



Effect of (Cu-Ni) Concentrations Ratio on Recoverable Strain and the Shape Memory Effect of Ternary Smart (Cu-Al-Ni) Alloys

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Abstract: -

The achieved results by this research determined the best ratio of (Cu-Ni) in the smart (Cu-Al-Ni) alloys that it achieve the best values of maximum recoverable strain and shape memory effect. These mechanical properties possess great technological and commercial importance in many smart applications such as aerospace and industrial applications. Three samples have different weight percentage S1(82.8%Cu, 13.5%Al, 3.7%Ni), S2(81.8%Cu, 13.5%Al, 4.7%Ni) and S3(82.4%Cu, 13.5%Al, 4.1%Ni) were manufactured from smart (Cu-Al-Ni) alloy using powder metallurgy method with heat treatment. The physical tests were performed using optical microscope, scanning electron microscope (SEM) and X-ray diffraction (XRD) to make sure the presence of the austenite phase before heat treatment and the martensite after heat treatment. The results showed, the presence of these two phases in all samples under study. Mechanical tests were carried out which they included shape memory effect (SME) and maximum recoverable strain. The results showed that the S3 (82.4%Cu, 13.5%Al, 4.1%Ni) sample gave the best values of SME%, the maximum recoverable strain is 5% at full shape recovery (i.e. SME =100%), the maximum recoverable strain is 10% at partially shape recovery.

Keywords: Powder metallurgy, recoverable strain (R.St.), shape memory effect (SME), smart (Cu-Al-Ni) alloys.

1. Introduction

The smart alloys have the capability to recover their premier shape when they stimulated by an electrical current, thermo-mechanical, magnetic effects or any outer affect leads to raise their temperature above

the transformation temperature, even if there are many plastic deformities [5]. These special characteristics of the smart alloys led to give them a great commercial and technological importance and use them in many diverse applications such as biomedical [9], aerospace [2] &

industrial applications [12]. The smart alloys can be classified into many kinds, the NiTi alloy possesses the greatest number of applications, because of the limitations of this alloy such as the high expensive, it requires non-conventional machining and low transformation temperature (less than 100°C) therefore the best replacement particularly at the high transformation temperature is Cu-Al-Ni smart alloy since, it has lower cost, the easier manufacturing higher transformation temperature (-200°C to 200°C) and the maximum recoverable strain is 4% however it has some disadvantages such as the brittleness and the low corrosion resistance to treat them, they used powder metallurgy technique in which changing the percentage of the alloying elements or addition new elements or changing the conditions of heat treatment [4 & 6]. The phenomenon of thermoelastic martensitic transformation is the main responsible for the unique characteristics of smart alloys. Smart alloy possesses two major phases, low temperature phase and high temperature phase named as the martensite and the austenite. The start and finish temperatures of the martensite and austenite transformation are M_s , M_f , A_s , A_f . The shape memory effect is the attractive phenomenon in smart alloys in which the austenite phase cooled under M_f temperature that

lead to produce twinned martensite (zig zag form), its transform to detwinned martensite (orthorhombic) when the stress has been applied, but when the alloy is heated to a temperature upward A_f after removing the stress, as a result that, the alloy returns to its original form as shown in Fig.1, [8].

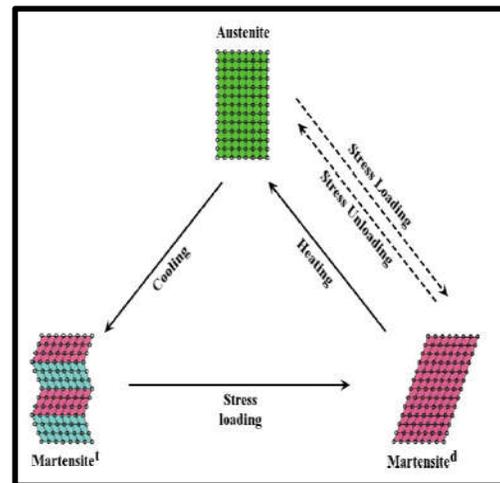


Fig.1 The SME phenomenon in smart alloys [8]

To study the Cu-Al-Ni smart alloys prefers selecting the ratios of Al between (13 to 14%) and Ni (1 to 6%) because they lead to obtain a single $\beta_3 \Rightarrow \beta'_3$ martensite transformation, it causes particularly for high temperature applications, more thermoelastic & thermally more stable, but when the ratio of Al increases above (14%) lead to obtain single brittle phase γ'_3 it causes high brittleness of the smart alloy and its failure to restore its former shape as shown in Fig. 2, [7]. The purpose of this research was to study the effect

of (Cu-Ni) ratio on the shape memory effect (strain recovery) and recoverable strain of ternary (Cu-Al-Ni) smart alloys, chosen ratios of the alloying elements depending on the comparison with the application of the new material CN-250X Nimesis alloy (single crystal wire) which it has (Cu 82.4wt%, Al 13.5wt%, Ni 4.1wt%) as shown in **Fig. 2**, recoverable strain (8 to 10%) for transformation temperature below 200°C was a better recoverable strain (6 to 7%) & transformation temperature (lower than 100°C) than NiTi [3].

2. Experimental work

2.1. Pure powders

The used pure powders in this research were copper, aluminum and nickel possess the purity of 99.9 %, the mean particle sizes 45 micrometer (-325 mesh) and other specifications were shown in **Table.1** [10], they imported and was verified from the chemical composition by emission spectrometer device after compacting them by the uniaxial press machine with die and punches to manufacture sample was (14mm diameter & 5 mm thickness).

Table.1 Specification of elements [10].

Specifications	Cu	Al	Ni
Density g/cm ³	8.94	2.685	8.902
Melting temp. °C	1082	660	1455

2.2. Sample Preparation

Three different weight percentages S1(82.8%Cu, 13.5%Al, 3.7%Ni), S2(81.8%Cu, 13.5%Al, 4.7%Ni) and S3(82.4%Cu, 13.5%Al, 4.1%Ni) samples were manufactured with dimensions (16.5 mm length & 11mm diameter) from the smart (Cu-Al-Ni) alloy by using powder metallurgy technique. The manufacturing process was carried out according to the stages:

2.2.1. Mixing by 4-digits sensitive balance and rotating drum mixing device (shown in **Fig.3**) at speed 72 rpm for 6 hrs. The mixture is placed in the glass container at 40% of its volume to achieve the best mixing performance [1], adding 1% acetone to prevent segregation between particles (because of the difference in the densities of the alloying elements) and for lubrication.

2.2.2. Compacting by a computerized uniaxial press machine (capacity of 100 KN) with floating die and punches (shown in **Fig.4**) at 650 Mpa, 4 minutes holding time (to prevent spring back) and 0.5 mm/min displacement rate to produce nine pieces of (16.5mm length * 11mm diameter) green compacts.

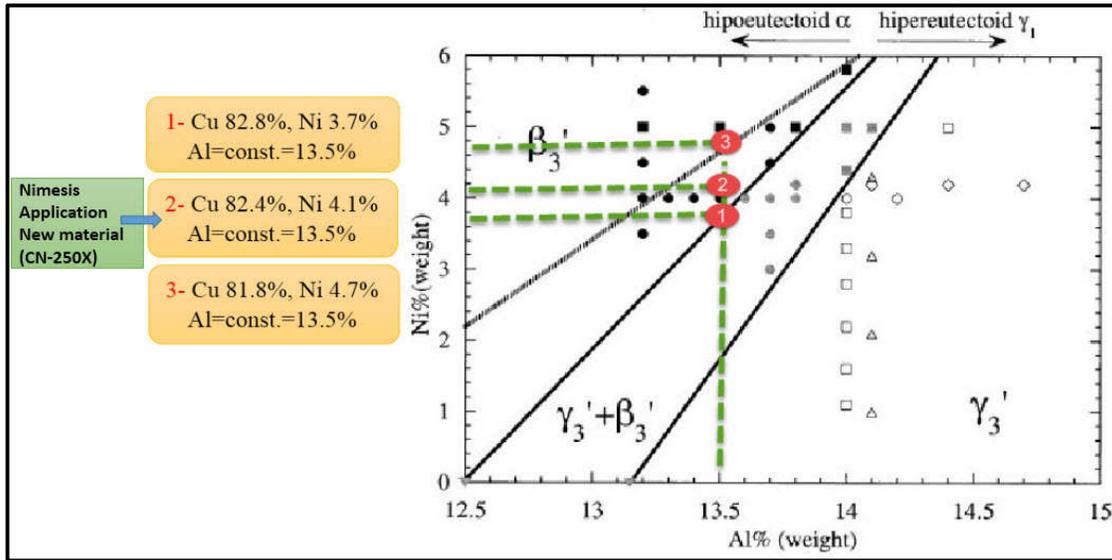


Fig. 2 Three chosen Concentrations ratios of (Cu-Ni) for (Cu-Al-Ni) smart alloys were shown on diagram of the type of thermally induced martensite vs Al & Ni ratios [7]

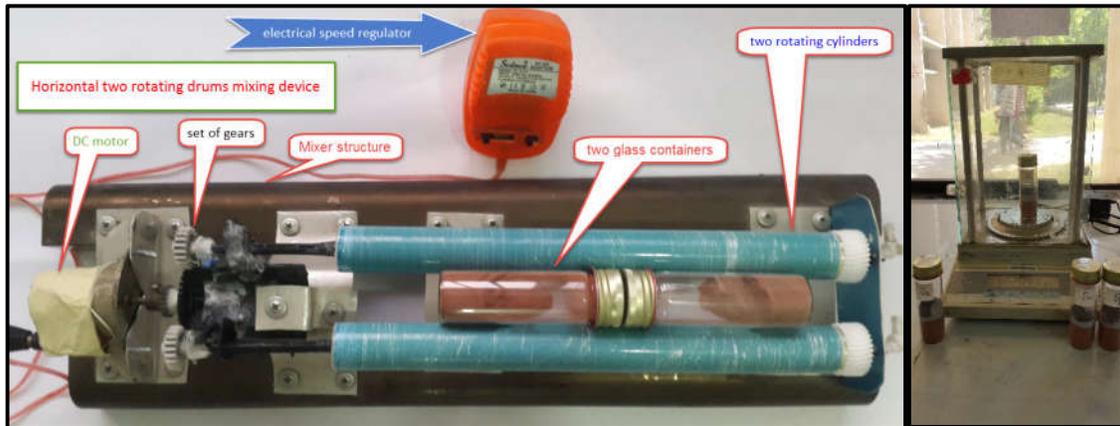


Fig. 3 Rotating drum mixing device & 4-digits sensitive balance

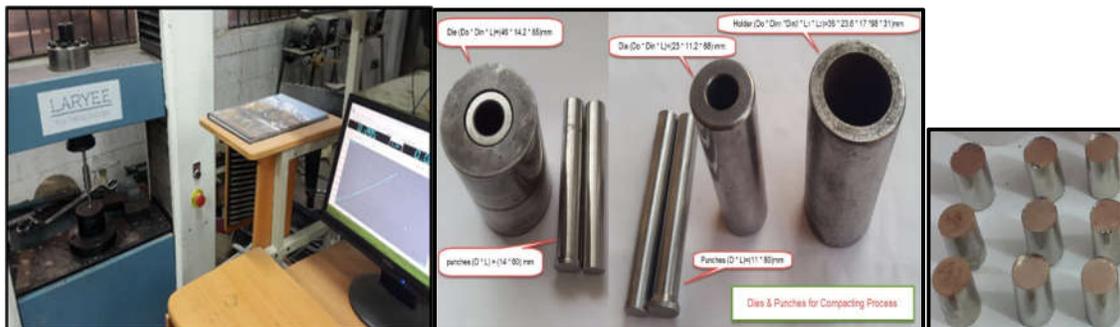


Fig.4 Computerized uniaxial press machine, Dies & punches & green compacts

2.2.3. Sintering by electrical furnace with a vacuum system to prevent the oxidation of the sample & disposal of harmful gases (shown in **Fig.5**). This process included two stages, it started at 500°C for one hour, then, it

continued until 850°C for five hours, then let go it to cool in the furnace for the purpose of bonding particles of the sample in the solid state as shown in **Fig.6** [1]



Fig.5 Electrical furnace with a vacuum system & sintered samples

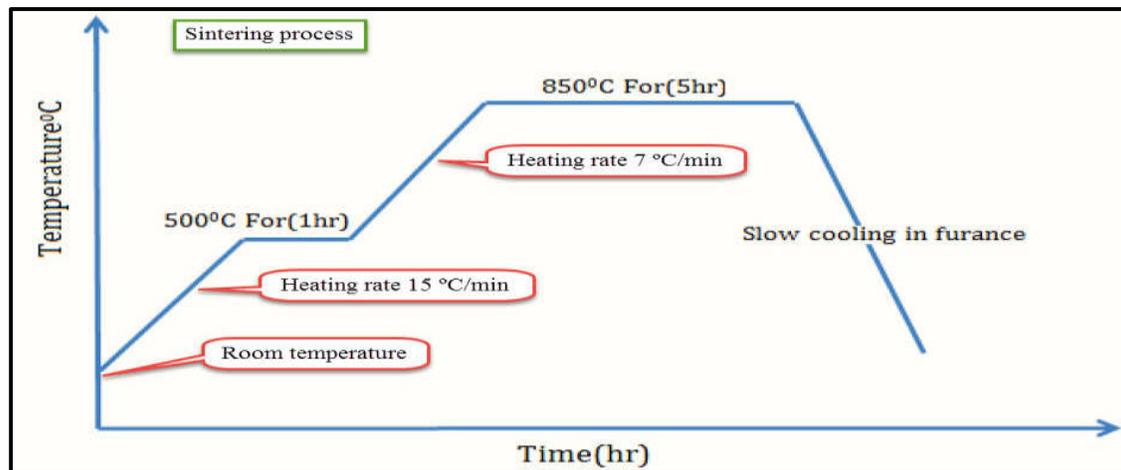


Fig.6 Temperature- time diagram of sintering process steps

2.2.4. Heat treatment using the same vacuum furnace system was used in sintering process. It included two stages: the first, quenching process in which the sample heated to 800°C, hold it for one hour, then cooled it

speedily in ice water to obtain martensitic phase, the second, aging process in which the sample heated to 100°C, hold it for two hours, then cooled it in the air to guarantee the

martensitic stabilization, as shown in Fig.7.

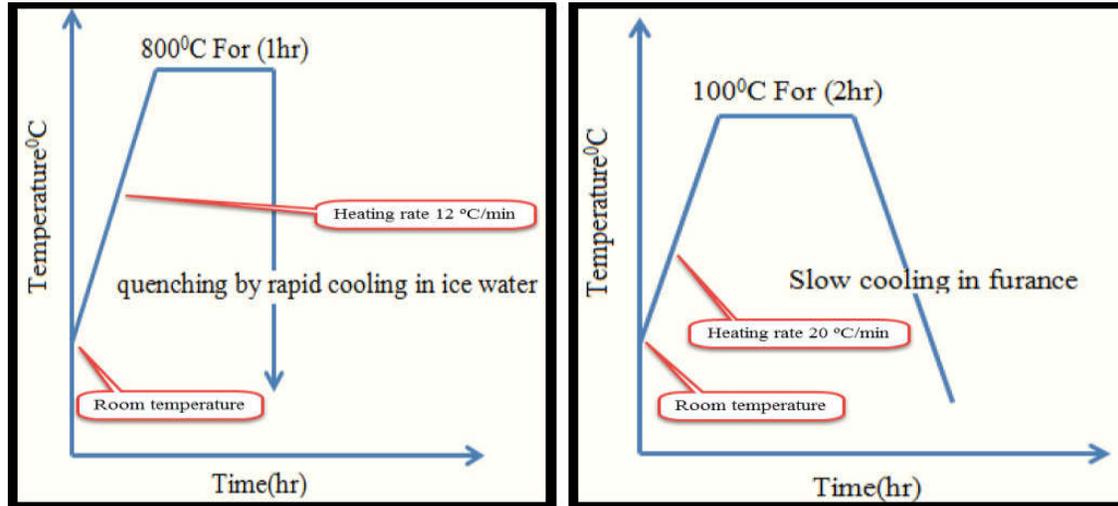


Fig.7 Temperature- time diagram of (a) Quenching process & (b) Aging process

2.2.5. Grinding, polishing and etching implemented on all samples to prepare them for physical and mechanical tests (shown in Fig. 8). The grinding was carried out by using a grinding device with two types of grinding paper and the water, the first, it was the waterproof aluminum oxide paper (400, 600, 800, 1200), the second, it was the waterproof silicon carbide paper (2000,3000) to

remove the scratches and adjust straightness of the samples faces. The polishing was carried out by using polishing device with cloth paper and diamond paste to remove very fine surface scratches. The etching was carried out by using the chemical etching solution consist of the mixing (2 mg Fe₃Cl, 5 ml HCl and 98 ml Ethanol).

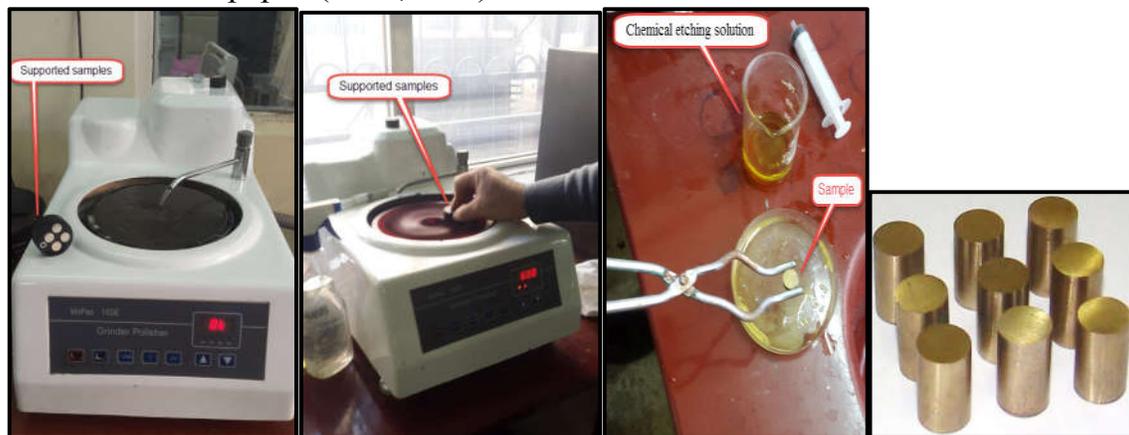


Fig.8 Grinding, polishing, etching & finished samples.

2.3. Testing

2.3.1 Physical Testing

The optical microscopic, X-ray Diffraction (XRD) & Scanning Electron Microscope (SEM) devices were used to determine the generated phases for each weight percentage of samples in order to make sure the austenite phase (Al₄Cu₉) present in the sintered samples, and the martensite phase (AlCu₃) present in the quenched samples, because the main responsible of the SME phenomenon is the martensite phase.

2.3.2. Mechanical Testing

Recoverable strain (R.St.) and shape memory effect (SME) tests using the same vacuum furnace system previously used in the sintering process and press machine previously used in the compacting process. Initially, the sample was compacted by 1% from the original length, raised the temperature of it to 250 ° C, held it for a five minutes and left to cool in the air, then this procedure repeated several times by increasing recoverable strain at 1% for each step until the sample failed in restore the former shape or cracking it. The strain recovery ratio (SME%) was calculated by the equation (1), [11].

$$(SME\%) = \frac{L_2 - L_1}{L_0 - L_1} * 100\% \quad \text{--- (1)}$$

Where: L_0 and L_1 were the lengths before and after compacting, L_2 was the length after strain recovery (as a result of heating the compacted sample).

3. Results and Discussion

3.1. Optical Microscopic and (SEM) Tests Results

The testing results of optical microscopic and (SEM) devices for three samples of smart (Cu-Al-Ni) alloys that have different (copper-nickel) ratios are shown in two sets of images, the first set displayed austenite layers before heat treatment, and the second set displayed martensite layers (zig-zag or V-shape) after heat treatment as shown in **Fig. 9** and **Fig. 10**.

3.2. XRD Test Results

Fig. 11 shows the XRD test results of (S1, S2 & S3) samples, this results were demonstrated the presence of the austenite phase (Al₄Cu₉) in the sintered samples and the martensite phase (AlCu₃) in the quenched samples.

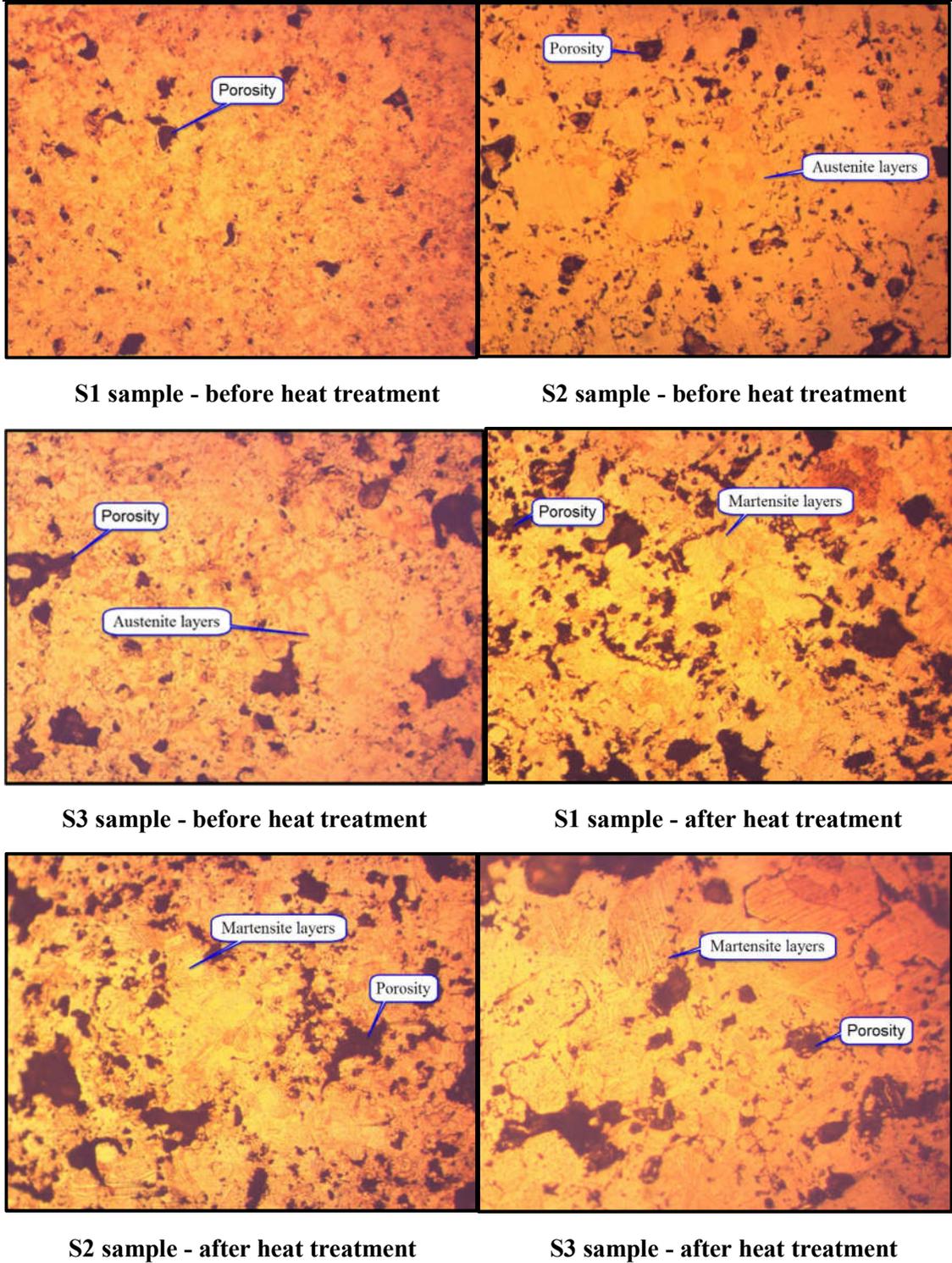


Fig. 9 Optical microscopic test images (magnification power 40X)

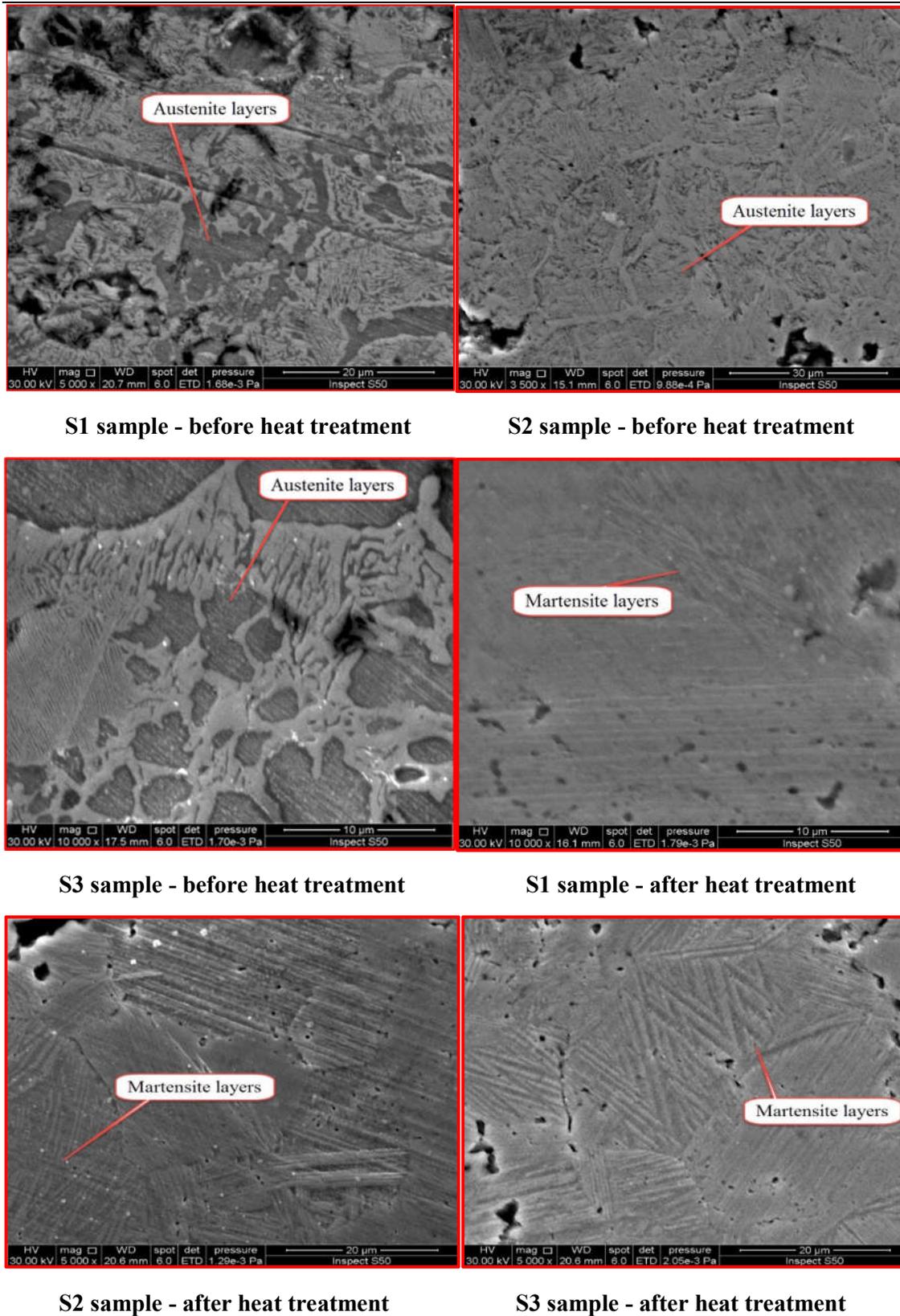
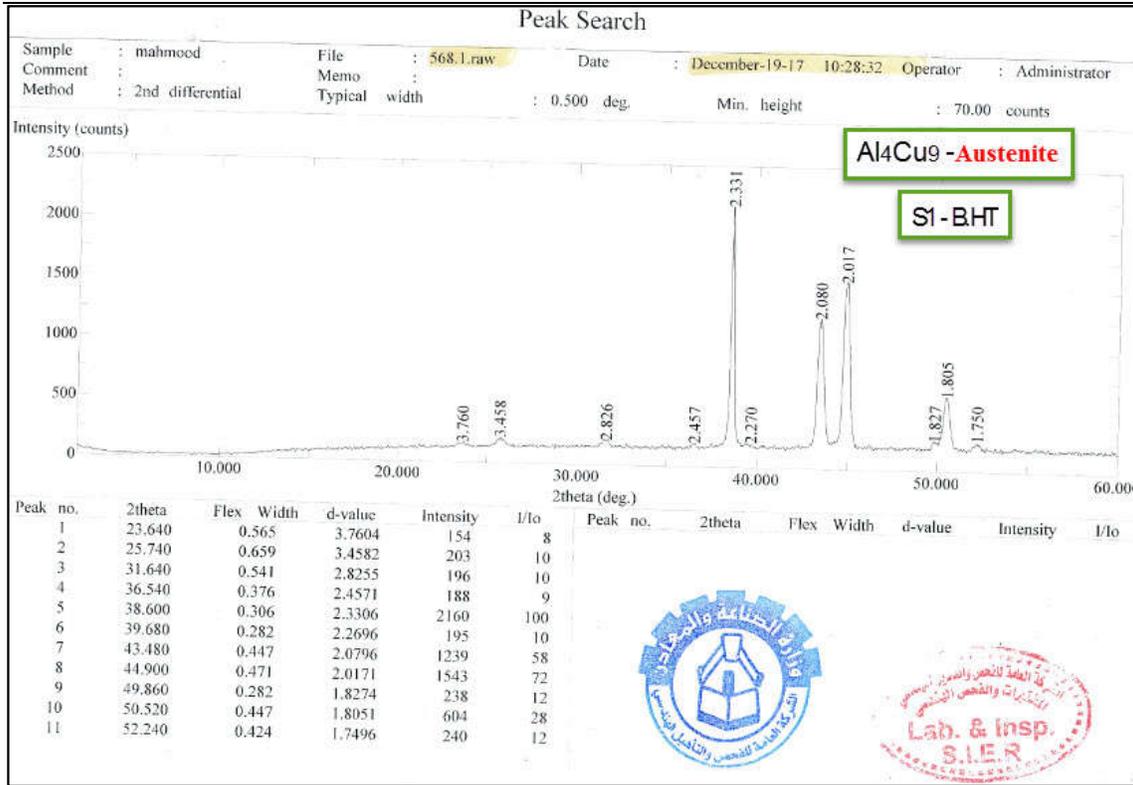
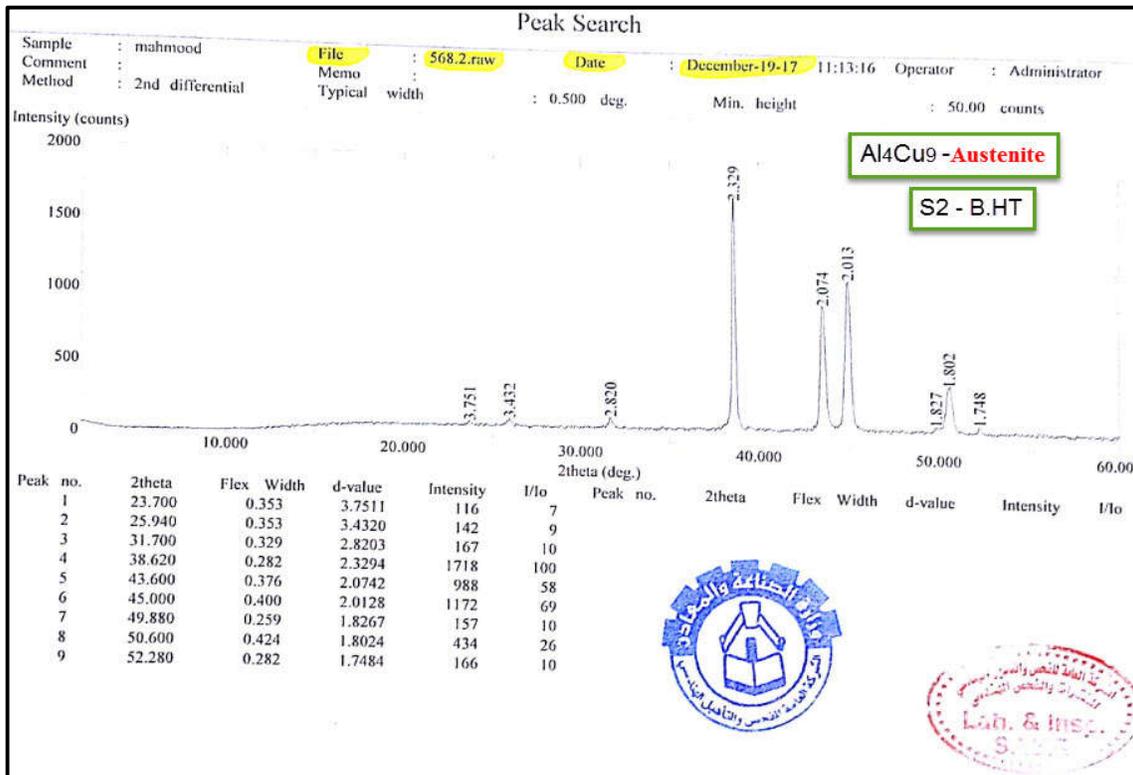


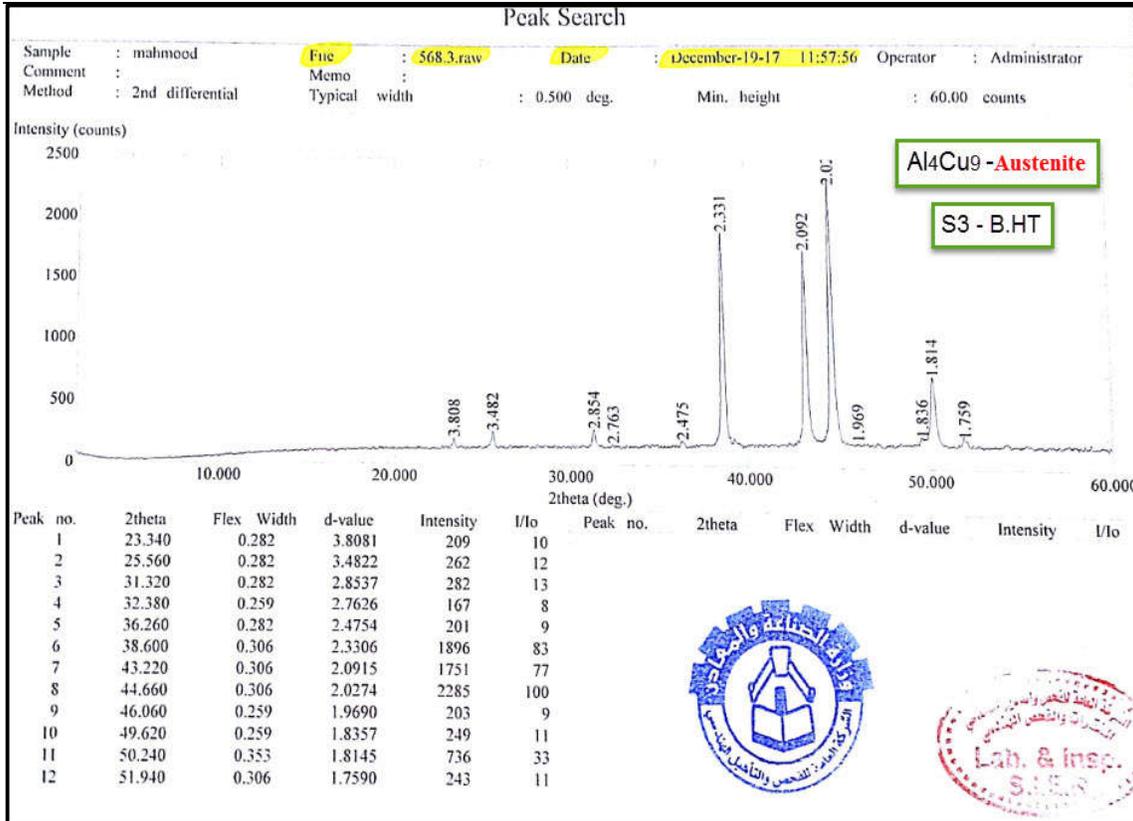
Fig. 10 Scanning Electron Microscope (SEM) test images (magnification 3500X-10000X)



S1 sample – XRD test, Austenite phase (Al4Cu9) before heat treatment

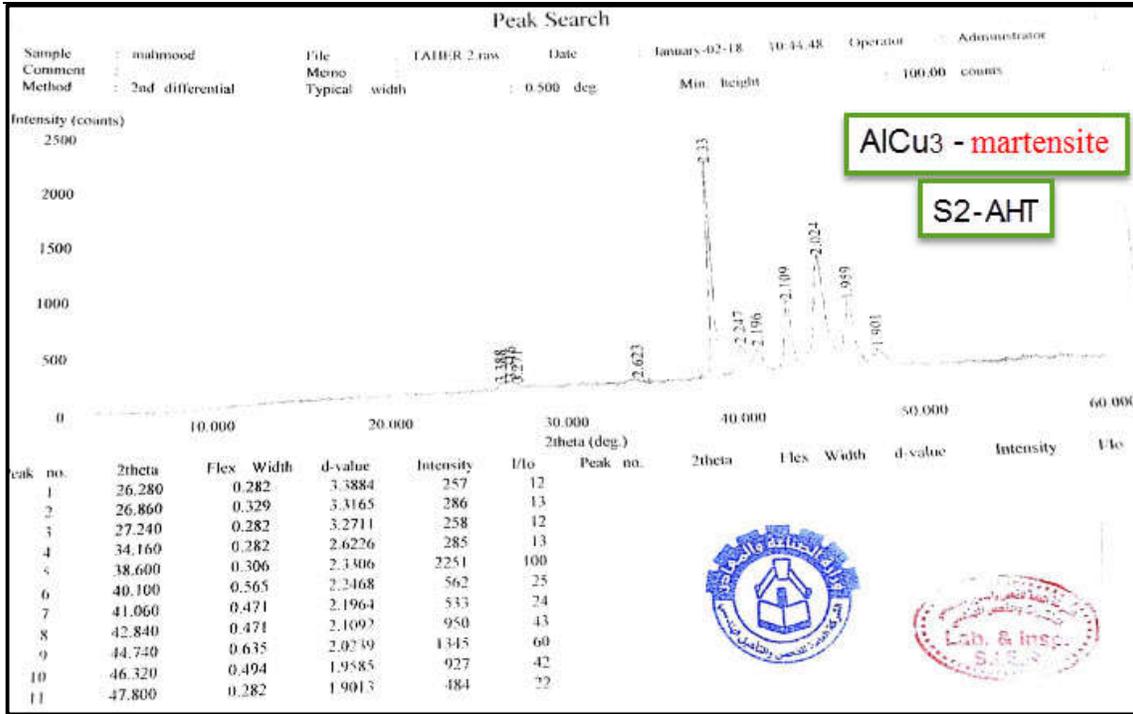


S2 sample – XRD test, Austenite phase (Al4Cu9) before heat treatment

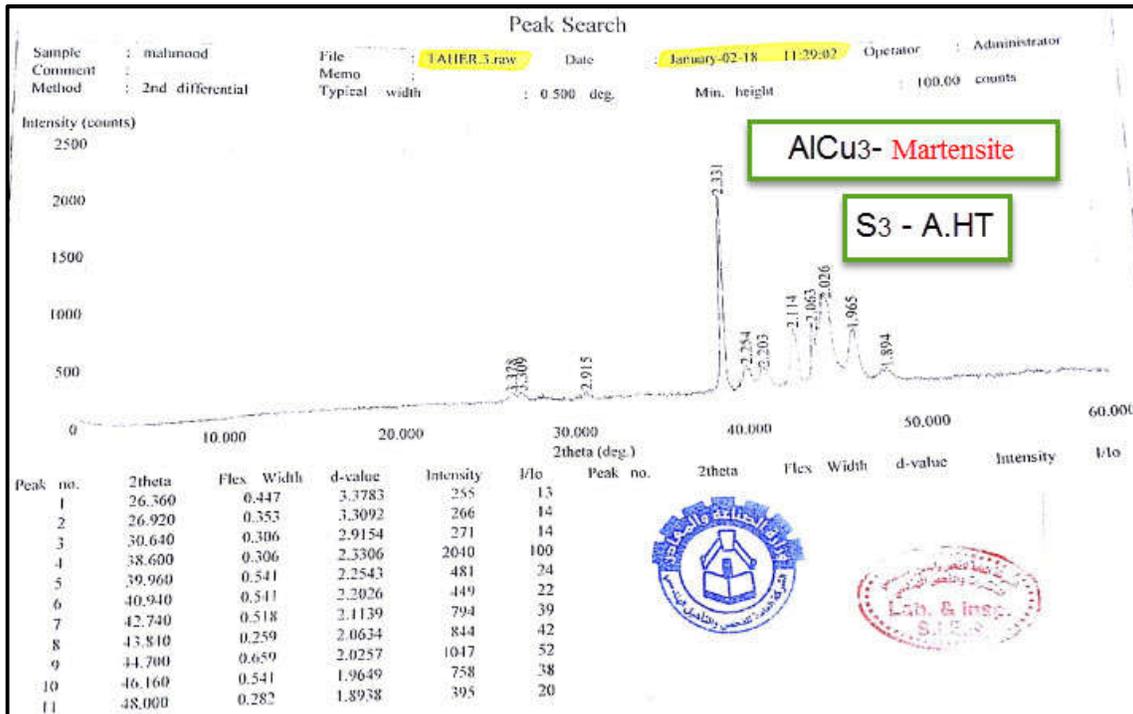


S3 sample – XRD test, Austenite phase (Al4Cu9) before heat treatment

S1 sample – XRD test, Martensite phase (AlCu3) after heat treatment



S2 sample – XRD test, Martensite phase (A1Cu3) after heat treatment



S3 sample – XRD test, Martensite phase (A1Cu3) after heat treatment

Fig. 11 X-ray Diffraction (XRD) test images

3.3. Recoverable strain and Shape Memory Effect Testing Results

The results of this testing were shown in the Table 2.

Table 2. Recoverable strain and shape Memory effect testing results

(S ₁) Sample – [82.8%Cu, 13.5%Al, 3.7%Ni]				
R.St.% (mm)	L ₀ (mm)	L ₁ (mm)	L ₂ (mm)	SME% (mm)
1 % L ₀	16.575	16.570	16.575	100
2 % L ₀	16.575	16.540	16.575	100
3 % L ₀	16.575	16.495	16.575	100
4 % L ₀	16.575	16.440	16.560	88.889
5 % L ₀	16.560	16.355	16.490	65.854
6 % L ₀	16.490	15.960	16.260	56.604
7 % L ₀	16.260	15.840	16.050	50
8 % L ₀	16.050	15.630	15.830	47.62
9 % L ₀	15.830	15.230	15.480	41.667
10 % L ₀	15.480	14.920	15.050	23.214
11 % L ₀	15.050	14.430	14.510	12.903
12 % L ₀	14.510	Failure	Failure	Failure

(S ₂) Sample – [81.8%Cu, 13.5%Al, 4.7%Ni]				
R.St. % (mm)	L ₀ (mm)	L ₁ (mm)	L ₂ (mm)	SME% (mm)
1 % L ₀	15.480	15.475	15.480	100

2 % L ₀	15.480	15.463	15.480	100
3 % L ₀	15.480	15.430	15.480	100
4 % L ₀	15.480	15.400	15.480	100
5 % L ₀	15.480	15.350	15.455	80.77
6 % L ₀	15.455	15.180	15.380	72.727
7 % L ₀	15.380	14.830	15.090	47.273
8 % L ₀	15.090	14.730	14.890	44.444
9 % L ₀	14.890	14.400	14.610	42.857
10%	14.610	14.120	14.290	34.7
11%	14.290	Failure	Failure	Failure

(S ₃) Sample – [82.4%Cu, 13.5%Al, 4.1%Ni]				
R.St. % (mm)	L ₀ (mm)	L ₁ (mm)	L ₂ (mm)	SME% (mm)
1 % L ₀	15.220	15.216	15.220	100
2 % L ₀	15.220	15.200	15.220	100
3 % L ₀	15.220	15.165	15.220	100
4 % L ₀	15.220	15.140	15.220	100
5 % L ₀	15.220	15.060	15.220	100
6 % L ₀	15.220	14.800	15.180	90.476
7 % L ₀	15.180	14.700	15.110	85.417
8 % L ₀	15.110	14.660	15.010	77.777
9 % L ₀	15.010	14.380	14.690	49.206
10 % L ₀	14.690	14.150	14.390	44.444
11 % L ₀	14.390	Failure	Failure	Failure

These results were represented in Fig. 12.

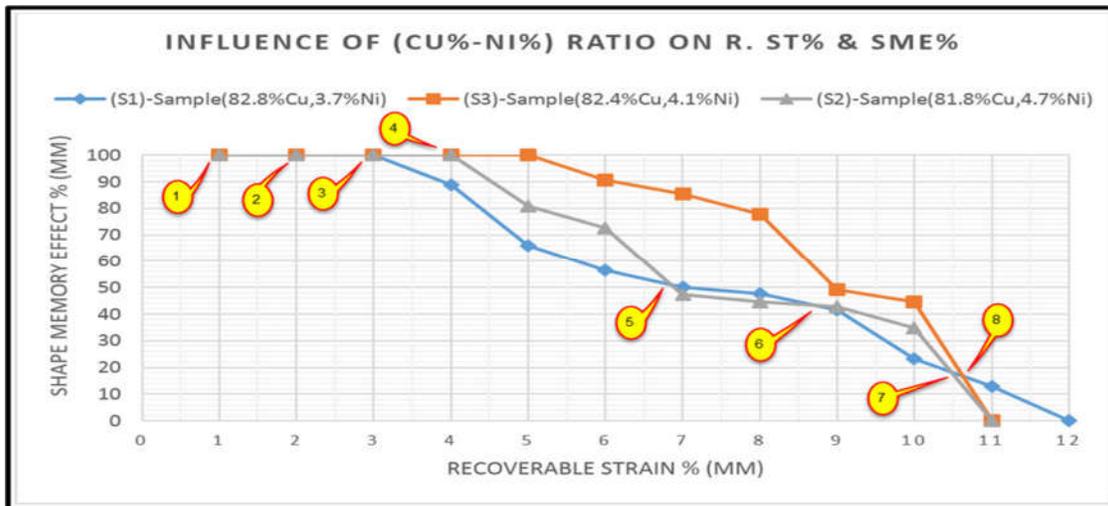


Fig. 12 Graph shows the effect of changing in (Cu-Ni) ratio with constant Al% on recoverable strain and shape memory effect in smart (Cu-Al-Ni) alloys

The (SME% – R.St.%) curves of the (S1, S2 & S3) samples in **Fig. 12** shows:

- 1- When increasing (R. St %) due to increasing the applied force, the (SME%) decreases for all samples.
- 2- The sample (S3) gave the (SME%) greater than the (SME%) of the samples (S₁ & S₂) when increasing the (R. St %).
- 3- The sample (S₃) gave the greatest (maximum recoverable strain = 5%) at full shape recovery, (maximum recoverable strain = 10%) at partially shape recovery
- 4- The intersection points (1,2,3,5,6 & 7) between the curves of S1 and S2 samples, the intersection points (1,2,3 & 4) between the S2 and S3 samples curves and the intersection points (1,2,3 & 8) between the S1 and S3 samples curves indicate that the samples behavior is similar at these intersection points.

In **Fig. 9** and **Fig. 10**, observed after heat treatment of S1, S2 and S3 samples appearance martensitic layers (V-shape) very weak in S1(82.8%Cu, 13.5%Al, 3.7%Ni) sample which it possesses least value of nickel concentration among three samples. In **Table 2** and **Fig. 12**, S1 sample gave the least value of (maximum recoverable strain = 3%) at full shape recovery and also gave the lowest values of (SME%) among the three samples. Because when

decreasing nickel concentration at constant aluminum concentration that lead to instability of single martensitic phase β_3 and decrease single martensitic layers β_3 (where the martensitic transformation changes from single ($\beta_3 \Rightarrow \beta'_3$) to mixed ($\beta_3 \Rightarrow \beta'_3 + \gamma'_3$) as shown in **Fig. 2**. The appearance of the brittle phase γ'_3 cause increasing the brittleness of the (Cu-Al-Ni) alloy and lead to decrease shape recovery characteristics (recoverable strain and shape memory effect). The major cause of the SME and other shape recovery properties is the stability of the β phase with a cubic structure. Where, it is believed that addition of nickel slows the diffusion of copper and aluminum.

4. Conclusions

From the results analysis for the samples testing (S₁, S₂ & S₃) we conclude

- 1- The values of (R.St.% & SME%) increased with increasing Ni% (from 3.7 to 4.1) and decreasing (Cu%). The (R.St.% & SME%) began to decrease after the Ni% increased to above 4.1%.
- 2- The sample S3 which it possesses the chemical composition (82.4%Cu, 13.5%Al, 4.1%Ni) was the best among the three samples because it achieved the greatest recoverable

strain ratio (5%) at full shape recovery (SME=100%).

3- The sample (S₁) gave the greatest (R.St.=11%) at partially shape recovery, but it not preferred because it gave the least value of (R. St.=3%) at full shape recovery and also gave the lowest values of (SME%) among the three samples when increasing the (R.St. %), because it has the least value of nickel concentration (3.7% Ni).

4- There is the greatest convergence of behavior between the S1 (82.8%Cu, 13.5%Al, 3.7%Ni) sample and S2 (81.8%Cu, 13.5%Al, 4.7%Ni) sample in the shape recovery property because the number of intersection points between their curves (6 points) are more than the intersection points (4 points) with other.

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تأثير نسبة تراكيز (نحاس-نيكل) على الانفعال القابل للاستعادة وتأثير ذاكرة الشكل لسبيكة (نحاس-المنيوم-نيكل) الثلاثية الذكوية

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جامعة بغداد

كلية الهندسة

قسم الهندسة الميكانيكية

الخلاصة:-

حددت النتائج المتحققة بواسطة هذا البحث أفضل نسبة (نحاس-نيكل) في سبائك (نحاس - المنيوم - نيكل) الذكوية التي تحقق أفضل القيم لأعظم انفعال قابل للاستعادة وتأثير ذاكرة الشكل. هذه الخواص الميكانيكية تملك أهمية تجارية وتكنولوجية كبيرة في الكثير من التطبيقات الذكوية مثل تطبيقات الفضاء والتطبيقات الصناعية. ثلاثة عينات لها نسب وزنية مختلفة S1 (82,8% نحاس, 13,5% المنيوم, 3,7% نيكل), S2 (81,8% نحاس, 13,5% المنيوم, 4,7% نيكل) و S3 (82,4% نحاس, 13,5% المنيوم, 4,1% نيكل) تم تصنيعها من سبيكة (نحاس-المنيوم-نيكل) الذكوية باستخدام طريقة ميتالورجيا المساحيق مع المعاملة الحرارية. تم إجراء الاختبارات الفيزيائية باستخدام المجهر الضوئي، المجهر الإلكتروني الماسح وحيود الأشعة السينية للتأكد من وجود طور الأوستينيت قبل المعاملة الحرارية و المارتنسايت بعد المعاملة الحرارية. النتائج بينت، وجود هذين الطورين في جميع العينات قيد الدراسة. تم إجراء الفحوصات الميكانيكية والتي تضمنت تأثير ذاكرة الشكل وأقصى أنفعال قابل للاستعادة. النتائج بينت أن العينة S3 (82,4% نحاس, 13,5% المنيوم, 4,1% نيكل) أعطت أفضل القيم لتأثير ذاكرة الشكل، أقصى أنفعال قابل للاستعادة (5% عند الاستعادة الكاملة) وأقصى أنفعال قابل للاستعادة (10% عند الاستعادة الجزئية).

الكلمات المفتاحية: ميتالورجيا المساحيق, الأنفعال القابل للاستعادة, تأثير ذاكرة الشكل, سبائك (نحاس-المنيوم-نيكل) الذكوية.