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# Influence of Machining Parameters on Surface Roughness in Chemical Machining of Stainless Steel 304

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## Abstract:

Chemical machining is a well-known nontraditional machining process and is the controlled chemical dissolution of the machined work piece material by contact with a strong acidic or alkaline chemical reagent. It is also called as chemical etching. The present work is aimed at studying the effect of machining time, machining temperature, etching solution concentration on the surface finish of stainless steel 304 using mixed of acids ($HCL+HNO_3+HF+ H_2SO_4+H_2O$). Alloy samples are of (33×33×6) mm dimensions. Three machining temperatures (45, 50 and 55 ºC) for each of which three machining times (3, 6, and 9 min) were used as machining conditions. Surface roughness increases with the machining temperature and machining time. An assessment of CHM was achieved by empirical models for selecting the appropriate machining conditions of the required surface finish. The models were designed based on multiple regression method via Mtb 16 software.

**Keywords**: CHM, stainless steel 304, roughness, regression.

# تأثير عوامل التشغيل على خشونة السطح في التشغيل الكيميائي

## الخلاصة:

يعد التشغيل الكيميائي احد طرق عمليات التشغيل اللاتقليدية, وهي عملية كيميائية مسيطر عليها لتشغيل قطعة معدنية بواسطة التماس مع حوامض قوية او محاليل كيميائية قلوية, وتسمى ايضا بالحفر الكيميائي. الغرض من هذا البحث هو دراسة تاثير زمن التشغيل , درجة حرارة التشغيل وتركيز المحلول المستخدم على عملية الانهاء السطحي لمعدن مقاوم للصدأ 304 بأستخدام محاليل مختلطة ( $HCL+HNO_3+HF+ H_2so_4+H_2O$) . العينات تمتلك ابعاد mm (33×33×6) مع ثلاث انماط من درجات الحراة (45, 50 and 55) مº عند ازمان تشغيل مختلفة (3, 6, and 9 min) حيث تستخدم كشروط للقطع. بينت الدراسة ان الانهاء السطحي يزداد مع زيادة درجة حرارة التشغيل وزمن التشغيل, وقد تحقق تقييما للعملية من خلال نماذج تجريبية لاختيار شروط التشغيل المثالية للانهاء السطحي المطلوب. النماذج صممت على اساس اسلوب الانحدار المتعدد باستخدام برنامج Mtb 16.

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## INTRODUCTION

Chemical machining (CHM) process is a controlled chemical dissolution (CD) of a workpiece material by contact with strong reagent (etchant). Special coatings called maskants protect areas from which the metal is not to be machined. CHM is one of the non-conventional machining processes. The advancement of technology causes the development of many hard-to-machine materials: stainless steel, super alloys, ceramics, refractories and fiber-reinforced composites due to their high hardness, strength, brittleness, toughness and low machinability properties. Sometimes, the machined components require high surface finish and dimensional accuracy, complicated shape and special size, which cannot be achieved by the conventional machining processes. Moreover, the rise in temperature and the residual stresses generated in the workpiece due to traditional machining processes may not be acceptable. These requirements have led to the development of non- traditional machining (NTM) processes [1]. In these processes, the conventional cutting tools are not employed. Instead, energy in its direct form is utilized.

Chemical energy is used in chemical machining. This process is the precision contouring of metal into any size, shape or form without the use of physical force, by a controlled chemical reaction. Material is removed by microscopic electrochemical cell action, as occurs in corrosion or chemical dissolution of a metal. This controlled chemical dissolution will simultaneously etch all exposed surfaces even though the penetration rates of the etch may be only 0.0005–0.0030 in./min. The basic process takes many forms: chemical milling of pockets, contours, overall metal removal, chemical blanking for etching through thin sheets; photochemical machining (PCM) for etching by using of photosensitive resists in microelectronics; chemical or electrochemical polishing, where weak chemical reagents are used (sometimes with remote electric assist) for polishing or deburring and chemical jet machining, where a single chemically active jet is used [2].

Chemical machining offers virtually unlimited scope for engineering and design ingenuity, to gain the most from its unique characteristics, it should be approached with the idea that this industrial tool can do jobs not practical or possible with any other metal working methods.[3].

**Fadaei and Imanian** [4] observed an improvement in surface finish by adding triethanolamine (TEA) which was able to decrease the etch rate. This was due to the ability of TEA to decrease the difference between the corrosion rate of grain and grain boundaries, thus reducing the pitting defects and producing better surface finish. Further agitation-isopropyl alcohol (IPA) will improve the surface finish. Besides, prolonged etching period is also known to increase the etch rate and cause irregular hillocks and defects to appear on the material surface. **Tambol Mabkha et al**. [5] discussed the pickling mechanism of HCl and H2SO4 based on the weight loss and the pickled surface qualities. It was found that the first step pickling efficiency directly affected the surface qualities of the final pickled sample. HCl solution showed much lower pickling efficiency than H2SO4 solution. This resulted in a high concentration of remaining oxide scales and intergranular attack at the Cr-depleted layer, which cannot be completely removed in the second pickling step. Increasing of HCl concentration and electrolytic current did not improve the pickling efficiency.

In **(2000), H. Sachdev [6]** studied the parameter effects during the etching process. According to the researcher, the parameters like temperature, time, and concentration can effect on the surface of the material. The MRR of the material was increased with increase in temperature, time and concentration. **Liu et al.** [7] reported that the surface roughness was closely related to photoresist's adhesive quality. Their results showed that better surface roughness was found in chromium photoresist, compared to fused silica wafer.

## Materials and Test Used in The Present Study

### Alloy under study

A stainless steel 304 of (750 ×350×6) mm with a chemical composition shown in table (2) was used in this work. The analysis of the chemical composition was carried out in the State Company for Inspection and Engineering Rehabilitation, S.I.E.R (Baghdad-Iraq).

**Table (1) Chemical composition of stainless steel 304.**

| Cr | Ni | Mn | N | S | C | Si | P |
|---|---|---|---|---|---|---|---|
| 18% | 8% | 2% | 0.1% | 0.03% | 0.08% | 0.75% | 0.03 |

**Table (2) Chemical composition of alloy under study.**

| Cr | Ni | Mn | N | S | C | Si | P |
|---|---|---|---|---|---|---|---|
| 16.38% | 9.17% | 1.31% | 0.1% | 0.02% | 0.06% | 0.32% | 0.04 |

### Maskent Material

Depending on the study alloy, polymer was selected to prepare the maskent material according to table (3).

**Table (3). Masking materials for various chemical machined materials [8].**

| **Workpiece material** | **masking material** |
|---|---|
| Aluminum and alloys | Polymer, Butyl rubber, neoprene |
| Iron based alloys | Polymer, Polyvinyl chloride, |
| | Polyetilien butyl rubber |
| Nickel | Neoprene |
| Magnesium | Polymer |
| Copper and alloys | Polymer |
| Titanium | Polymer |
| Silicon | Polymer |

### Etchant Solution

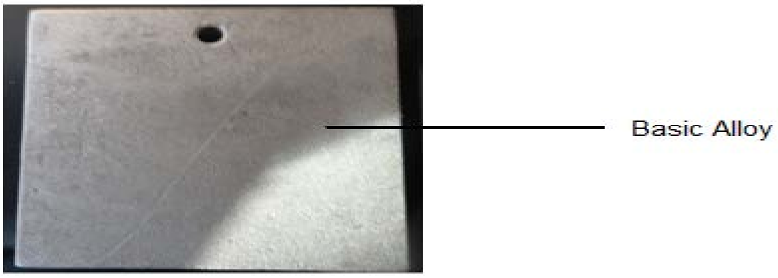
The used etchant was a mixture of acids with a chemical composition and concentration demonstrated in table (4).

**Table (4) Chemical composition and concentration of etchant solution.**

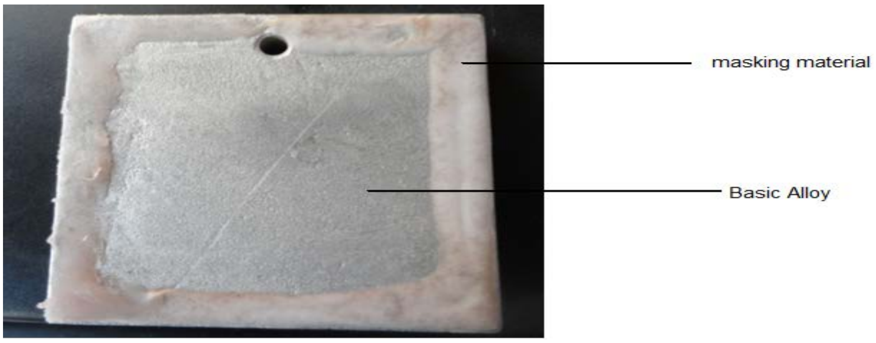
| Chemical composition | Etchant concentration |
|---|---|
| HCL+HNO3+HF+ H2so4+H2O | 5ml+4ml+4ml+5ml+82ml |

## Samples Preparation for Coating

The samples were cleaned from dirt, dust, fats, oil and organic compounds using alcohol (ethanol 99.5%) before coating with maskant material. After pouring the polymeric masking material, the mold (used for coating the samples) was kept in an oven at 70 ºC for 30 min for drying. One face of a sample was left without coating, which represents the area to be machined. A hole of 2 mm diameter was drilled in each simple for the purpose of suspension in the etchant solution. The prepared specimens have dimensions of (33×33×6) mm. Figure (1) shows the samples before and after the coating.



a. Sample before coating



B. Sample after coating

**Figure (1) Samples before and after coating**.

The machining process was achieved via magnetic stirrer thermostat (made in Germany). It contains a thermostat to regulate the temperature of etchant during the machining operation and controller on velocity of stirrer, as shown in fig (2).

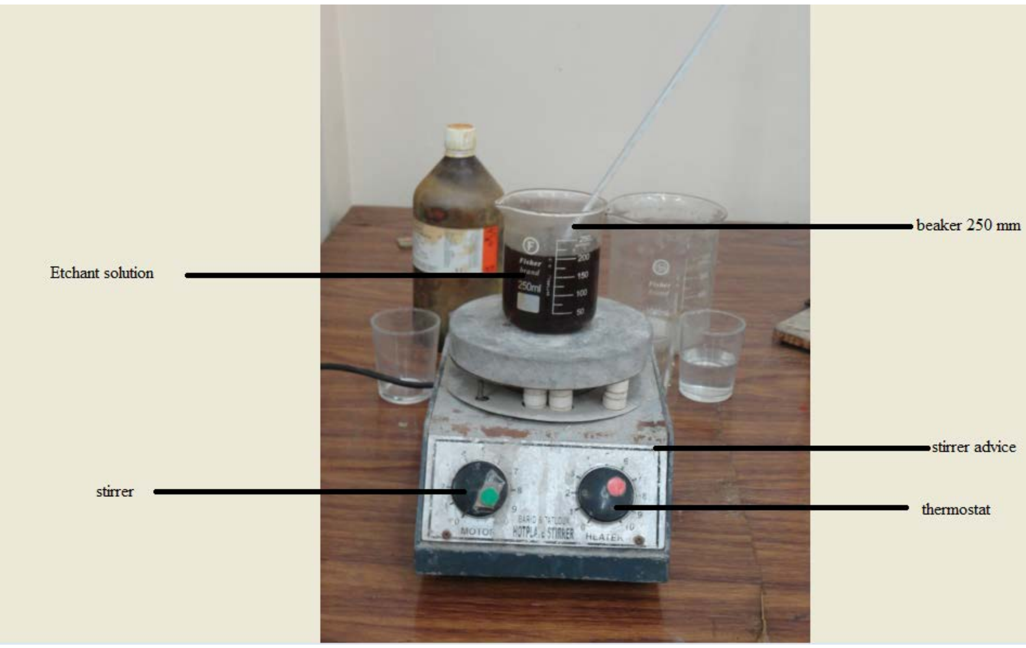


**Figure (2) Chemical machining system.**

**Hardness test**:

Digital display micro hardness tester, model FM800 was used. Suitable grinding papers (800, 1000, 1200 and 2000) grit and polishing with alumina. A load of 200 gm with 10 seconds period for testing was regarded. The hardness was measured in three different places on each specimen and the average of them was recorded.

**Microstructure test**:

microstructure tests of the samples were carried out by using scanning microscope (VEGA3LM) that used for surface topography.

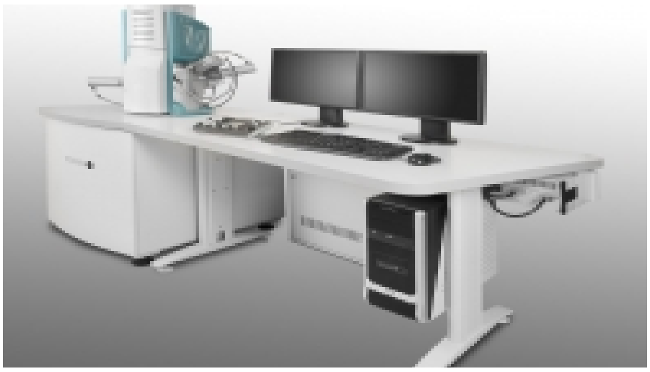


**Figure (3) Scanning Microscope (VEGA3LM).**

## Surface Roughness Test:

Pocket surf III ABSOLUTE MOBILITY, it contains digital micrometer with an accuracy of 0.001 mm.

**Figure (4) Surface roughness measurement**

## Chemical Machining Progra

The alloy samples were chemically machined according to a program included different machining conditions. Three machining temperatures (45, 50, and55 ºC) for each of which three machining times (3, 6, and 9min) were used as the machining conditions. After each machining process, the specimen was taken out of the etchant solution ,rinsed with water ,dried with air dryer, washed with alcohol and dried with air dryer again .for each experiment, the surface roughness was recorded.

## Results and Discussion

### Results of the Hardness Tests

The hardness tests were carried out for the base alloy sample before and after the machining process. The results are demonstrated in table (5).

**Table (5) Vickers hardness values of specimens before and after CHM.**

| Solution Type | Vickers hardness before CHM(gm/µm²) | Vickers hardness after CHM(gm/µm²) |
|---|---|---|
| HCL+HNO3+HF+ H2SO4+H2O | 330.5 | 332.23 |

The results indicated that the hardness of all samples was not affected by the chemical machining process, since no change in crystalline structure, no stresses induced by the process, and neither mechanical deformation nor exposure to high temperatures is involved.

## Effect of Machining Time on Surface Roughness

The effect of machining time on the surface roughness of the machined specimens at different machining temperatures is shown in figure (5).

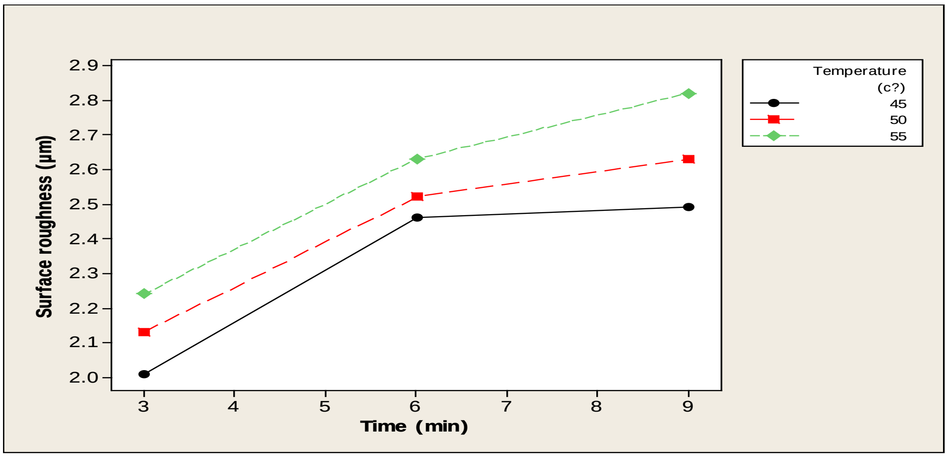


**Figure (5) Effect of machining time on the surface roughness at different machining temperatures.**

The figure indicates the following:

- For all specimens, it is clear that increasing machining time leads to increase in surface roughness (Ra). This can be explained on the basis of the variety of the elements in the composition of the alloy. Increasing the machining time lead to increase dissolving the alloy to ions, active meta (the anode) corrodes at an accelerated rate and the more noble metal (the cathode) corrodes at a retarded rate and both share in the chemical reaction with the components of the etchant.

During metallographic etching, twin and grain boundaries are preferentially attacked even the grain boundaries in very high purity metals are slightly grooved by appropriate etchants, the grain boundaries in impure metals and alloys are generally much more readily etched, primarily as are a result of segregation to them of the impurities and alloying additions. Grain boundary regions may be preferentially attacked either because segregation makes them more active or because segregation makes them more noble ; the grain boundary itself acts as a local cathode [8]. Smaller amount of a dissolved alloying element at grain boundaries(e.g., in SS).

- When using strong acid solution (HCL+HNO3+HF+ H2SO4+H2O) as etchant solution, it was noticed that the value of surface roughness decreases (2.01-2.77) at 3 min and 45 ºC (the surface brightness) and will increase with increase of time and machining temperature because (increase etch rate when addition HF to the solution of (HCL+HNO3+H2SO4+H2O) depending on the concentration of HF [10]. Hydrofluoric and nitric acids are most commonly used to etch stainless steel alloys because they form a highly acidic solution. The stronger the acid, the more metal removed. [11].

## Effect of Machining Temperature on Surface Roughness

Figure (6) shows the effect of machining temperature on the surface roughness of the machined specimens with different machining times.

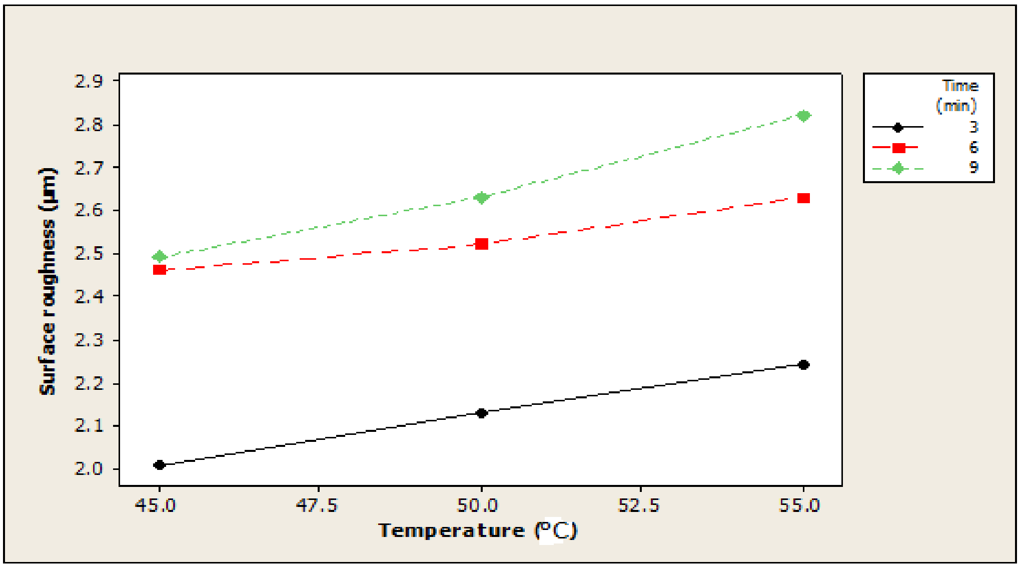


**Figure (6) Effect of machining temperature on the surface roughness at different machining times.**

•Figure (6) indicates that the increasing in machining temperature leads to increase in surface roughness, the surface roughness produced at 45 °C is lower than that produced at 50 °C for the same machining time and the solution type. In other words, an increase in machining temperature leads to increase in corrosion rate as a result of high power oxidizing agent and the mobility of ions will increase. Temperature also has an effect on etchant's ability to hold the dissolved metal content in solution. Temperature increases the kinetic energy for ions and gives the activation energy necessary to cross the energy barrier and delivery interaction more quickly.

## Optimization of The Machining Conditions

The results of the machining experiments showed that the machining temperature, machining time, and the concentration of solution have significant and interaction effects on the roughness of the machined surface. The optimum combination at these variables can be obtained by designing mathematical models representing their relations with the required output of the machining process-roughness.

Such model was constructed according to the statistical data of the carried out machining experiments. Data fit Ver.9 and Minitab 16 software were used for constructing this model based on the analysis of regression. Table (6) demonstrates the constructed mathematical models and the value of statistical coefficients.

**Table (6) Empirical model for Ra.**

| Empirical model | $R^2$(Square Root of SME) | F(Tests to fit) | S(stander Deviation) |
|---|---|---|---|
| **Ra**=1.75833-0.0406250×C+0.284444×t-0.00525×T+0.0003875×$C^2$ -0.0244444×$t^2$+0.00308333×C×t+0.000 | 91.13 | 42.3 | 0.05867 |

| | | | |
|---|---|---|---|
| 375×C×T | | | |

Where,
C: Concentration of Etchant.
t: machining time.
T: machining Temperature.

Figure (7) shows the matching between the experimental values of surface roughness Ra; the constructed empirical model indicates the following:

•Machining temperature has a greater effect on Ra in comparison with the machining time, and this is independent of the solution concentration.

•Machining for a larger duration increases the roughness of the produced surfaces and reduces the metal removal rate, and this is not affected by solution concentration.

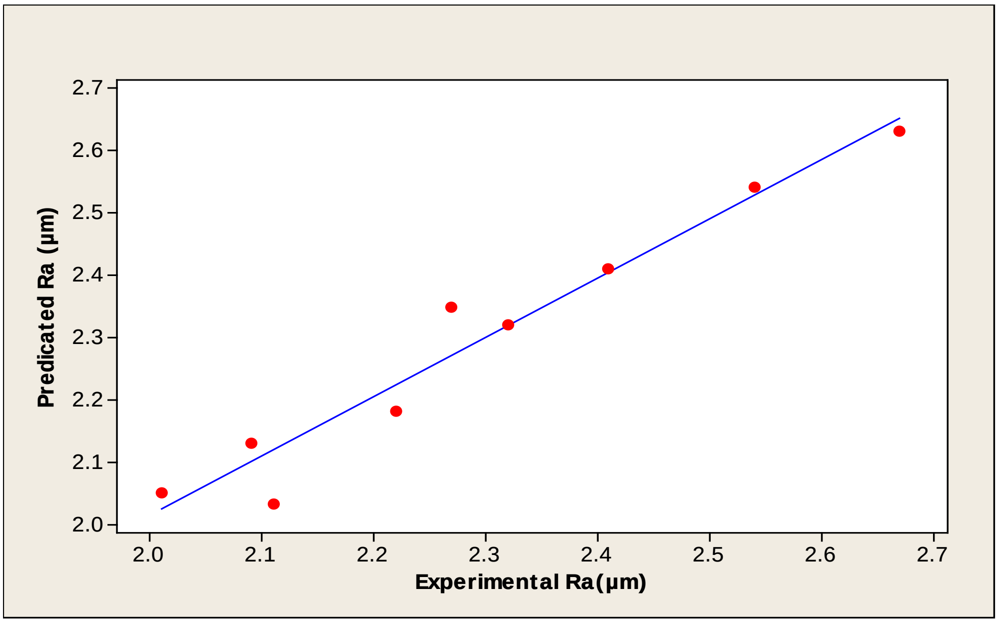


**Figure (7) The predicated values of the surface roughness based on the designed model are close match of their experimental values.**

### Estimation of Optimum Response Characteristics for (Ra)

Table (7) shows the Layout of the $L_9$ Orthogonal Array Based on Taguchi Design of Experiment, Tables (8) and (9) shows the results of ANOVA for raw Data from $L_9$ orthogonal array based on Taguchi method. As shown in this table, all the selected three parameters of machining time (t), machining Temperature (T), and Solution Concentration (C) significantly affect both the mean and the variation in the (Ra) values. The percentage contribution of machining time (59.88 %) followed by machining temperature (25.86 %) then solution concentration (6.84 %). It is clear that the optimal parametric combination for minimum Ra is $A_1$ $B_1$ $C_3$, i.e., at time 3 min, machining temperature 55ºC, and 10% concentration.

**Table (7) The Layout of the $L_9$ Orthogonal Array Based on Taguchi Design of Experiment.**

| No. | A | B | C | Time(min) | Temperature (°C) | Concentration | Ra(μm) |
|---|---|---|---|---|---|---|---|
| 1 | 1 | 1 | 1 | 3 | 45 | 10 | 1.94 |
| 2 | 1 | 2 | 2 | 3 | 50 | 20 | 2.11 |
| 3 | 1 | 3 | 3 | 3 | 55 | 30 | 2.12 |
| 4 | 2 | 1 | 2 | 6 | 45 | 20 | 2.35 |
| 5 | 2 | 2 | 3 | 6 | 50 | 30 | 2.33 |
| 6 | 2 | 3 | 1 | 6 | 55 | 10 | 2.38 |
| 7 | 3 | 1 | 3 | 9 | 45 | 30 | 2.42 |
| 8 | 3 | 2 | 1 | 9 | 50 | 10 | 2.36 |
| 9 | 3 | 3 | 2 | 9 | 55 | 20 | 2.60 |

**Table (8) Analysis of Variance for Surface Roughness.**

| Source | DF | Seq. SS | Adj. SS | Adj. MS | F | P | Percentage contribution |
|---|---|---|---|---|---|---|---|
| **time** | 2 | 0.22647 | 0.22647 | 0.11323 | 8.07 | 0.005 | 59.88 % |
| **Temperature** | 2 | 0.09780 | 0.09780 | 0.04890 | 3.48 | 0.023 | 25.86 % |
| **concentration** | 2 | 0.02587 | 0.02587 | 0.01293 | 0.92 | 0.310 | 6.84 % |
| **Residual Error** | 2 | 0.02807 | 0.02807 | 0.01403 | | | 7.42 % |
| **Total** | 8 | 0.37820 | | | | | 100 % |

**Table (9) Analysis of Variance for SN ratios.**

| Source | DF | Seq. SS | Adj. SS | Adj. MS | F | P |
|---|---|---|---|---|---|---|
| **Concentration** | 2 | 6.09045 | 6.09045 | 3.04523 | 322.69 | 0.003 |
| **time** | 2 | 1.13401 | 1.13401 | 0.56701 | 60.08 | 0.016 |
| **Temperature** | 2 | 0.05315 | 0.05315 | 0.02658 | 2.82 | 0.262 |
| **Residual Error** | 2 | 0.01887 | 0.01887 | 0.00944 | | |
| **Total** | 8 | 7.29649 | | | | |

Figure (8) shows that the optimal parametric combination for minimum Ra at time 3 min,machining temperature 55°C and 10% concentration.

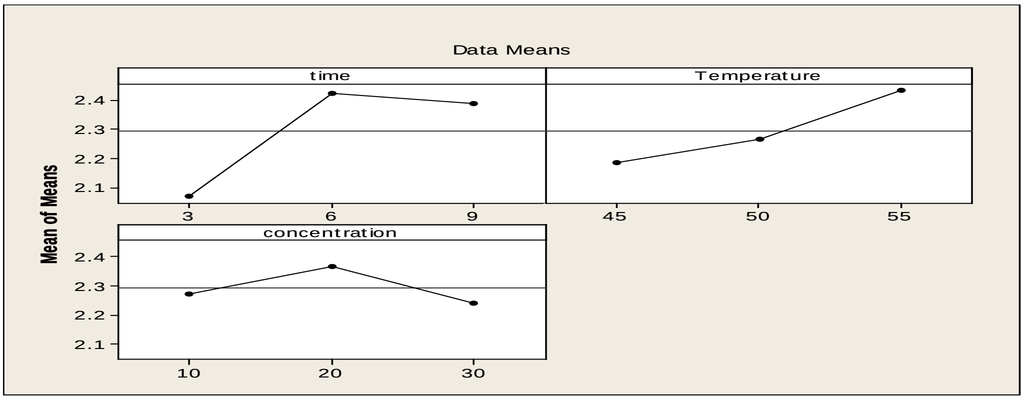


**Figure (8) Main effects Plot of Factors (Surface Roughness).**

Figure (9) indicates that the machining time has more effect and shows higher S/N ratio.

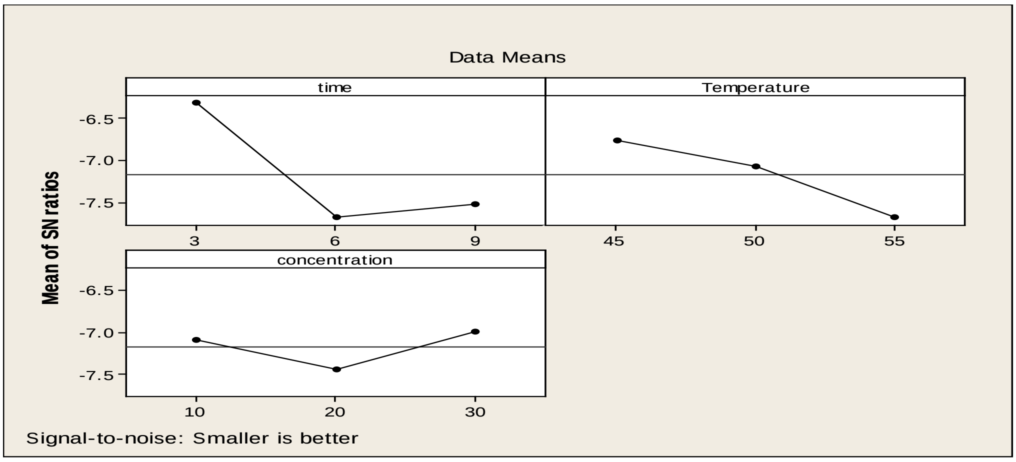


**Figure (9) Main effects Plot for signal to noise ratios**.

## Conclusions

Based on the detailed results the following conclusions can be stated:

1. Machining time, machining temperature and solution concentration are important variables that affect on chemical machining products; among these variables machining time has the largest effect.
2. Surface roughness of chemically machined parts decreases with the increase of the solution concentration.
3. Products of stainless steel 304 can be chemically machined in [H2O + HCl + HNO3 + HF + H2SO4] etchants in optimum conditions at temperature 55ºC, time 3 min and 10% concentration.
4. An assessment of CHM can be achieved by empirical models for selecting the appropriate machining conditions for the required surface roughness.

## References

[1]. Anusree.T. G and Govindan.P "Analysis of chemical machining for practical applications ", India /Government College of Engineering, Kannur Vol.2 pp.77-86, (2014).

[2]. Cakir, O.,"Study of Etch Rate and Surface Roughness in Chemical Etching of Stainless Steel", Key Engineering Materials, Optics Designed Precision Manufacturing Technologies, Vol.28, pp. 460-465, 2008.

[3]. Cakir, O.," Study of Etch Rate and Surface Roughness in Chemical Etching of Stainless Steel", Dicle University, Turkey, 2008.

[4]. A.Fadaei Tehrani and E.Imanian,"A new etchant for the chemical machining of St 304", Journal of Materials Processing Technology, 149, pp.404-408, 2004.

[5]. Tambol Mabkha,"Pickling Behavior of AISI 304 Stainless Steel in Sulfuric", Journal of Metals, Materials and Minerals, Vol.20, No.2, pp.1-6, 2010.

[6]. H. Sachdevand M. Strauß, “Selective etching of boron nitride phases” diamond and related materials, Journal of Biochemical Engineering pp. 614- 619, 2000.

[7]. Liu, J., "Etch rate and surface morphology of plasma etched glass and glassceramic substrates", Journal of Non-Crystalline Solids, 342: pp. 110-115, 2004.

[8]. Cakir, O.,"Reviw of Etchants for Copper and Its alloy in Wet Etching Processes", Key Engineering Materials, Optics Design and Precision Manufacturing Technologies, pp.364-366, pp.837-842, 2008.

[9]. Cakir, O.,"Photochemical Machining of Engineering Materials", Dicle University, Department of Mechanical Engineering Diyarbakir, Turkey, 2008.

[10].Li, L. F., Caenen, P. and Celis, J.P., "Effect of hydrochloric acid on pickling of hot-rolled 304 stainless steel in iron chloride-based electrolytes", Corros. Sci. 50(3), pp. 804-810, 2008.

[11]. Saito, Y. and B.Casady, "Mechanism of etching rate change of aluminosilicate glass in HF acid with micro-indentation", Applied Surface Science, 255: pp. 2290-4, 2008.

[12]. Xiuling Li, "Metal assisted chemical etching for high aspect ratio nanostructures: A review of characteristics and applications in photovoltaics", Electrical and Computer Engineering Department, University of Illinois, Urbana, IL 61801, United States,2012.

[13]. Li, L. F. and Celis, J.P.," Intergranular corrosion of 304 stainless steel pickled in acidic electrolytes", Scripta Mater, 51(10), pp.949-953, 2004.

[14]. DeJarld M. T, Shin J. C. Chern W, Chanda D, Balasundaram K and Rogers J.A." Formation of High Aspect Ratio GaAs Nanostructures with Metal Assisted Chemical Etching". Nano Lett, 11(12), pp.5259–63, 2011.