Investigations on Surface Integrity and Electrochemical Behavior of Machined Co-Cr-Mo Bio-implant Alloy

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Abstract: A decisive constraint for the long-term stability of the artificial joint is to minimize the release of debris particles. The wear/debris induced osteolysis and aseptic loosening are the result of failure of metal-on-metal joint implants. SPD processes have been used to adapt the surface integrity properties by generating ultrafine or even nano-sized grains and grain size gradients in the surface region of work materials. These fine grained materials often show enhanced surface integrity properties and improved functional performance (wear resistance, corrosion resistance, fatigue life, etc.) compared with their predictable coarse grained counterparts. To identify the implant material's post machined behaviour in biological environment, the experiments were planned by precision CNC turning process and accordingly post machined surfaces were analyzed by contact type and electrochemical measurement processes. The work includes effect of machining parameters on machined surface roughness and corrosion rate by an electrochemistry of Co-Cr-Mo bio-implant alloy. The minimum machined surface roughness value 0.450 µm shows minimum corrosion rate as 0.00002 mm/year. It is also shown that feed rate is having predominating effect on machined surface roughness and rake angle is on corrosion rate of Co-Cr-Mo bio-implant alloy.

Keywords: Co-Cr-Mo Bio-implant alloy, Corrosion rate, Electrochemistry, Surface roughness and Taguchi DOE

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1 INTRODUCTION

The most popular successful surgical treatments for patients suffering from arthritis and trauma, is the arthroplasty (artificial joint replacement). On average 1 million arthroplasties are annually performed worldwide [1]. Nowadays vivo metallic biomaterial used is the stainless steel followed by Titanium, Ti alloy and Cobalt–Chromium–Molybdenum, Co-Cr-Mo alloy [2], [3]. Metal-on metal bearings have a long history in total hip replacement. In the past, total hip replacements (THR) involved the use of metal-metal Co-Cr-Mo alloys as the implant material, largely due to their relatively high strength, bio-compatibility and corrosion resistance. However, limitations in the manufacturing methods adversely affected the performance of these joints [4].

In addition, young patients with dynamic lifestyles undergoing total hip arthroplasty will require improved performance over a period of 30-40 years from now. Co-Cr-Mo alloy is the most suitable alloy often used in sliding parts, such as artificial hip and knee joints. When it is to be used for the head of an artificial joint, a mirrored finish is necessary to extend the life of the joint by compact abrasive wear and improved chemical stability. The search for a longer wearing behavior has led to the development of metal on metal hip implants. These devices are commonly used today in patients less than 60 years of age [8]. Currently, more and more hip implants are machined from wrought Co-Cr-Mo alloys. There are very few precision processes which can be employed for machining of Co-Cr-Mo alloy such as grinding and turning. Precision turning is one of the important process producing the higher accuracy and surface finish on metal implants.

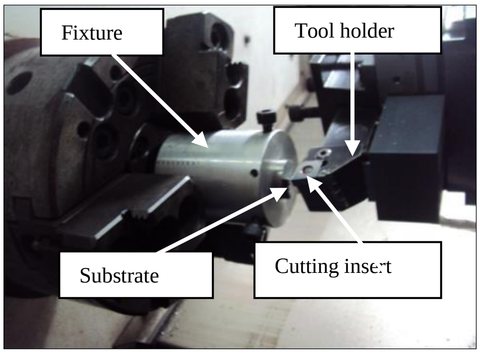
In the present investigation, hip implant alloy was simulated in physiological environment suitable for normal patients of 23-75 ages. Hip joint is lubricated by means of a viscous fluid known as synovial fluid. It also functions as a media for exchange of nutrients and regulatory cytokines [12]. Synovial fluid is found to have gained its lubricating and vibration damping capability from Hyaluronic Acid (HA) and is generally found in synovial membrane around the hip bone. It also gives high viscosity to the synovial fluid owing to hydrophilic nature of the polysaccharide bonds. It is found in several connective tissues including skin, umbilical cord, synovial membrane, etc. [14]. Surface treatment or surface modification is considered as one of the major concern in recent developments of metallic biomaterials. Surface characteristics of biomaterials have a vital effect on the adhesion process of adjacent cells. The recent review of some key publications which emphasizes on studying the Co-Cr-Mo hip implants based on laboratory as well as clinical experiences is presented below.

Geetharani et al. described the surface morphology and wear behavior of Co-Cr-Mo alloy by using polishing and coating methods (MPCVD) [1]. Author observed that “brain-coral” like surface morphology and rough surface morphology of 5 µm. Nevelos et al. tried to address the main metallurgical design issues in metal-on-metal bearing design [2]. Reclaru et al. reported that “inclusions” type microstructure was observed in electrochemical testing and polishing methods employed on Co-Cr-Mo alloy [3]. Howie et al. investigated twenty-four cobalt-chrome alloy McKee-Farrar matching acetabular and femoral components after 16 years in situ for wear and loss of sphericity [4]. The authors classified the wear on the components into four parts: polishing wear; fine abrasive wear; multidirectional dull abrasive wear and unidirectional dull abrasive wear.

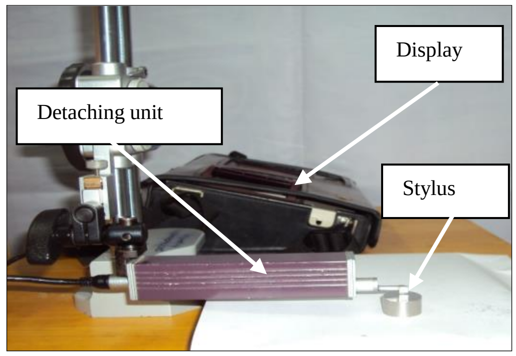
Affatato et al. investigated the effect of surface profile parameters on the amount of wear in metal artificial hip joints [5]. The surface roughness of the metallic ball heads was qualified in terms of average surface roughness R_a, total surface roughness R_t and skewness R_{sk}. Ohmori et al. observed the surface roughness (R_a) of 7 nm and also reported that surface roughness is superior than polished surface roughness [6]. Author used ELID grinding process in the experiments. Lee et al. found significant improvement in mechanical properties of Ni free Co-Cr alloy even under the as-cast condition [7]. The author investigated the hardness and tensile strength of Co-Cr-Mo alloy by preparing different chemical compositions and also used vacuum induction melting process for the experiments. Grgazka et al. analyzed the influence of chosen modifiers on mechanical properties of composite materials on the base of Co-Cr-Mo alloy [8].

Young Chan et al. machined Co-Cr-Mo alloy using elliptical vibration cutting process [9]. A fine mirrored surface (with a maximum surface roughness under 25 nm P-V) was maintained up to a cutting length of about 14 m. Shu Yang et al. have done systematic experimental study to investigate the influence of different burnishing parameters on distribution of grain size, phase structure and residual stresses of the processed material [10]. The wear performance of the processed Co-Cr-Mo alloy was tested via pin-on-disk wear tests. Uddin et al. carried out machining trials on Co-Cr-Mo femoral heads and optimized the cutting parameters in finishing of femoral heads [11]. Author reported that the cutting speed, depth of cut and feed rate influenced surface roughness significantly, while both the feed rate and depth of cut were the dominant factors impacting the sphericity of femoral heads.

Enhancement of the service life of implant alloys and its assessment is the main purpose of this research on Co-Cr-Mo implant alloy. Turning or various machining operations can significantly alter the surface morphological properties. Face turning operation causes the surface of the component to have bad surface finish owing to high stresses and deformations. This also increases effective surface area of the corroding sample. But this effect of morphology on the corrosion behavior of Co-Cr-Mo has not been addressed yet. It can be one of the methods of tailoring the surface characteristics of Co-Cr-Mo alloy. This lacuna is tried to be fulfilled in this investigation.

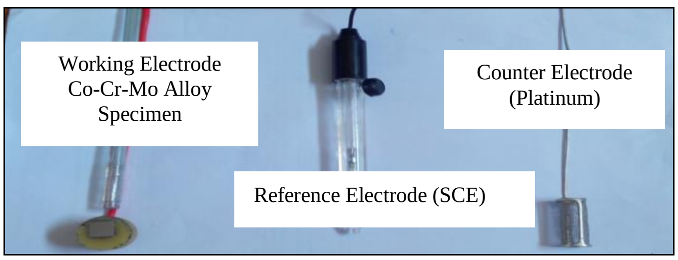


(a)



(b)

Fig. 1 (a) Closed view set up of precision turning and (b) surface roughness testing



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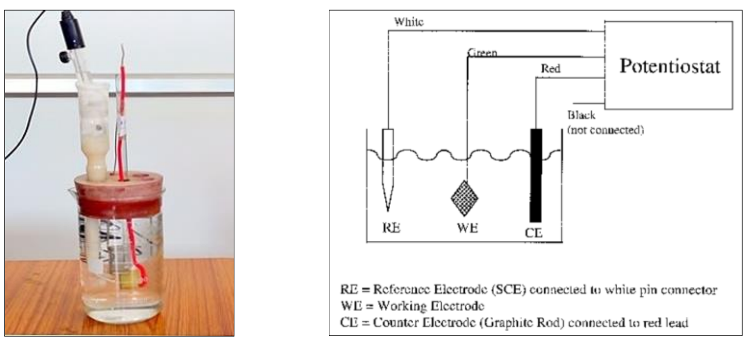


(b)

Fig. 2 (a) The components of an electrochemical cell for polarization test and (b) Assembly of an electrochemical cell

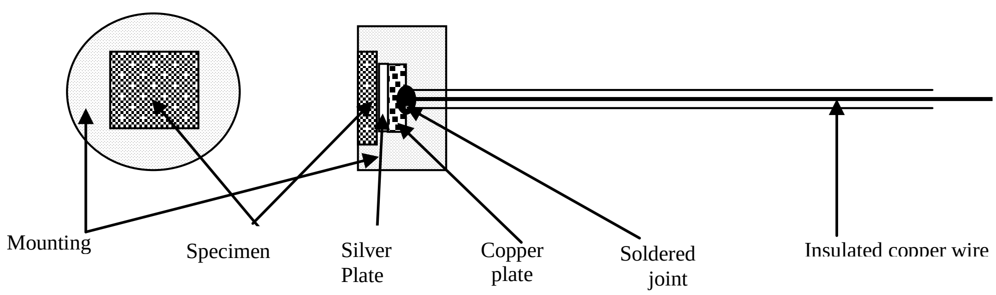


Fig. 3 The structure of the specimen for polarization studies

2 EXPERIMENTAL DETAILS

Precision turning process is employed for investigation on surface integrity of Co-Cr-Mo bio-implant alloy in dry cutting environment. A production type CNC turning precision lathe (Model DX-150 Make JYOTI India) having maximum spindle speed 4500 rpm and 20 KW capacity is used for conducting the experiments.

Table 1 The experimental design with actual standard L9 array and factor values for precision turning

Sr. No.	Cutting Parameters			
	DOC (mm)	Feed (mm/rev)	Cutting speed (m/min)	Rake angle (Degrees)
1	0.2	0.1	65	7
2	0.2	0.2	125	0
3	0.2	0.3	190	-11
4	0.3	0.2	65	-11
5	0.3	0.3	125	7
6	0.3	0.1	190	0
7	0.5	0.3	65	0
8	0.5	0.1	125	-11
9	0.5	0.2	190	7

Taguchi experimental design L9 orthogonal array is used for designing the parameter combinations for each experimental trial. In this orthogonal array, number of factors are 4 and number of levels are 3; Hence total numbers of runs are 9. The response variables are the arithmetic average surface roughness *Ra* and corrosion rate *Cr* for the experiments in precision turning. The input control factors selected for the experiments are: depth of cut (0.2-0.3-0.5 mm), feed rate (0.1-0.2-0.3 mm/min), cutting speed (65-125-190 m/min) and rake angle of cutting tool (7^0-0^0--11^0). Table 1 shows the experimental runs with the assigned factors to each of the columns of OA for precision CNC turning process.

The work material used in the present investigation is bio-implant alloy, which is a casted low carbon wrought version of ASTM F75 Co-Cr-Mo Alloy. The actual chemical composition (in wt. %) of Co-Cr-Mo alloy [Co, 28.61% Cr, 5.53% Mo, 0.10% C, 0.72% Si, 0.52% Mn, 0.01% P, 0.01% Ni, 0.007% S, 0.18% Fe] is provided by the supplier. A Co-Cr-Mo alloy bar (25 mm diameter) is used to prepare a sample of 20 mm in diameter and a thickness of 10 mm.

2.1 Machining Tests

Initially nine workpieces to the required length from a long rod of Co-Cr-Mo were cut as substrates. These substrates are exactly made to size of 20 × 10 mm thickness. An aluminium turning fixture was fabricated to size of 50 × 120 mm length to facilitate holding of substrates during turning operation. Three screws are used to hold the substrate tight against the fixture. Fixture along with substrate is mounted on the three jaw chuck of the machine. After proper mounting pressure, fixture is trued properly for perfect rotation as shown in Fig. 1 (a). To begin, a rough face cut of 0.5 mm is employed on each substrate and finish cut is taken on surface of 20 mm diameter. Finally each substrate is machined as per the experimental trials given in Table 1. After each experiment, the machined substrate is kept in a vacuum tight small container.

After machining trials, the machined surfaces were measured to analyze profile on surface roughness tester of Model SJ- 210 Mitutoyo, made in Japan shown in Fig. 1 (b). The 2D profiles are traced for the 10 mm assessment length with 0.25 mm sampling length.

2.2 Electrochemistry Tests

Electrochemical studies executed in this work are time-potential, electrochemical impedance spectroscopy and potentiodynamic polarization tests following ASTM standard G5-14 [18]. For simulating human body conditions, all of these tests were conducted for all machined samples at constant temperature of 37º C. Saturated calomel electrode (SCE) was used as the reference electrode via a luggin probe. Luggin probe is a tube with one narrow end (kept close to sample-working electrode) and one wide end enough to accommodate reference electrode. Lower half of its volume is filled with 3% Agar-Agar + 40% KCl jelly and other half filled with saturated KCl solution, in which reference electrode is dipped. A working electrode of surface area of 1 cm^2 and a platinum counter (auxiliary) electrode were used for polarization studies.

An individual electrodes and a typical cell setup is shown in Fig. 2 and 3 respectively. Polarization studies were carried out in Hyaluronic acid solution in stagnant conditions. The cathodic part of polarization plot reveals kinetics of cathodic reactions in the system and is used to find out i_{corr} and E_{corr}. This would supplement the data obtained in weight loss studies. The anodic part of the plot indicates whether test alloy undergoes passivation or not. Corrosion current density which was found from anodic part, then can be compared with corrosion current density obtained from cathodic part to ascertain which reaction is controlling overall corrosion process.

Specimens for polarization studies were prepared by soldering a piece of insulated copper wire at one flat face of copper plate (10 mm x 10 mm). This copper plate is then connected to the cut specimen with the help of conducting silver paste. Then this complete assembly is mounted using araldite to embed the solder joint and copper plate. The electrical connection between potentiostatic leads and copper wire was established by an insulated copper wire as shown in Fig. 3. A step by step procedure can be used for finding the corrosion rate as discussed below.

2.2.1 OCP (Open Circuit Potential) Measurement

A computer controlled scanning potentiostat (Biologic VMP 300) interfaced with computer is used for polarization studies. After the completion of assembly, electrochemical cell is allowed to reach a stable open circuit potential for 1 hour. Stable OCP was attained in less than 60 minutes in all the cases. The range of polarization potential was from -500 mV (Vs OCP) to +1000 mV (Vs OCP) at scan rate of 0.5 mV/s.

2.2.2 Electrochemical Impedance Spectroscopy (EIS)

EIS is an AC impedance technique of corrosion analysis. EIS is an abbreviation for Electrochemical Impedance Spectroscopy which provides an impedance spectrograph after analysis of the specimen. Impedance is the obstruction to current or electron flow. In the case of direct current (DC), only resisters provide this effect. However, for AC current other circuit elements such as inductors and capacitors also will influence the electron flow. AC impedance method is used for determining corrosion rate, inhibitor performance, coating performance, and passive layer characteristics. This provides data on both electrode capacitance and charge transfer kinetics.

2.2.3 Potentiodynamic Polarization Method

This technique involves holding a specimen's surface at a series of constant potentials versus a reference electrode, and then measuring the current necessary to maintain each of the applied potentials. This test is useful to measure dependence of the current on the applied potential of the sample and the parameters which are important for understanding the corrosion behaviour of the machined material in the environment.

3 RESULTS AND DISCUSSION

A satisfactory post machined performance of the Co-Cr-Mo alloy is vital for improving the life of the implant in a human body. The surface characteristics especially the surface roughness and its effect on corrosion rate of machined Co-Cr-Mo substrates are presented in this section. Table 2 shows the roughness values (*Ra*) and its corrosion rate *(Cr)* for each machined substrate.

Table 2 The Roughness and corrosion rate of machined substrates

Substrate No.	Roughness (μm)	Corrosion rate (mm/year)
1	0.663	0.00016
2	0.533	0.00016
3	0.926	0.00018
4	0.556	0.00017
5	0.936	0.00012
6	1.006	0.00023
7	0.450	0.00002
8	0.900	0.00018
9	1.056	0.00014

The main effects plots for surface roughness (ANOM) and the table of analysis of variance (ANOVA) are shown in Fig. 4 and Table 3 respectively. It is observed from the ANOVA that the feed rate shows statistical significance on surface roughness at 95% confidence level as the P-value in the ANOVA for any input variable is less than 0.05. The percentage contribution of the input variables influencing the surface roughness is depth of cut: 6.08 %, cutting speed: 7.22 %, feed rate: 68.95 % and rake angle: 17.75 % showing the higher impact of tool material on the surface roughness. The effect of each input factors on the surface roughness is presented using ANOM plots.

Table 3 ANOVA Table for Surface Roughness

Source	DF	SS	MS	F	P	% Cont
a_p	2	0.0256	0.0128	0.19	0.828	6.08
*f	2	0.2903	0.1452	6.66	0.030	68.95
V_c	2	0.0304	0.0152	0.23	0.799	7.22
a	2	0.0747	0.0374	0.65	0.556	17.75
Residual error	0	-	-	-	-	-
Total	8	0.4210	-	-	-	-

*Statistically significant factor

The main effects plots for corrosion rate (ANOM) and the table of analysis of variance (ANOVA) are shown in Fig. 5 and Table 4 respectively. It is observed from the ANOVA that none of the factors shows statistical significance on surface roughness at 95% confidence level as the P-value in the ANOVA for any input variable is not less than 0.05. But among the selected parameters, the rake angle is having more dominating effect on corrosion rate.

Table 4 ANOVA Table for Corrosion rate

Source	DF	SS	MS	F	P	% Cont
a_p	2	3E-8	2E-9	0.02	0.976	8.30
f	2	2E-8	1E-8	0.60	0.579	5.55
V_c	2	1E-8	4E-9	0.98	0.429	2.77
$*a$	2	3E-7	3E-8	4.13	0.074	83.33
Residual error	0	-	-	-	-	-
Total	8	3.6E-7	-	-	-	-

*Statistically significant factor

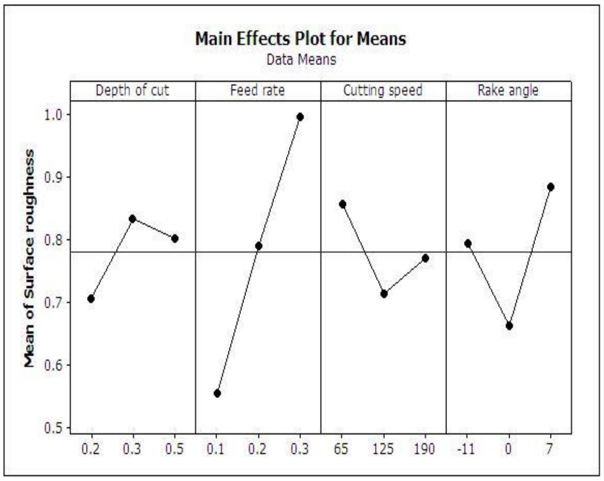


Fig. 4 The Main effect plots for surface roughness

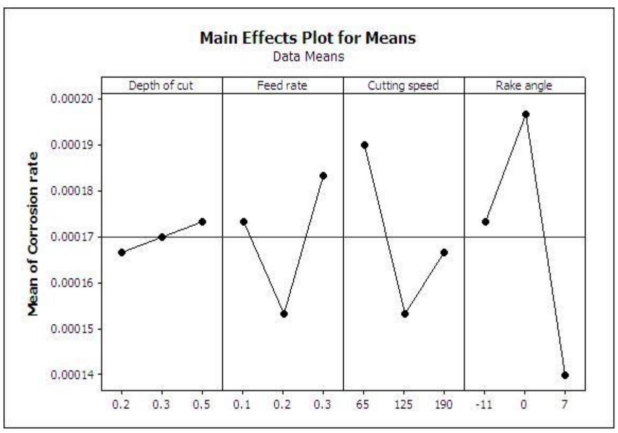


Fig. 5 The Main effect plots for corrosion rate

The percentage contribution of the input variables influencing the surface roughness is depth of cut: 6.08 %, cutting speed: 7.22 %, feed rate: 68.95 % and rake angle: 17.75 % showing the higher impact of tool material on the surface roughness. The effect of each input factors on the surface roughness is presented using ANOM plots.

3.1 Effect of depth of cut on roughness and corrosion rate

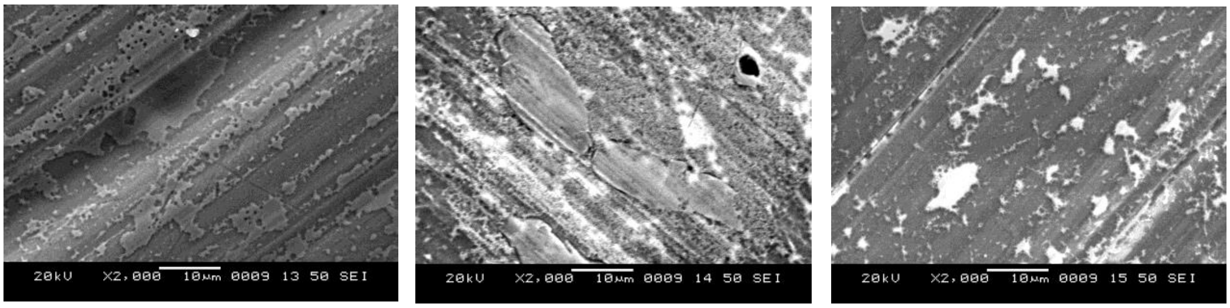
Depth of cut show non-linear and linear trend on the roughness of machined surface and corrosion rate respectively. When the depth of cut increases from 0.2 mm to 0.3 mm, surface roughness considerably increases from 0.70 µm to 0.85 µm. However, there is a drastic decrease in roughness up to 0.80 µm later at 0.5 mm depth of cut. In case of corrosion rate, when depth of cut increases from 0.2 mm to 0.5 mm, corrosion rate also increases from 0.000168 mm to 0.000175 mm. In dry cutting when the depth of cut is minimum, continuous chips are produced due to smooth shearing action. Therefore, these chips are generated by shear stresses, the former by separation along the simple shear plane, and later in shear zone. Therefore, the machined surface is smooth and accurate and it affects directly to the corrosion rate due to some machined surface irregularities

3.2 Effect of feed rate on roughness and corrosion rate

The effect of feed rate is linear and non linear on the roughness of machined surface and corrosion rate respectively. When the feed rate increases from 0.1 mm/rev to 0.2 mm/rev, surface roughness considerably increases from 0.55 µm to 0.80 µm. Further increment in feed rate up to 0.3 mm/min, the surface roughness again increases to 1 µm. It was observed that, when the feed rate is high, a sudden temperature rise between tool and chip interface prevails on account of higher friction. In this case, burned black chips are generated while machining. However, when the coolant is applied in machining there is a possibility of sudden temperature fall in machining zone and chips fragment remain same on the machined surface. Therefore at higher feed rate, the surface roughness value is also increased. Therefore, it is effectible on final surface integrity of machined surface. Due to this, the generated surface is having quiet higher corrosion rate than other machined surfaces.

3.3 Effect of cutting speed on roughness and corrosion rate

The cutting speed shows moreover similar trend on the roughness and corrosion rate of machined surfaces. When the cutting speed increases from 65 m/min to 125 m/min there is a decrease in surface roughness from 0.85 µm to 0.70 µm. However, a sudden increase in surface roughness from 0.70 µm to 0.76 µm is observed when the cutting speed increases from 125 m/min to 190 m/min. This can be attributed to the fact that the spindle speed has less effect on surface roughness.



(a_p=0.2, f=0.1 , V_c=65 , $a=7^0$) (a_p=0.3, f=0.2 , V_c=65 , $a=-11^0$) (a_p=0.3, f=0.1 , V_c=190 , $a=0^0$)

Fig. 6 SEM Micrographs of machined surfaces

The same effect is shown on corrosion rate like as on machined roughness. When the cutting speed increases from 65 m/min to 125 m/min there is a decrease in corrosion rate from 0.00019 mm to 0.00015 mm. However, there is a sudden increase in corrosion rate from 0.00015 mm to 0.000165 mm when the cutting speed increases from 125 m/min to 190 m/min. At medium cutting speeds due to temperature fall in shear zone it directly affects smooth breaking the chip from machined surface. Therefore, the surface generated at this condition is better and has less corrosion rate.

3.4 Effect of rake angle on roughness and corrosion rate

The rake angle shows opposite trend on the roughness and corrosion rate of the machined surface. When the rake angle increases from -11^0 to 0^0, there is a decrease in surface roughness from 0.80 µm to 0.65 µm. However, there is a sudden increase in surface roughness from 0.65 µm to 0.90 µm when the rake angle increases from 0^0 to 7^0. The effect is quiet similar on corrosion rate as non linear like as on machined roughness. When the rake angle increases from -11^0 to 0^0 there is increase in corrosion rate from 0.000172 mm to 0.00019 mm. However, there is a sudden decrease in corrosion rate from 0.00019 mm to 0.00014 mm when the rake angle is 7^0. Positive rake angle gives higher machined surface roughness and lower corrosion rates

4 CONCLUSIONS

Following conclusions can be deducted from the investigation carried out CNC turning process on Co-Cr-Mo alloy.

- As far as the effect of input factors is concerned, the feed rate shows dominating effect on surface roughness for the CNC turning operation, whereas the factor rake angle have nearly predominant influence on the machined surface roughness.
- Also in case of corrosion rate assessment, the input factor rake angle is having dominating effect on corrosion rate of machined surfaces.
- It is found that minimum feed rates and a positive rake angle gives the good surface quality with minimum corrosion behavior for preparing Co-Cr-Mo implants. It is also found that the tool material of CBN is effective for machining of Co-Cr-Mo implant material.

5 NOMENCLATURES

a_p= Depth of cut

f = Feed rate

V_c= Cutting speed

α = Rake angle

Ra= Surface roughness value

Cr=Corrosion rate

ASTM= American Society for Testing Materials

SPD= Severe Plastic Deformation

HA= Hyaluronic Acid

OCP= Open Circuit Potential

SCE= Saturated Calomel Electrode

ANOM= Analysis of Means

ANOVA= Analysis of Variance

OA= Orthogonal Array

CBN= Cubic Boron Nitride

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