



## **Optimization of Ni-Ti-Cu shape memory effect using minitab program**

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### **Abstract**

This study focuses on the manufacturing of Nickel-Titanium-Copper shape memory alloys using powder metallurgy (PM) method, 50% Ti, 47% Ni and 3% Cu powder mixture was prepared by mixing in a ball mill for two hours and compacted in a press machine using various compacting pressure (600, 700 and 800) MPa, samples was then sintered in an electrical tube furnace using three sintering temperature of (850, 900 and 950) °C. Optimization of the shape memory effect (SME) using Minitab program was conducted; the result showed increasing in the shape recovery with increasing of compacting pressure and reduction in sintering temperature.

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**Keywords:** Shape memory alloys; Powder metallurgy; Shape memory effect; Optimization by Minitab.

### **1. Introduction**

Shape memory alloys act as a very essential branch of clever or intelligence materials groups. It's a metallic material that can get back to their prior defined form and size when subjected to heat [1].

SMA's consist of two necessary phases called austenite and martensite; Austenite is the high temperature phase or called the parent phase, the crystal structure of austenite is (cubic). Martensite is the low-temperature phase which consist of (tetragonal or monoclinic) crystal structure [2].

There are four characteristic temperatures associated with the phase transformation of austenite and martensite phases expressed by; Ms, Mf, As, Af variables respectively [3]. The experimental research developed a range of the mechanical parameters that would be expected from shape memory alloy in its austenite and martensite phases and these mechanical properties are; pseudoelasticity or superelasticity effect and shape memory effect. Shape-memory effect (SME) is a phenomenon by which SMA's that have been permanently deformed can recover their original configuration after being heated above a certain transition temperature. Pseudoelasticity (also known as superelasticity) is the ability of the Ni-Ti SMA to return to its original shape upon unloading after substantial deformations, Ni-Ti alloy represents the most common shape memory alloy [4].

Shape memory alloys (SMA) provide high growth in aerospace, biomedical and mechanical engineering, the high success of these materials is due to their unusual properties, which makes them superior to conventional materials such as shape memory effects and superelastic effect [5].

Biological properties of the Ni-Ti SMA's can be improved by surface modification or by the addition of third element such as Cu. [6].

Ni-Ti-Cu shape memory alloys can be gained by the alteration of Ni by Cu atom in the equiatomic Ni-Ti alloy, copper placement as a third alloying element produce an increment in the characteristic temperatures of the martensitic transformation in contrast to a binary Ni-Ti alloy. Besides, copper addition result in good stability in the characteristic temperatures and increase the corrosion resistance, close transformation hysteresis and stop the Ti<sub>3</sub>Ni<sub>4</sub> precipitation, the composition susceptibility of martensite start temperature (Ms) can also be minimize by Cu addition. Unfortunately, the Cu addition, that is higher than 10 at % reduce the alloy formability [7].

This is the reason behind using of non-melting methods. Recently, high effort has been made to use the nonconventional production methods such as the powder metallurgy (PM), twin roll casting, melt-spinning (MS) for producing the Ni-Ti-based alloys.

The main feature of the powder metallurgy is to prevent thermo-mechanical treatment process that is needed after the conventional casting [8].

Minitab is statistical software used for different statistical analysis and data management, the program capable of performing different types of statistics methods, it can be used to do regression analysis also the basic statistical tests can be performed by it.

The program capable of generating graphs which are mainly used as a visual analysis for the statistic results, the program provides step-by-step guidance when performing statistical analysis. It even provides help when analyzing results and can offers an interactive decision tree that enables users to choose which approach is best for the provided data [9].

## 2. Experimental work

### 2.1 Samples preparation

Nickel-Titanium-Copper shape memory alloy samples were prepared using powder metallurgy method, a glass container was used to place the powder inside it, powder of high purity (>99%) and in the percent of 50% titanium with 47% nickel and 3% copper weight respectively and placed in the glass container as shown in Table 1 below. Electrical mixer was used to mix the powder for 2 hours according to reference [10] and the speed of rotating the drums are currently set to 75 rpm.

A press machine was used with single action tool steel mold for compacting the samples, the press machine compaction displacement rate was 1 mm/min with a holding time of 4 minutes to ensure from the required dimensions, cylindrical form samples of (11mm dia. X 16.5mm length) and (11mm dia. X 5 mm length) was obtained, the compact samples had strength called the green strength adequate for handling.

The green samples was then sintered using an electrical vacuum tube furnace at 850,900 and 950 °C for 5 hours as shown in Figure 1; and then slow called in furnace, sintered samples were then grinded and polished, Figure 2 show the samples after sintering.

### 2.2 Shape memory effect test

Shape memory effect (shape recovery) was measured by compacting the (11mm dia. X 16.5 mm length) samples Figure 3 by 0.06% from the fundamental length, the displacement rate was 1mm/min and then heating the samples to 110°C for 5 min and let to slow cool in furnace and then measuring the shape memory effect using the equation below [11]:

$$\text{Shape memory effect} = \frac{L_2 - L_1}{L_0 - L_1} * 100 \quad (1)$$

where; L<sub>0</sub>: Samples normal length, L<sub>1</sub>: Samples length after compaction by 0.06%, L<sub>2</sub>: Sample length after heating to 110°C for 5 min.

Table 1. 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
Samples dimensions (mm).	1. (11 dia. * 5 height)		
	2. (11 dia. * 16.5 height)		

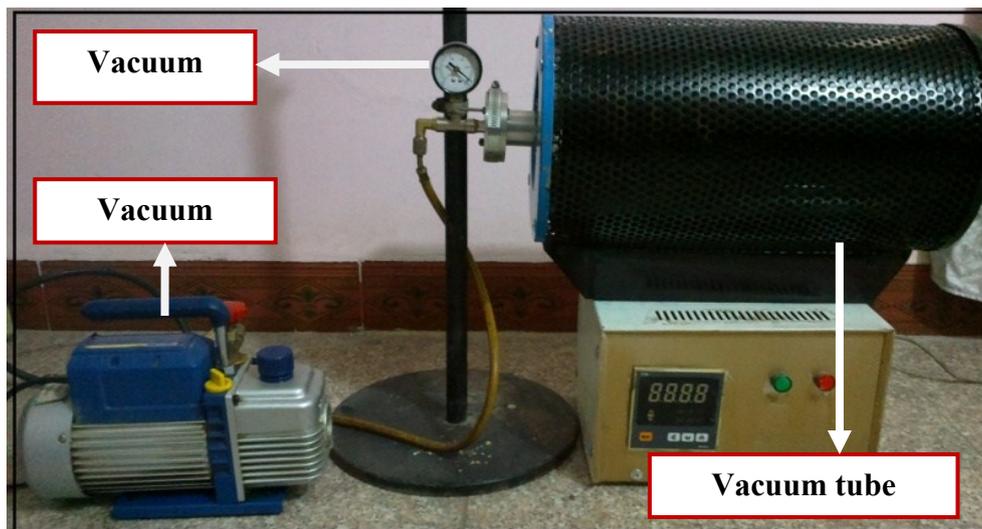


Figure 1. Electrical tube furnace.



Figure 2. Samples after sintering.



Figure 3. Shape memory effect test.

### 2.3 Optimization of the shape memory effect

Optimization of the shape memory effect was conducted using Minitab program especially Design of Experiment (DOE) tool and regression analysis, DOE help to investigate the effect of input variables on an output response using two variables (temperature and pressure) in the experimental work. After selecting DOE tool (Minitab > Stat> DOE) two variables was used (input variables) and three values for each variable the result was analyzed by; main effect plot to show the variables that had influence on the shape memory effect of the alloy, Pareto chart which help in analyzing the variable that have the most significant effect, response optimizer which helped to define the most suitable value that give the best shape memory effect. Regression analysis help to find the optimum shape memory effect.

After obtaining the conditions (compacting pressure and sintering temperature) that give the optimum shape memory effect the sample was manufactured using powder metallurgy method, shape memory effect test was conducted to make sure from the obtained result value.

## 3. Result and discussion

### 3.1 Shape memory effect

Shape memory effect test results shown in Table 2 appears an improvement in the shape retaining length with an increase in compaction pressure (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 increasing pressure cause the removal of pores and the creation of new contacts, and finally the homogeneous deformation of the whole compact) [12].

And with reduction in sintering temperature, the reason could be interpreted that increasing of the sintering temperature causes 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 [13]. And hence the best shape memory was (88%) at 800 MPa compacting pressure and 850°C sintering temperature.

Table 2. Shape memory effect test result.

No.	Pressure (MPa)	Temperature (°C)	Shape effect (%)
1	600	850	79.2
2	600	900	67
3	600	950	64
4	700	850	80
5	700	900	71.4
6	700	950	67
7	800	850	88.5
8	800	900	83.3
9	800	950	82

### 3.2 Optimization by minitab result

#### 3.2.1 Main Effect Plot

Main effect plot results show that the compacting pressure affect the shape memory of the alloy because the line is not horizontal and that's mean different level of pressure affect the shape memory differently, 800MPa compacting pressure has higher effect than 700 and 600MPa.

Sintering temperature also has influence on the shape memory effect, 850°C sintering temperature has higher effect than 900 and 950°C as shown in Figure 4.

#### 3.2.2 Pareto chart

Pareto chart gives help in analyzing the variables that have the most significant influence on the shape memory effect results, it was found that both temperature and pressure had effect on the obtained results but the most effective variables was the pressure as shown in Figure 5.

#### 3.2.3 Response optimizer

The method of response optimizer helped to define the most suitable values that gives the best shape memory effect as shown in Figure 6, the result showed that the best shape recovery value were (87.76%) at 800MPa compacting pressure and 850°C sintering temperature and this is nearly similar to the obtained results from the experimental works.

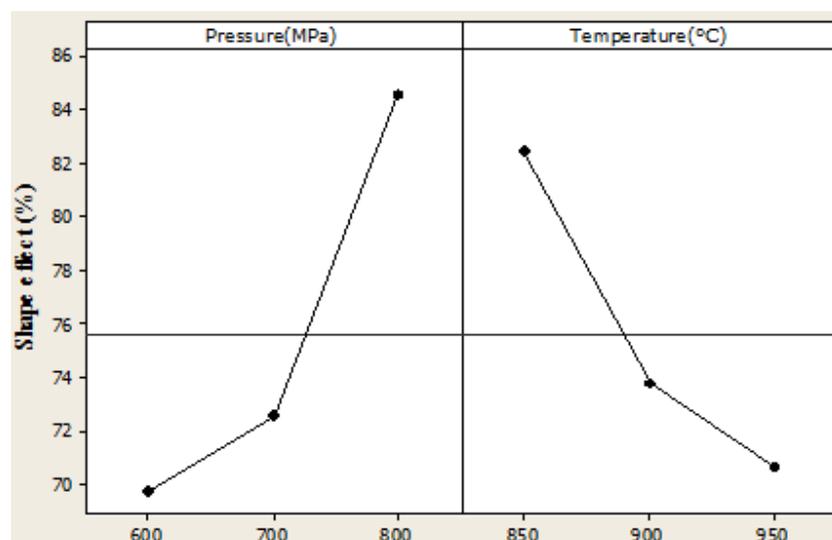


Figure 4. Main effect plot.

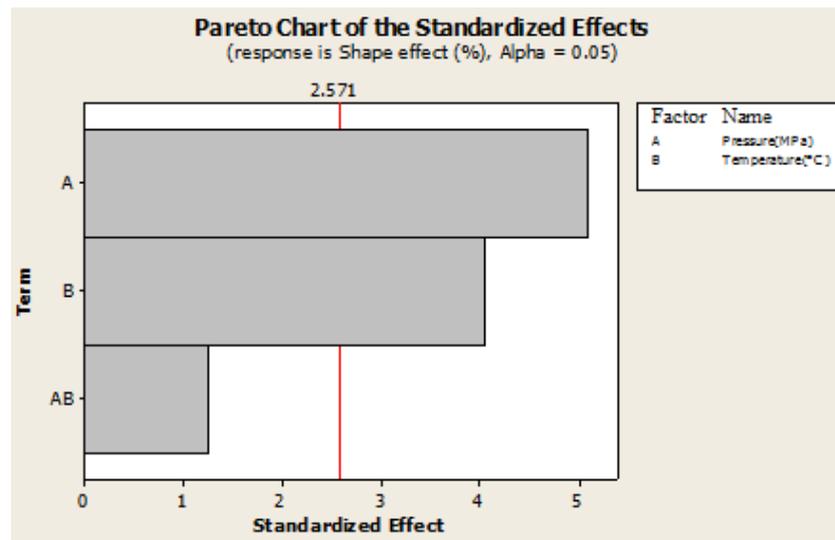


Figure 5. Pareto chart.

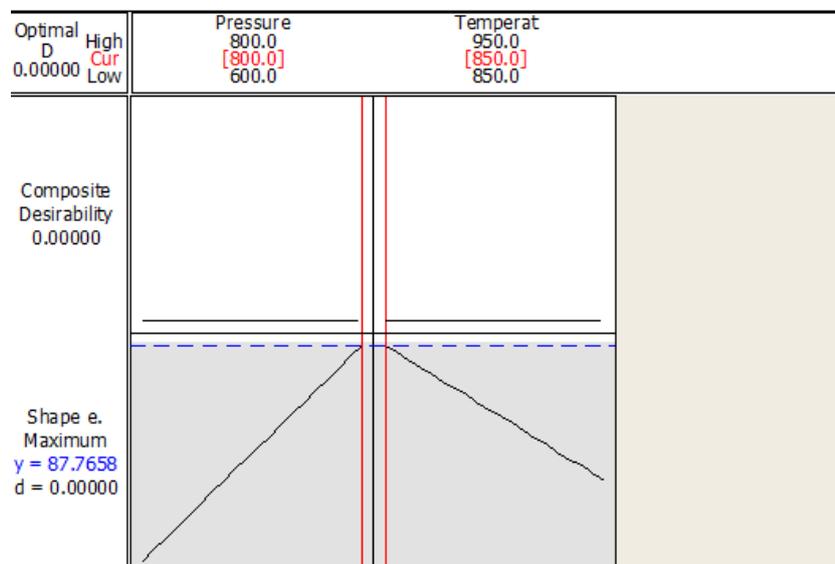


Figure 6. Response optimizer.

### 3.2.4 The regression equation

The equation was used to guess the values of the temperature and pressure that gives the optimum shape memory effect (100%).

The following result was reached:

$$\text{Shape memory effect (\%)} = 130 + 0.0736 \text{ Pressure (MPa)} - 0.117 \text{ Temperature (°C)} \quad (2)$$

For compacting pressure=865MPa, sintering temperature=800°C.

$$\text{Shape effect (\%)} = 130 + 0.0736 \times (865) - 0.117 \times (800^\circ\text{C}).$$

Shape effect =100%

This sample was manufactured by powder metallurgy technique mentioned above and the shape memory effect test was conducted and it was found that the shape recovery is actually 100%.

## 4. Conclusion

1. Shape memory effect test results show that the shape recovery increase with high compacting pressure and low sintering temperature since the porosity is less and hence the best shape recovery was (88%) at the highest compacting pressure (800MPa) and at (850°C) sintering temperature.
2. 100% shape memory effect was obtained at 865MPa compacting pressure and 800°C sintering temperature by optimization of sample using Minitab program.

**Nomenclature**

$A_S$	Austenite start temperature	SMA	Shape memory alloys
$A_F$	Austenite finish temperature	SME	Shape memory effect
PM	Powder metallurgy	XRD	X-ray diffraction
SMA	Shape memory alloy		

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