

Nanofinishing of OFHC Copper using Shape Adaptive Grinding

Gourhari Ghosh, Raj K. Dalabehera, Ajay Sidpara *

Mechanical Engineering Department, Indian Institute of Technology Kharagpur,

Kharagpur-721302, INDIA

Abstract

With the rapid development of nano-manufacturing, the demand of ultra-smooth surface is increasing in optical and electronic industries. Ultra-smooth oxygen-free high conducting (OFHC) copper is extensively used as metal mirror in optical and laser industries. In this present study, an advanced finishing process called shape adaptive grinding (SAG) is employed for nanofinishing of copper. Owing to the presence of elastically compliant medium in SAG tool, the numbers of active abrasive grain increases. As a result, smooth surface can be obtained due the lesser penetration of abrasive particles into the workpiece. The areal surface roughness (Sa) of as-received copper sample is found around 5.135 µm. Tool rotational speed and tool compression are selected as the process parameters and areal surface roughness (Sa) is considered as the output parameter. Full factorial design of experiment method is used to plan the experiments and finishing operation is carried out accordingly under the dry condition. Finishing is also performed with the assistance of benzotriazole (BTA) solution. It is observed that the BTA assisted finishing gives better result and around 10 nm surface roughness (Sa) is accomplished in 70 minutes without any substantial finishing marks on the workpiece.

Keywords: OFHC Copper, Shape Adaptive Grinding (SAG), Areal Surface Roughness (Sa), Nanofinishing, Benzotriazole (BTA).

__

1. INTRODUCTION

In the recent years, the demands of ultraprecision components are increasing progressively to meet the stringent requirements of electronics, space and optical industries. Ultra-smooth copper (Cu) mirror is extensively used in the optical systems to control the propagation and modulation of light beam [1, 2]. Moreover, it became an inevitable component of high-power continuous wave (C.W) and pulsed infra-red lasers for its favorable properties like high thermal conductivity, reflectivity and durability [3]. Besides, it has higher laser damage threshold which makes it suitable as the most promising reflector material in a high-power $CO₂$ laser [4]. It is difficult to achieve optical quality surface and to maintain good form accuracy by conventional finishing processes as copper is a soft material. Furthermore, residual finishing marks and subsurface damage (SSD) cannot be eliminated completely.

Chemical mechanical polishing (CMP) has been extensively used to planarize copper in the electronic industries for fabricating integrated circuits (IC) [5]. CMP of copper can be carried out either in acidic media using Fe $(NO₃)₃$ as an etchant and benzotriazole (BTA) as the inhibitor [6] or in alkaline media using silica slurry [7, 8]. Owing to the high selectivity and good corrosion resistance provided by BTA, CMP of copper in acidic media became more effective. However, the generation of micro scratches on the surface due to the mechanical abrasion of hard abrasive particles is the major limitation of CMP. This can be reduced by using a soft polishing pad or slurry without any abrasive particles or reducing polishing pressure that may deteriorate the planarity or reduce the material removal rate [9].

Single-point diamond turning (SPDT) is also an efficient and deterministic method for accomplishing mirror quality surface in soft material like OFHC copper [4, 10]. However, residual finishing marks cannot be avoided which restrict its application in fabrication of infrared (IR) optics owing to the possibility of scattering for shorter wavelength application [11]. Magnetorheological fluid based finishing is also getting attention in the recent years for achieving mirror quality surface. Khan et al. [2] have investigated the ball-end magnetorheological (MR) finishing of OFHC copper and surface roughness (Ra) is reduced to 38 nm from 65.9 nm in 30 minutes. Pandey et al. [12] have performed the magnetic abrasive polishing (MAP) and ultrasonic assisted magnetic abrasive polishing (UAMAP) of copper alloy. In UNAMP, addition of ultrasonic vibration increases the interaction between the abrasive particles of MAP tool and peaks of work surface which facilitates better shearing of the peaks. Hence, better surface finish can be obtained in UAMAP compared to the MAP.

In this present study, a newly developed finishing process called shape adaptive grinding is used to obtain nanolevel surface finish of OFHC copper. This technique has the ability to reduce the roughness of ceramic material below 0.5 nm (Ra) from 1 µm (Ra) [13]. Principle of the SAG process is based upon precession polishing [14]. SAG tool comprises of an elastically compliant medium covered with resin or nickel bonded diamond polishing pad [15]. This complaint tool described above increases the life of finishing tools, reduces surface errors and sub-surface damages [16].Owing to the presence of elastic medium in the SAG tool; it can undergo large scale elastic deformation which increases the numbers of active abrasive grains. Hence, smooth surface can be obtained. From the literature survey, it is perceived that SAG for finishing optical material such as OFHC copper is rarely addressed. Hence, in this present study, SAG is used to obtain nanolevel surface finish of OFHC copper. Final finishing operations are performed in both dry condition and with the assistance of BTA solution. It is observed that finishing with BTA solution gives better result than dry machining.

2. EXPERIMENTATION

A circular blank of OFHC copper $(Ø52 \times 10$ mm) is chosen as a workpiece material. The samples are prepared by facing operation. SAG experimental set up is shown in Fig.1. SAG tool consists of an elastic material and which is covered by the alumina polishing pad (average particle size 12 µm, FibrMet Abrasive pad PSA, Buheler, USA). Tool rotational speed (N) and tool compression (W) are selected as input parameters and areal surface roughness (Sa) is selected as output parameter. Full

l

 $^{\circ}$ Author to whom correspondence should be made. Email[: ajaymsidpara@mech.iitkgp.ernet.in](mailto:ajaymsidpara@mech.iitkgp.ernet.in)

factorial design of experiment method is used to plan the experiments by considering three levels of each parameter and finishing operations are carried out accordingly under the dry condition. Total 9 experiments are carried out to understand the effect of input parameters on the response. The areal surface roughness (Sa) is measured by 3D non-contact profilometer (CCI MP, Taylor Hobson, UK) and surface morphology is analysed by a scanning electron microscope (SEM) (EVO 18 Research, Carl Zeiss, Germany).

Fig.1. SAG Experimental setup

The experiments are carried out at a fixed feed rate of 50 mm/min which is finalized by performing some trial experiments. To and fro motion of SAG tool is given during finishing. To complete a single finishing experiment or finishing pass 7 min is required. After experimentation, the samples are ultrasonically cleaned and dried. For each sample, ten surface roughness measurements at different locations are taken and average of all is reported. The areal surface roughness (Sa) of as-received OFHC copper sample is found around $5.135 \mu m$. Table 1 shows the plan of experiments and corresponding surface roughness (Sa).

Table 1: Plan of experimentation and corresponding responses

No. of expt.	Tool speed (N) (rpm)	Tool compression	Areal surface	Standard deviation
		(W) (mm)	roughness	of 10
			(Sa) (μ m)	measurem
				ents
	500	0.8	2.445	0.873
\overline{c}	500	1.6	1.343	0.393
3	500	2.4	1.259	0.490
4	1500	0.8	1.852	0.718
5	1500	1.6	0.646	0.125
6	1500	2.4	0.707	0.341
	2500	0.8	1.114	0.324
8	2500	1.6	1.058	0.160
9	2500	2.4	0.808	0.350

The polishing pads are also analyzed by SEM after each experiment. The final experiment to achieve nanolevel surface finish of OFHC copper is carried out by considering the optimum parameters in dry condition. With the same parameter setting, another experiment is performed with the assistance of benzotriazole (BTA) solution as it is an eminent corrosion inhibitor for copper and copper alloys [17].

3. RESULTS AND DISCUSSION

Regression analysis is conducted to fit the response functions with the experimental data using Design-Expert software. The quadratic model for surface roughness (Sa) is given in Eq. (1).

Sa (µm) = 4.8921 - 0.00159 N - 2.7043W + 0.00028 NW + 0.00000027 N² + 0.5445 W² (1)

The coefficient of determination (R^2) is a measure of the degree of fit. It is desirable that its value should approach to unity and it is found that the value of \mathbb{R}^2 is 0.92. From the above observation, it is conceived that the proposed models can be used to predict the responses within the selected levels of the input parameters. The effects of process parameters on the response are represented using 3D response surface plots as shown in Fig. 2.

Fig.2. 3D response surfaces curve showing the interactions of process parameters with Sa

From the Fig. 2, it is observed that the combination of mid value of tool rotational speed and tool compression leads to good surface finish. It is desirable to increase the material removal rate (MRR) with the increase of tool speed and compression. However, during finishing of ductile material (i.e. copper), progressive loading of polishing pad becomes a major problem [18].

Fig.3. Topography of the polishing pad (a) Fresh, after (b) Expt. 1, (c) Expt. 5 and (d) Expt. 9 (1500X), subset of (c) shows EDX point analysis.

During dry machining, as the speed and compression increases, the heat generation at the finishing zone also increases which leads to more MRR and more possibility of polishing pad loading. The topography of fresh polishing pad and after each experiment are analyzed. The topography of fresh polishing pad is shown in Fig. 3 (a) and it is observed that alumina grits are clearly visible. Fig. 3(b), (c) and (d) represent the topography of

polishing pad after experiment no. 1, 5 and 9 as presented in Table 1, respectively. From the Fig. 3 (b) and (c), it is observed that the pads loading are not significant or pads are partially loaded. From the EDX point analysis as shown in subset of (c), it was found that the polishing pad surface had a substantial amount of copper which confirms the pad loading. It is perceived that loading increases with both the tool speed and compression. At higher values of speed and compression, the pad is too much loaded as shown in Fig. 3(d). Therefore, the fresh abrasive grit don't get chance to interact with the workpiece and Cu debris rub over the work surface which further deteriorates the surface finish. However, at the lower values of the parameters though the loading problem is not significant but material removal is also very less. Therefore improvement of surface finish is less at the lower parameter setting.

Based on the above preliminary experiments and responses, parameters are optimized to get minimum surface roughness in the Design-Expert software. Tool speed 1950 rpm and compression 1.9 mm are selected as the optimum parameters. To accomplish nanolevel surface finish, the final experiments are carried out based on the optimum parameters settings. OFHC copper has the high tendency to get oxidized. Therefore, final experiments are carried out under both dry condition and presence of BTA solution with the same optimum parameters setting. In dry machining, the polishing pad is loaded progressively after each experiment. During finishing with BTA solution, the solution is sprayed to the finishing zone to control the heat generation, to reduce oxidation and facilitate the removal of micro-chips from the polishing pad that reduces the chance of pad loading. Total 10 passes are completed for both the experiments and surface topography of the workpiece is analyzed to assess the surface roughness after each pass. Fig. 4 (a) shows the topography of as-received Cu where the facing marks are clearly visible. Fig. 4 (b) and (c) shows the topography of surface after 5 passes for dry finishing and with BTA solution, respectively. More residual facing marks are observed on the surface processed by dry finishing (Fig. 4(b)) compared to the wet condition (Fig. 4(c)) and Sa value are 131 nm and 97 nm for dry and wet finishing, respectively. Fig. 4(d) and (e) shows the topography of the surface having Sa value 50 nm and 10 nm after 10 passes for dry finishing and with BTA solution, respectively . Fig. 5 shows the variation of surface roughness with finishing passes for both dry and wet finishing. It is observed that the finishing rate is decreasing gradually as the no of passes increases. For dry finishing, at the end no significant improvement of finishing is observed as the polishing pad is loaded completely which is shown in Fig. 6 (a). It is perceived that the improvement rate during wet finishing after each finishing pass is greater than the dry one because the micro chips are flushed partially from the pad surface as shown in Fig. 6(b). Hence, it is conceived that the finishing with BTA solution has pronounced ability to achieve nanolevel surface finish of copper. Using this method, 10 nm Sa is achieved within 70 min. SEM micrographs of as-received and finished by BTA solution are shown in Fig. 7 (a) and (b), respectively.

Fig.4. 3D topography of the OFHC copper sample (a) As-received (Sa = 5.135 µm), (b) Dry machining after 5 passes (Sa = 131 nm), (c) With BTA after 5 passes (Sa = 97 nm), (d) Dry machining after 10 passes (Sa = 50 nm) and (e) With BTA after 10 passes (Sa = 10 nm)

Fig.5. Variation of surface roughness with the finishing passes for both dry machining and with BTA.

Fig.6. Topography of polishing pad after 10 passes (a) Dry machining and (b) With BTA solution (1500X).

Fig.7. SEM micrographs of OFHC Cu (a) As-received and (b) Finished using BTA solution after 10 passes (2000X).

The facing marks are noticeable in Fig 7 (a) but no substantial finishing marks are observed on the finished surface (Fig. 7 (b)).

4. CONCLUSION

From the preceding discussions, the following conclusion may be drawn.

- ➢ Tool speed and compression have profound effect on surface finish.
- ➢ High compression of tool as well as tool rotation contribute more material removal but loading of polishing pad is a major problem during finishing of ductile material like copper. The surface finish may deteriorate consequently. In lower value of parameter setting though the pad loading is not an issue but material removal is low. Therefore, surface finish improvement rate is low.
- ➢ The improvement rate (reduction in Sa) is greater in case of BTA solution than dry condition as the micro-chips from the polishing pad are flushed by the solution during finishing. The surface finish around 10 nm is achieved using this method in 70 minutes without substantial deep finishing marks.

ACKNOWLEDGEMENT

The authors acknowledge the funding support from the Institute under ISIRD grant and Board of Research in Nuclear Sciences (BRNS), Bombay under young scientist research award.

References

- [1] Ahmad A., Optomechanical Engineering Handbook, CRC Press LLC, Florida, USA, 1999.
- [2] Khan D.A. and Jha S., Synthesis of polishing fluid and novel approach for nanofinishing of copper using ball end magnetorheological finishing process, *Materials and Manufacturing Processes*, 2017 May 9(just-accepted).
- [3] Lee C.S., Koumvakalis N. and Bass M. A., Theoretical model for multiple‐pulse laser‐induced damage to metal mirrors. *Journal of applied physics,* **54** (1983) 5727-5731.
- [4] Zhang X, and Zhang Y., Study on the surface quality of a diamond-turned oxygen-free high-conductance copper reflector used in a high-power CO2 laser, *Optical Engineering*, **36(3)** (1997) 825-830.
- [5] Gutmann R. J., Steigerwald J.M., You L., Price D.T., Neirynck J., Duquette D.J. and Murarka S.P., Chemicalmechanical polishing of copper with oxide and polymer interlevel dielectrics, *Thin Solid Films*, **270 (1-2)** (1995) 596-600.
- [6] Luo Q., Campbell D. R. and Babu S. V., Proceedings of the 1st international VMIC specialty conference on CMP planarization (1996).
- [7] Steigerwald J. M., Duquette D. J., Murarka S. P. and Gutmann R. J., Electrochemical potential measurements during the chemical‐mechanical polishing of copper thin films, *Journal of The Electrochemical Society*, **142(7)** (1995) 2379-2385.
- [8] Luo Q., Campbell D. R. and Babu S. V., Chemical– mechanical polishing of copper in alkaline media, *Thin Solid Films*, **311(1)** (1997)177-182.
- [9] Ahn Y., Yoon J. Y., Baek C.W. and Kim Y.K., Chemical mechanical polishing by colloidal silica-based slurry for micro-scratch reduction, *Wear*, **257(7)** (2004) 785-789.
- [10] Mahajan K.A., Sadaiah M. and Gawande S.H., Experimental investigations of surface roughness on OFHC copper by diamond turning machine, *International Journal of Engineering Science and Technology*, **2 (10)** (2010) 5215–5220.
- [11] Dumas P., Golini D. and Tricard M., Improvement of figure and finish of diamond turned surfaces with magneto-rheological finishing (MRF), *Proc. SPIE*, **5786** (2005) 296-304.
- [12] Kala P., Kumar S. and Pandey P. M., Polishing of copper alloy using double disk ultrasonic assisted magnetic abrasive polishing, *Materials and Manufacturing Processes*, **28(2)** (2013) 200-206.
- [13] Beaucamp A., Namba Y. and Charlton P., Process mechanism in shape adaptive grinding (SAG), *CIRP Ann. Manuf. Technol.* **64** (2015) 305–308.
- [14] Walker D. D., Freeman R., Morton R., McCavana G. and Beaucamp A., Use of the "Precessions" TM process for prepolishing and correcting 2D & 2 1/2D form, *Opt. Express.* **14** (2006) 11787-17795.
- [15] Beaucamp A.T., Namba Y., Charlton P., Jain S. and Graziano A. A., Finishing of additively manufactured titanium alloy by shape adaptive grinding (SAG), *Surface Topography: Metrology and Properties*, 3(2) (2015) 024001.
- [16] Vogt C., Sinzinger S. and Rascher R., A study on elastic grinding tools, *Journal of the Optical Society of America* (2014) OTu3B.3.
- [17] Walker R., The use of benzotriazole as a corrosion inhibitor for copper, *Anti-corrosion methods and materials*, **17(9)** (1970) 9-15.
- [18] Samuels L. E., Metallographic polishing by mechanical methods, *Asm International* (2003).