Effect of Compaction Pressure and Sintering Temperature on Shape Effect and Compression strength of Cu-Al-Ni Shape Memory Alloy

Ahmed Abdulrasool
Asst.Prof.
Sarah Jalal Mosa
Mechanical Engineering Dept.
Engineering College
Baghdad University\Iraq

Abstract

Shape Memory Alloys (SMAs) are types of smart materials (SMs) which are used in many industries nowadays. The samples manufacturing smart alloy composed of copper alloy ,aluminum and nickel .The mixing materials weight percentage are 83% wt Cu - 13% wt Al-4% wt Ni .Has been followed by the compacting process .The powder compacted with three different value of the pressure (300,500,700)MPa. The sintering process has been in tube and vacuum furnace sintering environment at three different values of sintering temperature values (700,800,900)oC conducted the necessary tests, a screening of resilience and examination of compression for all samples. The experimental test for the samples showed that the highest value for the resilient be at a pressure of 700MPa and at a sintering temperature of 900oC either check compression straight was the highest value at a pressure of 300 MPa and temperature sintering 900oC.

KEYWORDS : Shape Memory Alloy, Cu-Al-Ni, powder metallurgy, shape effect, compression

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1-Introduction

Shape memory alloys (SMAs) are a fascinating group of metals that have two remarkable properties, the shape memory effect and superelasticity **[2].**Shape memory alloys are a unique class of metal alloys that can recover apparent permanent strains when they are heated above a certain temperature **[8].**The shape memory alloys have two stable phases , the high-temperature phase, called austenite (named after English metallurgist William Chandler Austen) and the low-temperature phase, called martensite (named after German metallurgist Adolf Martens) **[3].** Among a wide variety of shape memory alloys, copper shape memory alloys are considered as strong candidates to replace more expensive

Ni-Ti alloys because of their low cost and good properties. Copper based shape memory alloys succeeded to find their applications in Thermal actuators and Sensors **[1]**. Sintering is the basic method used in the preparation of the SMAs. Sintering's variables are mostly thermodynamic variables, such as temperature, time, atmosphere, pressure, heating and cooling rates. Many previous studies have examined the effects of sintering temperature and time on sintering of powder compacts. It appears, however, that in real processing, the effects of sintering atmosphere and pressure are much more complicated and important **[7]**. It is preferred to have minimum porosity in the material which can be obtained at high temperature and short time **[4]**. Unfortunately, further increase in sintering temperature may lead to the evaporation of one of the elements that has the lowest melting point and therefore sometimes the density of the sintered samples does not show notable changes with increasing sintering temperature **[5]** Sintering atmospheres must be controlled for proper production since they affect the densification, microstructure, and the properties of the products **[6].** found that sintering of (Al-Zn-Cu-Mn and Al-Cu-Mn-Si) alloys under vacuum gives better properties than sintering with nitrogen, hydrogen, and argon.

2. EXPERIMENTAL PROCEDURES

Powder metallurgy technique was used in manufacture throughout this work. ,cupper powder with 99.9% purity(-325 mesh) , aluminum powder with 99.9%purity (-325mesh) ,and nickel powder with 99.9% purity(-325 mesh) respectively were imported from *SkySpring Nanomaterials, Inc. USA* and used the main mixture prepared in this study included (83%wt Cu+13%wt Al+4%wt Ni). Mixed in a horizontal barrel mixer , and the powders were compacted by using a single action tool steel mold to form cylindrical shape green compacts (11 dia. X 16.5mm length) with different compacting (300,500,700)MPa used for compaction as shown in **Fig .1** which was located in Central Organization for Standardization and Quality Control.

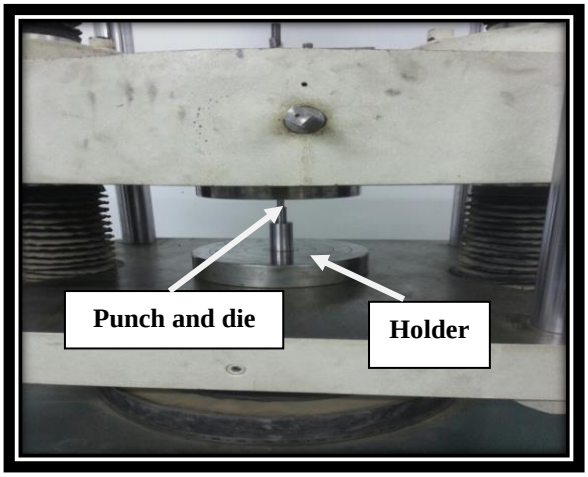


Fig 1: Uniaxial press machine

The sintering process of green compacts was performed in an electrical tube furnace supplied with a quartz tube and vacuum equipment as shown in **Fig.2**

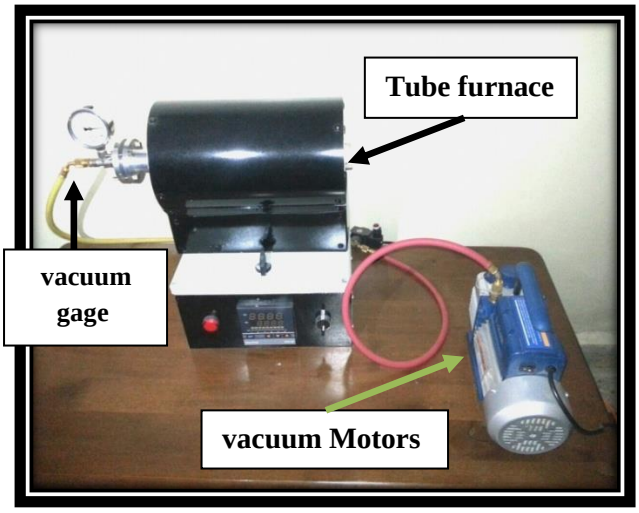


Fig 2: Vacuum tube furnace used in the sintering process.

Sintering process consists of a primary stage of heating to 500°C hold 1 hr and then the second stage is by heating up to (700,800,900) °C for 5 hrs and slow cooled in furnace as shown in **Fig.3**,**Fig.4** and **Table 1**.

Fig 3: Samples after sintering (after different sintering temperature)

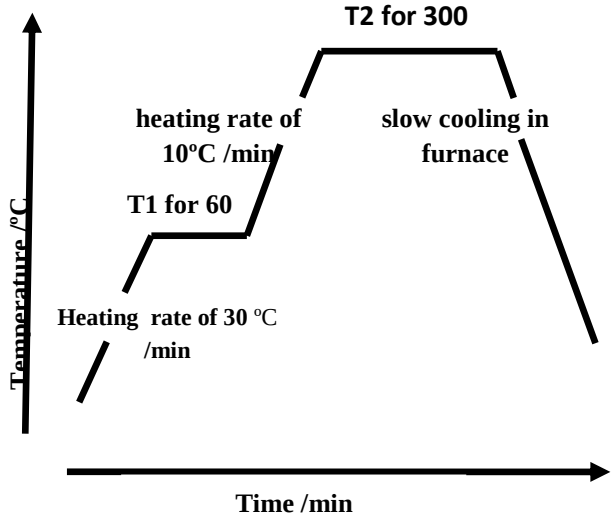


Fig 4: Diagram of sintering procedure

Table 1:Time and temperature of two stage sintering

The alloys were then heated to 800 °C for one hour and quenched by ice water and aged at 100°C for two hours and cooled in furnace , 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 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 hours. The quenching and ageing process was also implemented in vacuum atmosphere to prevent the oxidation.

After the heat treatment the sintered samples grinding with grinding paper grit size (800,1000,1200) and polishing the sample with cloth paper and alumina powder to prepare it for the microstructure and mechanical tests.

To measure the compression strength of samples and to investigate the effect of compacting load and sintering temperature on this behavior, this test was conducted by using the same device as shown in **Fig 1** .

Exp.	Compacting load (MPa)	T_1(ºC)	t_1(hr)	T_2(ºC)	t_2(hrs)
1	300	500	60	700	300
2	300	500	60	800	300
3	300	500	60	900	300
4	500	500	60	700	300
5	500	500	60	800	300
6	500	500	60	900	300
7	700	500	60	700	300
8	700	500	60	800	300
9	700	500	60	900	300

To measure the shape effect of samples and to investigate the effect of compacting load and sintering temperature on this behavior, this test was conducted by using the same universal test device mentioned earlier in compression test as shown **Fig .1** . By compacting the (11mm dia. X 16.5 mm length) sample at 4% of its length with1mm/min displacement and then heating the sample to 250°C for 5min and left to cooling in air cooling. Then , the shape effect (the return length) was calculated by applying the Equation.1 ,shape effect shown in **Table 3.**

$$\text{Shape Effect\%} = \frac{L2 - L1}{L0 - L1} \ldots [1]$$

where :

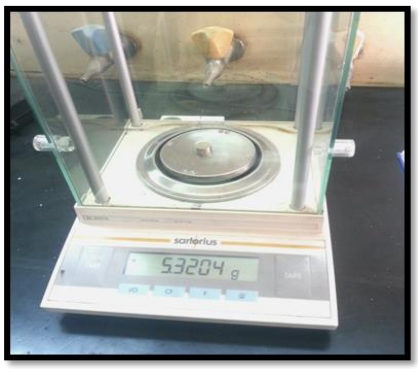
L0: the original sample length

L1: sample length after 4% compact
L2 : return length after heating
To measure (L0, L1, L2) used vernia .

Due to the use of powder metallurgy method to produce samples, this test is necessary to see the effect of this method on the density of samples and the effect of density on shape memory property. density was done according to ASTM B 328 which is implemented by weighing the samples in three different cases , the first is weighting the sample in air .The second includes immersion of the sample in container filled with oil and the pressure over it is reduced to be not more than 7 KPa for 30 min . After cleaning the sample from excessive oil, it was weighed. The third case is weighting the oil-impregnated sample in water by using a hook balance. To calculate the density apply Equation.2
The same vacuum pump which was used for sintering was used also to evacuate the sealed sample chamber as shown in **Fig.5**,the Measerment and calcalation for density &porosity shown in Table7

$$\mathbf{D} = \left(\frac{\boldsymbol{B}}{\boldsymbol{B}-\mathbf{C}+\mathbf{E}}\right)\ \mathbf{D_w} \qquad \ldots[2]$$

where:
D = Density, g/cm^3,
B = Mass of oil-impregnated specimen, g,
C = Mass of oil-impregnated sample immersed in water, g,
E = Mass of wire in water (which handling the sample) ,g,
Dw = density of water ,g/cm^3



a

b

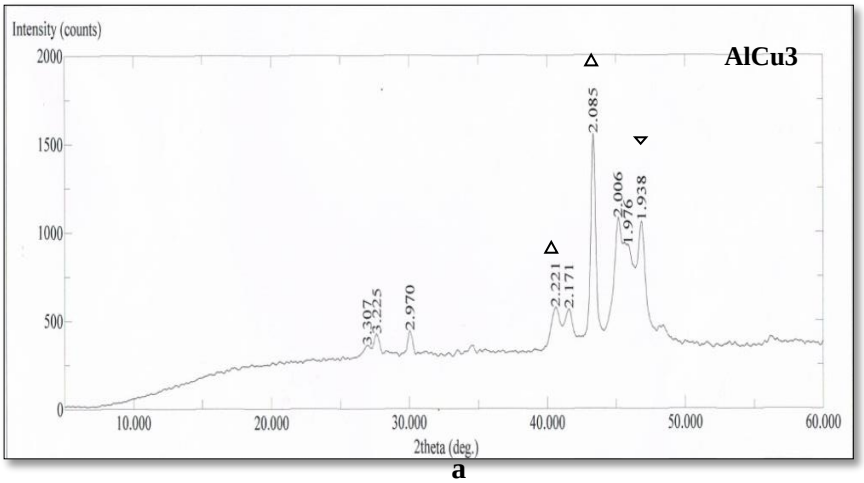
Fig 5: a) Sensitive hock balance, b)samples in vacuum chamber

3-RESULTS AND DISCUSSION

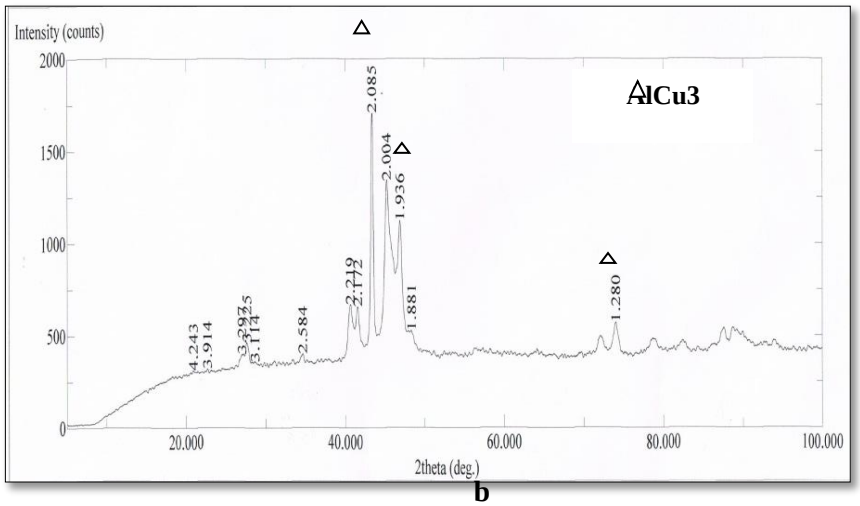
Fig. 6 (a ,b ,c) shows four x-ray charts for alloy with different sintering temperature the result peaks was compared with the standard cards with the possible known phases which will be appear.
All samples have shown the martensitic phase after different sintering temperature and quenching which

indicate the existence of the shape effect and no effect of change temperature of sintering to the martensitic phase.

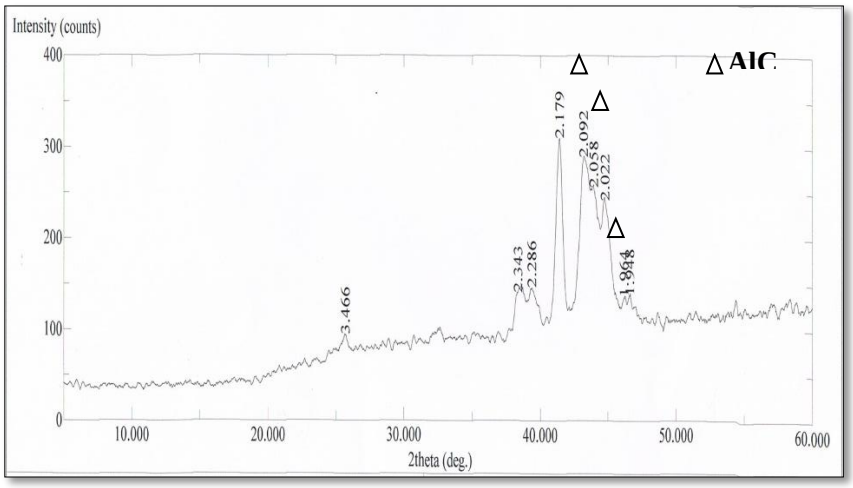
Fig.7 show the optical microscope pictures for the microstructures of samples which reveal the grain size and boundary after etching. As it is seen there is a clear grain boundary for all samples but since the powder metallurgy technique was used to produce these sample, it has been so difficult to compute the average size of grain size (because there is differences in grain due to existence of pores)



a



b



C

Fig 6 (a,b,c) : X-ray diffractions

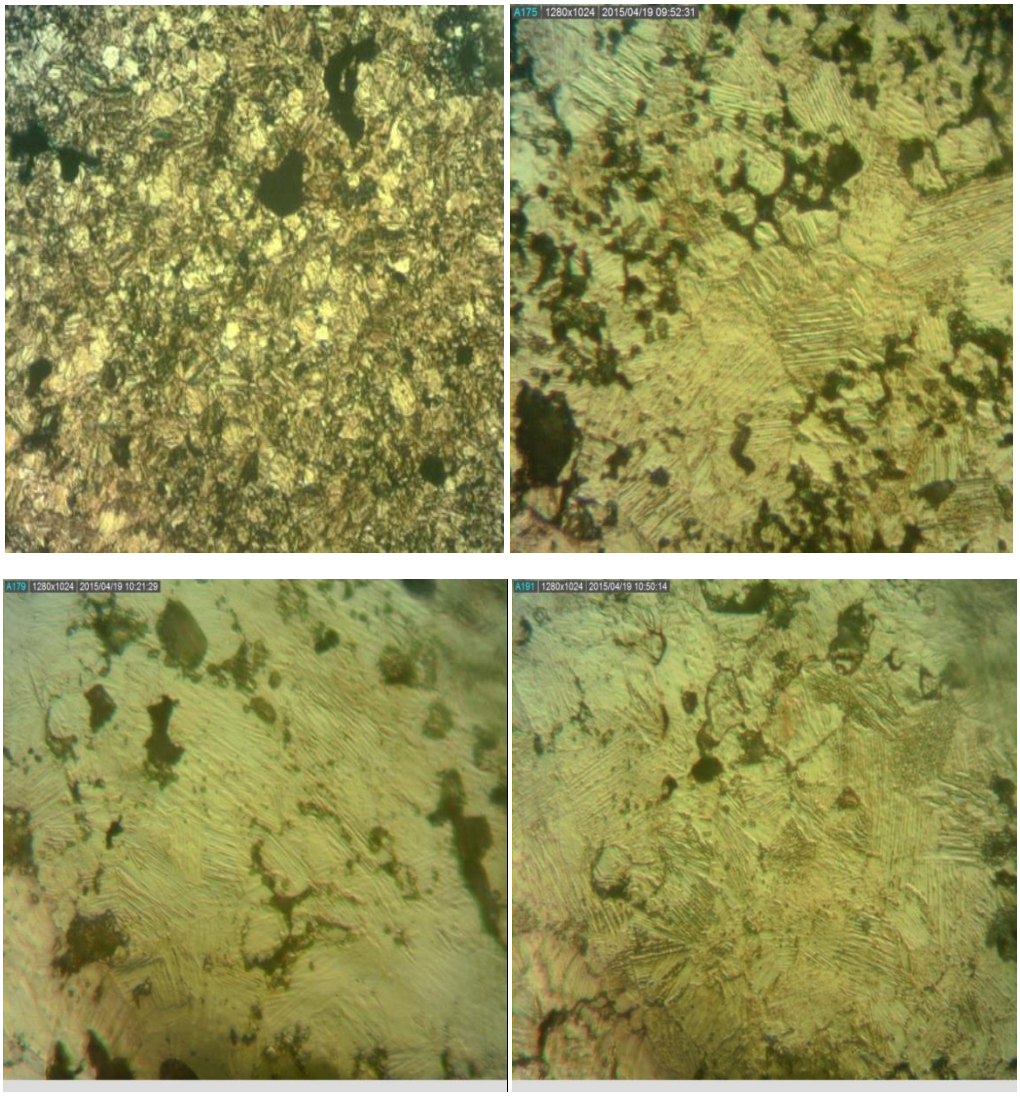
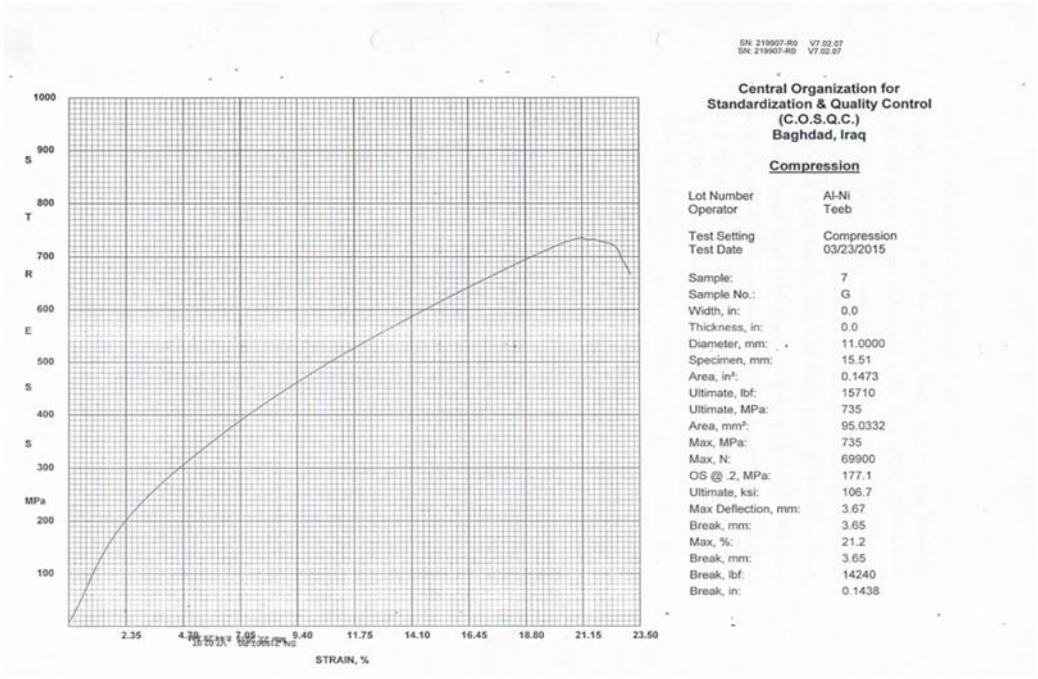


Fig.7: Microstructure for some of samples

The compression strength test of shape memory alloy done at room temperature and compression results shown in **Fig.8**and **Fig.9.Table 2** give experimental results of compression specimens of powder metallurgy for shape memory alloy (compression strength , mean). And the ultimate compression strength for each compacting pressure with three sintering temperature are shown in **Fig.10.**

Fig.13 show the sample densities after quenching which was done .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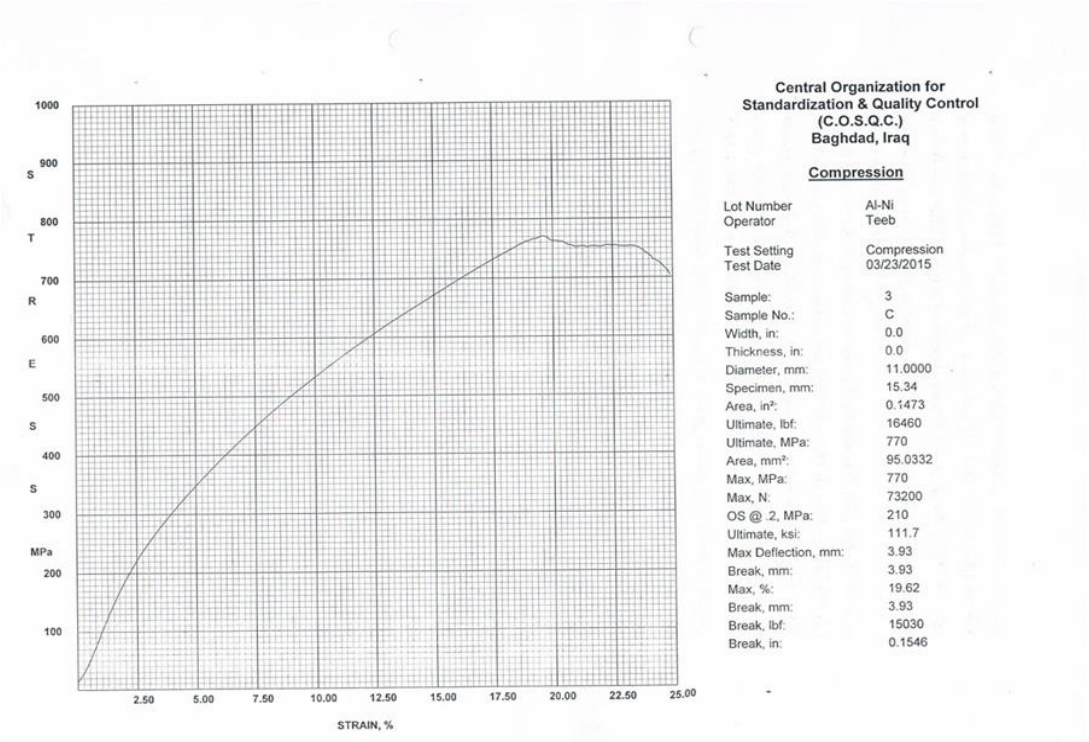
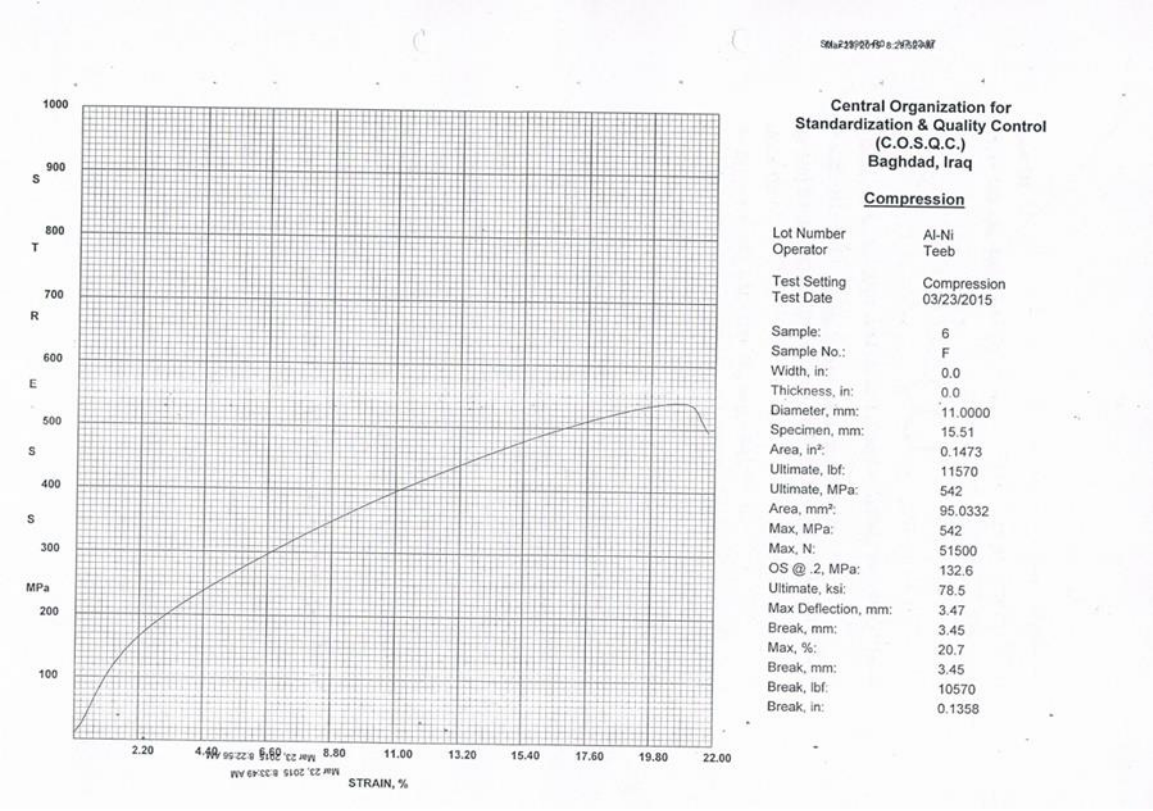
SN: 219907-R0 V7.02.07
Central Organization for
Standardization & Quality Control
(C.O.S.Q.C.)
Baghdad, Iraq
Compression
Lot Number Al-Ni
Operator Teeb
Test Setting Compression
Test Date 03/23/2015
Sample: 7
Sample No.: G
Width, in: 0.0
Thickness, in: 0.0
Diameter, mm: 11.0000
Specimen, mm: 15.51
Area, in²: 0.1473
Ultimate, lbf: 15710
Ultimate, MPa: 735
Area, mm²: 95.0332
Max, MPa: 735
Max, N: 69900
OS @ .2, MPa: 177.1
Ultimate, ksi: 106.7
Max Deflection, mm: 3.67
Break, mm: 3.65
Max, %: 21.2
Break, mm: 3.65
Break, lbf: 14240
Break, in: 0.1438
STRESS MPa
1000
900
800
700
600
500
400
300
200
100
2.35
4.70
7.05
9.40
11.75
14.10
16.45
18.80
21.15
23.50
STRAIN, %


Central Organization for
Standardization & Quality Control
(C.O.S.Q.C.)
Baghdad, Iraq
Compression
Lot Number Al-Ni
Operator Teeb
Test Setting Compression
Test Date 03/23/2015
Sample: 3
Sample No.: C
Width, in: 0.0
Thickness, in: 0.0
Diameter, mm: 11.0000
Specimen, mm: 15.34
Area, in²: 0.1473
Ultimate, lbf: 16460
Ultimate, MPa: 770
Area, mm²: 95.0332
Max, MPa: 770
Max, N: 73200
OS @ .2, MPa: 210
Ultimate, ksi: 111.7
Max Deflection, mm: 3.93
Break, mm: 3.93
Max, %: 19.62
Break, mm: 3.93
Break, lbf: 15030
Break, in: 0.1546
STRESS MPa
1000
900
800
700
600
500
400
300
200
100
2.50
5.00
7.50
10.00
12.50
15.00
17.50
20.00
22.50
25.00
STRAIN, %



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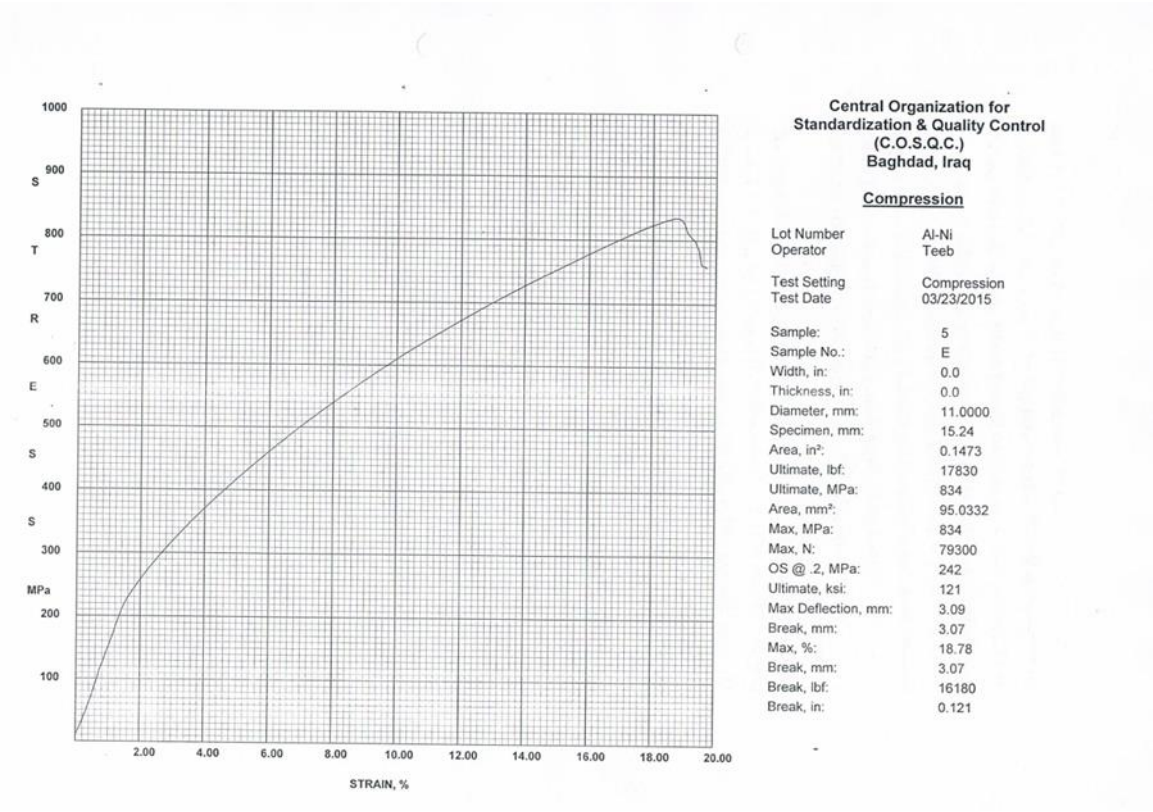
Compression

Lot Number	Al-Ni
Operator	Teeb
Test Setting	Compression
Test Date	03/23/2015
Sample:	6
Sample No.:	F
Width, in:	0.0
Thickness, in:	0.0
Diameter, mm:	11.0000
Specimen, mm:	15.51
Area, in²:	0.1473
Ultimate, lbf:	11570
Ultimate, MPa:	542
Area, mm²:	95.0332
Max, MPa:	542
Max, N:	51500
OS @ .2, MPa:	132.6
Ultimate, ksi:	78.5
Max Deflection, mm:	3.47
Break, mm:	3.45
Max, %:	20.7
Break, mm:	3.45
Break, lbf:	10570
Break, in:	0.1358



Central Organization for Standardization & Quality Control (C.O.S.Q.C.) Baghdad, Iraq

Compression

Lot Number	Al-Ni
Operator	Teeb
Test Setting	Compression
Test Date	03/23/2015
Sample:	5
Sample No.:	E
Width, in:	0.0
Thickness, in:	0.0
Diameter, mm:	11.0000
Specimen, mm:	15.24
Area, in²:	0.1473
Ultimate, lbf:	17830
Ultimate, MPa:	834
Area, mm²:	95.0332
Max, MPa:	834
Max, N:	79300
OS @ .2, MPa:	242
Ultimate, ksi:	121
Max Deflection, mm:	3.09
Break, mm:	3.07
Max, %:	18.78
Break, mm:	3.07
Break, lbf:	16180
Break, in:	0.121

Fig 8: Stress-strain curve (Compression strength result)

Table 2: Ultimate compression strength

Exp.	Compacting load (MPa)	Sintering temperature (ºC)	Ultimate compression (MPa)	Ultimate compression (MPa)	Average ultimate compression
1	300	700	542	579	560.5
2	300	800	631	604	617.5
3	300	900	846	855	850.5
4	500	700	735	726	730.5
5	500	800	770	775	772.5
6	500	900	838	886	862
7	700	700	716	724	720
8	700	800	825	830	827.5
9	700	900	834	829	831.5

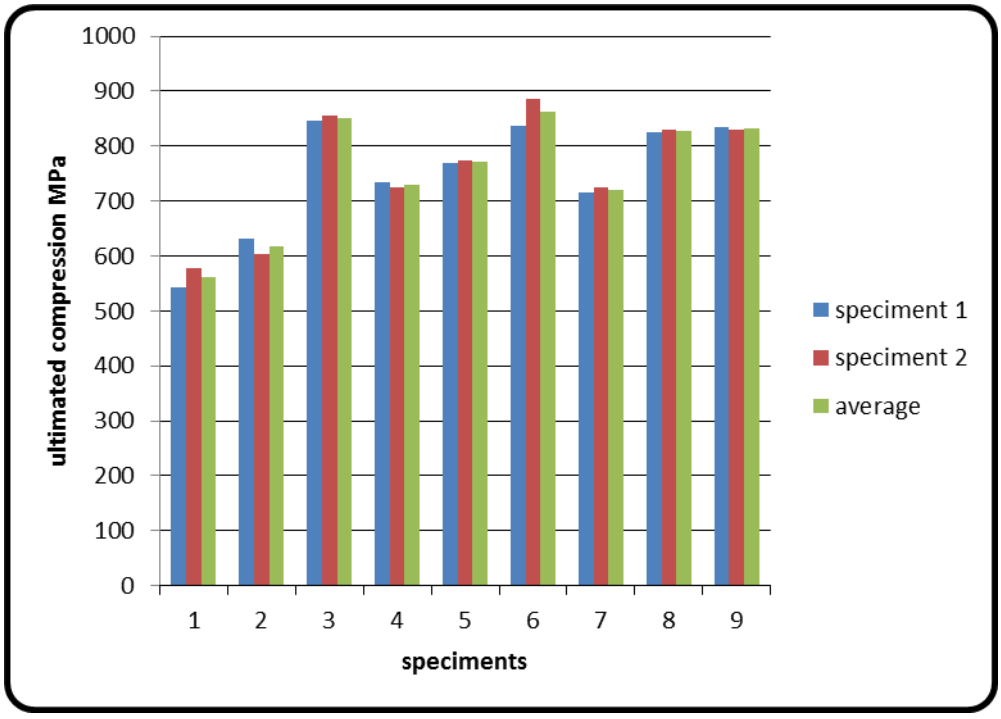


Fig 9: Ultimate compression test results

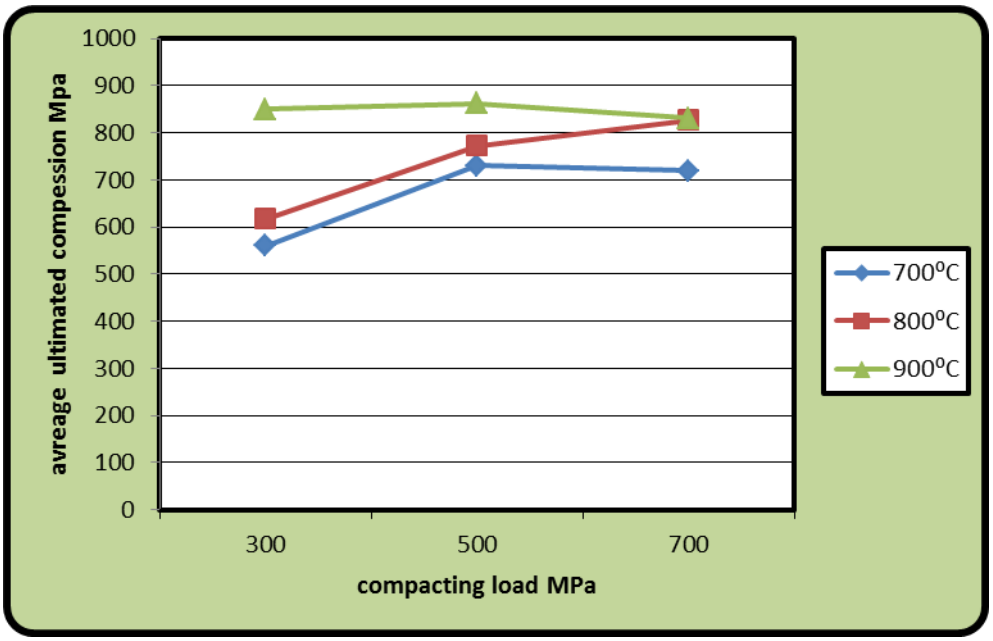


Fig 10:Results of average ultimate compression test at different compacting pressure and sintering temperature

The shape effect test of shape memory alloy results shown in **Fig.11**.**Table 3** give experimental results of compressed specimens of powder metallurgy for shape memory alloy (shape effect , mean). And the ultimate shape effect for each compacting pressure with three sintering temperature are shown in **Fig.12.**

Table 3: Measurement and calculate shape effect

Compacting load (MPa)	Sintering temperature (°C)	Shape effect)1 (%)	Shape effect)2 (%)	Average shape effect %
300	700	76.92	70.96	73.94
300	800	67.74	63.49	65.61
300	900	83.33	79.41	81.37
500	700	66.66	69.81	68.23
500	800	80.55	72.41	76.48
500	900	83.92	80.59	82.25

700	700	72.54	76.19	74.36
700	800	78.94	71.42	75.18
700	900	89.47	83.33	86.40

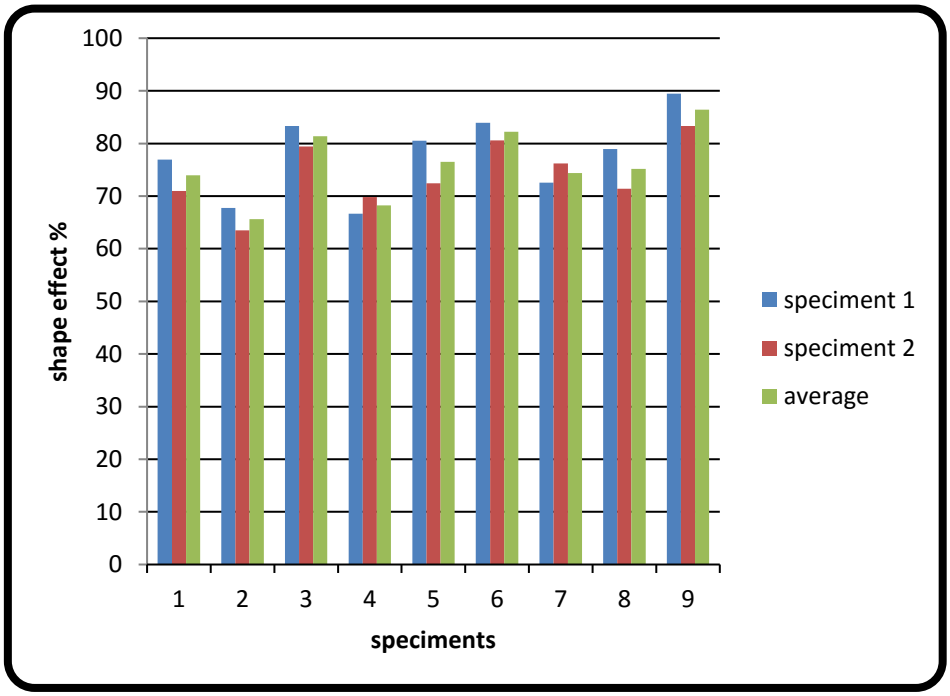


Fig 11: Shape effect test results

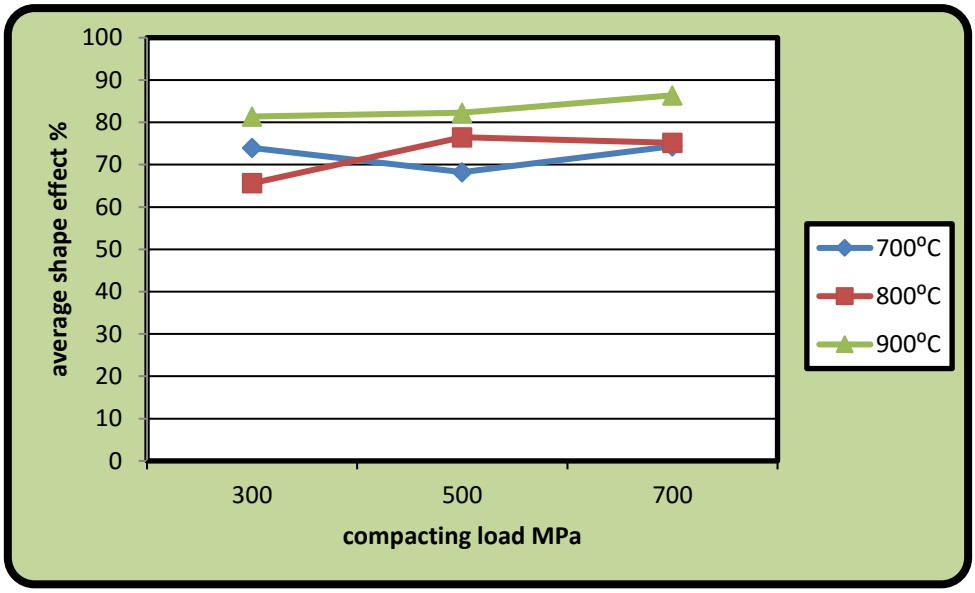


Fig 12:Result of average shape effect test at different compacting pressure and sintering temperature

The density test of shape memory alloy results shown in **Fig.13**.**Table 3** give experimental results of compressed specimens of powder metallurgy for shape memory alloy (density , mean).

Table 3: Measurement and calculate density

Compacting load (MPa)	Sintering temperature (°C)	Density)1 (g/cm^3)	Density)2 (g/cm^3)	Average density g/cm^3
300	700	5.037	4.167	4.602
300	800	4.999	5.259	5.129
300	900	5.578	5.589	5.583
500	700	5.329	5.004	5.166
500	800	5.294	5.303	5.298
500	900	5.294	4.870	5.082
700	700	5.268	4.933	5.100
700	800	5.380	5.426	5.403
700	900	5.317	5.699	5.508

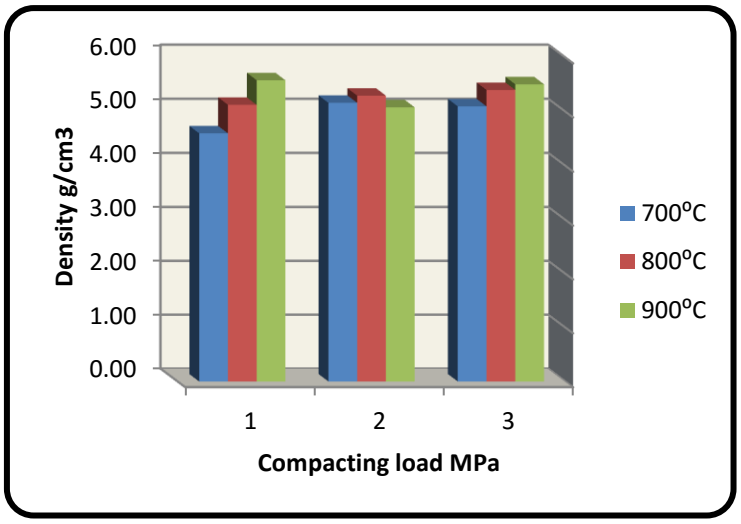


Fig 13:Result of average density test at different compacting pressure and sintering temperature

4- CONICLUSIONS

1-Using different pressure and sintering temperature will not effect on x-ray diffraction results or the shape memory effect.

2-Increasing sintering temperature will:

(a) Increase shape effect.

(b) Increase ultimate compression strength for the shape memory alloy.

3- The experimental test for the samples showed that the highest value for the resilient be at a pressure of 700MPa and at a sintering temperature of 900°C

4-Either check compression strength was the highest value at a pressure of 300 MPa and temperature sintering 900°C.

5- ACKNOWLEDGEMENTS

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تأثير الضغط ودرجة حرارة التلبيد على خاصية الشكل ومقاومة الانضغاط لسبيكة Cu-Al-Ni ذات خاصية تذكر الشكل

احمد عبد الرسول
استاذ مساعد
سارة جلال موسى
قسم الهندسة الميكانيكية
كلية الهندسة – جامعة بغداد /العراق

الخلاصة

السبائك الذاكرة للشكل هي نوع من السبائك الذكية التي تستخدم في العديد من التطبيقات الصناعية في الوقت الحالي.تم تصنيع عينات لسبيكة ذكية تتكون من نحاس وألمنيوم ونيكل بطريقة مساحيق المعادن ,تم خلط المواد بنسب وزني Cu83%wt – Al 13%wt- Ni 4%wt بعد ذلك تم كبس العينات بثلاث قيم للضغوط مختلفة MPa((300,500,700 , تمت عملية التلبيد في فرن انبوبي كهربائي مفرغ من الهواء حيث ان درجات الحرارة للتلبيد ثلاث قيم مختلفة oC((700,800,900.اجريت الفحوصات اللازمة وهي فحص الرجوعية وفحص الانضغاط لجميع العينات. النتائج العملية اظهرت انه اعلى قيمة للرجوعية تكون عند ضغط MPa700وعند درجة حرارة التلبيد oC900 اما فحص الانضغاطية كانت اعلى قيمة لها عند ضغط MPa 300 ودرجة حرارة التلبيد .oC900

الكلمات الرئيسية : سبيكة ذاكرة للشكل ، نحاس –المنيوم- نيكل ,ميتالورجيا المساحيق , الرجوعية ,الانضغاط