The Effective of Pressure and Sintering Temperature for Hardness Characteristics of Shape Memory Alloy by Using Taguchi Technique

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ABSTRACT

This paper presents the Taguchi approach for optimization of hardness for shape memory alloy (Cu-Al-Ni) . The influence of powder metallurgy parameters on hardness has been investigated. Taguchi technique and ANOVA were used for analysis. Nine experimental runs based on Taguchi's L9 orthogonal array were performed (OA),for two parameters was study (Pressure and sintering temperature) for three different levels (300 ,500 and 700) MPa ,(700 ,800 and 900)^{o}C respectively . Main effect, signal-to-noise (S/N) ratio was study, and analysis of variance (ANOVA) using to investigate the micro-hardness characteristics of the shape memory alloy .after application the result of study shown the height hardness at the level 2 of pressure and level 1 of temperature (A_2B_1) by taguchi technique at magnitude value 500MPa and 700 ºC. The best effective factor at ANOVA has pressure 36.39%. the interaction given the best pressure 500 MPa and Temperature 800 ºC.

Keywords :shape memory alloy , Cu-Al-Ni , taguchi method , optimization, powder metallurgy , hardness.

تأثير الضغط ودرجة حرارة التلبيد على الصلادة لسبيكة ذكية باستخدام تقنية تاكوشي

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الخلاصة:

هذا البحث بين استخدام طريقة تاكوشي للوقوف على امثلية الصلادة لسبيكة ذكية وتضمن المتغيرات المؤثرة على صلادة نماذج مصنعة بطريقة ميتالورجيا المساحيق.تقنية تاكوشي و تحليل التباين, استعملت في عملية تحليل تسع عينات تمت تصنيعها اعتمادا على خوارزمية تاكوشي لمتغيرين اساسين للدراسة (ضغط ودرجة حرارة التلبيد) لثلاث مستويات مختلفة (300,500and 700) Mpaودرجة حرارة التلبيد℃ (700,800 and 900) على التوالي تم دراسة تأثير العامل الرئيسي ونسبة الضوضاء وكذلك تمت تحليل التباين للصلادة المايكروية للسبيكة الذكية.نتائج الدراسة بينت بعد التطبيق ان اعلى صلادة كانت في (A_2B_1)اي عند المستوى الثاني للضغط والمستوى الاول للحرارة وكانت قيمة الضغط 500Mpaوحرارة التلبيد 700 ºC تحلل التباين بين ان افضل عامل مؤثر هو الضغط بنسبة 36.39 %. العامل المؤثر ضمن مستويات مختلفة(interaction) بينت ان افضل ضغط 500MPa ودرجة حرارة التلبيد 800 ºC.

الكلمات الرئيسية: سبائك ذكيةCu-Al-Ni, , طريقة تاكوشي, امثلية , ميتالورجيا المساحيق , الصلادة.

1. INTRODUCTION

During the past years, smart materials and structures have received increasing attention because of their great scientific and technological significance **,Wezz, et al.,1998.**Shape memory alloys are the most important branch from the smart and /or intelligence materials**,Noecker,2004 .**Smart materials and their technologies are still in the beginning stages of the implementation phase even though they have undergone extensive research, especially during the last two decades ,**Dunn, et al., 1999.** Shape memory materials (SMMs) are smart materials that "remember" their original shapes **, Huang, et al .,2010**.Some shape memory alloys (SMAs) when exposed to plastic deformation, it returns to their original shape when heated. These unusualeffects are called a thermal shape memory alloys and super elasticity (elastic shape memory). Both effects depend on the occurrence of aspecific type of phase change known as thermoelastic martensitic transformation **,Otsuka .,et al., 2002.**The shape memory alloys have two stable phases: the high–temperature phase, called austenite and the low–temperature phase, called martensite as shown in **Fig .1, Kneissl,et al.,2000.**The austenite phase is characterized by a cubic crystal structure ,while the martensite phase has a monoclinic (orthorhombic) crystal structure, shape memory applications as hydraulic couplings, force actuators as fire safety valves, proportional control as fluid flow control valve

2. THE TAGUCHI METHOD

In this work, analysis based on the Taguchi method is performed by utilizing the Minitab software to estimate the significant factors of the P/M process parameters on shape memory alloy . Taguchi's orthogonal array is highly functional design, used to estimate main effects using few experimental tests only . These designs can investigate main effects when factors have more than two levels. In Taguchi method, the analysis of variation is performed using Signal – to – Noise ratio (S/N). There are three S/N ratio approaches of common interest for optimization **,Wen, ,2008.**

1. Smaller – the better (for making the system response as small as possible).
2. Larger – the better (for making the system response as large as possible).
3. Nominal – the best (for reducing variability around the target).

In this work, the objective is to maximize the P/M parameter. Therefore, the S/N ratio for each experiment of L9 calculated using larger- the better approach. The objective of using S/N ratio as a performance measurement is to develop a product and process insensitive to noise factor. In Taguchi method, the term "signal" represents the desirable value (mean) for the output characteristic, and the where *n* is the number of experiments in the orthogonal array andy_i the i^{th} value measured.term "noise" represents the undesirable value (square deviation) for the output characteristic . Therefore, the S/N ratio is the ratio of the mean to square deviation.
S/N ratio of the overcut can be calculated by **Taguchi, et al.,2000.**

$$S/N = -10\log\left[\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_i^2}\right] \quad (1)$$

Where: Yi: is the ith observed value of the response.
(n: number of observations).
S/N ratio is used to measure the quality characteristic deviating from the desired value.

3. EXPERIMENTAL PROCEDURE

The test samples are produced using powder metallurgy of Cu , Al , Ni powders , which consist of mixing , compacting and sintering processes . Copper powder with 99.9% purity (-325 mesh), nickel powder with 99.9% purity(-325 mesh) and aluminum powder with 99.9%purity (-325mesh)respectively were imported from Sky Spring Nanomaterials, Inc. USA used to prepare samples of the master alloy with a composition of (83% Cu weight 13%AL weight and 4% Ni weight as shown in **Table 1**. These powders were weighed accordingly and placed into cylindrical containers which were then mixed in a horizontal barrel mixer as shown in **Fig.2** . These elements were put into a glass cylindrical container which is 50% filled and 1% (acetone) was added in order to get better properties as lower friction at compaction and increase the segregation and prevent the separation of the components (since there is a difference in the densities) . Alumina balls for assisting the segregation has not been used in order to prevent the milling process and contamination of powder , the speed of rotating drum was set to 80 rpm and the time of mixing was 6 hour . Samples from powder were prepared in the same die with a cross section of 14 mm in diameter and approximately 5 mm length in average. The samples were pressed at (300MPa , 500MPa and 700MPa) in a 100 ton Hydraulic computerized press Machine as shown in **Table.2**. **Fig.3** shows samples from each composition after sintering in an electrical tube furnace supplied with a quartz tube and vacuum equipment as shown in **Fig.4** in order to get a fine samples without cracks or defect ,Two stage sintering has been implemented since there is difference in melting point of the alloy components and to prevent the appearance of liquid phase sintering. The first stage is to sinter the sample at 500ºC for 1 hour and followed by the second stage which is raising the temperature to (700 ºC) with soaking time of 5 hour then leaving the sintered sample to cool in furnace. A heating rate of 20ºC/min was maintained for the first stage and 20ºC/min for the second stage. The vacuum pressure was always allowed to reach $3x10^{\wedge -6}$ bar before sintering and during the whole sintering process and cooling. The dual stage vacuum pump is allowed to run for the entire sintering time to suck the harmful gases which will be produced during the diffusing of particles which might effect the sintering efficiency .After sintering , all sample have been quenched to get ß phase(shown the ß phase by X-Ray diffraction) which is AlCu3 (martensite) by heating the sintered sample to 800 ºC and holding it at this temperature for 1 hour then rapidly quenched into iced water . After the quenching process, an ageing heat treatment is implemented to stabilize the ß phase by heating the sample to 100ºC and holding at this temperature for 2 hour**.**The quenching and ageing process was also implemented in vacuum atmosphere to prevent the oxidation **.**Vickers micro hardness testing has been carried out on allsamples,the device shown in **Fig.5** . More than three values of hardness for each sample have been taken to get the mean value represents hardness.

4. RESULTS AND DISCUSSION

The results, in terms of average micro-hardness were obtained after conducting the hardness test for all nine sample . Each test sample , indeed represented one experiment in the orthogonal array **Table 3.** The experimental results for hardness test in **Table 4**. In the latter, the results were analyzed using main effects, ANOVA, and the signal-to noise ratio (S/N) analyses. **Fig. 6 (a ,b ,c)** shows four x-ray charts for samples with different sintering temperature(700 ,800,900)^{o}C samples respectively the result peaks was compared with the standard cards with the possible known phases which will be appear. All samples have shown the martensite phase after quenching which indicate no effect of pressure and sintering temperature to the martensite phase (AlCu3).

4.1 Main Effects

The average value of micro-hardness for each factor pressure and sintering temperature (A and B) at each level (level 1, level 2 and level 3) was obtained and the result is summarized in **Table 6**. It can be seen from **Fig.(7,8)** that the combination of parameters and their levels A_2B_1 yield the optimum quality characteristic. the other capability for ANOVA analysis its present to the effect of one parameter for any level (interaction). **Fig.9** shows that for the case under study the best parameter are (500 MPa and 800 ^oC). **Fig. 10** shows the Vickers micro hardness with different pressure and sintering temperature.

4.2 Signal-to-Noise Ratio (S/N Ratio)

The program (MINTAB 16.1) calculates the S/N ratio for each level of each variable by using Eq.(1). The results are shown in **Table 5.** According to **Figs.7 and 8,** second level of pressure (A), first level of sintering Temperature (B) are optimal levels in this test. They have highest S/N ratios.

4.3 Analysis of Variance (ANOVA)

The parameters which significantly affected the hardness were investigated using the analysis of variance (ANOVA). The contribution percentage of different parameters on Cu-Al-Ni as obtained by ANOVA are presented in **Table 7** and it can be seen from this Table that the pressure (A), and sintering temperature (B) affect hardness by 36.39% and 4.19% in the shape memory alloy respectively.

5. CONCLUTION

On the basis of the results obtained from the present case study the following points can be concluded:

- The combination of parameters and their levels for optimum micro-hardness and also for shape memory alloy Cu-Al-Ni is A_2B_1(pressure 500 MPa and temperature 700°C) .
- The contribution of pressure and temperature to hardness of shape memory alloy are 36.39% and 4.19% respectively.
- The value obtain from correlation between process parameters and hardness by using an optimum parameter combination (A_2B_1) was consistent with maximum hardness obtained by using the analysis of S/N ratios.
- The X-ray diffraction shown the AlCu3 (martensite) which indicate no effect of pressure and sintering temperature to the martensite phase
- from the ANOVA table ,pressure is the most significant parameter for minimum micro hardness
- This research gives how to use Taguchi's parameter design to obtain an optimum condition with lowest cost , therefore minimum number of experiments is required for industrial applications.

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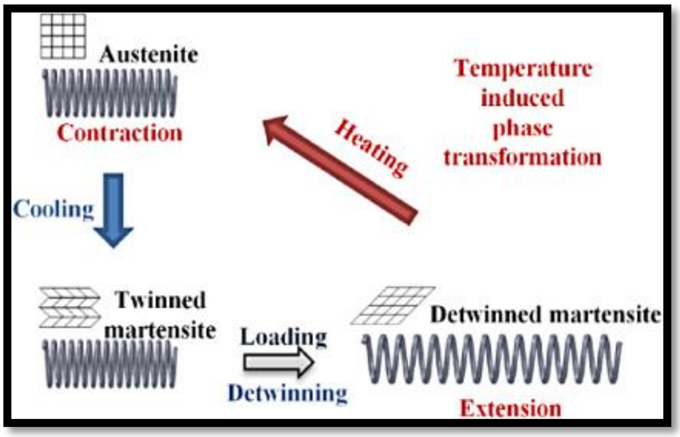


Figure 1. Phases of a shape memory alloy**, Mel Schwartz,2002.**

Figure 2. Horizontal barrel mixer.

Figure 3. Sintered samples after grinding and polishing

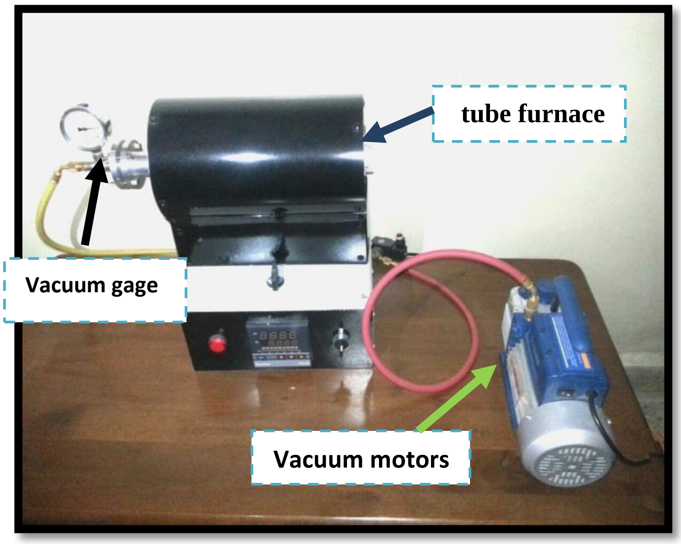


Figure 4. Electrical tube furnace.

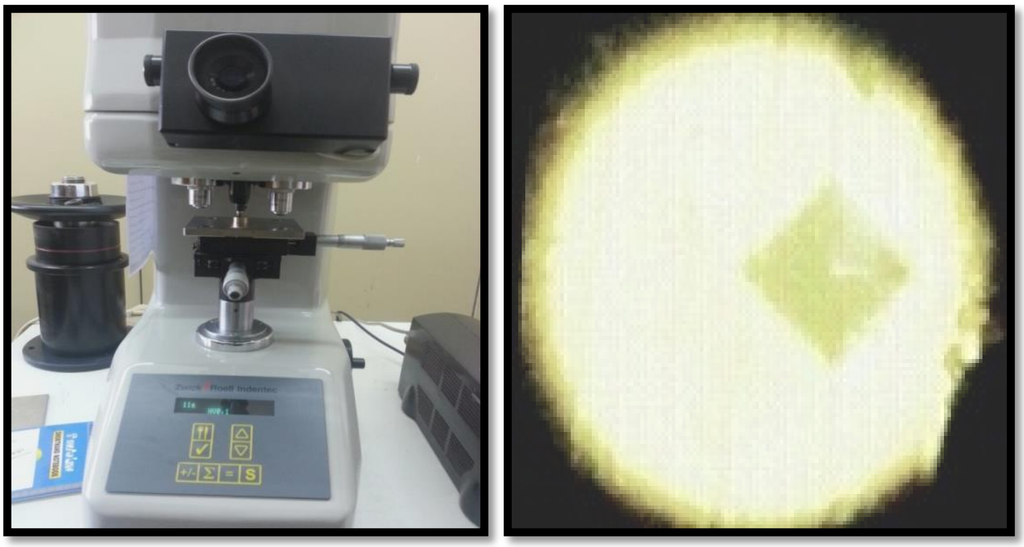
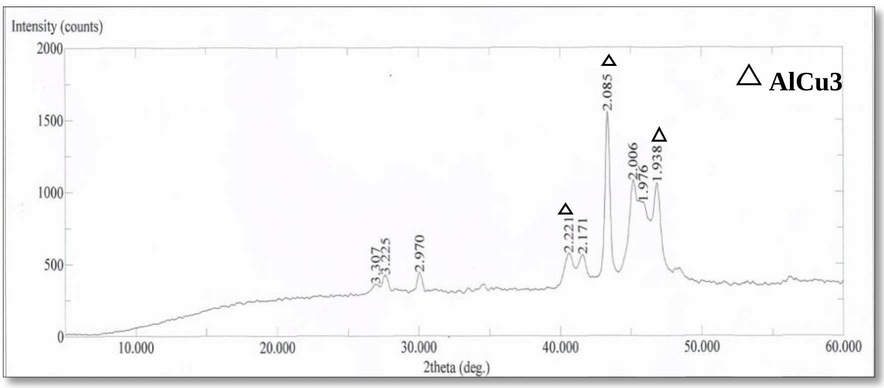
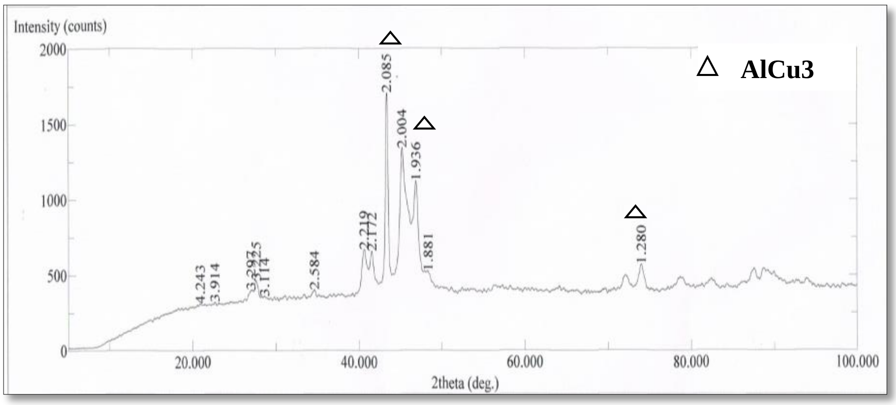


Figure 5. Micro-hardness device.



a



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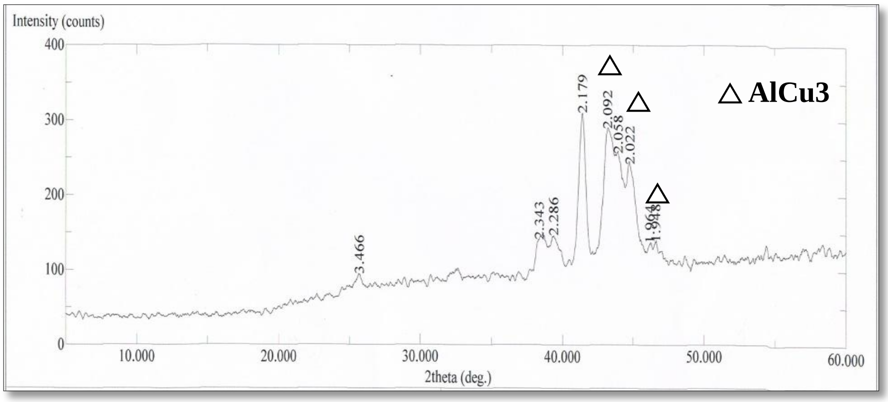

c

Figure 6. (a,b,c) X-ray diffractions.

Table 1. Notation for sample with alloying element.

Powder element	**Copper**	**Aluminum**	**Nickel**
Weight percentage	83%	13%	4%

Table 2 . Shape memory alloys parameters and their levels.

Factors	**Factors Code**	**Levels**		
		1	**2**	**3**
Pressure(MPa)	**A**	300	500	700
Temperature(˚C)	**B**	700	800	900

Table 3.Experimental plan using *L*9 orthogonal array.

Parameter / Level		
Experiment number	**Code No.for pressure (A)**	**Code No.for temperature (B)**
1	1	1
2	1	2
3	1	3
4	2	1
5	2	2
6	2	3
7	3	1
8	3	2
9	3	3

Where : A=Pressure(MPa),B=Temperature(^{o}C)

Table 4. Experimental results for micro-hardness test.

Exp. No	(A)	(B)	Average microhardness (HV)
1	300	700	137.66
2	300	800	114
3	300	900	116
4	500	700	135
5	500	800	166
6	500	900	133.66
7	700	700	148
8	700	800	123
9	700	900	148.33

Table 5. S/N ratio response for micro hardness test.

Experiment no.	Average Micro hardness (HV)	S/N ratio
1	137.66	42.77
2	114	41.13
3	116	41.28
4	135	42.60
5	166	44.40
6	133.66	42.52
7	148	43.40
8	123	41.79
9	148.33	43.42

Table 6. Levels average for main effects.

Average S/N Ratio					
Factors	**Level 1**	**Level 2**	**Level 3**	**Delta**	**Rank**
A	41.73	43.18	42.88	1.45	1
B	42.93	42.44	42.41	0.52	2

Where : A=pressure(MPa),B=temperature(^{o}C).

For Factor (A) for S/N ratio

(S/N) ratioA_1 = ((42.77+41.13+41.28)/3)

(S/N) ratioA_1 = (125.18/3) = 41.73

(S/N) ratioA_2= ((42.60+44.40+42.52)/3)

(S/N) ratioA_2 = (129.52/3) = 43.18

(S/N) ratioA_3= ((43.40+41.79+43.42)/3)

(S/N) ratioA_3 = (128.61/3) = 42.88

The same procedure for factor B
Delta =Max-Min

Delta=43.18-41.73=1.45 for factor A

Delta =42.93-42.41=0.52 for factor B

Table 7. Analysis of variance (ANOVA) table for the S/N ratio of hardness.

Where : A=pressure(MPa),B=temperature(^{o}C)

Source	Degree of freedom (DOF)	Sum of Squares (SS)	Mean Square (MS)	Model F-Value	Percentage Contribution, (%)
A	2	821.5	410.8	1.22	36.39%
B	2	94.5	47.3	0.14	4.19%
Error	4	1341.6	335.4		59.42%
Total	8	2257.6			

Total Degree of Freedom (DOF_T)= *n–1*

Where:*n = Number of experiments.*

Degree of Freedom of Factor A (DOF_A) = NO. of levels – 1

Degree of Freedom of Factor B (DOF_B) = NO. of levels – 1

Degrees of freedom of error($\mathbf{DOF}_e$)=DOF_T–DOF_A–DOF_B

Where: DOF_A=Degree of Freedom of factor pressure , DOF_B=Degree of Freedom of factor temperature

$$\hat{Y} = \sum_{i=1}^{n} \mathbf{Y_i} / \mathbf{n}$$

Where: $\hat{Y}$ = the average value of Yi.

$$SS_T = \sum_{i=1}^{n} (\mathbf{Y_i} - \hat{Y})^2$$

Where: SS_T = Total *sum of squares*, Y_i = *Sum of all results*, n = *Experiment number* ,
$\hat{Y}$= the average value of Yi

SS any Factor = $N1(x1-X)^2 + N2(x2-X)^2 + N3(x3-X)^2$

Where: $X = (x1+ x2 + x3) / 3$ and x1,x2,x3 are mean of parameter as per level, N1,N2,N3= number of experiments at level A1,A2andA3.

$SS_e = SS_T - SS_{(For\ all\ Factors)}$

Where:SSe = Sum of square of error.

$MS_A = SS_A / \mathbf{DOF}_A$

Where: MS_A = *Mean squares (variance)* **for** *one of factor* ***A,***DOF_y= Degree of freedom of any factor.

$MS_e = SS_e / \mathbf{DOF}_e$

Where:MS_e = Mean squares *of* error,SS_e = *Sum of square of error terms*

$MS_T = SS_T / \boldsymbol{DOF}_T$

Where: MS_T = Total mean squares.

$F = MS_A / MS_e$

*Where:*F = Variance or fisher ratio

P Percentage of contribution = SS/SS_T

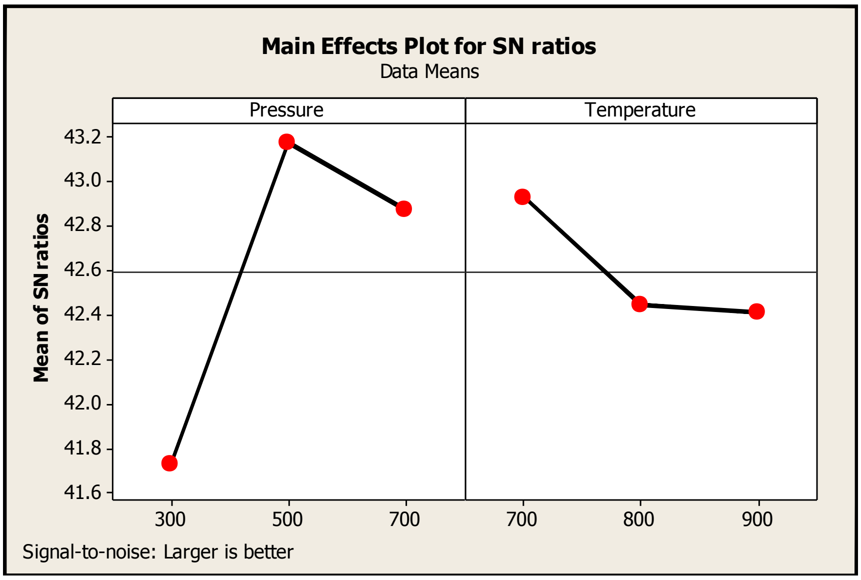

Figure 7. Main effects plot for S/N ratio.

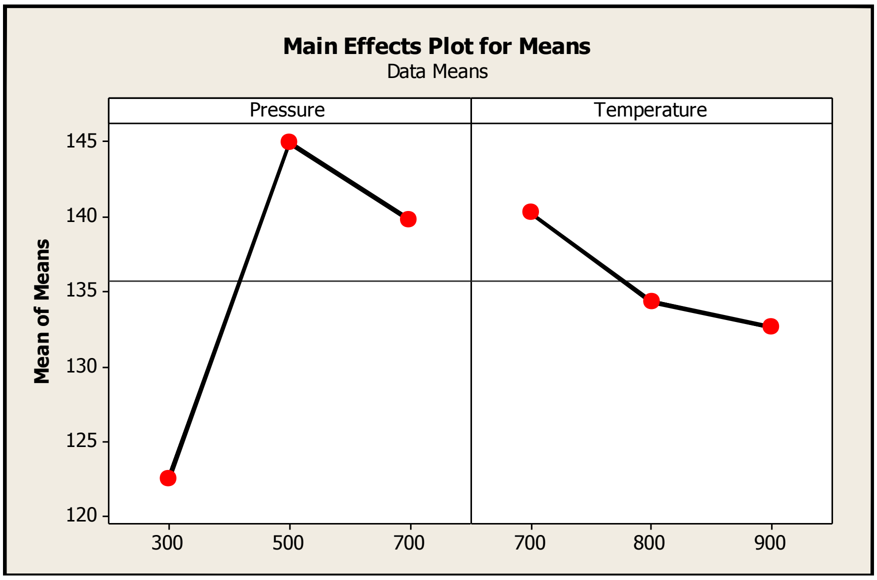

Figure 8. Main effects plot for means.

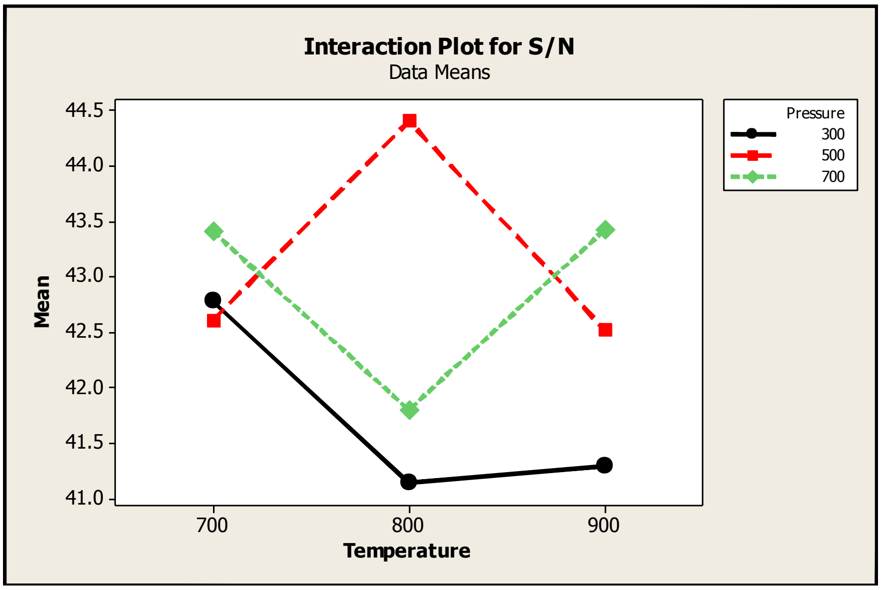

Figure 9. Interaction plot for S/N ratio (temperature and pressure).

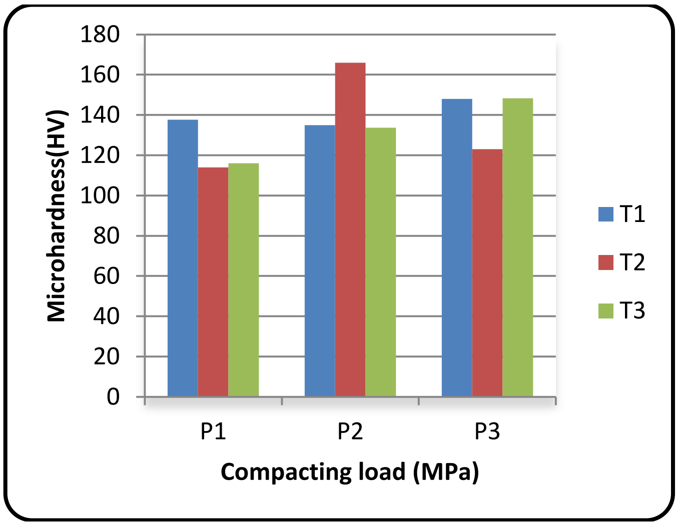

Figure 10. Vickers micro hardness.