The Effect of Laser Energy on the Rate of Corrosion for Ni46-Ti50-Cu4 Shape Memory Alloy

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Abstract. The microstructure, mechanical and corrosion properties of Nickel-Titanium-Copper shape memory alloy were inspected in this research. The Ni46-Ti50-Cu4alloy was prepared based on the powder metallurgy method, then mixed and cold-pressed at (600, 700, and 800) MPa, respectively, to form cylindrical samples of 11mm diameter × 16.5mm length. After pressing, the samples got sufficient green strength for handling. The specimens were consequently sintered at (850, 900 and 950) °C for five hours in an electric vacuum tube furnace. The optimum compacting pressure and sintering temperature were identified using the Minitab program. The effect of optimum parameters on the shape memory was 83.5% at a temperature of sintering of 850°C and a pressure of compacting 800 MPa. Samples prepared under the optimum process conditions were then irradiated utilizing a pulsed ND: YAG laser at 300mJ, 400mJ, 500mJ, and 600mJ. Multiple tests were conducted on the alloys, including shape memory effect, porosity measurements, scanning electron microscopy (SEM), and corrosion rate test. The results indicate that decrease the corrosion rate after laser processing. The lowest value of the rate of corrosion was obtained when the energy of laser irradiation was 500 mJ, which corresponds to the structural and physical properties.

Keywords: ND, YAG laser, Corrosion, SEM, Powder metallurgy, Shape memory alloy.

1. Introduction
Shape memory alloys (SMAs) comprise one of the most influential groups of smart materials [1]. Two phases (austenite and martensite) are observed in SMAs. The austenite phase, which exhibits a crystalline cubic structure, exists at a high temperature. By contrast, the martensite phase, which exhibits a crystalline tetragonal or monoclinic structure, exists at a low temperature. The change from the austenite phase to the martensite phase may lead to twinned or detwinned martensite [2]. Ms and Mf are variables used to represent the martensite start temperatures and forming of finish, whereas As and Af represent those for austenite. If the values of the four variables are increased, then the loading amount is located on a portion of the SMA [3]. SMAs have extensively been used for various technological applications because of their good properties, such as superelasticity and shape effect (SE) [4].

The shape effect of memory refers to the recovery of the “memorized” shape after deformation [5]. SMAs have different types, such as NiTi, CuZnAl, CuAlNi alloys, and other metallic alloy systems [2]. Nitinol is a well-known SMA. At present, SMAs are based on Ni, and Ti, which exhibit the best arrangement of material properties for most traditional applications given their excellent properties.
The abbreviation “Nitinol” was coined by [3]. SMAs are exposed to several surface treatment techniques to improve their resistance to corrosion and increase their surfaces. These techniques contain electrochemical and mechanical processing, such as heat treatments, chemical etching, plasma ion immersion implantation, conventional and electron irradiation of beam [6]. Modification methods by laser surface are the most generally used engineering tools of the surface because they are melting that subsequently extend the solution of solid and enable rapid heating, microstructures refining, and alloy composition of homogenizing to yield products with excellent metallurgical interfaces [7]. The processing of a laser surface is a viable means to change the properties of the surface of materials [8] due to its high precision, flexibility, and the intense beam power [9]. The radiation of electromagnetic of a laser beam is absorbed within the layer of the surface of metals. Thus, laser energy can be deposited precisely at the point where it is required. The bulk properties of NiTi alloy are unaffected by laser surface treatment [10]. This research aims to obtain the best shape memory effect for Ti-Ni-Cu alloys, improve the corrosion resistance of the alloy by using different laser energies, and study its effect on the corrosion rate of the Ti-Ni-Cu alloy and the structural and surface properties of this alloy. Then select the best energy that can reduce the rate of wear.

2. Experimental method

The experimental work includes manufacturing the required testing samples [11-17] and then testing them to calculate the behavior required [18-24]. The samples must be manufactured according to the ASTM standard [25-30]. Then, depending on the experimental results, the effect of various parameters can be calculated [31-37]. Samples were obtained based on the powder metallurgy method. The powder mixture includes 46% Ni, 50%Ti, and 4%Cu, which were set to a pipe glass holder. The powders are mixed at a rotational speed of 70 rpm for two hours using a parallel electric mixer, depending on reference [38]. The mixture of powder was subsequently compressed by a pressing machine at (600,700 and 800) MPa inside steel mold to get cylindrical specimens of 11mm diameter × 16.5mm length. After compaction, the green specimens have got sufficient strength to be handled. A vacuum pipe electric furnace sintered the green specimens at 850,900 and 950 °C for five hours. SME test was conducted by compressing the (16.5 mm length × 11 mm) specimens by 0.06% of the main length and then utilizing temperature at 120°C for 10 minutes. Lastly, slow cooling in the oven was performed, and then the effect of the shape memory was calculated by applying the formula below [39].

\[
\text{SME} = \left( \frac{L_2 - L_1}{L_0 - L_1} \right) \times 100
\]  

(1)

Where; L0: The normal specimen length, L1: Length of specimens after compression at 0.06% and L2: Length of specimens after applying the temperature at 120°C for 10 minutes. Based on the ASTM C373-38 [40], the investigation of porosity was performed using the rule of Archimedes by utilizing digital balance. The specimen was weighted in the air at that point and then weighted again in a bushel hanging from a holder filled with purified water. Afterward, the samples were soaked in purified water for 24 hours and weighed immediately after drying with fabric to compute porosity and bulk density, as indicated by Equations (2) and (3), respectively [41].

\[
\text{Apparent porosity} = \left( \frac{w_1 - w_2}{w_1 - w_3} \right) \times 100
\]  

(2)

\[
\text{Bulk density} = \left( \frac{w_2}{w_1 - w_3} \right) \times \text{pd}
\]  

(3)

Where; W2: The dried specimen weight, W1: The specimen weight in the basket, W3: The specimen weight after 24 hours soaking in water and pd : Density of water (1 g/ cm³).

The shape memory effect test was shown to confirm the value of the obtained result by applying the conditions (i.e., sintering temperature and compacting pressure) are determined from the Minitab software that can provide the optimum shape memory effect for specimens prepared using powder metallurgy technology. The ideal specimens were exposed to the laser and testing it. The heating of the surface by laser, i.e., quick hardening process, is a laser modification of surface technology to improve the resistance of corrosion. In the current work, the laser used is a Q-switched Nd: YAG
energies of a laser of 300, 400, 500, and 600 mJ and laser with 1064 nm. The laser duration is 300 ms, and the spot size is 11 mm. In this research, the distance between the specimen and the laser tool is 100 mm laser irradiance multi-pulse for treated alloys. The laser type Nd: YAG is highly popular due to its essential features; it provides various output energies and is consistent. The temperature of the surface of the specimen should not be higher than 300 °C to avoid thermal damage [6]. Thus, to find the amount of laser energy that extend the best effect on the surface without getting thermal damage, Equation (4) was used [42].

\[
T(z, t) = \frac{2H}{K} \left[ \sqrt{kT} \text{erfc} \left( \frac{Z}{2\sqrt{kT}} \right) \right]
\]

(4)

\[
\text{erfc} (0) = \frac{1}{\sqrt{\pi}}
\]

(5)

Where; H: Power density of surface, k: Diffusivity of Thermal (\( \frac{K}{\rho \cdot C} \) m²/s), K: NiTi alloys thermal conductivity, C: Specific heat capacity (322 J/kg for NiTi alloy) and ρ: Density of materials (6.5 g/cm³ for alloys of NiTi).

Microscopy by scanning electron was utilized to get the microstructure of the specimen. Corrosion investigation was achieved to measure the rate of corrosion for the specimens through open circuit potential (OCP), duration estimation. The extrapolation of Tafel was used to determine the operation variables (Icorr, Ecarr). The resistance of corrosion of the specimens was considered using the solution of the hank. The expected electrochemical cell of the utilization of cathodes, the first anode is the electrode of working (WE). The second anode is a bar of platinum utilized as an auxiliary electrode (AE) to load the circuit current. The third electrode is named as a reference electrode, and it is used for measurement of the voltage between the working electrode and the electrolyte. The temperature cell was set at 37 °C, which is the human body temperature. Therefore, the calculated experimental results can be compared with the analytical results [43-49], numerical results, [50-55], or other publication results, [56-61], to give the discrepancy for the calculated results [62-67]. Also, it can be depending on the experimental results without comparison with other results [68-81].

3. Results and discussion

3.1. Shape effect test
The percent of the effect of shape memory was shown in Figure 1. It was measured at sintering temperature and pressure of compacting. The results in Figure 1 were used to obtain the optimum compacting pressure and sintering temperature. The results revealed that the effect of shape was improved as the pressure of compacting increased at low temperatures of sintering. The optimum of effect shape memory was 83.5 % at a temperature of sintering of 850 °C and a pressure of compacting 800 MPa

3.2. Density and measurement of porosity
The density and porosity of the optimum specimens were measurement according to Equations (2) and (3). Weight of sample (g): Wd = 5.901, Wn =4.682, Ws =5.931 , Density (g/cm³) = 5.202 and Porosity (%) = 2.4.

3.3. SEM test results
This test offered more details about the microstructure of alloy, as shown in Figure 2. The specimens with the grain boundary formation and the pores were apparent before laser irradiation is seen in Figure 3.

The scanning electron microscope figure of the specimens microstructures after irradiation with various laser energies are seen in Figures 4a, b, c, d, which show decreased porosity after irradiation of laser. The irradiated region was homogeneous and uniform. These properties have increased hardness. Subsequently, comparing the various images of SEM based on the laser energy, the energy of laser at 500 mJ was determined to get the optimum microstructural features.
3.4. Chemical tests (Corrosion Test)
The time was measured of the open circuit potential on all the optimum specimens after and before laser exposition with various laser energies. The results provide significant data about the polarization of initial curves. The inspection of polarization has used a solution of hanks at 37 °C. The results showed that the rate of corrosion was decreased with increasing the laser energy on account of the decrease in the sizes of the pore. The external area was reduced due to the solution of hank and found a decrease in the rate of corrosion measurement. The curve of electrochemical of the Ti-Ni-Cu specimen as shown in Figure 5, before laser irradiation. The anodic and cathodic reactions are also shown in this figure. The rate of corrosion of the specimen was 44.8 mpy. After the samples irradiating with changed energies of laser s (300, 400, 500, and 600 mJ), as shown in Figures 6 to 9, the corrosion rate was decreased to 18 mpy at 300 mJ, 25.4 mpy at 400 mJ, 8.3 mpy at 500 mJ, and 38.4 mpy at 600 mJ. These outcomes show which the potential of corrosion was decreased after exposure of laser. The lowest value of the corrosion rate was obtained after the laser irradiation energy was 500 mJ, which corresponds to the structural and physical properties. All calculations were shown in Table (1), and Figure (10) shows the relation between corrosion rate and laser energy.

![Figure 1. Shape memory effect test result.](image1)

![Figure 2. SEM and Eds device.](image2)

![Figure 3. Scanning electron microscope of the samples as received.](image3)
Figure 4. Scanning Electron Microscope of samples after irradiation with different laser energies.

Figure 5. Current density (Amp/Cm$^2$) vs. Potential (V) for as received sample.

Figure 6. Current density (Amp/Cm$^2$) vs. Potential (V) for sample after irradiated with laser energy 300mJ.

Figure 7. Current density (Amp/Cm$^2$) vs. Potential (V) for sample after irradiated with laser energy 400mJ.

Figure 8. Current density (Amp/Cm$^2$) vs. Potential (V) for sample after irradiated with laser energy 500mJ.
Figure 9. Current density (Amp/Cm$^2$) vs. Potential (V) for sample after irradiated with laser energy 600mJ.

Figure 10. Relation between corrosion rate and laser energy.

Table 1. Corrosion test results

| Samples         | Beta A (V/decade) | Beta C (V/decade) | Icorr (A/cm$^2$) | Ecorr (mV) | CR (mpy) |
|-----------------|-------------------|-------------------|------------------|------------|----------|
| Before laser    | 8.4               | 6.3               | 1.210 $\mu$A     | 205.0      | 44.7     |
| After irradiated with different laser energies (mJ) |                   |                   |                 |            |          |
| 300             | 8.9               | 6.6               | 489.0 $\mu$A     | 266.0      | 18.0     |
| 400             | 9.5               | 9.2               | 689.0 $\mu$A     | 265.0      | 25.4     |
| 500             | 5.4               | 4.04              | 225.0 $\mu$A     | 266.0      | 8.3      |
| 600             | 2.9               | 3.2               | 1.040 $\mu$A     | 214.0      | 38.4     |

4. Conclusions
1. The optimum shape memory effect is 83.5% under the temperature of sintering of 850 °C and the compacting pressure of 800 MPa.
2. The scanning electron microscope test (SEM) revealed that the martensite layers formation was appreciated obviously. Fracture toughness results can be divided into two groups, the first one showed a slight increase in the fracture toughness above the fracture toughness of PMMA resin, but the second group showed a slightly decreasing in the fracture toughness below the PMMA resin.
3. The corrosion rate results revealed that the corrosion rate was varied after irradiated the samples with different laser energy and the higher value with laser energy 500 mJ, on account of the decrease of pores size, which reduces the external area exposed to the hank solution and this directed to decrease the measured corrosion rate.

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