. This range has been used widely in different fields of manufacturing i.e. mechanical (Chenga, et al., 2008), electronics (Luo, et al., 2006), magnetic (Arias, 2006), optical (Jai, et al., 2011) and chemical/biological (Li, et al., 2014) devices having applications ranging from IC controller to sensors and space. Today world desire more efficient, reliable, less maintenance and longer wear to fatigue life systems in our top to bottom approach, the process of manufacturing micro-nano fabrication with precision has a lot of challenges. Almost all micro/nano mechanical processes required less friction, which leads to better performance and longer life span. Surface finish is the critical parameter when technology is proceed toward micro/nanofabrication. Friction can only be reduces effectively by increasing the surface finish of the work piece. Less scratches means more conductive and having better corrosion resistance (Ahn, et al., 2004). As the world entering in the domain of nanotechnology the demand of ultra-precision nano finishing is increasing in present micro/nano manufacturing processes. Highly advanced manufacturing processes have been developed in last few years for manufacturing of highly precise miniature devices with required size, surface roughness and tolerances for various applications. As shown in Figure 1.1. Taniguchi states that these different manufacturing processes can be classified by achieved precision value.

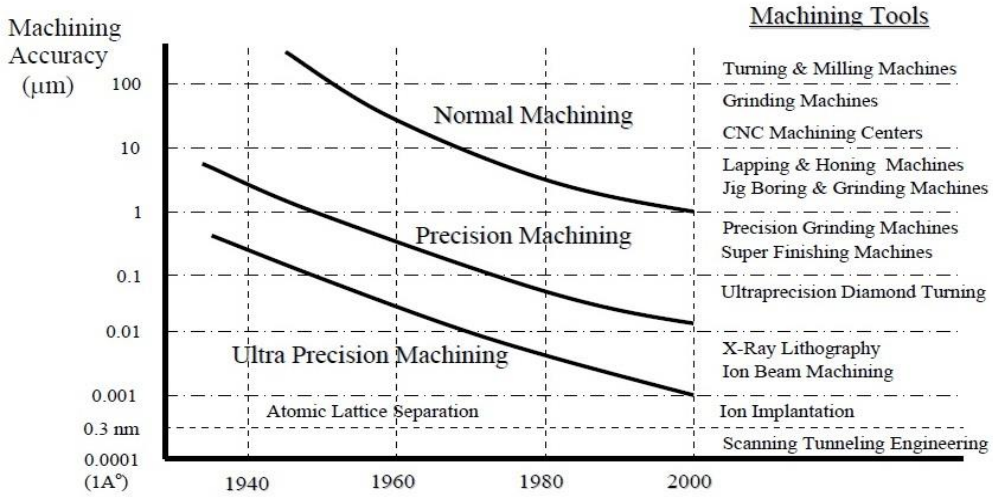

Figure 1.1: Taniguchi Precision Processes (Taniguchi, 1983)

Miniaturization machining also known as micro machining frequently use ultraprecision machining processes for manufacturing of microparts and microcomponents. Micromachining has been evolved drastically from last few years, yet the definition of micromachining is lacking. According to Masuzawa (Masuzawa, 2000) "micro" is having range of 1-999 μm, but according to researchers micro must be the range of machining of difficulties so the proposed and accepted range of micro is 1-500 μm. But it is common to shift the range to 0.1-100 μm with inclusion of its accuracy, size and surface texture (Madou, 2002; Alting, et al., 2003). Beyond 0.1 μm (100 nm) the region defined as nanotechnology. The rage of nanotechnology is defined similar to that of micromachining as 0.1 nm to 100 nm (Corbett, et al., 2000). Theoretically the nanotechnology limits up to the processing of atoms, as diameter of an atom is 1 Å. For better understanding the size scale of micro/nano manufacturing is shown in Figure 1.2.

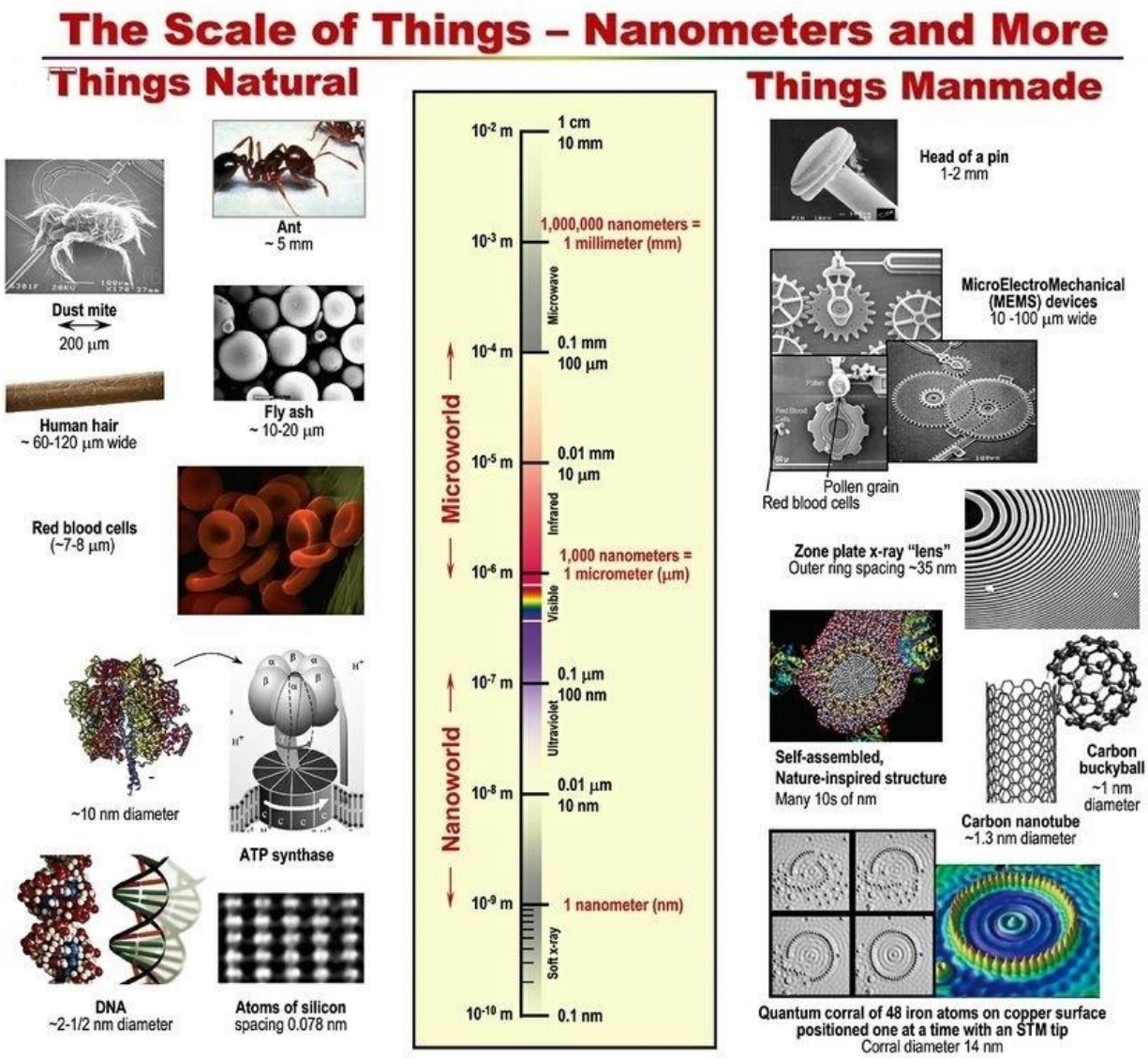

Figure 1.2: Size scale and examples of micro and nanocomponents (Kipper & Murphy, 2013).

1.2 Background of the Problem

As the development in the technology modern industries require high precision components with better quality and longer life. The life and functionality of micro/nanoparts depends directly on the surface finish. There are many materials like aluminium, copper etc. which are widely used in the industries these days, having properties of highly conductive, ductile and light weighted make them special than others materials. But some of their properties also cause problem in the finishing of these metals such as malleability and poor resistance to scratch. Recent a successful attempt has been made for nano finishing of copper alloy, but there has been no lead in finishing of aluminium alloys up to atomic metric level till date.

As aluminium is most conductive and light weighted metal, silver and gold has been used in optical mirror coatings a lot, but now days high quality mirrors use aluminum coatings instead of silver because it tarnishes with exposure to air, specifically toward sulphur and this cause appearance of brown tarnish spots (Boccas, et al., 2005) as time pass so have to polish regularly. These spots cause reduction in reflectivity and an increase in the emissivity. Aluminium also have constant reflectance over the visible spectrum as shown in from 400 nm to 700 nm silver varies from 80 to 95 percent of reflectance while aluminium stays between 90 to 95 percent as shown in the Figure 1.3. Silver metallic mirrors absorb light very strongly at 315 nm, that means if used in telescope and have inserted in UV light form a star, than it will be unable to show anything and there is blank data appears not only this light gathers at wavelength less than 310 nm would require longer processing times. Gold is very costly as compare to aluminium so preferred to use aluminium.

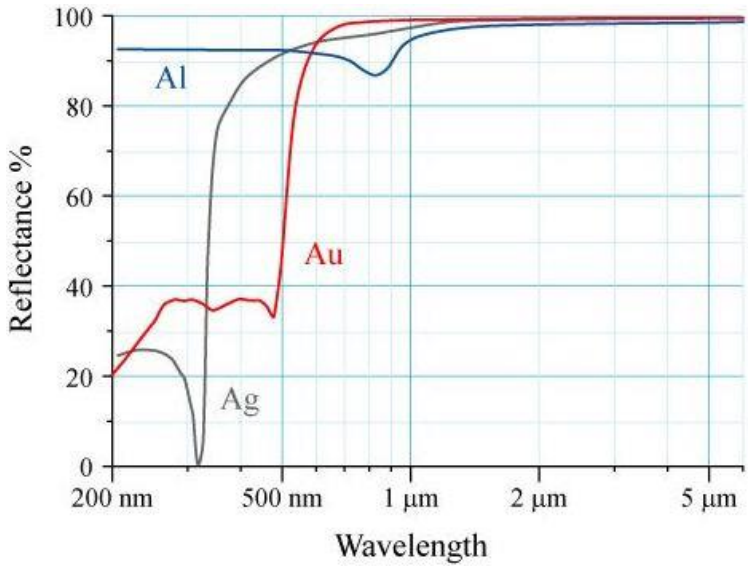

Figure 1.3: Comparison of Au, Ag and Al (Carlos, 2014).

Application of nano finished aluminium alloys arises as the human desire of space exploration. Metal Mirrors of aluminium alloys finish (Newswander, et al., 2013) by DTM process (Gebhardt, et al., 2014; Casstevens, 2011) has been to install in the telescope. These are having properties of light weight, highly conductive, high corrosion resistance and longer wear to fatigue life. Many other major applications of nano finished aluminium is in Optical sensors as diaphragm, high speed rotors, aerospace industries in nozzles, fuel control bodies, turbine sections, combustion liners, fuel spray nozzles and diffusers.

As aluminium have low density, light weighted and have highly conductive 1.85 times more than that of copper so it can be used in energy distribution systems like high-tension wires etc. (Pryo, et al., n.d.). It has high use in HVAC systems because of its highly thermal conductive nature. As its corrosion resistance is directly proportional to surface roughness, so decrease in surface roughness increases its corrosion resistance (Ahn, et al., 2004). With high corrosion resistance make aluminium alloys to use even on water in marine transportation (Tec, 2014).

1.3 Statement of the Problem

As discussed above there are abundant applications of nano finished aluminium alloys, but the problem arises in finishing of aluminium, because it is having properties of ductility as well as malleability. With conventional finishing processes like lapping it is not possible to finish soft metal like aluminium up to atomic level, only way mirror finished aluminium surface is achieved by diamond turn machining (DTM) in order of 10-20 nm. But with highly precision the R_a by DTM process on RSP Al alloys has been achieved up to 1 nm (Horsta, et al., 2012). DTM metal mirror surface have problem of tool feed marks associated with it. Because of this different coatings of nano finished materials like nickel, silver (Boccas, et al., 2005) etc. has been applied on the DTM surface for most of its applications. But there exist problem in bonding of coatings to that of parent aluminium metal occurs over time.

Even when finishing operation is performed with soft abrasive/rubber pad scratches and digs appears on the surface of aluminium alloys which cause reduction in finish. There has been very few studies in obtaining mirror finished up to atomic level on the aluminium alloys, but no one has reported the process till date. Because of its abundant applications the problem of atomic level finishing of aluminium alloy will be addressed in this study.

1.4 Purpose of the Study

Nano finished aluminum alloys having immense applications in optics etc., but no process has been developed for atomic level finishing of aluminum. Purpose of study is to obtain the high quality atomic level surface finished aluminum alloy. So that these can be directly used in different application. Atomic level finishing will improve the efficiency, corrosion resistance and longer wear to fatigue life, comparing to the current scenario of using different coating on aluminum alloys.

1.5 Significance of the Study

Study would be very beneficial for telescope primary mirror construction, as the study will solve most of the problems associated with the telescope metallic mirror construction. Which leads human to explore universe with vast precision and accuracy. The study will also increase the life of the telescope used in space against different odd conditions compare to currently used telescopes. This study not only deals with telescopes but also have a huge influence in increasing the capability of optical sensor systems, micro/nano electronics systems and micro mechanical systems as high speed rotors etc. So the study will open the new phase of technology for betterment of human kind.

1.6 Hypothesis

As aluminium is soft comparing to other metals with properties of ductility and malleability cause it hard to finish. With improvement in hardness by heat treated process and selecting proper chemical for removal of oxide layer formed on the surface of aluminium, and making a proper balance of oxidizer and abrasive is very important. It has been studied that with proper selecting varying variables like type of chemical, concentration of chemicals, type of abrasives, concentration of abrasives, concentration of carbonyl iron particles, pH value, zeta potential, feed, rotational speed, & working gap the atomic level finishing of aluminum alloy is possible.

1.7 Structure of the thesis

Thesis contains six chapters in total having first chapter of introduction, chapter second on literature survey of nano finishing of aluminum alloys with different finishing processes. Chapter third covers the different aluminum grades used and method of finishing by Chemo-Mechanical Magneto Rheological Finishing (CMMRF) process and experimental setup. Chapter fourth describes the procedures and the results, Conclusions and suggestion for future work are presented in chapter fifth. Last chapter covers all the reference of thesis.

Chapter 2

Literature Survey

2.1 Introduction

Aluminum having properties like light weight, high strength, high corrosion resistance, highly malleable and ductile, high electrical and thermal conductivity, cheap, easily available, reuse, make it special comparing to all other metals. Metal mirror of Al is possible by polishing it up to the subatomic level with advanced finishing process and that can be used in different applications in optics like telescope etc.

This chapter gives the fundamentals of nano finishing as a whole with a focus on nano finishing techniques for aluminum alloys. Planarization of Al alloys performed for many applications on MRF and CMP process has also been discussed in this chapter.

2.2 Nano-Finishing Techniques

As shown in the Figure 2.1 conventional finishing processes like lapping, honing and grinding are not capable of producing desired surface characteristics for achieving nano finished metallic mirrors. Even in some of cases these are being used but because of high labour cost and expensive equipment's they are not used for ultra-finished nanocomponents. For overcoming the limitations of these conventional processes many advance processes for achieving nano finish has been developed in last decades. These processes can also finish random surfaces because of abrasive particles use in some of the processes, which leads to finish even very harder metals with better control over surface quality of workpiece.

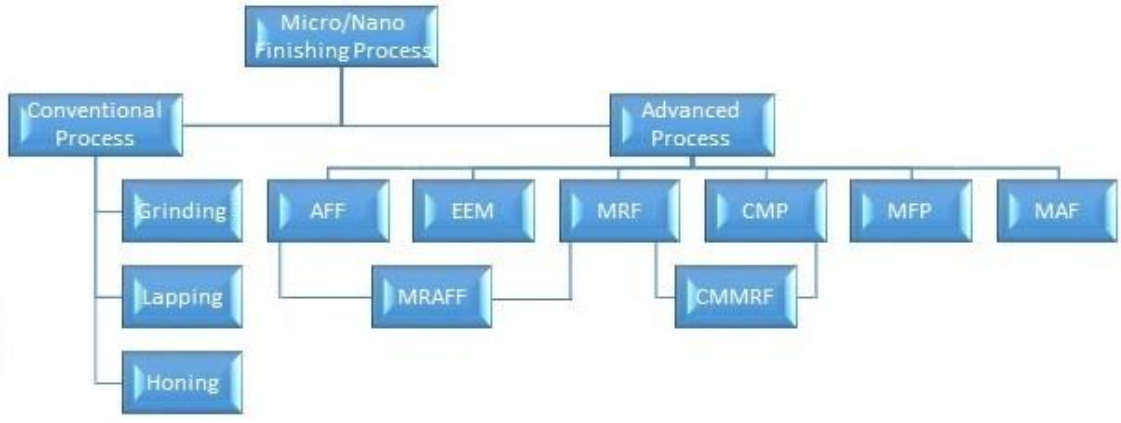

Figure 2.1: Nanofinishing processes

2.2.1 Conventional Finishing Process

1. **Grinding:** - It is the commonly used production process for finishing (Shiou & Hsu, 2008; Peia & Strasbaughb, 2001). In this process workpiece is finished by rotating cylinder containing abrasive particles on periphery. These are two type of grinding processes one which are used for material removal from the workpiece and other for improvement of surface finish on the surface. The significant process parameter (Shaji & Radhakrishnan, 2003) are feed, force, abrasive particle bonding with wheel there size etc. The applications of grinding are in the finishing of basic shapes like cylinder etc. where size is a concern.
2. **Lapping: -** This process generally deals with finishing of workpiece, material removal is negligible compare to other conventional processes. Lapping is performed by applying pressure on workpiece opposite to lap, with different mesh size emery papers.
3. **Honing:** - This process has been used widely for finishing internal surfaces of cylinder (Nagel, 2000) etc. Abrasive particles in shape of stones inserted in oscillating mandrel which also expands has been used for finishing process. The pressure applied in honing process in more than that of lapping.

2.2.2 Advanced Finishing Processes

Advanced nano finishing processes means finishing of workpiece up to sub atomic scale. That leads to removing of material in form of atoms or molecules at individual or in groups. Mostly the nano finishing processes use abrasive particles either missed in liquid to form slurry or held by carbonyl iron particles, viscoelastic materials or by magnetorheological fluids. 3D complex shapes can be finished by (AFM, MRAFF and MRF) some of the processes.

The Advanced abrasive flow finishing processes are mainly classified in two categories i.e.

(a) No controlling of forces externally.
(b) Full controlling of forces externally.

In second case forces can be controlled by varying current in electromagnetic coil or by varying working gap between permanent magnet and workpiece. The basic advances nano finishing processes leads to the development of some of new processes to overcome the limitations of these processes.

(a) Advanced nano finishing processes having no external control of forces:-

1) **Abrasive Flow Finishing (AFF):-** AFF process was used for deburring and finishing the fuel system and critical hydraulic components of aircrafts/shuttles in aerospace industries. Air, fuel and liquid flowing surfaces cab be polished by AFF process. With AFF process EDM and milling surfaces R_a has been improved significantly (Kohut, 1989). Surface finish achieved by AFF process has been found 50 nm. Process can be applied to both macro/micro components only viscosity of medium has to be low in case of micro components. The ability of AFF to finish complex surfaces, leads to number of applications in aerospace, automobile, medical components, precision dies, mould manufacturing and electronics industries. In AFF it is difficult to achieve better finishing rate on intake ports internal passage because of it is complexity (Lam, 2000). AFF uses two vertical opposite cylinders and extrudes pressure is applied on abrasives (SiC, Al_2O_3, Diamond, Boron Carbide) mix in viscoelastic medium as shown in Figure 2.2 (b). Back and forth movement of slurry from a passage formed between workpiece and tooling leads to nano finishing of that area. The viscoelastic abrasive particles are forced to pass from a restrictive passage shows change in viscosity. Material is being removed in the form of micro/nano chips from the workpiece surface by axial force and radial forces causes indentation of abrasive particles on the workpiece as shown in Figure 2.2 (a). Abrasive action can be increased by changing the rheological properties of medium (Rhoades, 1988; Rhoades, 1991). The forces acting on the workpiece is enough to remove a small micro/nano chip and the finally nano level surface finish can be achieved.

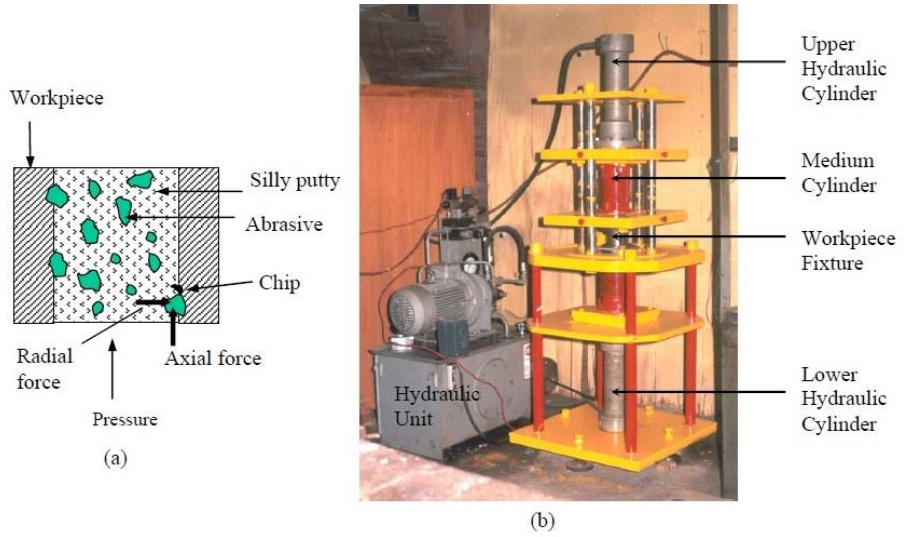

Figure 2.2: AFF (a) Forces on abrasive particle in AFF process (b) Experimental setup (Jha & Jain, 2006)

2) **Elastic Emission Machining (EEM):-** EEM has been developed in Process gives complete mirrored, crystallographically and physically undisturbed (Mori, et al., 1987), ultrafine surface finish by removing material at atomic level with mechanical means. Super fine abrasive of few nanometer size strikes the workpiece surface and remove individual atom/group of atoms by elastic fractures (Mori & Yamauchi, 1987) from the parent metal for giving nano finish up to sub atomic level (Mori, et al., 1988). The surface finish obtained by this process is close to the order of 2 Å to 4 Å. In this process the type and size of abrasive used is critical as it is responsible of material removal which directly leads to finish.

3) **Chemical Mechanical polishing/ Planarization (CMP):-** CMP process has been used widely for higher surface finishing and planarization in semiconductor industries. The process gives better surface finish as it is having both mechanical abrasive based finishing and chemically etching (Stokes & Orent, 982). Which further improves the finishing rate and surface finish. As the workpiece used is cylinder in this process, so relative velocity of both rotating pad and workpiece should be match or it should be varying slight from center to the periphery of workpiece. In CMP chemical slurry composes of chemical used to form passivation layer for softening the parent metal layer and abrasive particles remove this soft layer by mechanical action, CMP process has been shown in Figure 2.3. So combined technique has been used widely for obtaining better surface finish at higher rate. CMP process having polishing pad made of polyurethane in general, abrasive slurry delivery

system, workpiece holder system, polishing slurry compose of abrasive and etchants of different type for PH control.

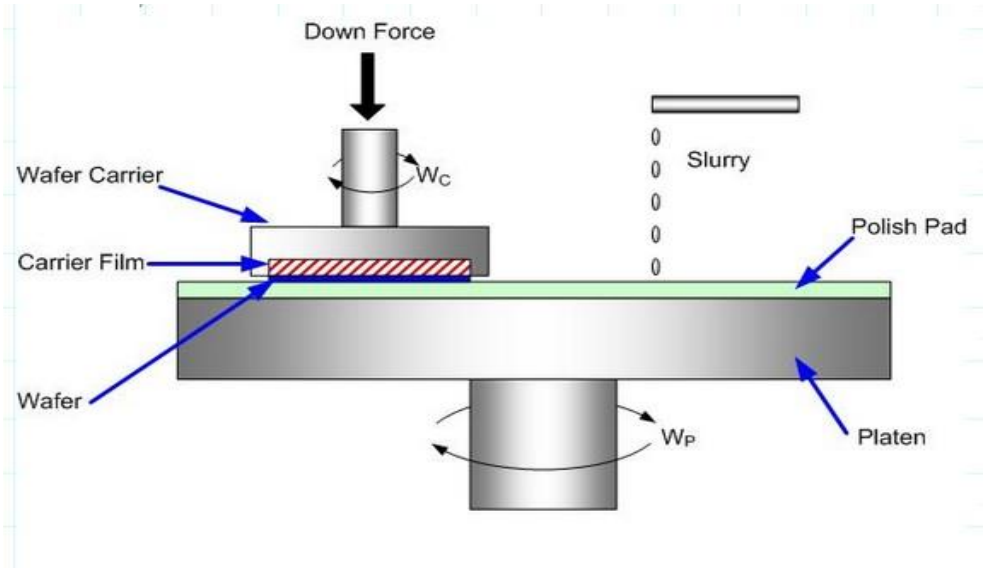

Figure 2.3: CMP Process (Butler, n.d.)

The complex system between workpiece and pad in the presence of slurry is important to understand. In CMP the slurry and workpiece made a reaction product which is then removed by abrasive particles (Nanz & Camilletti, 1995). The CMP process was earlier used for glass polishing than with further enhancement of process leads it to be used for other metal surfaces and mainly for silicon wafer. Comparing to mechanical polishing CMP produces defect free surface (Jhansson, et al., 1989). In 1960 first time CMP process was commercially available. In late 70's and 80's silica based slurry can finish surfaces at higher rates and with high quality surface finish with much reduction in process time. It is expected that CMP will overcome number of problems associated with it like pitting because of brittle fracture, scratches caused by abrasion and surface damage occurring due to hard abrasive particles (Komanduri, et al., 1997). Now days with a lot of research in CMP leads it to finish any material and (flatness and finish) is even better than silica based slurry. CMP is also used in the field of thing film transistor (TFT) technology (Chang, et al., 1996).

(b) Advanced nano finishing processes with external control of forces:-

Magnetorheological Fluid: - After ER fluids reported in 1948 by Wilson, Rainbow reported magnetorheological fluids (Rabinow, 1948). After that research has been focused on understanding the behavior of MR fluids, how to develop better quality and high strength fluids is undergoing. Klingenberg (Kingenberg, 2001) has reported different properties and

applications of MR fluids for commercial use. Oxidization of iron particles, sedimentation of particles and there cost has been area of higher priority.

4) **Magnetic Abrasive Finishing (MAF):-** For finishing metals and ceramics newly developed magnetic abrasives are emerging as most efficient way. MAF can finish tubes (vacuum and sanitation pipes (Shinmura, 1987)) and flat surfaces with high level of finishing. MAF can finish both magnetic as well as nonmagnetic materials.

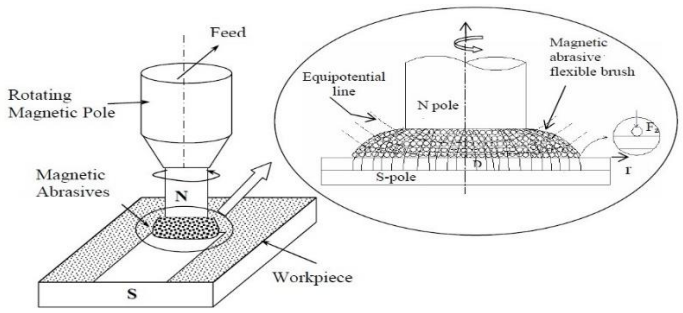

Figure 2.4: MAF Process (Jha & Jain, 2006)

As shown in Figure 2.4 ferromagnetic material particles used with different abrasives (SiC, CBN, Al_2O_3 or diamond) for finishing. As the force acting by soft pad formation by ferromagnetic particles and abrasives is very less, but it is controllable leads to mirror like finish obtained. The surface finish obtained by this process is of few nano meters, the Ra of 7.6 nm has been reported on stainless steel rollers (Fox, et al., 1994). Surface finish, deburring (burrs has been removed from the surface without degrading accuracy of shape). Magnetic field acting as a binder, keeps the powder in gap causes the slurry to be pressed against the workpiece (Kremen, 1994). Magnetic force can be controlled by controlling the magnetic coil (Jain, et al., 2001). MAF is highly efficient process and the removal rate and surface finish depends upon the volume, shape and size of abrasive particles, workpiece to be finished and magnetic flux density etc.

5) **Magnetic Float Polishing (MFP):-** MFP is mainly used for finishing ceramic, as ceramics are very hard to machine the applications in the field of ceramic are limited. Machining of ceramics conventionally is done by diamond abrasive particles. These cause defects on the ceramics surface like pits, micro scratches and micro cracks. For planarization and finishing MFP is the best choice existing. For removing these defects ferromagnetic based abrasive particles which are gentle compare to diamond are used in high magnetic field with a non-magnetic float as shown in Figure 2.5.

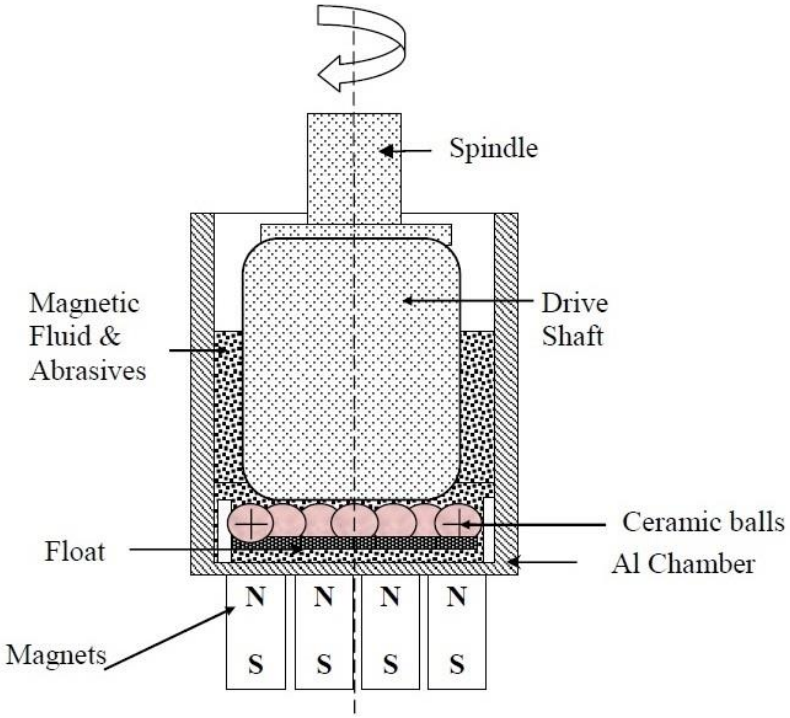

Figure 2.5: MFP Process (Jha & Jain, 2006)

In MFP balls are polished because of relative motion between them and magnetic abrasive under resistance and levitation force (Tani & Kawata, 1984). With finer ferromagnetic abrasive particles and stronger magnetic field higher MRR and surface finish can be achieved. Slurry is made of fine abrasive particles and with ferromagnetic particles in carrier medium like water and kerosene which are filled in Al chamber. The surface finish obtained with MFP is 4 nm Ra on Si_3N_4 free from many surface defects like scratches, pits and cracks. These are being used in ultra-high speed spindles and jet turbine in aircraft as high speed bearing.

6) **Magnetorheological Finishing (MRF):-** MRF process contains smart fluid known as magnetorheological (MR) fluids. MR –fluids contains micron sized suspended carbonyl iron particles. These particles are dispersed in different carrier mediums such as water, silicon oil and mineral oil. Without a magnetic field MR-fluid having Newtonian behavior (random distributed particles), with apply magnetic force particles form dipole moment which is proportional to the applied magnetic field strength and then all get aligned in the field direction. Again when the magnetic field is removed particles again come to their original random state and show Newtonian behavior. This behavior of MR-fluids has been defined by Bingham Plastic model (Kordonski, et al., 2001) as shown in Figure 2.6. As the

MR- fluids properties can be manipulated electronically, this focused the attention of industry and researcher's for using MR-fluids (Klingenberg, 2001). These having abundant applications in the fields like breaks, shock absorbers, damping devices, actuators, clutches and in artificial joints.

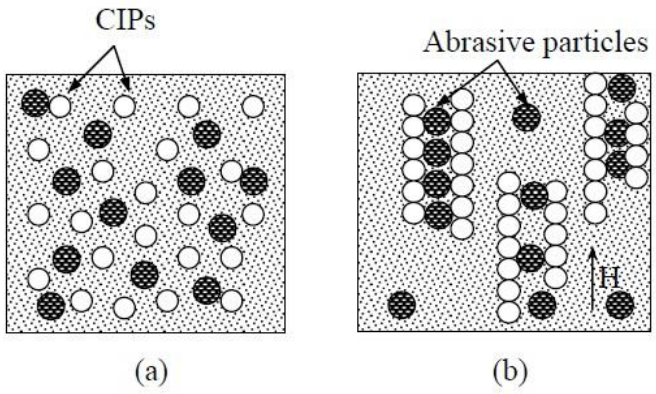

Figure 2.6: Behavior of MR-fluid (a) MRP-fluid without magnetic field (b) MRP-fluid after applying magnetic field (Jha & Jain, 2006).

Finishing rate can be controlled by controlling the magnetic field strength and direction. MR-fluid is very effective in finishing lens, optical glasses, plastics, glass ceramics and non-magnetic materials (Lambropoulo, et al., 1996), it was first developed by Kordonski and Jacobs they also calculated MRR of the process (Kordonski & Jacobs, 1996) shown in Figure 2.7.

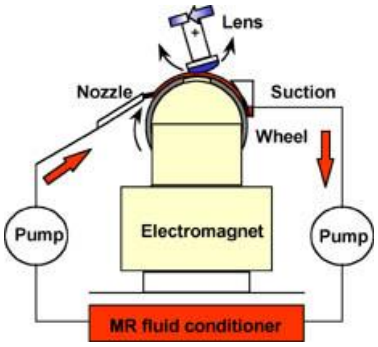

Figure 2.7: MRF Process (Jacobs, n.d.)

Flat, convex and concave surfaces can be finished by this process. The material removal in MRF is through shear stress. Using numeric controller (NC) aspheres, spheres and flat surfaces can be finished by mounting the workpiece on rotating spindle, MRR can be determined by dwell time. In MRF surface finish of order 10-100 nm has been obtained without any surface defects which were there by conventional processes.

7) **Magnetorheological Abrasive Flow Finishing (MRAFF):-** As the abrasive force in AFM is least controllable by external means because the behavior of polymeric medium slurry is not known. For overcoming from this limitation magneto rheological fluids have been used of MRF process. In MRF process force can be controlled by changing the magnetic flux density, strength of magnets etc. So the new hybrid process named as MRAFF (Jha & Jain, 2004) has been develop having properties of both AFM and MRF. This new process shown in Figure 2.8 having the capability to finish complex internal geometries to sub nano metric level.

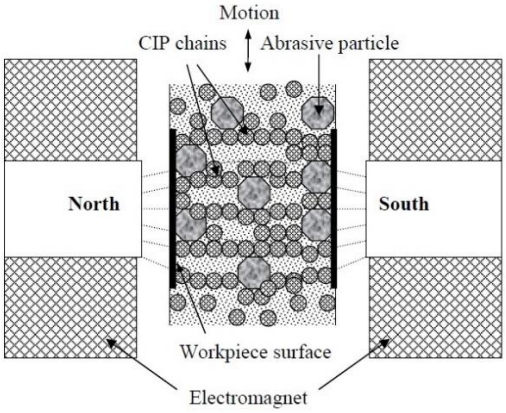

Figure 2.8: MRAFF process (Jha & Jain, 2006)

The magnetic force provides bonding between CIP and abrasive particles as CIP's form chains holding abrasive particles in it. The magnitude and bonding strength is a function of CIP concentration, magnetic permeability of particles, magnetic flux density and size of particles. The material is removed only from the area where magnetic field is applied and leaving other areas unaffected. There are many other process variables that have an effect on the performance of MRAFF process are extrusion pressure, MR-fluid composition and number of finishing cycles etc. In MRAFF the size of CIP and abrasive is having important role in the finishing process.

8) **Chemo-Mechanical Magnetorheological Finishing (CMMRF):-** CMP process having the limitation of polishing only circular workpiece as it does not apply uniform pressure in any other workpiece throughout. And also the abrading forces in CMP are difficult to control externally which leads to an uncontrollable finishing rate and surface finish. Where as in MRF because of development of high level of stress, surface damages are common in this process. With the combination of both the processes the controllable pad of magnetic particles has been achieved which controls the finishing rate and surface finish with the

direction and strength of magnetic force applied. Newly hybrid process named as chemo-mechanical magnetorheological finishing (Jain, et al., 2010) formed by combination of CMP & MRF shown in Figure 2.9 has removed the weakness of both the processes and gain the strength of both so having high degree of surface flatness and finish by externally controllable system.

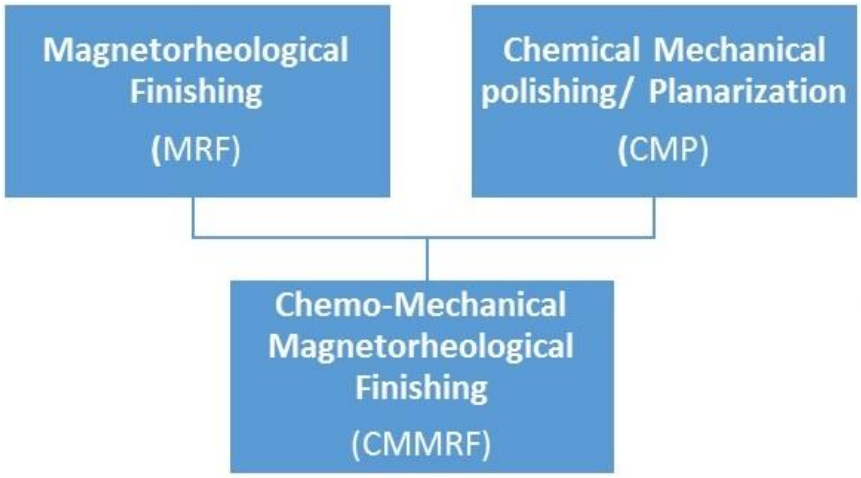

Figure 2.9: CMMRF Process

Abrasive cutting force and holding forces can be calculated in real time in CMMRF process by controlling magnetic field strength and working gap of workpiece and slurry. This helps in removing and etching the material from selective areas on workpiece. The Setup of CMMRF is almost similar to MRF so it can finish any kind of flat, convex and concave surfaces. In CMMRF process higher finishing rate with high surface finish in order of 4.6 Å in silicon (Jain, et al., 2010) and 2 nm (Ranjan, et al., 2013) in copper can be achievable. With CMMRF process all kind of materials can be finished up to sub nano metric level by selecting proper slurry. CMMRF process has finished Silicon, Copper (Ranjan, et al., 2013) and stainless steel to nano level surface finish till date. The Figure 2.10 showing the schematic diagram of CMMRF set-up.

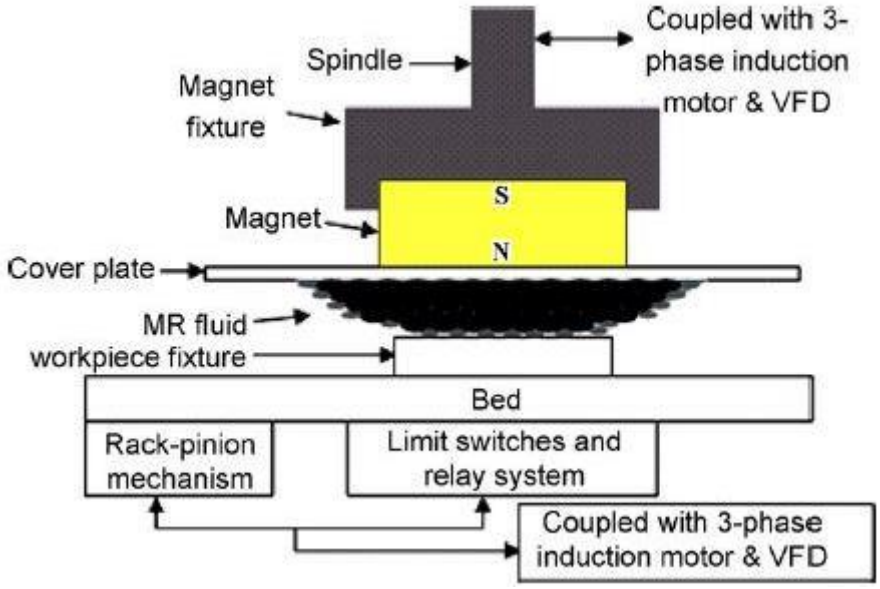

Figure 2.10: CMMRF set-up (Jain, et al., 2010)

2.3 Literature Review

Wang et al. (Wang, et al., 1998) studied the CMP polishing of Al alloy thin sheets for Damascene. As aluminum having low resistivity, patterning and ease of deposition it has been widely used material in VLSI for conductive wires. Different Al sheets prepared for experimental work are pure Al and aluminum alloys having composition of Al±0.5% Cu, Al±1.0% Si±0.5% Cu, Al±1% Si and Al±1% Cu. Aluminum films has been deposited on oxidized silicon wafers. Slurry for polishing used is hydrogen peroxide as an oxidizer for formation of passivation layer on Al. Abrasive used for the experimental work is alumina having 5% vol. and 0.05 µm average size of particles. Slurry PH is maintained by controlling potassium hydroxide value and mixed in DI water. Abrasive removes the protective layer formed by hydrogen peroxide, while H_2O_2 again form the layer again. As the cycle of formation

and removal of protective layer continues the planarization achieved. Removal rate increases when Al is incorporated with copper. As the grain size increases removal rate decreases and vice versa. H_2O_2 should be between 2.5 to 3.75%, higher removal rates has been achieved in this range. Higher H_2O_2 cause lower PH results in acceleration of corrosion rate. More refined grain size and higher concentration of copper is recommended for better CMP polish rate.

Smialowska (Smialowska, 1999) studied the metastable and stable pitting corrosion of aluminum, chemistry of pitting and inhabitation was discussed. It was found that pit depth is a function of time, results shows that pit depth increases as square root of time. There are two types of solid salts formed in the pits i.e. aluminum chloride ($AlCl_3$) at PH 1 and aluminum oxychlorides $Al(OH)_2Cl$ and $Al(OH)Cl_2$ at PH 3. Chromates as an inhibitors were studied, it was found that chromates undergo reduction inside the oxide film. With addition of chromates the pit size of metastable pits also starts decreasing. Inhibitor process involves absorption of chromates on defective area, so to reduce surface area to increase passive properties of film. Also chromates after absorbed by defective areas form chromium oxide, which trends to increase the protective property of film against chlorine attack. Chromate ions can protect the surface by decreasing corrosion potential as they can increase the PH in neutral solution. It has been observed that high concentration of chromates are required to decrease pit growth in Al.

Kuo and Tsai (Kuo & Tsai, 2000) studied the effect of potential applied on the Al CMP finished process in phosphoric based slurry. Results shows that at 3 and 9 psi contact pressure cause enormous change in passivation layer. Results also indicated that the MRR depends on potential applied and contact pressure, i.e. with increase in contact pressure MRR increases. Slurry in rotating condition can have increased passive current density of order two and having a decreased corrosion potential of 360 mV. At catholic polarization chemical removal is absent and MRR is caused by only mechanical wear so low MRR is observed. Results also shows that at large anodic potential the chemical and mechanical removal rate of passivated film trends to equal almost giving a higher MRR, at this stage MRR becomes independent to applied potential.

Kuo and Tsai (Kuo & Tsai, 2000) studied Al electrochemical behavior in CMP having phosphoric based slurry. Abrasive particles of Al_2O_3 having size 50 nm has been used to explore the effect of contact pressure and slurry flow rate on electrochemical behavior of Al. Results indicated that contact pressure and platen rotating speed is inversely proportional to the corrosion potential while dissolution rate having direct proportionality with both. Citric acid is used to maintain the PH of the slurry, having composition of 5 vol. % H_3PO_4 + 0.5M citric

acid. The results shows that removal rate is 700 Å/min where only 13% is contributed by mechanical polishing.

Kuo and Tsai (Kuo & Tsai, 2001) studied the effect of alumina and hydrogen peroxide on CMP of Al alloy in phosphoric acid based slurry. Al alloys having composition 0.1 Si, 0.2 Fe, 0.01Ti was used as workpiece material. Slurry contains 5 vol. % H_3PO_4 + 0.5M citric acid. Potassium hydroxide was used to adjust PH level at 4 for slurry. Al_2O_3 having average abrasive particle size of 0.05 µm has been used. Process runs on 100 rpm and have a down force of 5 psi. Results shows that removal rate of passivation layer is slower than it is formation rate i.e. even when large amount of abrasive is present in the absence of oxidizer like hydrogen peroxide. Results shows that 1 wt. % of alumina is enough to remove passivation layer in CMP process. Material rate in aluminum free solution is 42 $Åmin^{-1}$. Maximum removal rate achieved when alumina is 10 wt. %, after that saturation in MRR is observed. Reason on this saturation of MRR even with increase in abrasive quantity is not understood. Maximum repassivation rate is achieved at low quantity of H_2O_2 i.e. 1 vol. %. Analysis is done by electrochemical impedance spectroscopy (EIS) and X-ray photoelectron spectroscopy (XPS).

Chiu et al. (Chiu, et al., 2003) studied the CMP of Al with Ti barrier layer for application in electrochemical metrologies. Study is focused on finishing of Al alloys coupled with titanium in phosphoric acid and H_2O_2 based slurries. Slurry has been prepared by 5 wt. % γ-Al_2O_3 having size of 0.05 µm with H_3PO_4 and H_2O_2. Required PH level is maintained by citric acid and potassium hydroxide. The MRR of CMP process depends on H_2O_2 concentration. If it is more than 3 vol. % than polishing rate is less than oxidation rate. Removal rate of Al decreases with the increase in PH value and vice versa for Ti. Pressure while polishing Al should be less as 4 kPa. Results indicates that 6 vol. % H_2O_2 at 4 PH level can mitigate Al dishing and corrosion attack.

Cho et al. (Cho, et al., 2003) studied the effect of polishing pressure and abrasive concentration on CMP for better planarization of Al thin films. Thing Al of thickness 1.8 µm is deposited on silicon oxide coated wafer. Slurry was prepared by 5% mixture of acids (he slurry was made by adding 5 vol.% of a mixed acid solution (10 vol.% HNO_3, 25 vol.% H_2SO_4 , and 65 vol.% H_3PO_4) in de-ionized water. To maintain the PH of slurry near 2.0 $K_2Cr_2O_7$, citric acid and phosphoric acid with oxidizer as H_2O_2 is used. Results shows that with increase in polishing pressure roughness decreases but waviness and MRR increased. It is observed that with smaller size abrasive particles R_a improves, also concentration of abrasive effect R_a i.e. when concentration is lower as 0.1 wt. % R_a deteriorates a bit. But at low concentration such

as at 0.1 wt. % waviness improves. It was observed that at 10 kPa and 5wt. % abrasive concentration better R_a, waviness and reflectivity can be achieved.

Ahn et al. (Ahn, et al., 2004) studied the effect of PH, abrasive and oxidizer concentration on MRR and surface roughness. Results of fumed silica based slurry compare to the traditional alumina based slurry comes better. Size of abrasive particles used is 20 nm and concentration is 5 vol. %. While H_2O_2 optimal concentration found at 1-3 vol. %. Rv of 6.08 nm has been observed with colloidal silica compare to alumina of 18.15 nm. Less number of micro scratches observed at 1-2 PH level. Micro scratches size decrease with smaller size of abrasive particles and concentration. Measurements were carried out using sample rate of 10 mm and cutoff length of 0.08 mm having scanning speed of 25 µm/s with Dektak. Removal rate of Al was measured with four-point probe.

Hernandez et al. (Hernandez, et al., 1999) studied CMP of aluminum thin films with Al_2O_3 abrasive. Aluminum has been polished using two different pads, which vary in hardness as well as structure. Results shows that MRR depends upon the polishing pad pressure, linear velocity applied and type of pad used. For study of surface with different polishing conditions scanning electron microscope (SEM) and X-ray photoelectron spectroscopy was used in the process. Pad surface found saturated after experimentation without any aluminum on it. Results also shows that strong oxidizer (H_2O_2) has no effect on the material removal rate. It has been found that Preston's equation is not describing correctly the dependence of pressure and velocity on the removal rate. Pressure is more significant parameter compare to linear velocity in the process. The pad used in the polishing has high effect on the removal rate, hard pad causes more MRR. It has been found that strong oxidizer like H_2O_2 not required for polishing of Al like reactive metals.

Wrschka et al. (Wrschka, et al., 1999) studied the different polishing parameter on Al thin films by CMP process. Pressure of 19 to 47 kPa with linear velocity 26 to 48 mm/min and Al removal rate of 80 to 250 nm/min are used for conducting the experiments. Study revealed that Preston's equation fails to explain the process that's why power function is being proposed. It been found that oxidant has no effect on polishing rate in case of Al. For examining the surface of Al before and after the experimentation X-ray photoelectron spectroscopy has been used. The results shows the relation between the thickness of passivated layer and removal rate. Different size and shape of abrasive particles having different and significant effect on the surface of Al. Growth and removal of layer should be at same rate for achieving better surface quality. It has been found that thickness of passivation layer is inversely proportional to the removal rate.

Wang et al. (Wang, et al., 2005) studied the CMP of Al thin films, there mechanism and slurry chemistry and found with increase in PH of slurry the removal rate decreases. The experiments were conducted on two kind of slurries having PH<4 and PH≥4. Results shows addition of a strong oxidizer like H_2O_2 of concentration of 0 to 15 vol. % at PH of 2 have both formation of passivation layer and mechanical abrasion at equal rate, leads to an optimal removal rate. When PH is maintained at 4 it has been observed that material removal rate depends mainly on concentration of H_2O_2, and chemical and mechanical part of CMP process are not equating each other cause non uniform removal rate. Slurry of PH low seems scratching on the surface and also material loss, while too high in PH of slurry cause non uniform removal rates. α-Al_2O_3 has been used as an abrasive particles of size 0.3 and 0.05 µm, Ra of 7.7 and 6.5 nm is achieved. Larger removal rate was observed by smaller abrasive particles because of increase in contact area.

Jain et al. (Jain, et al., 2010) studied polishing of silicon for electronics applications with newly develop process called as chemo mechanical magneto rheological finishing process (CMMRF). Process has been developed by combining two different finishing processes i.e. CMP and MRF. The process has advantages to both process, in this chemical portion is used to enhance the surface finish quality and magnetic part helps to control the forces externally. Different abrasives (SiO_2, Al_2O_3 or CeO_2) mixed with ferromagnetic particles also called as CIP. Glycerol added to reduce agglomeration and sedimentation of slurry. Finishing with oil and water based carrier has been used and found better results with water. Optimizing of different process parameters have been conducted by CCD model. Ra increases with spindle speed and decrease with finishing time. Finishing time, working gap and rpm of the process has been optimized. And found 20% error comparing with DOE. The best surface finish obtained on silicon wafer is 4.8Å.

Skubal et al. (Skubal & Walters, 2013) studied chemical polishing of Al in vacuum chambers. Alumina and DI water in 1:1 ratio has been used to finish the internal surfaces of small aperture undulators. Time. Temp and composition has been controlled for finishing operation to run smoothly. 40% reduction in RMS value of surface has been obtained by finishing. Temp from 110 to 120°C, and time 0.5 to 4 min used. It has been observed that a hotter solution used has high MRR i.e. it dissolve more mass compare to a cooler solution. The samples rinsed with DI water were having more surface finish comparing to the samples rinsed in nitric acid.

Ranjan et al. (Ranjan, et al., 2013) studied the CMMRF process for obtaining super finished copper alloys. Mechanism for finishing of copper by chemically forming soft brittle

layer of oxide and hydroxide with ammonia and nitric acid has been obtained. The layer then removed by mechanically Zeta potential has also been controlled up to 30mV because less zeta potential causes agglomeration of particles. Corrosion inhibitor BTA was added and workpiece was fully submerged in slurry. It has been found that at 3.7 PH value best surface finish of 2.5 nm obtained. Large quantity of ammonia causes Ra to decrease but even less quantity of ammonia would give large porous surface and decrease the reflectivity of the surface. Even ammonia trends to zero more surface defects are visible. Nitric acid has been added causing removal of peaks of workpiece whereas the valleys are being protected by BTA. Results show that CMMRF process can finish the monocrystalline materials up to sub nano level.

Ranjan et al. (Ranjan, et al., 2014) studied the effect of polishing pressure with effect of different process parameters. Some of the parameters are finishing time, Working gap, PH value, rotation speed of spindle, magnetic particles etc. Finite element simulations are performed to understand variation of magnetic force, magnetic field and pressure applied with working gap. Higher working gap less magnetic force is applied Results show that with increase in volume of MR fluid pressure applied at same working gap increases up to an optimum value than start decreasing. This is because at higher working gap low magnetic field is available. At fixed working gap in the process will not give more pressure than the optimal. Also decrease in gap leads to lesser optimum volume of slurry. So both gap and volume of slurry must be controlled efficiently for better results.

2.4 Scope and objectives of the present study

The new hybrid process CMMRF has ability to finish all kind of surfaces i.e. flat, convex and concave etc. It has overcome the limitations of both the processes CMP and MRF. In this process finishing rate and surface finish both can be controlled by varying strength of magnetic field applied. Following are the specific objectives of present study.

1) Synthesis of the magnetorheological polishing fluid for finishing of Aluminum workpiece.

2) Study the effect of process parameters (abrasive mesh size, abrasive concentration, rotational speed of magnet, working gap and finishing time) on surface finish improvement.
3) Conduct of experiments to understand the effect of magnetorheological polishing fluid composition on surface finish improvement.

Chapter 3

Materials and Methods

3.1 Introduction

This chapter contains information about experimental setup, different machines and measuring instruments used for nano finishing. Primary experimentation done for polishing of Al alloys, also been reported in this chapter and results are discussed. Magnetorheological polishing fluid required for Al alloy finishing has been reported. Chapter also contains mechanism of finishing of Al alloys. And the chemical reactions that are governing finishing up to sub atomic level. Different process parameters has been discussed in this chapter and technique used for optimization of those process parameters is described.

3.2 Material Used Al 7075-T6

Material used for initial experiments for finishing is Al alloy is 6061. After initial experiments has been performed on 6061, with the results of primary experiments 7075-T6 has been chosen as the material to be worked on. It is chosen because of its hardness nearly equal to SS304 i.e. it is a high strength material also having higher corrosion and crack resistance. Composition and properties of Al 7075-T6 has been reported in Table 1 and Table 2.

Table 1: Composition of Al 7075-T6

Component	**Wt. %**
Al	87.1-91.4
Cr	0.18-0.28
Cu	1.2-2
Fe	Max 0.5
Zn	5.1-6.1
Mg	2.1-2.9
Mn	Max 0.3
Si	Max 0.4
Ti	Max 0.2

Table 2: Properties of Al 7075-T6

Properties	Metric
Density	2.81 g/cc
Hardness, Brinell	150
Hardness, Knoop	191
Hardness, Rockwell B	87
Ultimate Tensile Strength	572 MPa
Tensile Yield Strength	503 MPa
Elongation at Break	11 %
Modulus of Elasticity	71.7 GPa
Poisson's Ratio	0.33
Fatigue Strength	159 MPa
Fatigue Strength	29 MPa-mΩ
Machinability	70 %
Shear Modulus	26.9 MPa
Shear Strength	331 GPa
Electrical Resistivity	5.15e006 cmΩ
Thermal Conductivity	130 W/m-K
Melting Point	477-635°C

Applications of Al 7075-T6:- Aerospace, defense, gear and shafts, missile parts, worm gear, bike frames, all-terrain vehicle (ATV), fuse parts, aircraft fitting, keys, meter shaft and gears, regulating valve parts and extensively in Boeing aircraft.

3.3 Experimental Setup and Meteorology Equipment's

3.3.1 Micro tools Nano-finishing machine

As shown Figure 3.1 in Micro tools Nano-finishing machine has been used for finishing the Al workpiece. The machine is available at Precision Engineering Division (PED) in BARC, Mumbai. Machine is having 3-axis CNC control with a rotary spindle. Slurry circulation, slurry control and finishing tank are the part of the machine. The magnet used for holding the magnetic particles (CIP) is of 6 Tesla. Magnetic field has been measured by Gauss meter and found 5.9 Tesla at center and 3.2 Tesla at sides of rectangular magnet.

Figure 3.1: Micro tools Nano-finishing machine

3.3.2 Workpiece, Fixture and Template

For holding workpiece shown in Figure 3.2 (b), the fixture has been made by same material having thickness of 15mm. For placement of workpiece in square cavity has been created and for proper placement and tightening of workpiece in the cavity screw has been provided as shown in Figure 3.2 (a).

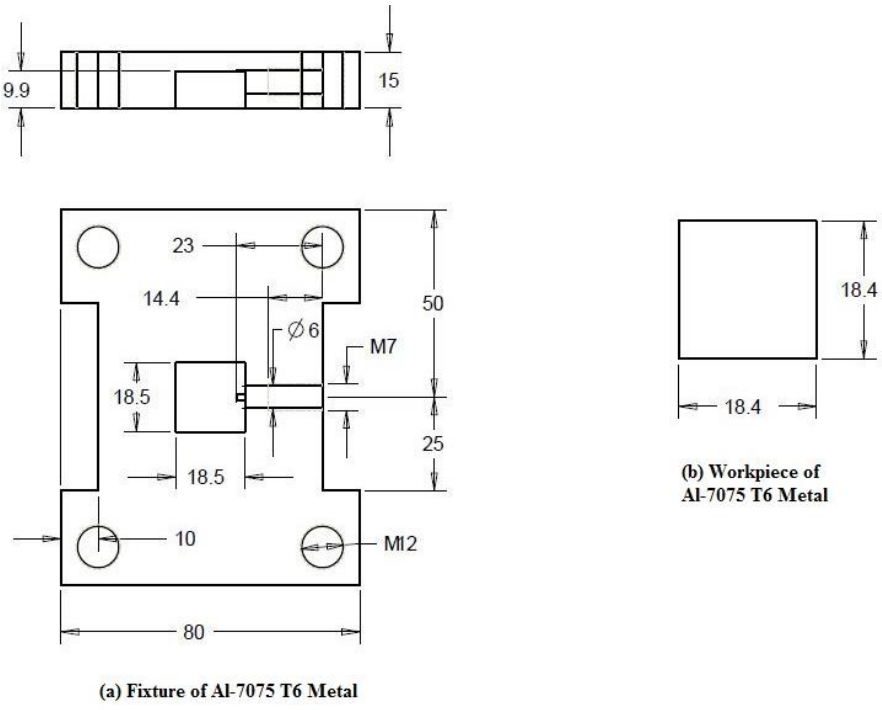

Figure 3.2: Fixture and Workpiece

For calculating Ra of surface of workpiece at same area template has been prepared as shown in Figure 3.3. Fixture has been made on Perspex material on DT-110 with micro milling and micro drilling operations.

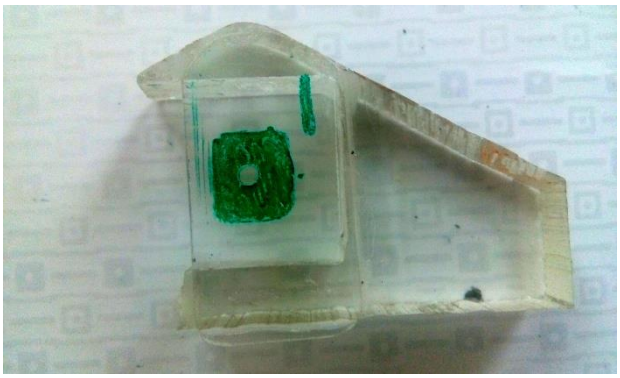

Figure 3.3: Template for Ra Measurement

3.3.3 Micro tools DT-110

Micro tools DT-110 has been shown in Figure 3.4 is used. The other name of machine is universal micro machining center because of the capability of machine to perform different operation like micro drilling, micro turning, micro milling, micro EDM, micro wire EDM, micro wire EDG, micro ECM. In this study by using micro milling operation width of 500 µm and depth of 25± 2 µm slot has been made. The slot is made to study the material removal rate from the surface of workpiece. The machine facility is available at Precision Engineering Division in BARC, Mumbai. The machine has positional accuracy of ±1 µm.

Figure 3.4: Micro tools DT 110, Singapore. Model: Multipurpose Micro machine

3.3.4 Taylor Hobson precision, Taly-surf CCI

Taylor Hobson precision, Taly-surf CCI (coherent correlation interferometry) machine as shown in Figure 3.5 has been used for metrological purpose. The surface roughness parameters such as Ra and Sa were measured before and after finishing operation. For determining MRR of the process slot made on DT110 has been investigated on CCI. CCI machine used for all the measurement is having Z resolution of 0.01Å. 3-D and 2-D profiles of surface has been observed. Different type of analyzing techniques has been applied with the help of Taly-Map-Gold software. Some of them are leveling of surface, removal of form error, generation 3-D surface, extracting profile for surface measurement in 2-D i.e. Ra. Measurement of slot depth before and after, calculating scratch to dig ratio etc.

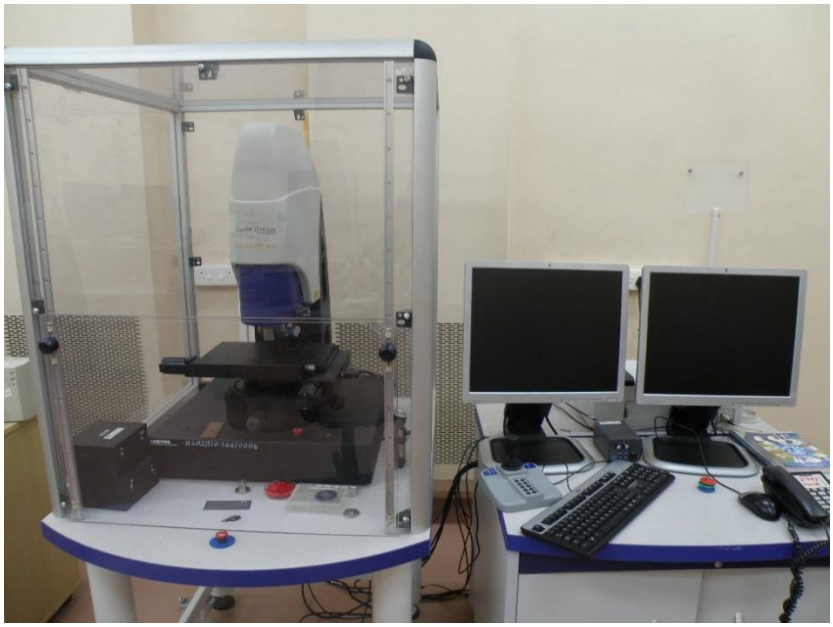

Figure 3.5: Taylor Hobson precision, Taly-surf CCI

3.4 Magnetorheological Polishing fluid

Water based MR fluid deionized water containing carbonyl iron particles (magnetic particles for creating soft pad) with abrasive particles of fumed silica (SiO_2, Al_2O_3) are used. As magnetic particles are denser than abrasive so settling of CIP occurs commonly. Also to protect the CIP from forming of large particles can be avoided by using glycerol in the water. Glycerol form a protective layer around the particle which reduce the inter particle interaction. Which also leads to reduction of Van Der Waals forces of attraction.

Abrasives: - There are two type of abrasive available which are chemically active in reaction and other are non-active.

Magnetic Particles: - Carbonyl iron particles and Fe particles are mainly used.

DI water: - Deionized water activates the chemical reaction to undergo.

Glycerol: - To avoid agglomeration of particles glycerol has been used.

3.5 Initial Sample Preparation

- Al 7075-T6 workpiece used of size 18.5 X 18.5 mm square workpiece has been prepared by facing operation on conventional lathe.
- Lapping operation has been performed on all the work pieces with different grit size emery papers i.e. 600, 1000, 1500, 2000 and 3000 size.
- Lapping operation has been done after creating the micro cavity on workpiece by diamond paste having average grain diameter of 250 nm.
- Samples prepared has been cleaned with acetone, for removal of all the dirt, oil and abrasive particles remaining after lapping on Al surface.

3.6 Mechanism of Finishing

In CMMRF finishing take place by the combination of both chemical as well as mechanical erosion. Magnetic particles form a soft brush holding abrasives causing mechanical removal of material, chemicals in the DI water based slurry causes the formation of passivated layer which is being removed by abrasive particles and magnetic particles.

Both the processes i.e. chemical action and the mechanical action acting on the Al workpiece has been explained.

Chemical Reaction: - In Al slurry of DI water based glycine, glycol, citric acid, ammonia and fumed silica has been added.

1) Glycine, ammonia and citric acid acting as a ligands reacts with aluminum surface.
2) There is a formation of methylglycinediacetic acid (MGDA) forming a yellow colour layer on the workpiece.
3) In the ratio of 1:1 mol MGDA chelates binds with the 1 mol of metal ions forming 1:1 complex, as these are being stable on a wide range of PH i.e. 2 to 13.5 and high temp even up to 100°C, especially in alkaline medium (BASF Aktiengesellschaft Performance Chemicals for Detergents and Formulators, 2007).

4) As aluminum is quickly corrode in strong base and it is also not resistant to MGDA so it is used for AL-7075-T6 (BASF Aktiengesellschaft Performance Chemicals for Detergents and Formulators, 2007) .
5) As shown in Figure 3.6 Al bonding with glycine and citric acid formation (Rao & Kumar, 2014).

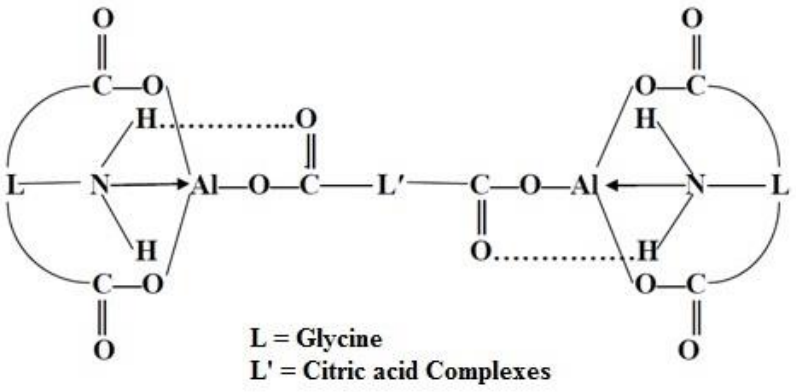

Figure 3.6: Chemical bonding of Al (Rao & Kumar, 2014).

6) Glycine in alkaline solution form glycinate anion and bound with Al cation forming a layer on Al surface (Bonaccorsi, et al., 1984), glycine is also used for potentially causing the redistribution of metals and mobilizing them, especially in the case of aluminum (CureZone, 2009).
7) With high surface energy at peaks than that at valleys, aqueous ammonia react at peaks converting brittle layer by passivation on to the surface.

Mechanical Action: - As explained below the removal of aluminum and chemicals from the surface by mechanical action with abrasive particles and CIP.

1) The brittle layer formed is removed i.e. silica forming bond with aluminum and remove it as shown in with similar cycle peaks are being cut down up to the level of surface. CIP remove the chemical slurry and clean the surface of workpiece.
2) No more reaction occurs when surface having sub nano level finishing that corresponds to very less surface energy available to form chemically active bounding, so no further removal of metal happen.

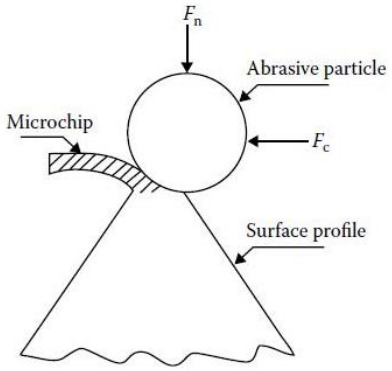

Figure 3.7: Mechanically Removal of Micro Chip (Jain, 2013).

3.7 Preliminary Experimental Study

3.7.1 Process Parameters

Process Parameters has been studded to know the effect of key parameters on MRR and surface finish Ra. The main parameters which influences the process are:-

1) MR fluid 2) Composition 3) Size of Magnetic particles 4) Size of abrasive particles 5) Viscosity of the fluid 6) Magnetic field in working zone 7) Working gap 8) Rotational speed of the magnet 9) Feed rate to the workpiece and 10) Finishing time

3.7.2 Experimentation

A lot of experiments has been performed with the variation of different process parameters. Mainly MR fluid variation has been studied i.e. varying oxidizer, abrasive and chemicals etc. All the experiments performed are studied and best results of surface finish Ra and MRR has been used for designing the experiments.

Abrasive Study:-

Table 3: Varying different abrasives in slurry

Sr No.	Grade	Slurry	Working Gap	Stock Length	Feed	Spindle Speed	Time	Ra Final	Sa Final
1	6061	SiO_2+H_2O_2+Citric Acid+Glycol+Buffer Sol.	0.5 then 0.8 then 1mm	40	400	259	3	31.6	46.2
2	6061	Al_2O_3+SiO_2+Glycol+ H_2O_2+Buffer Sol.+Cirtic Acid	0.5 then 0.65 then 0.9	40	200	259	3.4	58.1	105
3	6061	SiO_2+H_2O_2+Citric Acid+Glycol+Buffer Sol.	0.8	40	400	259	1.5	5.11	33.4
4	6061	Al_2O_3+SiO_2+Glycol+ H_2O_2+Buffer Sol.+Cirtic Acid	0.5 then 0.65 then 0.8	20	200	259	3.4	2.75	57.9
5	6061	Initial for 1 hr, then very less SiO_2+H_2O_2+Citric Acid+Glycol+Buffer Sol…..Final SiO_2 replaced by Chalk	0.8	40	400	259	2	14.8	20.6
6	6061	Chalk+Glycol+H_2O_2 +Buffer Sol.	0.8	40	500	200	1	29.9	242
7	6061	Al_2O_3+Glycol+H_2O_2 +Buffer Sol.+Cirtic Acid	0.8	40	500	259	1	49.4	239
8	6061	Glycol+Citric Acid+H_2O_2	0.8	40	500	259	1	16.9	52
9	6061	Alumina+Glycol+H_2O_2	1	40	400	259	0.5	51.9	168

As shown in Table 3 and Figure 3.8 best results obtained with SiO_2 + Al_2O_3 experiment number 3 and Sa has been improved with SiO_2 slurry only. Large no. of pits has been observed with slurry number 3 comparing to 4. So SiO_2 has been chosen as an abrasive in Al polishing operation.

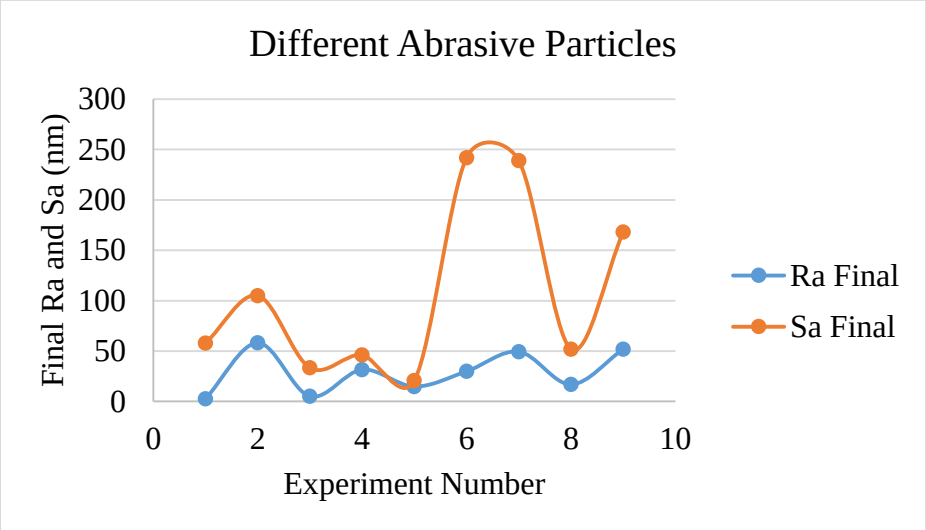

Figure 3.8: Ra and Sa with different abrasive

PH Study:-

Different PH of solutions has been investigated as shown in Table 4.

Table 4: PH Study

Sr no.	PH	Ra (nm) Initial	Ra (nm) Final	% Change in Ra	Time (hr)
1	4	83.6	58.5	30.0239	1
2	7	102	41.2	59.6078	1
4	7.3	78	4.1	94.7436	1
3	10	125	69.6	44.32	1

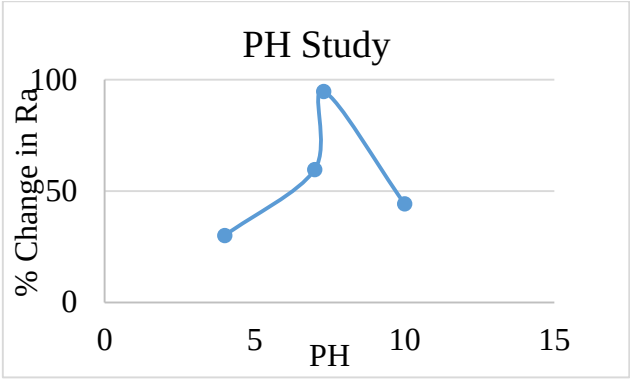

Figure 3.9: % Change in PH

Results shows in Figure 3.9 that DI water having PH of 7.3 has 94% change compare to all buffer solutions of 4, 7, and 10. So DI water has been selected for the finishing of Al.

NH_3 Study:-

- NH_3 has been selected comparing to H_2O_2 because of formation of corrosion by hydrogen peroxide when reacting with CIP in slurry.
- Different experiments has been performed by taking 1, 3, 5, 7 wt. % of NH_3 are shown in Table 5. The results shown in Figure 3.10 shows maximum Ra % change occurs at 1% so it has been selected for experiments.

Table 5: NH_3 Study

Sr no.	**% of NH_3**	**Ra (nm) Initial**	**Ra (nm) Final**	**% Change in Ra**	**Time (hr.)**
1	1	48.5	11.8	75.6701	1
2	3	40.1	30.5	23.9401	1
4	5	28.1	15.4	45.1957	1
3	7	32.7	15.9	51.3761	1

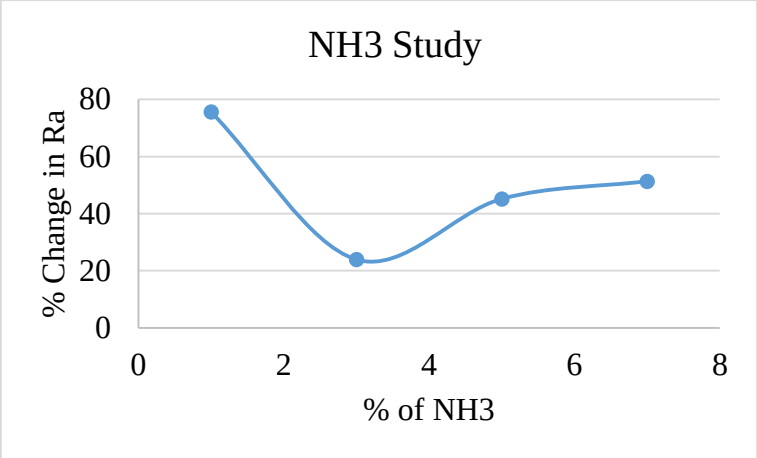

Figure 3.10: NH_3 Study

Time Study:-

- Ra has been measured with time, taking 10min of interval.
- Ra has been measured on CCI.
- Behavior of finishing rate has been studied as shown Table 6.

Table 6: Time Study

Sr No.	Time (min)	Ra (nm)	MRR (nm)
1	0	45.6	
2	10	12.2	326
3	20	11.5	137
4	30	10.6	92
5	40	9.93	73
6	50	9.24	49
7	60	8.4	52

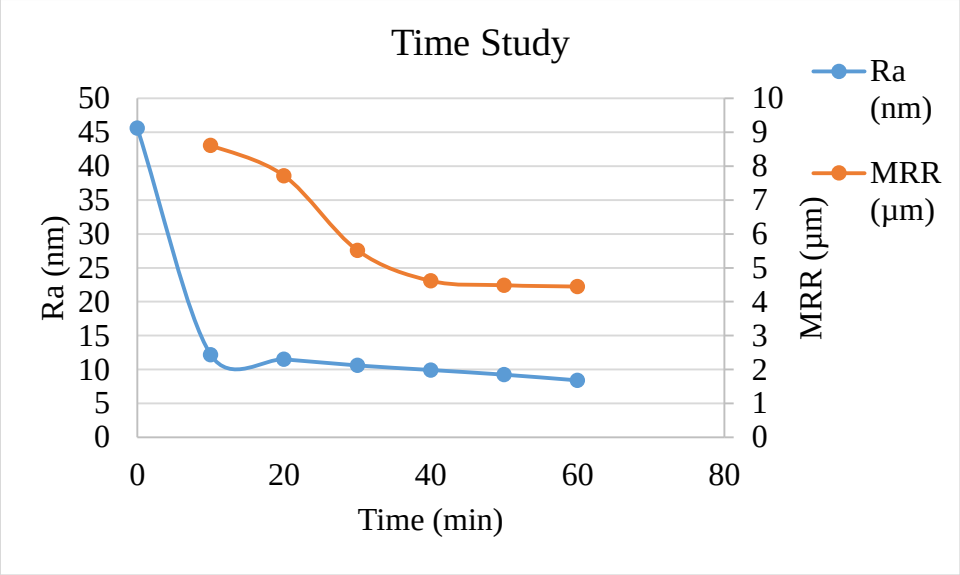

Figure 3.11: Time Study

Constant Parameters by Pilot Experiments: - As shown in Table 7 with the results of pilot experiments some of the parameters which were kept constant for DOE.

Table 7: Constant Parameters

Name	Citric Acid (g)	Glycerol (g)	Ammonia (g)	DI Water (ml)	Spindle Rotation (rpm)	Stock Length (mm)	Feed (mm/min)
Quantity	0.2	0.2	0.14	5	250	40	400

Contact Angle: - Change of contact angle of water droplet with change in finishing has been studied. As shown in Figure 3.19 Figure 3.13, Figure 3.12, Figure 3.15, Figure 3.14 and Figure 3.16 with increasing surface finish contact angle starts increasing from hydrophilic region i.e. 45° up to 86° then starts decreasing at a very sharp rate as shown in Table 8.

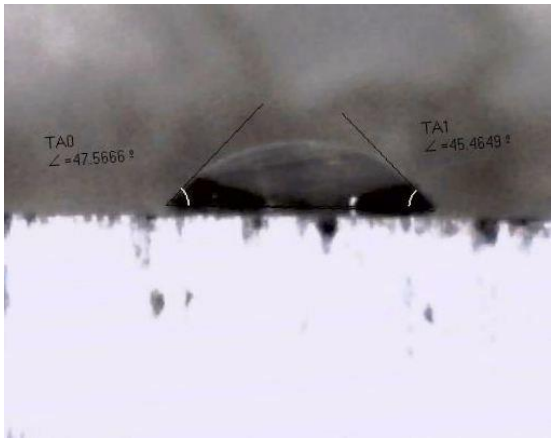

Figure 3.13: Initial Surface

Figure 3.12: Lapped Surface

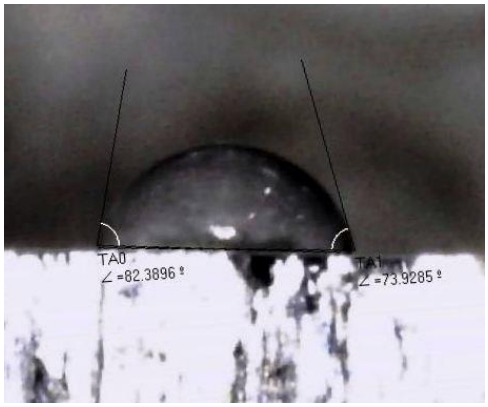

Figure 3.15: Finished surface after one hour

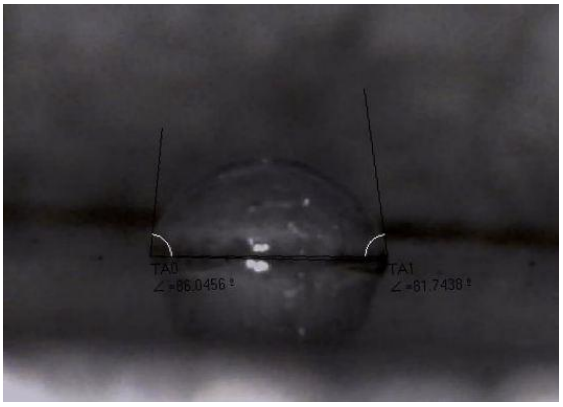

Figure 3.14: Finished surface after two hour

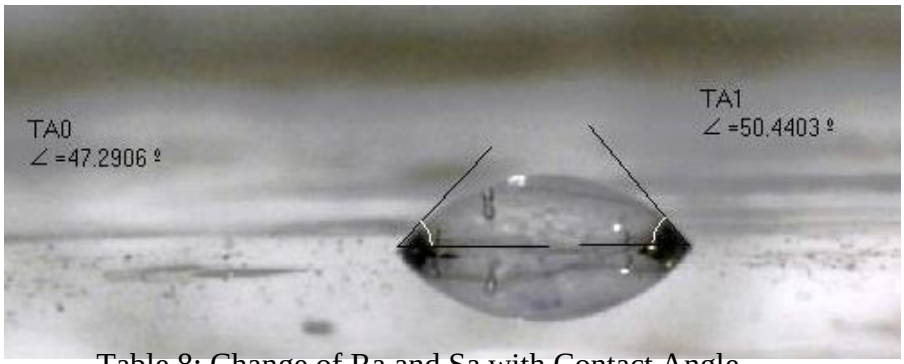

Figure 3.16: Finished surface after three hour

Table 8: Change of Ra and Sa with Contact Angle

Operation	Ra (nm)	Sa (nm)	Contact Angle (degree)	
			Left	Right
Conventional Facing	933	2460	47.5666	45.4649
Lapping Diamond Paste of particle size 250 nm	52.1	45.2	66.7821	67.4871

CMMRF (After one hour)	6.83	8.39	82.3896	73.9285
CMMRF (After two hour)	2.84	5.59	86.0456	81.7438
CMMRF (After three hour)	1.62	3.6	47.2906	50.4403
CMMRF (After three hour)	0.69	0.82	16.023	24.006

3.7.3 Result and discussions

Pilot experimental results shows that best combination shown in Table 9 is fumed silica, ammonia, citric acid, glycerol, CIP mix in DI water for nano finishing of aluminum. For optimization of process full factorial experiment has been conducted. For these experiments SiO_2, NH_3, glycerol, citric acid and DI water values has been kept constant according to the pilot study results.

Table 9: Results of Pilot Study

CIP (g)	**SiO_2 (g)**	**Glycine (g)**	**Citric acid (g)**	**Glycerol (g)**	**DI Water (ml)**	**NH_3 (g)**
1	0.015	0.3	0.2	0.2	5	0.2

- Mean diameter of CIP particles is 1µm, black in colour.
- Mean diameter of SiO_2 particles is 0.005 µm (5 nm), white in colour.
- For conversion of the mean diameter of particles with mesh number formula is
  Mean diameter (µm) = 15200/ (Mesh number of particles).

Different surface initial, after lapping operation and after finial finishing are shown in Figure 3.17, Figure 3.18 and Figure 3.19.

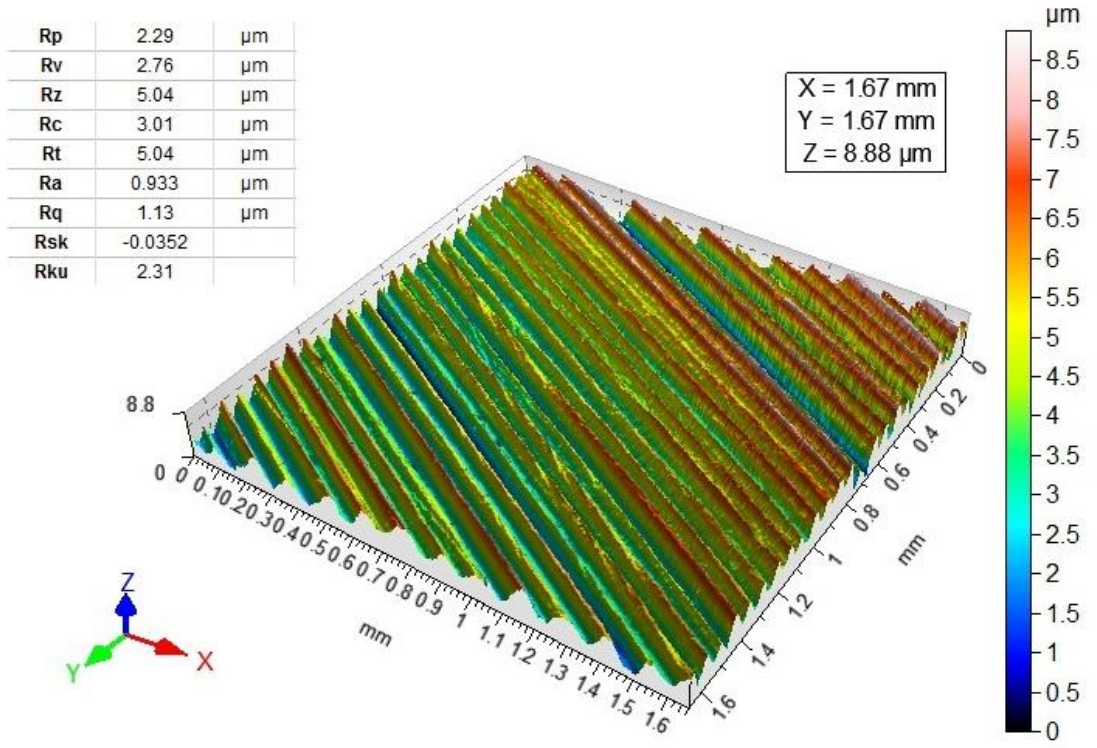

Figure 3.17: Initial Surface

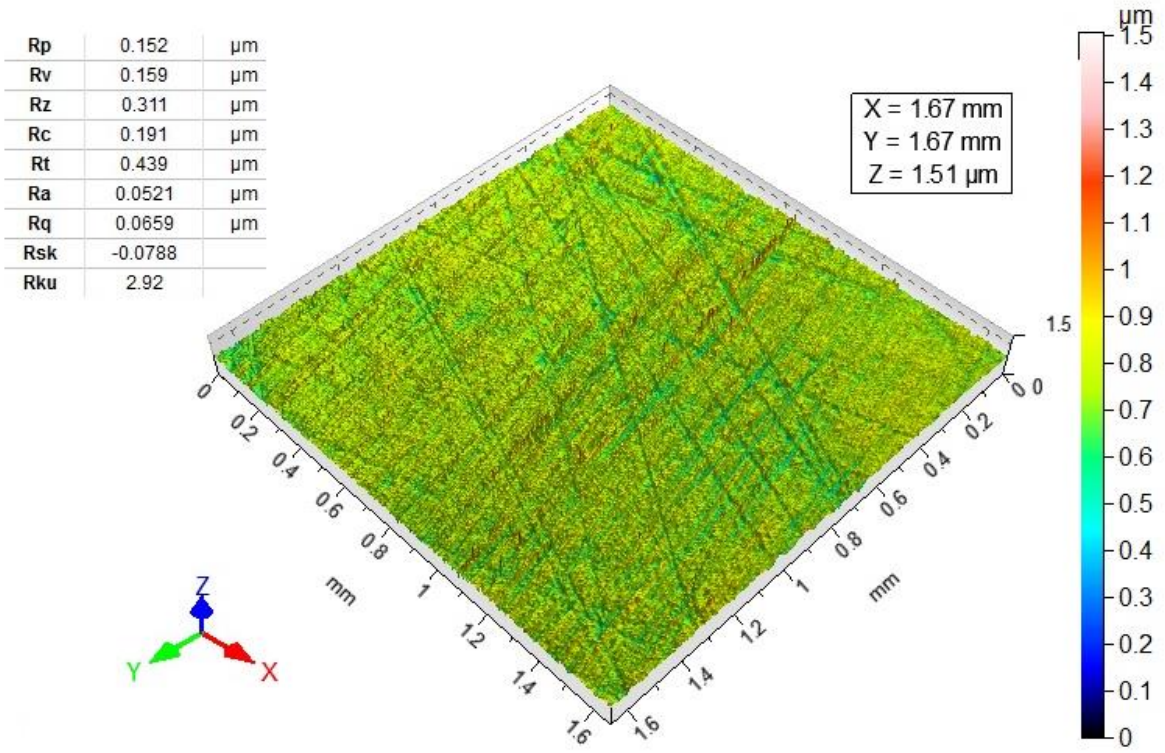

Figure 3.18: Surface after Lapping

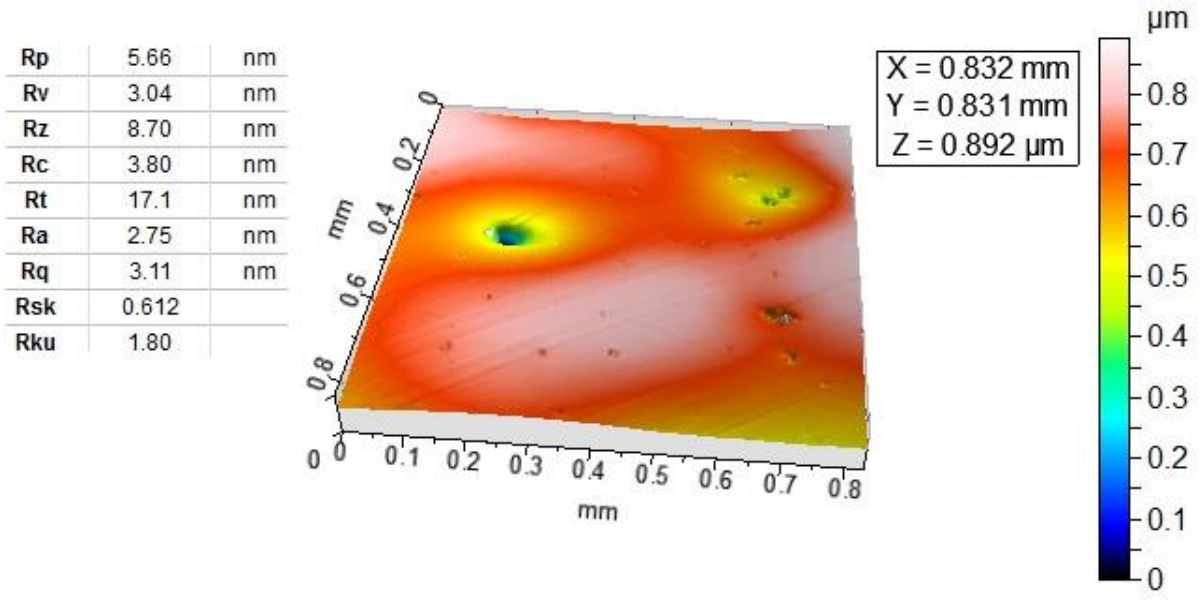

Figure 3.19: Finished Surface

3.8 Design of Experiments and Analysis

3.8.2 Central Composite Design

For second order model most powerful design is CCD (central composite design). Two level axil point, factorial points and central runs has been compared in CCD model. Two factors interactions or any kind of interaction present and linear terms can be studied by factorial points. Quadratic terms can be estimated by axial and central runs. Pure error can also be estimated by calculating central runs in the design. Factorial points mainly depends on number of factors (k) in the process and type of factorial design used. 2k factorial runs are there for any full factorial design. Axial points are 2^k, and central points are dependent upon the number of factors chosen for any full factorial design. Axial points are having a range of $\pm\alpha$ from 0 level of every point or from central point. Depending upon the value of α as shown in Table 10 the full factorial designs.

Table 10: α values in full factorial design

Number of Factors	Factorial Portion	Value of α
2	2^2	$2^{2/4} = 1.414$
3	2^3	$2^{3/4} = 1.682$
4	2^4	$2^{4/4} = 2.000$

3.8.3 Analysis of Variance

Four factors were selected for the study of CMMRF process. Full factorial was performed, names, units, abbreviations and levels are given below in

Table 11. "Design Expert® 7.0.0" software was used for design of experiments and analysis study.

Table 11: Factors and Levels

Sr. No	Parameter	Unit	Levels				
			-2	-1	0	1	2
1	Fumed Silica	g	0.01	0.015	0.02	0.025	0.03
2	Glycine	g	0.2	0.3	0.4	0.5	0.6
3	Working Gap	mm	0.8	0.9	1	1.1	1.2
4	CIP	g	0.75	1	1.25	1.5	1.75

Experiments were conducted by using CCD methodology with four factors i.e. fumed silica, glycine, working gap and CIP. Different response factors have been studied as shown in Table 12 they are final Ra, % change in Ra, % change in depth. Workpiece used is Al 7075-T6.

Table 12: Responses

Std	Run	SiO_2 (g)	Glycine (g)	Working Gap (mm)	CIP (g)	Ra Final (nm)	% Change in Ra (%)	% Change in Depth
18	1	0.02	0.4	1	1.25	7.37	87.8383	1.45773
15	2	0.015	0.5	1.1	1.5	16.7	53.0899	2.34742

2	3	0.025	0.3	0.9	1	5.45	77.6639	5.65966
17	4	0.02	0.4	1	1.25	7.84	35.2066	3.03867
3	5	0.015	0.5	0.9	1	12.4	57.9661	4.2365
8	6	0.025	0.5	1.1	1	9.63	72.4069	1.0582
12	7	0.025	0.5	0.9	1.5	24.4	52.4016	1.68971
14	8	0.025	0.3	1.1	1.5	3.1	81.7647	4.08719
9	9	0.015	0.3	0.9	1.5	6.07	73.4934	8.98204
5	10	0.015	0.3	1.1	1	1.84	96.1181	12.9512
11	11	0.015	0.5	0.9	1.5	15.1	44.2804	2.22222
20	12	0.02	0.4	1	1.25	7.71	77.9083	1.2
6	13	0.025	0.3	1.1	1	2.2	94.6341	11.9134
7	14	0.015	0.5	1.1	1	11	61.8056	5.27859
13	15	0.015	0.3	1.1	1.5	13.9	20.5714	3.05344
1	16	0.015	0.3	0.9	1	9.72	58.8462	8.57143
19	17	0.02	0.4	1	1.25	7.52	76.2025	1.49502
16	18	0.025	0.5	1.1	1.5	12.4	4.6154	1.6883
10	19	0.025	0.3	0.9	1.5	11.1	59.3407	3.85906
4	20	0.025	0.5	0.9	1	14	45.9459	9.09091
25	21	0.02	0.4	0.8	1.25	38.8	64.4068	9.85507
24	22	0.02	0.6	1	1.25	31.3	21.069	1.96078
30	23	0.02	0.4	1	1.25	8.46	27.069	3.84615
22	24	0.03	0.4	1	1.25	10.1	63.4058	1.0101
28	25	0.02	0.4	1	1.75	35.9	20.56	7.69231
26	26	0.02	0.4	1.2	1.25	13	52.5547	14.5658
23	27	0.02	0.2	1	1.25	12.8	31.1828	7.65172
21	28	0.01	0.4	1	1.25	24.7	50.4016	4.7956
29	29	0.02	0.4	1	1.25	6.88	81.2534	3.09524
27	30	0.02	0.4	1	0.75	29.3	28.0098	6.6667

Following procedure was used for nano finishing of aluminum alloys as described below.

- Workpiece of Al 7075-T6 has been loaded on machine, X, Y and Z has been set as per desire.

- Slurry as given in DOE has been prepared by mixing different quantity of chemicals, CIP and abrasive together.
- Process running time has been made fix to one hour, stock length was also made constant of 40mm.
- After one hour cleaning in water has been done and analyzed in CCI.

3.8.4 Response Surface Regression Analysis

1. **Sequential Model Sum of Squares:** In sequential model sum of squares model it is name describes how increasing complexity terms contributes to the full model. The sequential model is having additional terms in the model that does not get aligned and is used to select higher order polynomial. Having value more than 0.05 in Probability > F must not be select for better and correct results. From the below given analysis quadratic model arrives as suitable model for optimizing the factor Final Ra and % change in depth. For % change in Ra 2FI has been suggested as better model. As in CCD model unique point's quantity is less so model is not able to calculate all the cubic points, that's why cubic model has been aliased in the analysis shown. So it is not recommended to add any cubic terms in the model. Hierarchy of the model has been given as:-

a) **Linear vs Mean:** It gives importance of adding the linear terms to mean of model.

b) **FI vs Linear:** As linear and mean terms are there in the CCD model, with FI the significance of addition of two factor interaction i.e. 2FI in to the model can be studied.

c) **Quadratic vs 2FI:** Model having mean value, linear terms and two factors interaction present in the model. With addition of quadratic terms in to the model importance of these terms can be studied.

d) **Cubic vs Quadratic:** There is a presence of linear, mean, two factors interaction and quadratic terms i.e. all lower order terms already in the model. Then addition of quadratic terms gives there importance to the model.

Calculating sum of deviation from mean of the model is termed as sum of square of the model. Degree of freedom of the model depends upon the number of additional terms added into the model and residual degree of freedom of the model. Whereas the degree of freedom for mean is always 1. For every source, the mean sum of square has been divided to the degree of freedom. This has been used to calculate the F value of the term and that described the significance of adding new terms to the terms which are already present in the model. That

gives the probability of adding these terms to the model. Model must be chosen whose values are not aliased and with higher order having significant additional terms in to it.

Table 13: Sequential Model Sum of Squares for Final Ra

Sequential Model Sum of Squares For Ra						
Source	**Sum of Squares**	**df**	**Mean Square**	**F Value**	**P value Prob > F**	
Mean vs Total	5622.2092	1	5622.2092			
Block vs Mean	833.890807	2	416.945403			
Linear vs Mean	821.167617	4	205.291904	4.43110561	0.00841512	
2FI vs Linear	69.3391875	6	11.5565313	0.19720162	0.97312954	
Quadratic vs 2FI	523.360451	4	130.840113	3.59691032	0.03478761	Suggested
Cubic vs Quadratic	265.283433	8	33.1604292	0.79865927	0.63056617	Aliased
Residual	207.600602	5	41.5201203			
Total	8342.8513	30	278.095043			

Table 14: Sequential Model Sum of Squares for % Change in Ra

Sequential Model Sum of Squares For % Change in Ra						
Source	**Sum of Squares**	**df**	**Mean Square**	**F Value**	**P value Prob > F**	
Mean vs Total	0.03374	1	0.03374			
Block vs Mean	0.00731	2	0.00365			
Linear vs Mean	0.02221	4	0.00555	1.55311	0.22035	
2FI vs Linear	0.03947	6	0.00658	2.61484	0.05548	Suggested
Quadratic vs 2FI	0.00275	4	0.00069	0.2231	0.92069	
Cubic vs Quadratic	0.0366	8	0.00457	6.6809	0.02561	Aliased
Residual	0.00342	5	0.00068			

Total	0.1455	30	0.00485			

Table 15: Sequential Model Sum of Squares for % Change in Depth

Sequential Model Sum of Squares For % Change in Depth						
Source	**Sum of Squares**	**df**	**Mean Square**	**F Value**	**P value Prob > F**	
Mean vs Total	801.041	1	801.041			Suggested
Block vs Mean	13.8513	2	6.92564			
Linear vs Mean	124.233	4	31.0582	2.53235	0.06795	
2FI vs Linear	21.2598	6	3.54329	0.23094	0.96066	
Quadratic vs 2FI	156.454	4	39.1135	4.87178	0.01272	Suggested
Cubic vs Quadratic	100.928	8	12.616	18.3199	0.00266	Aliased
Residual	3.44326	5	0.68865			
Total	1221.21	30	40.707			

Sequential model sum of square had been shown in Table 13, Table 14 and Table 15. It has been observed that there is significant improvement in the model fit with accumulation of additional terms in to the model. Linear, mean terms having a significance with the addition of terms, whereas quadratic are showing improvement by adding mean and linear terms.

2. **Lack of Fit Test:** From replicated points of design pure error has been compare to residual error in this fit test. It is not a good idea to use the given model for predicting response if the Prob > F is less than 0.05, i.e. less than 95 % of confidence level for analyzing. Below given tables represents the lack of fit of all response factors. As shown in
3. Table 16 linear, 2FI are not selected because there value is less than 0.05. Quadratic model has been suggested as its value is more than 0.05 which is 0.14, as there is also addition of significant terms in the given model that leads to better approximation.

Table 16: Lack of Fit Tests of Final Ra

Lack of Fit Tests for Ra						
Source	**Sum of Squares**	**df**	**Mean Square**	**F Value**	**P value Prob > F**	
Linear	1064.20697	20	53.2103487	115.951947	0.00113756	
2FI	994.867786	14	71.0619847	154.852876	0.00074951	
Quadratic	471.507335	10	47.1507335	102.747295	0.14075487	Suggested
Cubic	206.223902	2	103.111951	224.693726	0.00054003	Aliased
Pure Error	1.3767	3	0.4589			

Table 17: Lack of Fit Tests for % Change in Ra

Lack of Fit Tests for % Change in Ra						
Source	**Sum of Squares**	**df**	**Mean Square**	**F Value**	**P value Prob > F**	
Linear	0.08171	20	0.00409	23.043	0.01239	
2FI	0.04224	14	0.00302	17.0165	0.01945	Suggested
Quadratic	0.03949	10	0.00395	22.2735	0.01339	
Cubic	0.00289	2	0.00145	8.15552	0.06123	Aliased
Pure Error	0.00053	3	0.00018			

Table 18: Lack of Fit Tests for % Change in Depth

Lack of Fit Tests for % Change in Depth						
Source	**Sum of Squares**	**df**	**Mean Square**	**F Value**	**P value Prob > F**	
Linear	280.51	20	14.0255	26.7127	0.00999	
2FI	259.25	14	18.5179	35.2687	0.00673	
Quadratic	102.796	10	10.2796	19.5784	0.01613	Suggested
Cubic	1.86811	2	0.93406	1.77898	0.30941	Aliased
Pure Error	1.57515	3	0.52505			

As discussed above in the Table 17 and Table 18 2FI and quadratic model has been suggested been selected because as the value of linear is less than that 2FI in % Ra and both linear and 2FI less in % depth so that's why quadratic model has been selected.

4. Analysis of variance (ANOVA):

Table 19: Analysis of variance (ANOVA) for Final Ra

Source	**Sum of Squares**	**df**	**Mean Square**	**F Value**	**P value Prob > F**	
Block	833.891	2	416.945			
Model	1413.87	14	100.991	2.77632	< 0.0001	significant
A-SiO_2	47.1801	1	47.1801	1.29702	0.275	
B-Glycine	410.44	1	410.44	11.2834	0.005	
C-Working Gap	260.503	1	260.503	7.16145	0.019	
D-CIP	103.045	1	103.045	2.83279	< 0.0001	
AB	13.8943	1	13.8943	0.38197	< 0.0001	
AC	48.1983	1	48.1983	1.32501	< 0.0001	
AD	0.52926	1	0.52926	0.01455	< 0.0001	
BC	1.48231	1	1.48231	0.04075	< 0.0001	
BD	2.73076	1	2.73076	0.07507	< 0.0001	
CD	2.50431	1	2.50431	0.06885	< 0.0001	
A^2	0.40671	1	0.40671	0.01118	< 0.0001	
B^2	45.2394	1	45.2394	1.24367	< 0.0001	
C^2	138.459	1	138.459	3.80636	0.072	
D^2	421.859	1	421.859	11.5973	0.004	
Residual	472.884	13	36.3757			
Lack of Fit	471.507	10	47.1507	102.747	0.00140	significant

Pure Error	1.3767	3	0.4589			
Cor Total	2720.64	29				

Table 20: Analysis of variance (ANOVA) for % Change in Ra

Source	**Sum of Squares**	**df**	**Mean Square**	**F Value**	**P value Prob > F**	
Block	0.00730856	2	0.00365428			
Model	0.06168409	10	0.00616841	2.45184965	0.04985431	significant
A-SiO_2	0.00342427	1	0.00342427	1.36109763	0.25945314	
B-Glycine	0.00596692	1	0.00596692	2.37176199	0.1419541	
C-Working Gap	0.00530876	1	0.00530876	2.11015012	0.16453354	
D-CIP	0.00751341	1	0.00751341	2.98646621	0.10208236	
AB	0.00870312	1	0.00870312	3.45936025	0.0802874	
AC	0.00525001	1	0.00525001	2.08679783	0.16675667	
AD	0.00539485	1	0.00539485	2.14437089	0.16134138	
BC	0.00540332	1	0.00540332	2.14773647	0.16103157	
BD	0.00559025	1	0.00559025	2.22203956	0.15437443	
CD	0.00912919	1	0.00912919	3.62871551	0.07385089	
Residual	0.04276892	17	0.00251582			
Lack of Fit	0.04223704	14	0.00301693	17.0165183	0.01944512	significant
Pure Error	0.00053188	3	0.00017729			
Cor Total	0.11176157	29				

Table 21: Analysis of variance (ANOVA) for %Change in Depth

Source	**Sum of Squares**	**df**	**Mean Square**	**F Value**	**P value Prob > F**	
Block	13.851281	2	6.92564048			
Model	301.94668	14	21.56762	2.68635497	0.041773	significant
A-SiO_2	10.8910467	1	10.8910467	1.35653435	0.265067	

B-Glycine	76.4956358	1	76.4956358	9.52791414	0.008665	
C-Working Gap	2.3360372	1	2.3360372	0.29096512	0.59872	
D-CIP	34.5101621	1	34.5101621	4.29841335	0.058583	
AB	3.49800613	1	3.49800613	0.43569416	0.520732	
AC	0.08566027	1	0.08566027	0.01066942	0.919307	
AD	0.24139706	1	0.24139706	0.03006721	0.865009	
BC	8.70220203	1	8.70220203	1.0839028	0.316802	
BD	3.42003391	1	3.42003391	0.42598233	0.525341	
CD	5.31245128	1	5.31245128	0.66169238	0.43061	
A^2	0.21857823	1	0.21857823	0.02722501	0.871482	
B^2	4.09904972	1	4.09904972	0.51055715	0.487531	
C^2	137.334587	1	137.334587	17.1057099	0.001172	
D^2	26.3367067	1	26.3367067	3.28036858	0.093272	
Residual	104.37156	13	8.02858156			
Lack of Fit	102.796407	10	10.2796407	19.5783654	0.016133	significant
Pure Error	1.57515306	3	0.52505102			
Cor Total	420.169521	29				

The ANOVA of all the cases are shown in Table 19, Table 20 and Table 21. Ra probability of every term is given in the tables. The terms having Prob > F i.e. less than 0.05 are significant and other having more than 0.05 are insignificant and not included in the model.

Table 22: Other ANOVA Parameters for Ra

Std. Dev.	7E-04	**R-Squared**	0.97936599
Mean	0.017	**Adj R-Squared**	0.94524481
C.V. %	4.215	**Pred R-Squared**	0.91623917
PRESS	0.008	**Adeq Precision**	17.0057693

Table 23: Other ANOVA Parameters for % change in Ra

Std. Dev.	0.00585054	**R-Squared**	0.96016574
Mean	1.26677882	**Adj R-Squared**	0.92267316
C.V. %	0.46184416	**Pred R-Squared**	0.82427524
PRESS	0.57718697	**Adeq Precision**	14.9945282

Table 24: Other ANOVA Parameters for % change in Depth

Std. Dev.	0.01680128	**R-Squared**	0.866946014
Mean	2.22594838	**Adj R-Squared**	0.831058018
C.V. %	0.75479181	**Pred R-Squared**	0.829214717
PRESS	9.88469374	**Adeq Precision**	13.92281086

Other parameter are also shown in Table 22, Table 23 and Table 24 for all the response factors. Standard deviation represents the standard deviation related to the experimental error. Mean is average of response data. Percentage coefficient of variation is the error associated with mean. It is calculated by dividing standard deviation of response to mean and multiply by 100. PRESS represents how well each points fit in the design. Variance around the mean can be calculated in term of R-Square. Pred R-Square and Adj R-Square are found close in each case with less difference. Both values must be within 0.20 range otherwise there might be problem in model selected or data. Adeq Precision measure signal to noise ration value greater than 4 required for model to fit. In each case the value satisfies the requirements.

Final Regression Equation with Actual Factors: Final regression equation has been calculated after ANOVA with actual factors. Represents the responses Ra, % change in Ra and % change in depth as actual factors.

Ra Final = +7.63 -1.402(A) +4.135(B) -3.294(C) +2.072(D) +0.931(AB) -1.735(AC) +0.181(AD) -0.304(BC) +0.413(BD) +0.395(CD) +0.121(A^2) +1.284(B^2) +2.246(C^2) +3.921(D^2)……..Eq. (3.1).

% Δ Ra = +7.8464 +0.044(A) -0.599(B) -0.179(C) -0.655(D) -0.551(AB) +0.034(AC) -0.072(AD) -0.137(BC) -0.040(BD) -0.722(CD)….. ……Eq. (3.2).

% Δ Depth = 2.355 -0.673(A) -1.785(B) +0.311(C) -1.199(D) +0.467(AB) -0.0731(AC) -0.122(AD) -0.737(BC) +0.462(BD) -0.576(CD) -0.089(A^2) +0.386(B^2) +2.237(C^2) +0.979(D^2)……Eq. (3.3).

Chapter 4

Results and Discussions

4.1 Introduction

On the basis of the ANOVA results and different models, results are obtained in terms of four factors i.e. silica, glycine, working gap and CIP. The results have been plotted by the Eq.1, 2 and 3 by using DOE Expert® software. Optimization study has also been carried out and results has been obtained. Optimization study has been done on aluminum metal only in CMMRF process.

4.2 Parametric Analysis of Responses

In parametric analysis all the four factors i.e. SiO_2, glycine, working gap and CIP has been plotted against final Ra, % change in Ra and % change in depth according to the model suggested and with the help of Eq. 1, 2 and 3. Only significant factors in all models i.e. quadratic and 2FI has been plotted and discussed below.

4.2.1 Final Ra

As shown in Figure 4.1 with increse in working gap Ra improves for large quantity of silica and get worse at low quantity of silica. Similarly finish improves with decrease in silica at higher working gap and roughness increases at low working gap. This is because of high pressure at low working gap and high quantity of silica on the surface causing scratches and surface defectes.

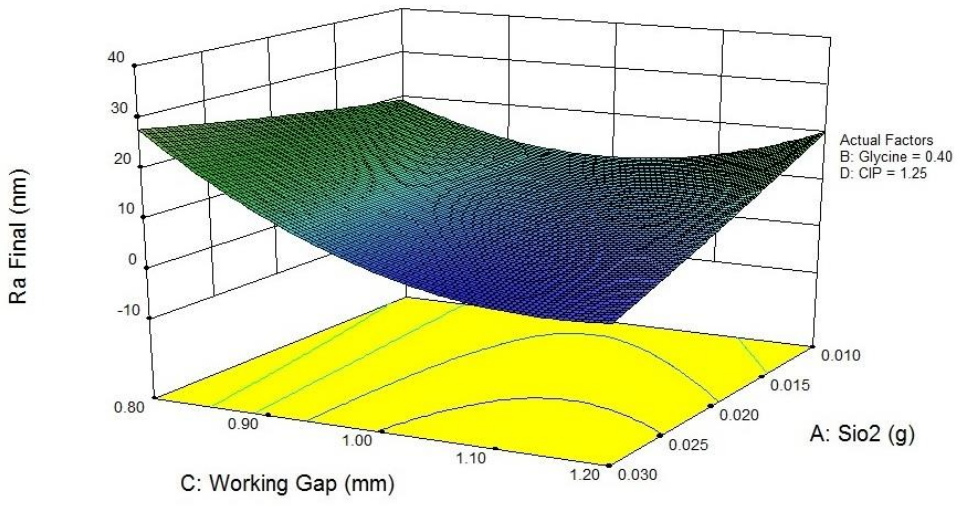

Figure 4.1: Variation of Silica and Working Gap with Final Ra

As shown in Figure 4.2 with decrease in silica less abrasive are active to finish the surface compare to 0.03 g of silica used to obtain improved surface quality. Results also shows that finish decrease at both lower and higher values of CIP in the slurry, which causing very less pressure and very high pressure for finish the surface. So proper pressure is needed for having high quality surface finish, as shown at 1 g CIP Ra improves with higher silica in slurry.

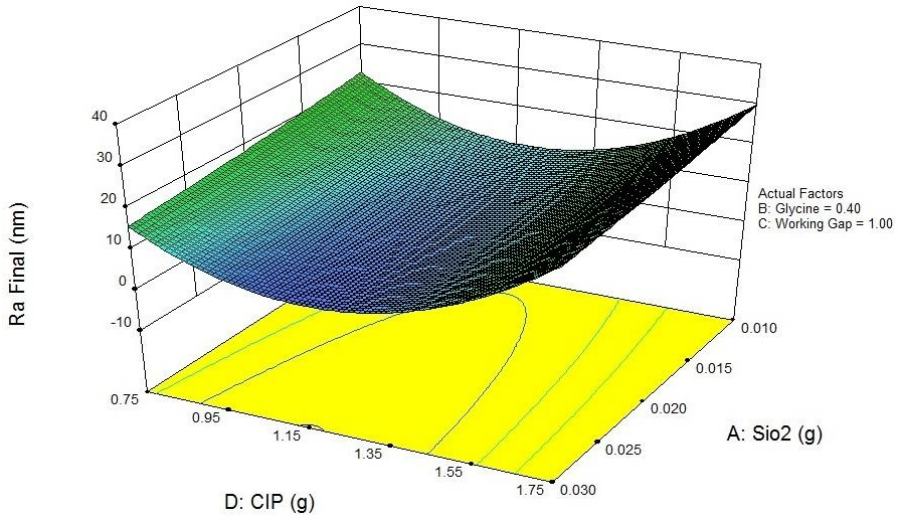

Figure 4.2: Variation of Silica and CIP with Final Ra

As shown in Figure 4.3 Ra decrese with large increase or decrease in CIP, because for higher cutting force at high CIP in slurry and low force and abrasive binding ability at low CIP causing reduction in Ra. With glycine increase finish decrese so low quantity of glycine is recommended.

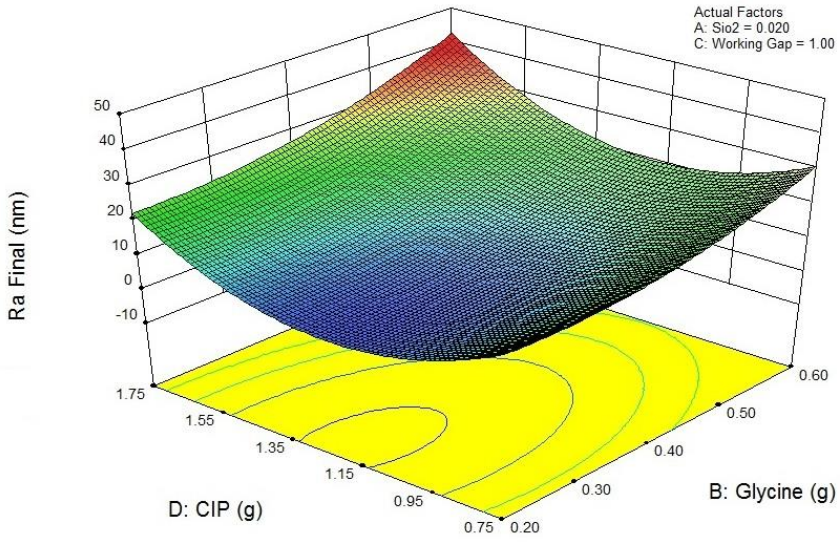

Figure 4.3: Variation of Glycine and CIP with Final Ra

4.2.2 % Change in Ra

As shown in Figure 4.4 with increase in glycine surface finish decreases, so % change in Ra also decrease as with increase in glycine protective layer formed of glycinate anion causing less MRR. Results shows that variation in % change in Ra is less with working gap varying.

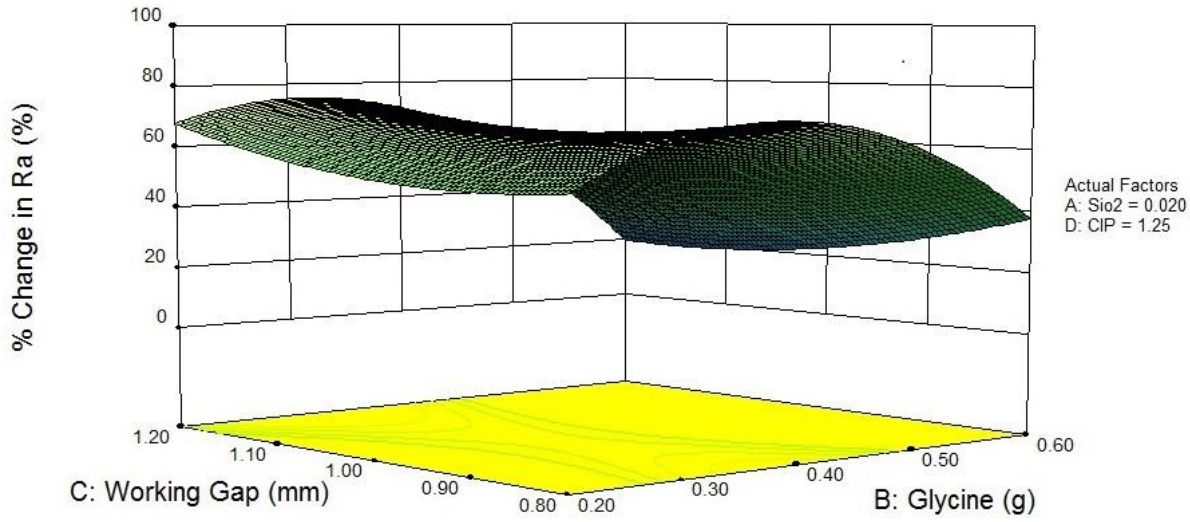

Figure 4.4: Variation of Glycine and Working Gap with % Change in Ra

As shown in Figure 4.5 with both increase and decrese in CIP % change in Ra decerese, because at higher and lower CIP force exerting on the workpiece is very high and low causing deteroation of surface finish. At a proper pressure and working gap force is enough to remove only the upper form passivitation layer causing improvement in surface finish. At large CIP and working gap less surface finish is obtained because on pressure is exerted on the Al. even at low working gap and CIP high pressure is there causing silimar effect of worst surface finish.

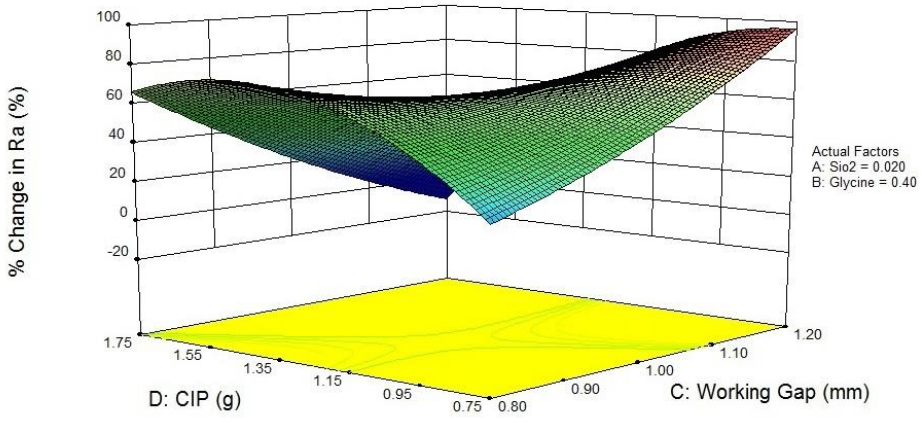

Figure 4.5: Variation of CIP and Working Gap with % Change in Ra

As shown in Figure 4.6 CIP having a significant effect on % change in Ra, at 1.25 g better surface finish is achieved. At higher and lower sides less change in Ra has been observed. Silica is not as significant as compare to CIP.

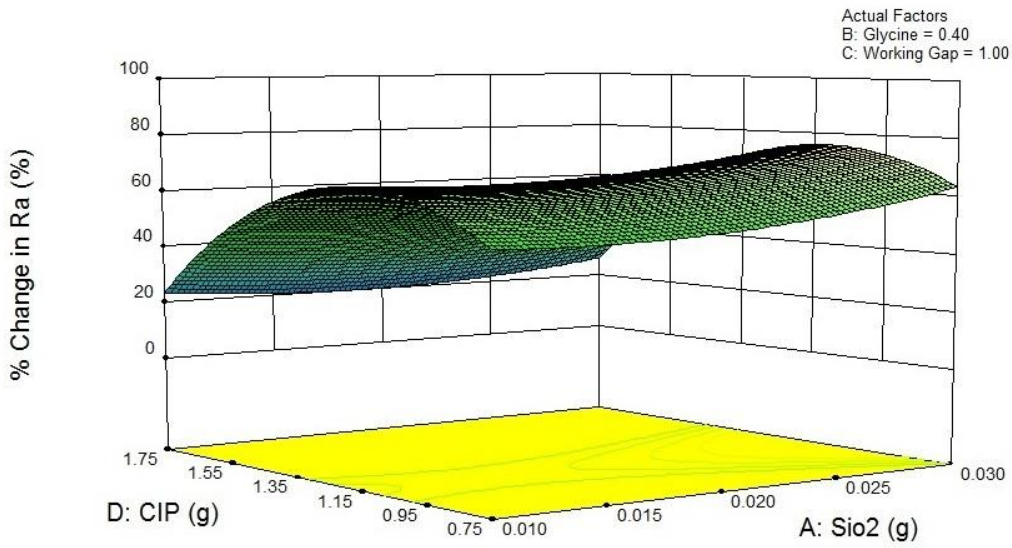

Figure 4.6: Variation of CIP and Silica with % Change in Ra

4.2.3 % change in Depth

As shown in Figure 4.7 with increase in glycine % change in depth decreases because of formation of protective layer on the Al workpiece. Whereas with increase in CIP % change in depth decrease as starts increasing and at low CIP also it increases because of higher abrasive holding capability of CIP at low quantity.

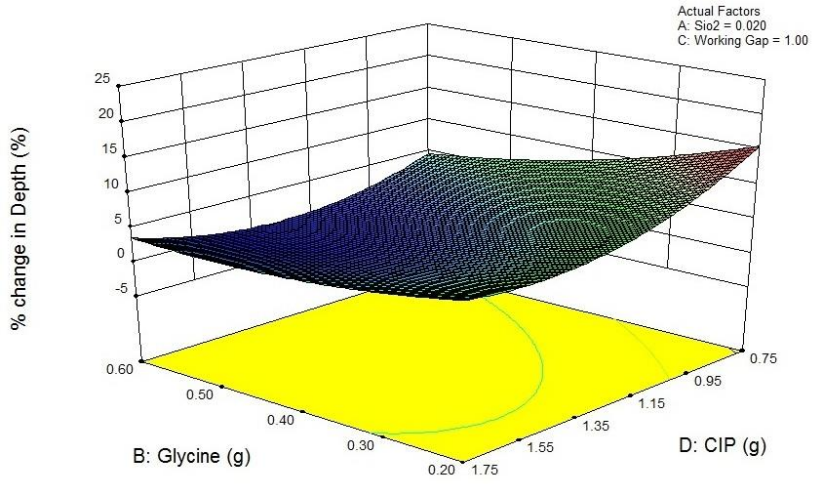

Figure 4.7: Variation of CIP and Glycine with % Change in Depth

As shown in Figure 4.8 with increase in silica at higher CIP less % change in depth occurred because of low holding capability of CIP of abrasive particles. But at lower CIP more abrasive can be bound to the CIP at same working gap causing more removal of material from the surface.

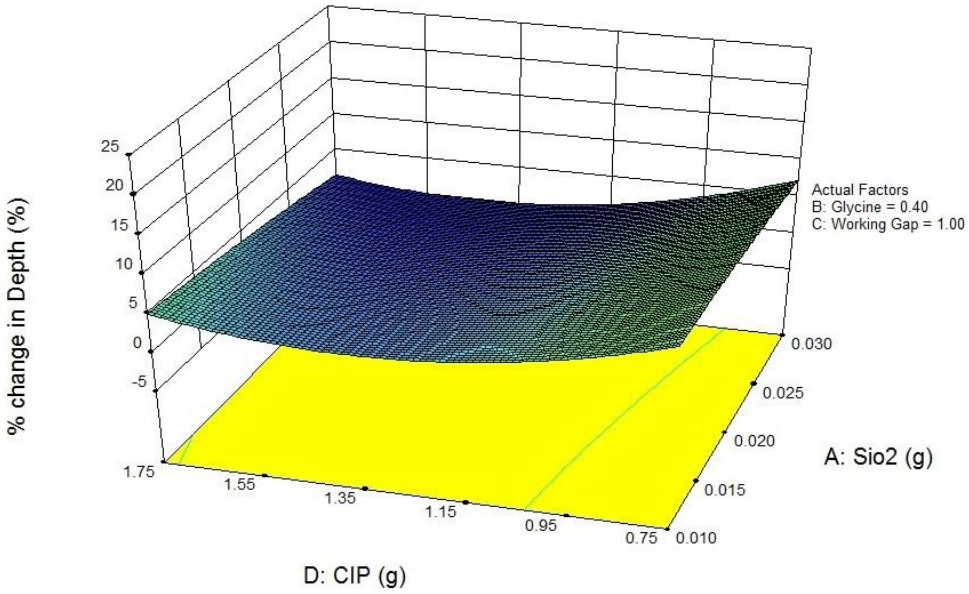

Figure 4.8: Variation of CIP and Silica with % Change in Depth

As shown in Figure 4.9 with increase in working gap reduction in % change in depth has been seen because at higher working gap more force is required to remove the protective layer. At low glycine less force is require so higher MRR is obtained.

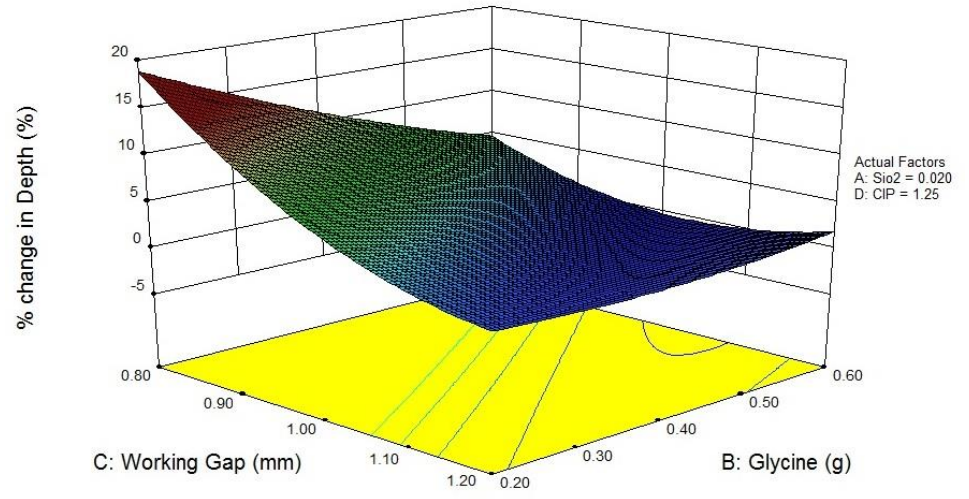

Figure 4.9: Variation of Glycine and Working Gap with % Change in Depth

4.3 Optimization of Al workpiece by CMMRF process

Optimization study was carried out by varying the each and every independent parameters with full selected range of experiments. Goals were set according to the required output i.e. for higher or lower value of output. For obtaining the optimum values Design Expert ® V0.7 was used. Desirability was calculated, which is having a range from 0 to 1. One represents the ideal case for fully optimized process, whereas zero represents that may be any value or data is wrong or out of order. Theoretical values, optimization conditions and there equivalent desirability was calculated.

All the independent parameters like silica, CIP, working gap and glycine were kept in the rage defined in DOE. For Response-I i.e. Final Ra minimum is selected i.e. lowest the best. All the selected response and different parameters are shown in Table 25.

For Response –II and III i.e. % change in Ra and % change in depth maximum is selected, i.e. higher the best. Theoretical optimized results obtained with Design Expert ® V0.7 are shown in Table 26 gives four solutions the solution having higher desirability was selected.

Table 25: Constraints for parameters

Name	Goal	Lower Limit	Upper Limit
SiO_2	Is in range	0.015	0.025
Glycine	Is in range	0.3	0.5
Working Gap	Is in range	0.9	1.1
CIP	Is in range	1	1.5
Ra Final	Minimize	1.84	38.8
% Change in Ra	Maximize	4.6154	96.1181435
% change in Depth	Maximize	1.010101	14.5658263

Table 26: Solutions for parameters

Number	SiO_2 (g)	Glycine (g)	Working Gap (mm)	CIP (g)	Ra Final (nm)	% Change in Ra (%)	% change in Depth (%)	Desirability	
1	0.024	0.3	1	1	2	87.1	9.92	0.96	Selected
2	0.024	0.3	1	1	1.98	85.67	9.88	0.953	
3	0.024	0.3	1	1	2.1	84.54	9.86	0.953	
4	0.024	0.3	1	1	1.99	83.39	9.84	0.953	

Experiment has been performed for validation of the result obtained at 0.024 g of silica, 0.3 g of glycine, 1 mm working gap and 1 g CIP for one hour. Other parameter are kept constant as same as in DOE according to the Table 7. Ra found 2.83 nm shown in Figure 4.10 and Figure 4.11 , % change in Ra and depth were 86.4% and 9.98%.

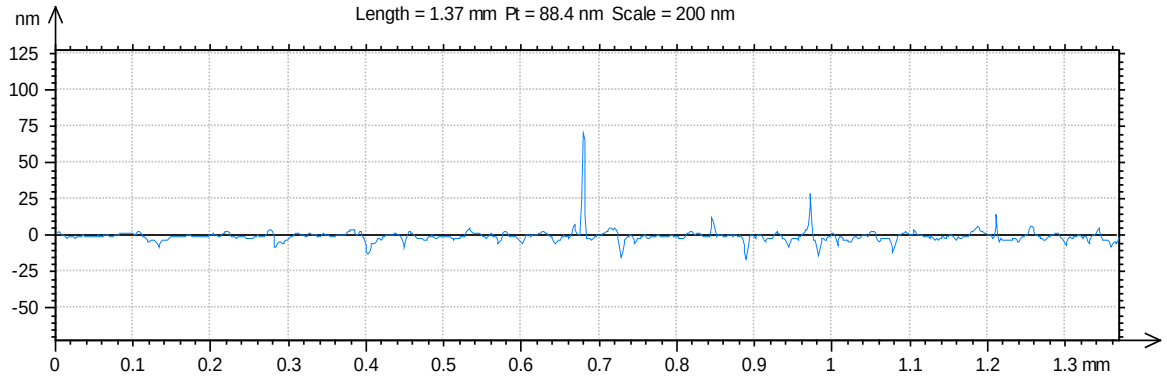

Figure 4.10: Optimization Experiment

Rp	5.71	nm
Rv	8.88	nm
Rz	14.6	nm
Rc	8.00	nm
Rt	18.0	nm
Ra	2.83	nm
Rq	3.49	nm
Rsk	-0.605	
Rku	2.63	

Figure 4.11: Final Ra of Optimized Experiment

Chapter 5

Conclusions

5.1 Introduction

The demand of super polished Al for optics and other major fields is unmatched. The problem in finishing of Al is because of its properties like malleability and ductility, which also make it special than any other metal. For outer space research today world needs telescopes having high accuracy and precision, Al is only solution for telescope mirrors. Defects of coatings of other material like silver and nickel has already been published at a large number. This research work mainly focused on polishing of Al up to sub nonmetric level for optic applications. In this Al-7075-T6 has been used as a workpiece and CMMRF process was used for finish. The process was optimised by varying parameters. Pilot study and full DOE was conducted by using CCD model and results were obtained. In this chapter remarks the conclusion of thesis work based on results obtained. This chapter also focused on the future scope of present work.

5.2 Conclusions

Following main conclusions are made on the basis of results obtained:-

- Al workpiece can be finished with CMMRF process, because of having combination of both mechanical and chemical action in single process.
- No oxide layer formed on finished Al surface as the surface energy of the atom is very small at atomic level to form any kind of bond. So surface can be preserved having atomic level finishing without any coating.
- Surface obtained is scratch free having no defect other than having sub nano size pits.
- From all different abrasives defect free high quality surface finish obtained by using fine fumed silica of size less than 5 nm.
- Results shows that Al can be finished only in alkaline solution, comparing to all other metals finishing in acidic region.
- Best surface finish obtained on aluminium workpiece is 8 Å.
- Finishing of Al causes its surface, shift from hydrophilic to hydrophobic region. Having contact angle greater than 90°.

5.3 Scope for future work

There is a large scope of work to be done for make metal mirror aluminium as a product for all kind of applications. Some of the areas where one should be focused in finishing of Al is:-

- Different process parameters may have significant effect on the finishing rate and quality of surface like size of iron particles, rpm of spindle, feed rate, stock length, time of finishing, magnetic field intensity. These parameters can be optimised and their effect can be studied.
- Problem of pitting effect in finished aluminium can be studied and solved.
- Switching ON/OFF magnetic field in same setup can be studied, this causes new abrasive to be used in finishing operation. This can finish the surface at much higher rate comparing to present.

Chapter 6

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