Investigate the Microstructure and the Mechanical Properties of Ni-Ti-Cu Shape Memory Alloys

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Abstract

In this study a Nickel-Titanium-Cupper shape memory alloys was manufactured by powder metallurgy (PM) technique, powder mixture of 50% Ti , 47% Ni and 3% Cu was prepared by mixing for two hours and compacted in a press machine using various compacting pressure (600, 700 and 800) MPa , sample was then sintered for 5 hrs in an electrical tube vacuum furnace using sintering temperature of (850˚C, 900˚C and 950˚C) .phase analysis of samples was conducted by X-ray diffraction test, the effect of different sintering temperature and compacting pressure on the porosity, microhardness ,compression strength and the shape memory effect (SME) was studied, the result showed decrease in the porosity and increasing in the shape recovery ,compression strength and microhardness with increasing compacting pressure and at lower sintering temperature and hence the best results was at 800MPa compacting pressure and 850˚C sintering temperature.

Key words: shape memory alloys, powder metallurgy, porosity, compression strength, shape memory effect.

Introduction

Shape memory alloys are the most important branch from the smart or intelligence materials . The term “Shape memory alloys” (SMAs) refers to that group of metallic materials that have the ability to return to some previously defined shape or size when subjected to appropriate thermal cycle. Among the many alloy systems which exhibit Shape Memory Effect (SME), Ni-Ti, Cu-Al-Ni and Cu-Zn-Al SMAs have been studied extensively and are commercially exploited [1].

Shape memory alloys have two phases namely austenite and martensite; Austenite is the high temperature or “parent” phase and exhibits a (cubic) crystalline structure while martensite is the low temperature phase that exhibits a (tetragonal or monoclinic) crystalline structure . The transformation from austenite to martensite may lead to twinned martensite in the absence of internal and external stresses or detwinned martensite if such stresses exist at a sufficient level [2]. The temperatures at which martensite start and finish forming and austenite start and finish forming are represented by the following variables accordingly: Ms, Mf, As, Af. The amount of loading placed on a piece of shape memory alloy increases with the values of these four variables [3].

Nitinol represent the most common type of shape memory alloys and it is based on Ni and Ti, most frequently used in commercial applications because they combine good mechanical properties with shape memory [4].

Two significant effects are related with the phase change: the shape memory effect and the superelasticity. The superelasticity occurs when the martensitic phase transformation is stress induced at a constant temperature. The transformation is characterized by a plateau and a hysteresis upon unloading. The magnitude of reversible "pseudo-elastic strain" can be as high as 8 % or even more for single crystals. The shape memory effect (SME) refers to the ability of the material, initially deformed in its low-temperature phase (martensitic), to recover its original shape upon heating to its high temperature phase (austenic or "parent phase").[5]

Powder metallurgy (PM) is a promising method for the production of net shape or near-net-shape components of Ni-Ti. Its advantages include avoiding expensive thermomechanical working needed after casting, the machining of cast alloys and high losses of material.[6]

Nitinol shape memory alloys have achieved high growth in the biomedical area due to their an usual properties, it can be used in orthopedic application such as plates for bone fracture repair, load-bearings, vertebral spacers, spinal correctors, and bone distraction devices, the cardiovascular application like stents, heart valves, vena cave filters. Orthodontic applications such as in the correction of malocclusions .Eyeglasses frame and can be used for making advanced surgical instruments. [7].

The goal of this research is to produce a Ni-Ti-Cu shape memory alloy using powder metallurgy (PM) technique and investigate the effect of different sintering temperature and compacting pressure on the porosity, compression strength, shape recovery and microhardness of the alloys.

Experemental Work

Ni-Ti-Cu shape memory alloys was prepared by powder metallurgy technique , to prepare the samples the first step was the preparation of the powder which consist of 50% Titanium with 47% Nickel and 3% Copper respectively was used with high purity (>99.9%) and (325 mesh) particle size. These powders were weighed and placed in cylindrical containers then mixed by an electrical barrel mixer, the time of mixing the powder was 2 hrs [8].

Table1: SMAs chemical composition with compacting pressure and sintering temperature.

Alloy composition (%)	(50% Ti + 47%Ni + 3% Cu)		
Compacting pressure (MPa)	600	700	800
Sintering temperature (˚C)	850	900	950

Powder mixture were then compacted in a press machine at 600,700 and 800 MPa using single action tool steel mold to form a cylindrical shaped sample (11mm dia. X 16.5mm length) and (11mm dia. X 5 mm length), the samples after compacting had sufficient green strength to be handled .

The green compacted samples were then sintered at 850,900 and 950 ºC for 5 hours in an electrical vacuum furnace as shown in “Fig. 1."

Porosity measured from the actual and theoretical density of the samples [9].

$$\text{Porosity\%} = [1-(\frac{\text{actual density}}{\text{theoretical density}})] \text{ x100} \quad \ldots (1)$$

$$\text{Actual density} = \frac{weight}{volum} \quad \ldots (2)$$

Where the volume of green samples = $\pi R^2 L$...(3)

R=Radius.

L=Length of cylinder.

While the volume of sintered samples was measured using water displacement method.

The theoretical density equal 6.7 g/cm³ and obtained from equation below:

$$\rho s = \sum_{i=1}^{n} \text{Wt} * \rho_1 + \text{Wt} * \rho_2 + \text{Wt} * \rho_3 + \cdots + \text{Wtn} * \rho n \quad \ldots (4)$$

ρs = theoretical density of sample (g/cm^3). , n= no. of elemental powders.

Wt= weight percent (%)

$\rho_{1,2,3,....n}$ = density of elemental powder (g/ cm^3).

Microhardness test was conducted on all the samples by taking three values of hardness for each sample to get the mean value which represents hardness.

Shape memory effect test was conducted by compacting the (11mm dia. X 16.5 mm length) samples to 0.06% of its original length then let to cool in air and calculating the shape effect (the return length) by applying equation below[10] :

$$\text{Shape memory effect} = \frac{L2-L1}{L0-L1} * 100 \quad \ldots (5)$$

Where:

L_0: normal sample length

L_1: sample length after compacted

L_2: sample length after heating.

The compression strength of the samples was measured using the same press machine.

The phases that formed by sintering process were detected using the X-Ray diffraction (XRD) and scanning electron microscope (SEM) for all the samples.

Result and Discusion:

- Porosity percentage decrease with increasing compacting pressure as shown in table (2), Rearrangement of powder particles occur when the compacting operation starts then as the pressure is applied a localized deformation occurs at powder contacts. Increasing in the pressure results in an increase in the proportional volume of all particles undergoing plastic distortion furthermore increases in pressure cause the removal of more pores and the creation of new contacts, and finally the homogeneous deformation of the whole compact, Work hardening necessarily comes along with plastic distortion. Applying sintering temperature on the green samples table (2) reduce the porosity due to the shrinkage in the original pores . With further increasing of sintering temperature the porosity increased slightly table (3) The reason could be interpreted that increasing of the sintering temperature cause differences in shape and distribution of pores and the average diameter of the pores increased, in result of that most of the small pores were joined into large one forming their irregular shapes.

Table 2: Porosity measurement test result of green samples

No.	Pressure (MPa)	Porosity (%)
1	600	25.7
2	700	23.82
3	800	23.43

Table 3: Porosity measurement test result of sintered samples.

No.	Pressure (MPa)	Temperature (˚C)	Porosity (%)
1	600	850	11.26
2	600	900	12.06
3	600	950	13.74
4	700	850	10.89
5	700	900	11.06
6	700	950	11.32
7	800	850	8.16
8	800	900	8.62
9	800	950	9.65

- The microhardness test results shown in “Fig. 2." demonstrate an improvement in hardness with increasing of compacting pressure since high pressure eliminate the pore and with decreasing of sintering temperature , high sintering temperature increase the porosity as it was explained in the results of porosity test above.
- The results of shape memory effect percent (the retaining length) shown in “Fig. 3." as a function of different compacting pressure and sintering temperature showed that the best shape recovery was (88.5%) at the highest compacting pressure (800MPa) and the lower sintering temperature (850˚C) since the porosity is less this show agreement with (Tomasz Goryczka) increasing sintering temperature cause grain growth and increase the porosity and this was clear at XRD test since high intensity of austenite and martensite was formed at this condition and they are responsible on the shape memory affect.
- Compression test results “Fig. 4." shows the improvement in compression strength with high compacting pressure and lower sintering temperature , the best compression strength was at 800MPa and 850˚C,these improvements were due to the decrease in the defects, such as pores and microcracks which act as a stress concentration regions and may cause the early failures of the alloys.
- Scanning electron microscope gives important information on the microstructures of the samples, SEM images show the appearance of the martensite layers with the pores distribution. The shape and distribution of pores depend on the sintering temperature and it increases with an increase in sintering temperature, for the samples compacted at 800MPa the martensite layers formed clearly at 850˚C sintering temperature with some small pores ,as the sintering temperature increase the pores volume and distribution increase as shown in “Fig. 5."
- XRD was conducted to all sintered sample , the result showed that NiTi alloys with different phase composition can be achieved by controlling the sintering conditions time and temperature . All Ni and Ti are transformed to NiTi monoclinic phase, Ti_2Ni cubic phase and rhombohedral Ni_4Ti_3 phase. They show the high intensity of NiTi monoclinic which is known as martensitic phase and Ti2Ni cubic phase which is known as austenitic phase. These two phases are responsible of the shape memory effect in Ni-Ti shape memory alloys. The formation of Ni_4Ti_3 might be attributed to the slow cooling of samples within the furnace, it is difficult to completely remove Ni_4Ti_3 from sintered sample so the conditions for sintering are sufficient to complete the transformation. “Fig. 6.

Conclusion

1. Porosity percentage reduced with increasing of compacting pressure and at lower sintering temperature.
2. Different phase composition can be achieved by controlling the sintering conditions time and temperature.XRD test shows the formation of two main phases (austenite and martensite) in Ni-Ti-Cu shape memory alloys.
3. Shape effect test results show the presence of shape memory in the alloys.
4. Compression strength of the samples improved with raising compacting pressure (low porosity).
5. Microhardness increase with increasing of compacting pressure and at lower sintering temperature since the porosity decreased.

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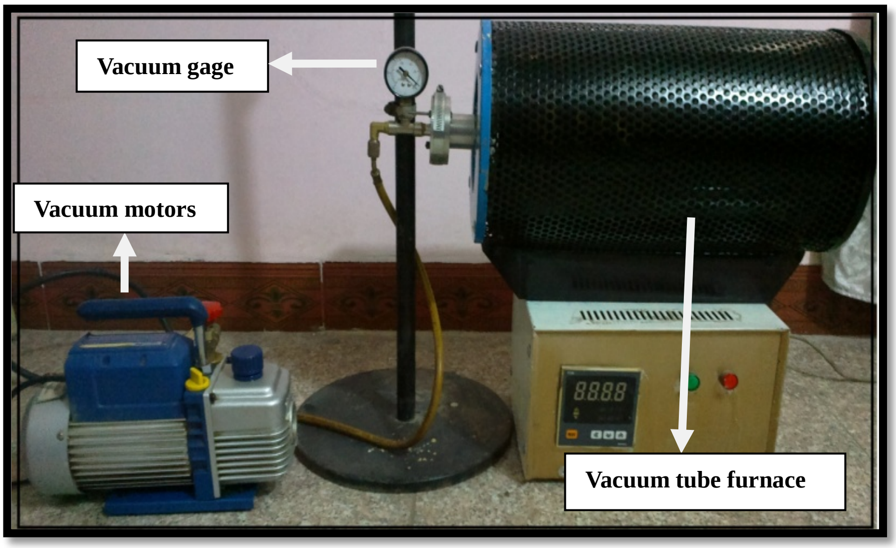
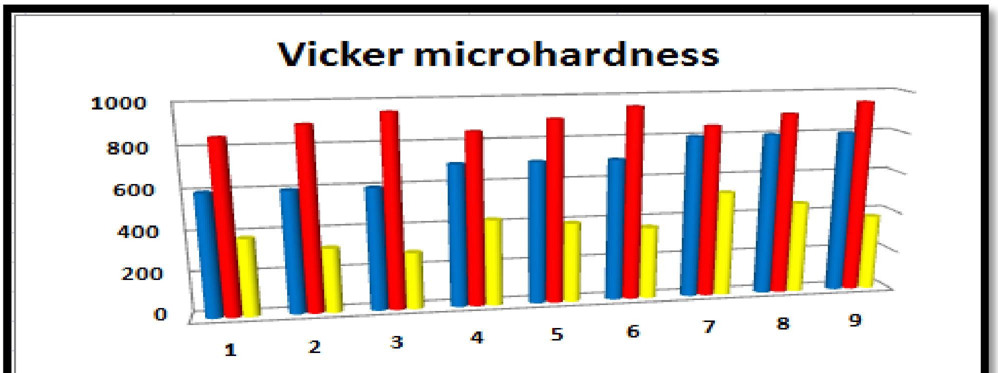
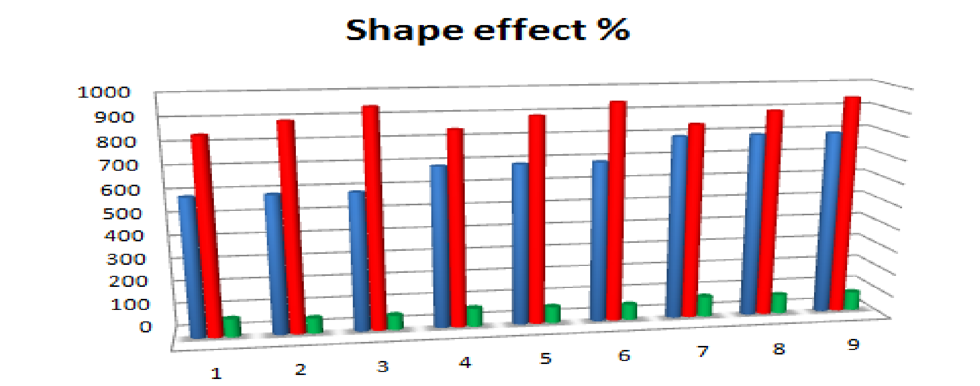


Figure1: Electrical tube furnace.



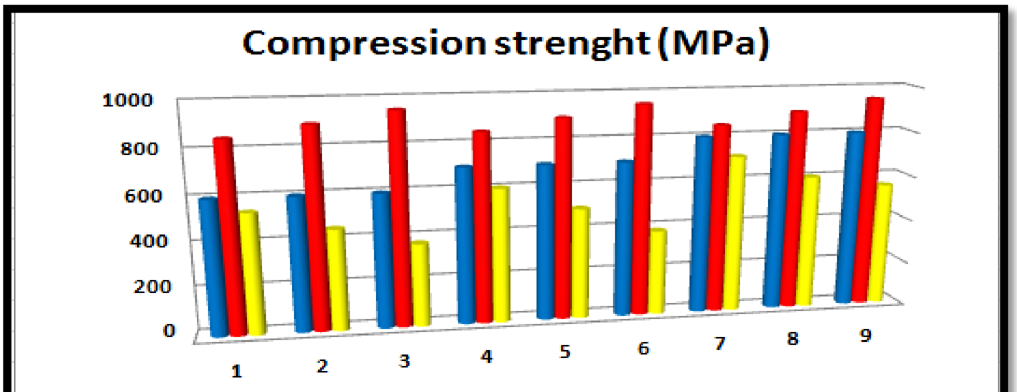
	1	2	3	4	5	6	7	8	9
Pressure	600	600	600	700	700	700	800	800	800
Temperature	850	900	950	850	900	950	850	900	950
Vicker microhardness	379	317.8	279	426.23	394.03	359.36	520.2	454.03	374.5

Figure 2: Vicker microhardness test results.



	1	2	3	4	5	6	7	8	9
Pressure (MPa)	600	600	600	700	700	700	800	800	800
Temperature (˚C)	850	900	950	850	900	950	850	900	950
Shape effect %	79.2	67	64	80	71.4	67	88.5	83.3	82

Figure 3: Shape effect test results.



	1	2	3	4	5	6	7	8	9
Pressure (MPa)	600	600	600	700	700	700	800	800	800
Temperature (˚C)	850	900	950	850	900	950	850	900	950
Compression strenght (MPa)	538.25	452.37	372.29	603.49	499.66	383.75	708.22	603.97	555.78

Figure 4: Compression strength test results .

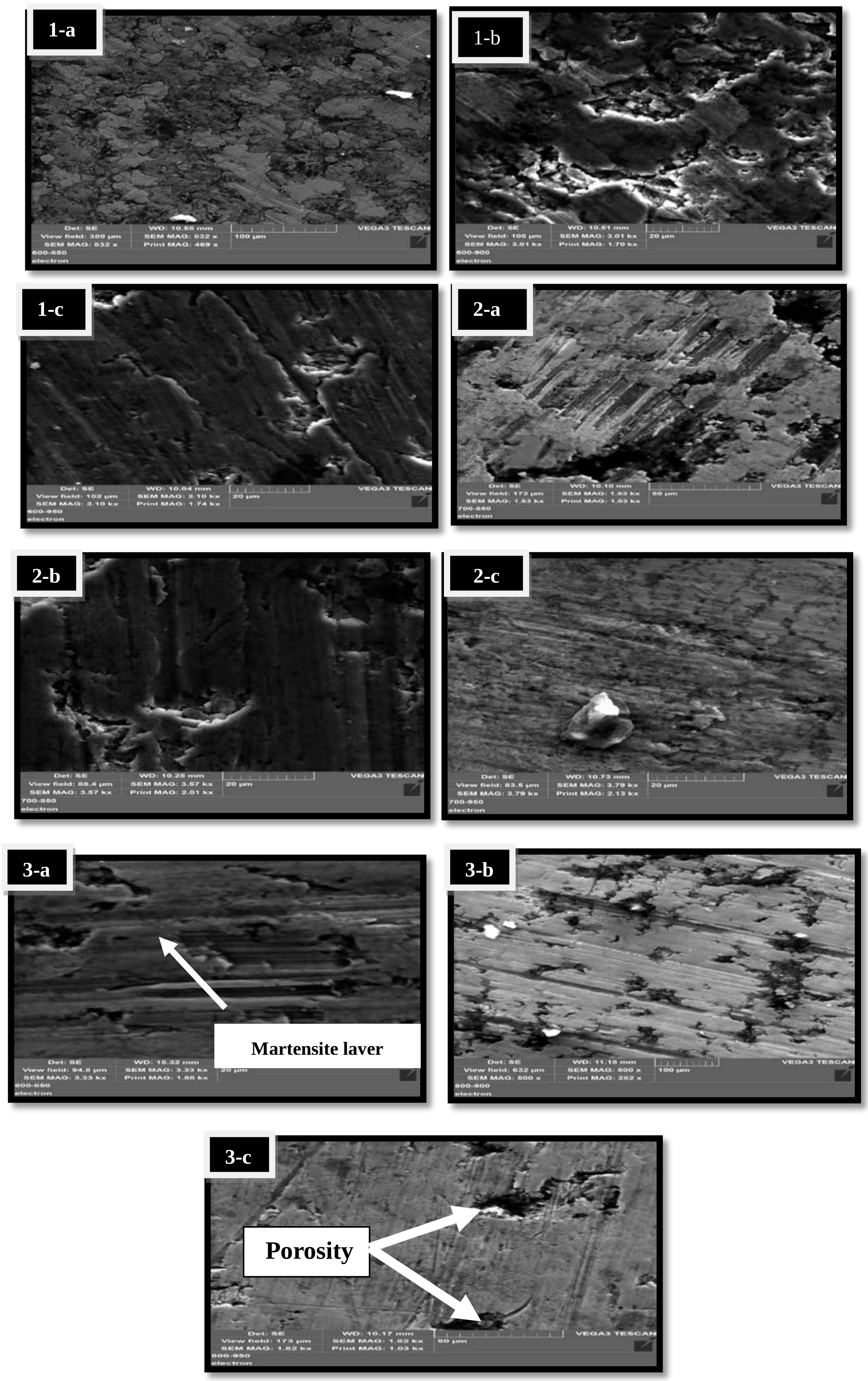
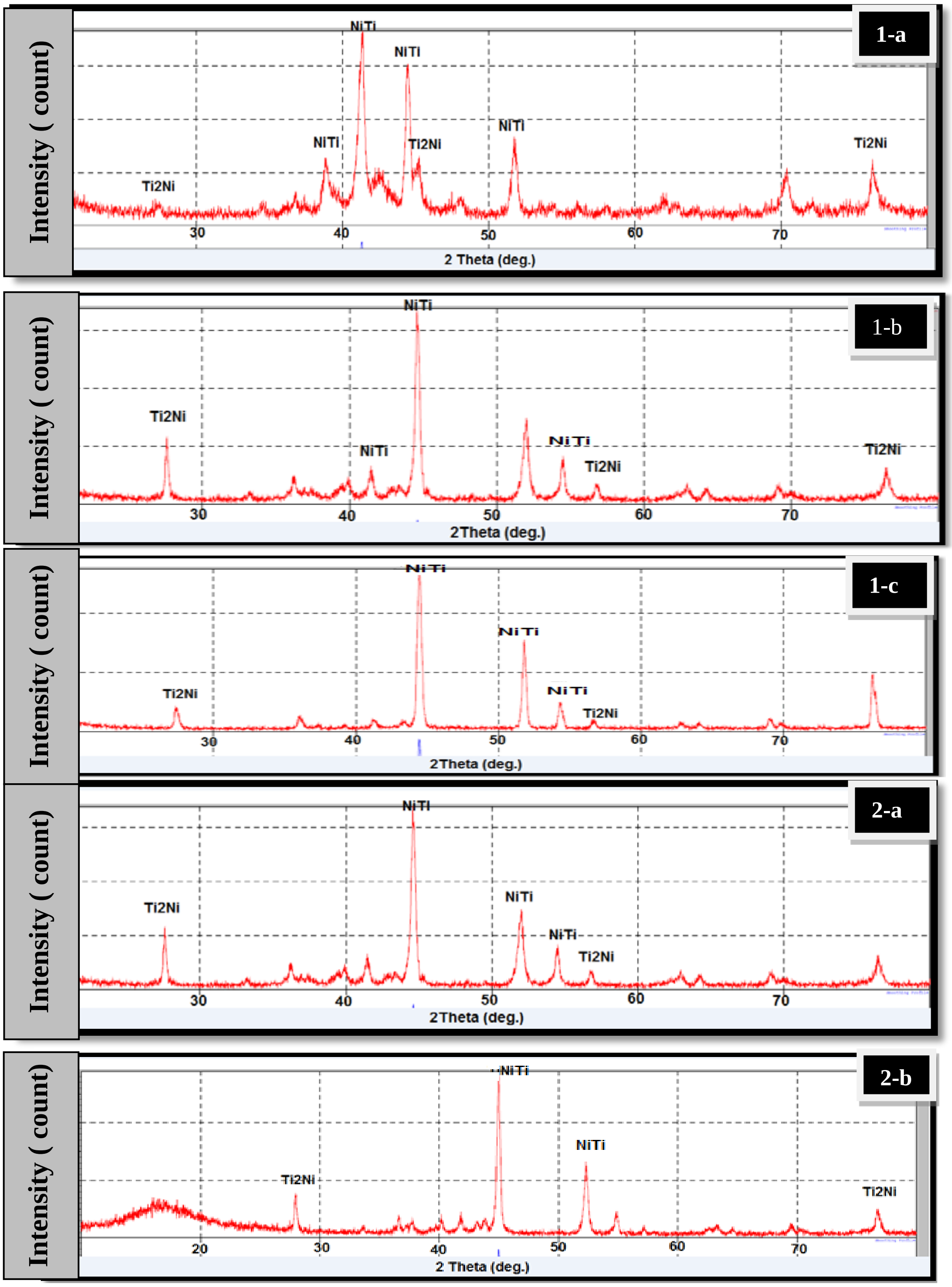


Figure 5: Result of SEM test at (1-600MPa 2-700MPa 3- 800MPa) compacting pressure with (a-850˚ C, b-900˚C, c- 950˚C) sintering temperature.


1-a
1-b
1-c
2-a
2-b
Intensity (count)
2 Theta (deg.)
NiTi
Ti2Ni
20
30
40
50
60
70

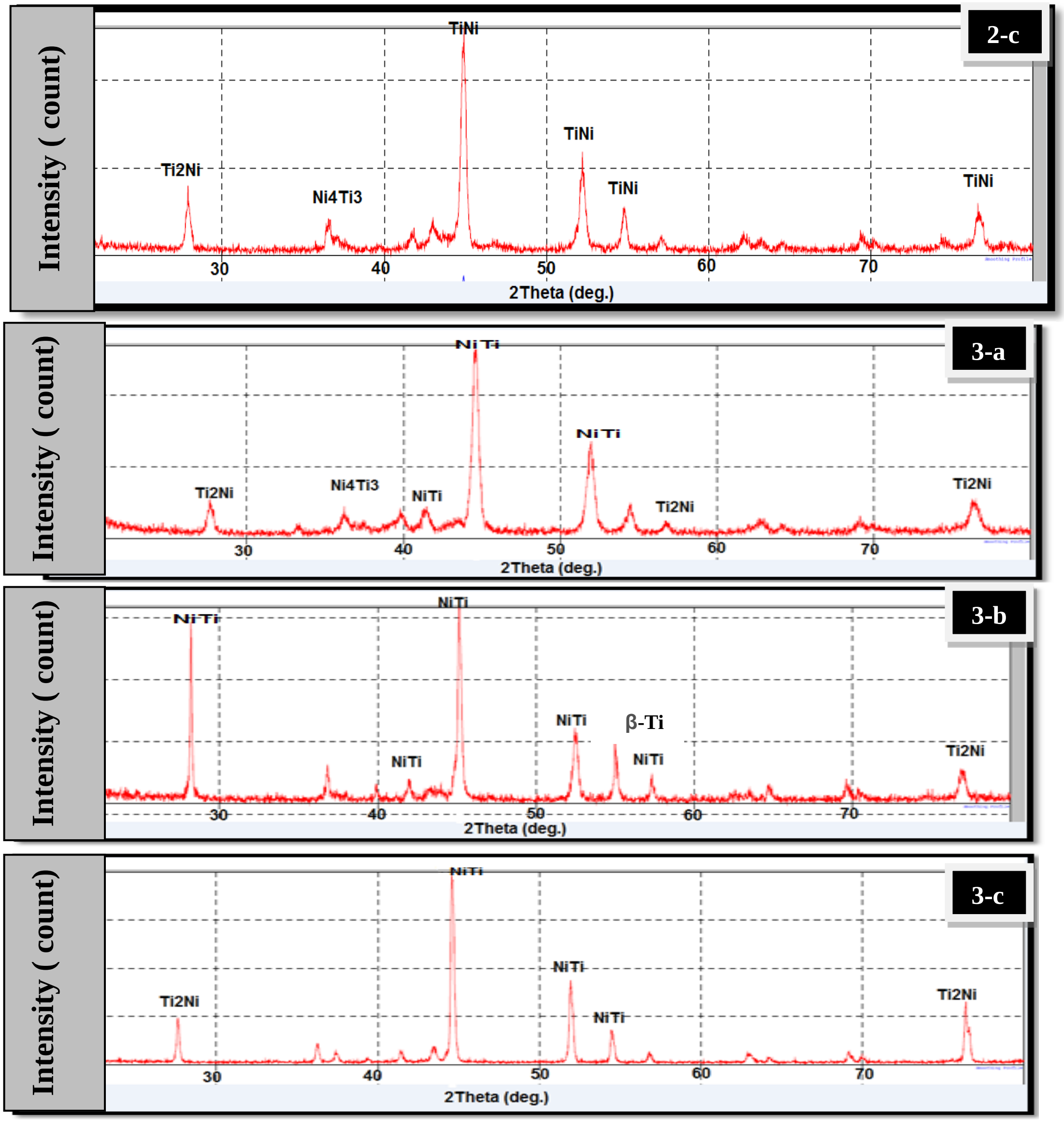


Figure 6: Result of XRD test at (1-600 MPa 2-700MPa 3- 800 MPa) compacting pressure with (a- 850˚ C,b-900˚C, c- 950˚C) sintering temperature.

التحقيق في البنيه المجهرية والخواص الميكانيكيه لسبائك Ni-Ti-Cu الذاكرة الشكل

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الخلاصة

في هذه الدراسة تم تصنيع سبائك ذاكره الشكل (نيكل – تيتانيوم – نحاس) بواسطة تقنيه ميتالورجيا المساحيق وتم تحضير النماذج بخلط المساحيق بنسب وزنية (50%Ti)، (47%Ni) و(3%Cu) لمدة ساعتين. تم كبس الخليط بضغط 600، 700و 800 ميكا بسكال وبعدها أجراء عملية التلبيد باستخدام فرن كهربائي و لمده 5 ساعات وبدرجات حرارة 950,900,850 ˚م. تم دراسة تكون الأطوار باستخدام مطياف الأشعة السينية , ودراسة تأثير حرارة التلبيد وضغوط الكبس المختلفة على المسامية، الانضغاط،الصلادة وقابليه التذكر للسبائك.
النتائج العملية أظهرت زيادة قابليه التذكر،الصلادة، قوة الانضغاط ونقصان المسامية بزيادة الضغط ونقصان درجه حرارة التلبيد وبالتالي فأن أفضل النتائج كانت عند ضغط كبس 800 ميكا بسكال وحرارة تلبيد850˚ م..

الكلمات الرئيسية : سبائك ذاكره الشكل ،ميتالورجيا المساحيق ، مسامية ، متانة الانضغاط ، قابليه التذكر.