# Study of Compression Behaviour of a Cu-13Al-4Ni-xCr Shape Memory Alloys Prepared by powder metallurgy process

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## Abstract

In this study (Cu-13Al-4Ni-xCr) shape memory alloys have been prepared by using Powder metallurgy technique with added Cr element (x=0.3,0.6,0.9) . After compacted , sintering process has been done in vacuum pressure ($10^{-4}$torr) at ( 550°C for 120 minute) and ( 900°C for 60 minute) then cooling inside furnace.Quenching heat treatment carry out at (850°C for 60 minute) then cooling in cold water to (3-4°C) .

The microstructure has been studied by optical microscope and scanning electron microscope (SEM) , porosity , compression behavior(stress-strain) for quenched specimens also have been studied .

The results have been approached that the hardness increases with percent chrome while the porosity decreases . The compression stresses to fracture , have been increased with respect to (0.3%C) had porosity (27%) and decreased for (0.9%) has porosity (22%) . from the previous results , quenched alloy (Cu-13%Al-4%Ni-0.3%Cr) is the more compression strength

**Keywords :** Shape memory alloys, Powder metallurgy, (CuAlNi), compressibility , mechanical behavior .

## الخلاصة

في هذه الدراسة تم تحضير سبيكة (نحاس- 13%ألمنيوم-4%نيكل) باستخدام تقنية ميثالورجيا المساحيق مع اضافة عنصر الكروم بنسب محددة (0.3%، 0.6%، 0.9% ) فبعد عملية الكبس أجريت عملية التلبيد في جو مفرغ من الهواء بضغط ($10^{-4}$ تورر )عند درجة حرارة (500$^0$م) لفترة (120 دقيقة) وعند (900 $^0$م ) لمدة (60 دقيقة) ثم التبريد داخل الفرن ، بعد ذلك اجريت المعاملة الحرارية (الاخماد) عند درجة حرارة (850$^0$م) لمدة(60 دقيقة) ثم الاخماد بماء بارد بدرجة حرارة (3-4$^0$م).

تم دراسة البنية المجهرية بالمجهر الضوئي والمجهر الالكتروني الماسح(SEM) وكذلك تم دراسة المسامية والسلوك ألانضغاطي (الجهد-انفعال) للسبائك المخمدة و من خلال النتائج التي تم التوصل اليها وجد أن الصلادة تزداد مع زيادة نسبة الكروم بينما المسامية انخفضت اما بالنسبة لجهود الانضغاط حتى نقطة الكسر فأنها ازدادت بالنسبة لنسبة الكروم(0.3%) البالغة مساميتها (27%) وانخفضت بالنسبة لنسبة الكروم(0.9%) مساميتها (22%) واظهرت السبيكة (نحاس – 13%ألمنيوم-4%نيكل-0.3%كروم) المخمدة أكثر مقاومة للانضغاط.

**الكلمات الدالة**: سبائك ذاكرة الشكل (نحاس-المنيوم-نيكل) ، ميثالورجيا المساحيق ،السلوك الميكانيكي ، السلوك ألانضغاطي (جهد-انفعال) .

## Introduction

Shape memory alloys (SMAs) are a fascinating group of metals that have two remarkable properties, the shape memory effect and superelasticity. Shape memory refers to the recovery of shape after apparent ''permanent'' deformation by heating above a characteristic transformation temperature. Superelasticity refers to the isothermal recovery of relatively large strains during a mechanical load–unload cycle. A large number of SMAs have been discovered since the mid-1900s to late 1900s, and the list continues to grow[**Shaw *et.al.*,2008**].Cu-Al-Ni shape memory alloy one of these alloys that is the only alternative if high transformation temperatures are required and martensitic transformation temperatures can be "adjusted" between -200°C and 200°C .[**Lojen *et.al.*,2005**]. Many scientists have been studied shape memory alloys , some of these ,[ **Zhao *et.al.*,2004]** studied Compression behavior of porous shape memory alloy to predict effective elastic and superelastic behavior of a porous SMA based on the assumption of stress–strain curve. The analytical results are compared with experimental data for porous alloy with 13% porosity resulting in a reasonably good agreement. **Picornell (2004)** studied Stress-Temperature Relationship in Compression Mode in Cu-Al-Ni Shape Memory Alloys . The results of systematic mechanical experiments in compression, performed at different temperatures and from different initial states (β-parent phase, β -martensitic phase or a mixture of both) are reported together with the data obtained from the calorimetric measurements.

**Sari *et.al.*,2007** studied Effects of deformation on microstructure and mechanical properties of a Cu–Al–Ni shape memory alloy . The alloy exhibits good mechanical properties with high ultimate compression strength and ductility after annealing at high temperature . The results show that the ultimate compression strength increases with increased of annealing time and the fracture behavior of the alloy changes from brittle fracture to ductile fracture as the heat treatment temperature increases .

**Suresh *et.al.*, 2007** have studied effect of aging on mechanical behavior of single crystal Cu–Al–Ni shape memory alloys in tensile and compression responses and their variation with aging. In the Compression loading of the aged samples shows large mechanical hysteresis and stabilization of $\gamma_1$ martensite which results in permanent strain after unloading. **Gonga *et.al.*,2011** studied property of cellular CuAlMn shape memory alloys produced by sintering–evaporation process , It was found maximum stress of the cellular CuAlMn shape memory alloys increased with the decrease in porosity, and the energy absorption per unit volume approached the maximum value of 35.81 $MJ/m^3$ (the compression direction parallel to the cross-section ) and 25.71 $MJ/m^3$ ( the compression direction perpendicular to the cross-section) as the porosity of the alloys was 60% and the pore size was between 355 and 800μm .

**Nigam *et.al.*,2013** have studied Effect of heat treatment on tensile and compression strength of nickel aluminium bronze (Cu-10%Al-5%Ni-5%Fe). During compression loading, higher stress was recorded with increasing strain prior to specimen failure. In this case, the rate of increase in stress was high initially and reduction in the rate of increase in stress and ultimately specimen fracture . Moreover, the compressive strength of the heat treated alloy samples was somewhat inferior to that of the as cast alloy while the aged samples attained higher strength compared to that of the solutionized ones and it emerges from the study that it is possible to obtain desired combinations of properties through optimizing the heat treatment type and parameters . **Gargarella *et.al.*,2015** have studied the mechanical properties of the rods were measured by compression tests using an Instron 5869 and the results appear that the samples exhibit a large plasticity in compression (around 15±1%) with a typical “double-yielding” behavior.

Also it has been reported that alloying additions play a very significant role in the properties of Cu-based shape memory alloys and pre requisite properties can be achieved by proper designing/selection of the alloying elements **Kumar *et.al.*, 2015**].The present work aims to investigate the correlation between porosity and compressibility of a Cu-13Al-4Ni-xCr shape memory alloy (when x=0.3%,0.6%,0.9%-from the standers ) produced by powder metallurgy processing.

**xperimental:**

**Materials and Preparation of Samples**

The powders (Cu , Al , Ni, Cr) with average particle size, purity and origin as shown in table(1) have been used to prepare several alloys provided by powder metallurgy process and the mixtures alloys shown in the table (2). Process of preparation consist of mixing, compacting and heat treatments. Electric rolling mixer have been used to mix and refine metal powders for (4) hours. Acetone has been used in mixing, mixtures have been compacted with best compression (520Mpa) that proceeds from repeated attempts by using different compressions comparison with green density and select the compression just at maximum green density . After compaction , the samples sintered by using vacuum tube furnace (GSL 1600X / MTI) with pressure $10^{-4}$torr at (550°C for 2 hr.and 900°C for 1hr) adopted from previous researches , then slow cooling in furnace . Samples have been heat treated at 850°C for (1hr.) then quenched in cold water (4–7°c).The samples wet ground using 180, 400, 600,800 , 1200,2000 , 2500,3000 grit silicon carbide papers , then cleaned with water and dried with hot air in drying instrument . After drying , stored in polyethylene zip–lock bags with silica gel , etched with etching solution (5gram $FeCl_3$,10ml HCl,100ml $H_2O$) for microstructure test[Ricksecker].

**Microstructure**

Microstructure of the specimen is the key to its behavior and it has studied by optical and Digital camera microscope type (Mercury, Cyberpix S-550V), and scanning electron microscopy (SEM with 1kx and 5kx) .

**Porosity**

Porosity can occur in the form of both open and closed types, depending on the volume fraction, material, and the specific processing method. The method has been used according to the ASTM-CB 328 – 96 standard by using the suspension balance .

Calculate the interconnected porosity can be determine by the following equation:

$$P = \left( \frac{B-A}{(B-C+E)*D_o} *100* D_W \quad \ldots \right) \quad .(1)$$

Where: P = interconnecting porosity by volume, % , A = mass in air of oil-free specimen, g, B = mass of oil-impregnated specimen, g, C = mass of oil-impregnated sample immersed in water, g, E = mass of wire in water, g, $D_o$ = density of the oil, g/cm$^3$, F = mass of oil impregnated specimen in water with mass of wire tared, g, and $D_w$ = density of water at the immersion temperature, g/cm$^3$. The True porosity has been evaluated for all the specimens.

**Compression Test**

The compressibility studied by using computer control electronic universal testing machine model (WAW-200) . The Compression test was performed according to ASTM (D695-85) at room temperature. Samples have been compressed , their dimensions with (10mm diameter and 14-16 mm in height) and the test achieved with strain rate (0.1mm/s).

**Vickers Hardness**

Macrohardness Rockwell-B tester was also used to measure hardness with 588N load adopted form the standers , for exactly , three readings are recorded for each sample.

**Results and Discussion**

**Light optical microscope analysis**

The results have been reached appear phases , grain boundary and porosities in the microstructure. The analysis of microstructure for not etched specimen doesn't appear anything in figures (1a,2a) while there are dark area on grain boundary named for ($\gamma_2$) phase and bright area described ($\alpha$) phase shown in figures (1b,2b) for alloys (Cu-13%Al-4%Ni) and (Cu-13%Al-4%Ni-0.9Cr) respectively . The martensite phase represent as lines shape in figures (1c,2c) as well as cavities in alloy (Cu-13%Al-4%Ni-0.9%Cr) shown in figure (2c) and these cavities effect in the mechanical properties of the shape memory alloy. The specimen preparation can also induce small alterations of the results for example, the presence of strain-induced martensite generated during mechanical polishing can induce martensite needle formation[**Melo *et.al.*, 2010**].

**Scanning electron microscope (SEM) analysis**

Also the microstructure has studied by scanning electron microscope shown in fig.(3) to understanding the microstructure that is essential for the proper interpretation of the general behavior of the material. Scanning electron microscope image for etched specimens of alloys shown in fig.(4a,b) reveal invariant lines of martensite , grain boundaries , porosities and their distribution. SEM examination gave us the previous features, Presumably some large elements powders particles are clustered, some of which may have converted to unwanted brittle intermetallics .

**Hardness**

Rockwell hardness have been evaluated for sintered and quenched alloys with added (Cr) element as shown in fig.(5) and the results have been reached that the hardness for quenched samples greater than that for sintered samples due to hard phase (martensite ) formed after quenching treatment while less hardness phases (α , γ ) have formed after sintering state. Also Rockwell hardness values refer to increasing with alloying element (chrome) due to concentrated the element at the grain boundaries as reinforcement.

**Porosity and Compression correlation**

Porosity and Compression tests have been studied for Cu-based shape memory alloys with and without (Cr) produced by sintering and quenching processes. The porosity values have been achieved shown in table(3) and recorded for average of the values. It seems from Fig.(6) porosity decreases with (Cr%) so that the alloys (1,2,3 and 4 ) have the porosities ( 33% , 27% ,22.8% and 22% ) respectively , these behavior may be due to intersect the particles of Cr with other elements particles caused blocking to the pores between particles and resulting in elevating the density and lowering the porosity portion.

Compression behavior have been achieved and plotted as shown in fig.(9) that refer to stress-strain curves for alloys (1,2&4) with porosities 33% , 27% and 22% respectively and the results of alloy (3) with (22.8%) porosity approached to that obtained from alloy(4) . When stress is large enough, collapse of imperfect necking structure among large Cu based shape memory alloy particles**[Zhao,2004]**. The fracture of a specimen after compression shown in Fig.(8b) and a large fracture strain was observed in compression , which is related to the small grain size obtained in the samples . From the figure (9) , alloy(1) with porosity (33%) exhibit much lower flow stresses than that in alloy (2) with porosity (27%) that exhibit large flow stresses and high ductility , the main reason of this behavior for alloy (2) specimen due to continuous connectivity between adjacent particles , while in the case of 33% porosity specimen there is non-uniform connectivity**[Zhao,2004]**. The results of compressive stress-strain curve of alloy (4) referred to decline values of flow stress through crystal structure although its porosity was lower (22%) and these behavior might be return to stress razor caused by increasing Cr particles, therefore smaller additive percent would be sufficient to get better results.

**Conclusions:**

The conclusions can be drawn as follow

- Decreases porosity with increasing alloying element .
- SEM test appear amount of porosity and its distribution as well as martensite lines.

- Hardness values increased with increasing Cr element .
- The alloy (Cu-13Al-4Ni-0.3Cr) with (27%) porosity exhibit higher flow stresses to fracture than that of other samples.
- The alloy (Cu-13Al-4Ni-0.9Cr) samples have been exhibited lower flow stresses to fracture due to stress razor by Chrome element.

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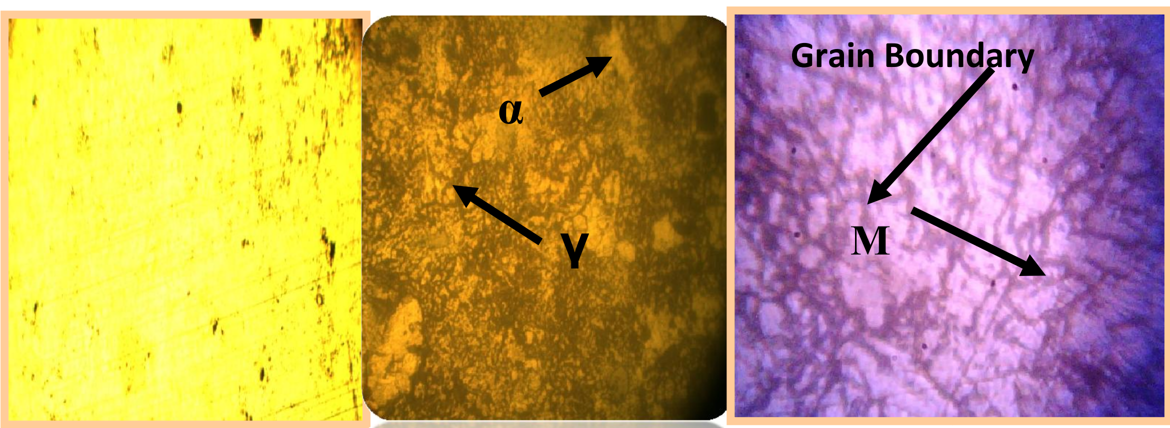
**Tables and diagrams**

Table (1): Powders used in this study

| Powder | Average particle size (μm) | Purity% | Origin (country). |
|---|---|---|---|
| Cu | 45 | 99.98 | Skyspring nanomaterials.inc./USA |
| Al | 50 | 99.90 | Skyspring nanomaterials.inc./USA |
| Ni | 45 | 99.98 | Skyspring nanomaterials.inc./USA |
| Cr | 45 | 99.9 | Skyspring nanomaterials.inc./USA |

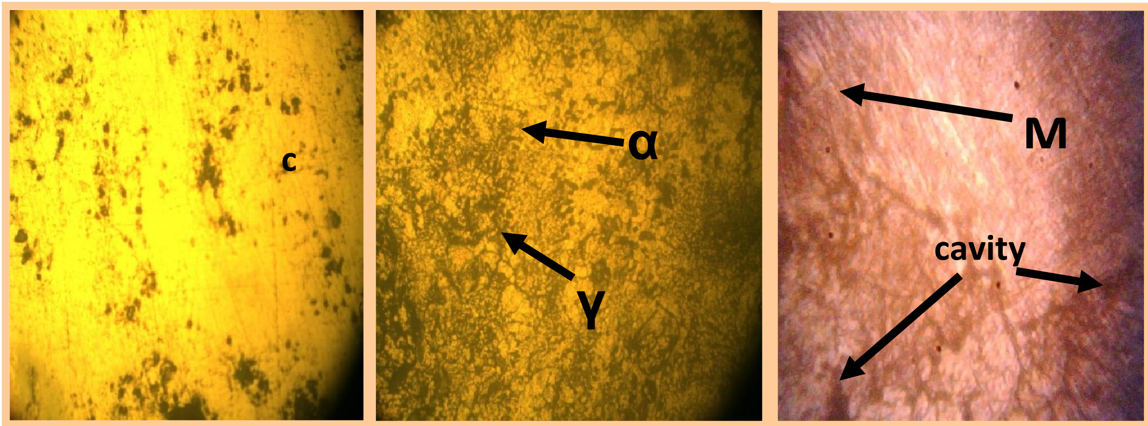
Table ( 2 ) : The mixtures of alloys.

| element | Alloy number | | | |
|---|---|---|---|---|
| | 1 | 2 | 3 | 4 |
| Cu | Bal | Bal | Bal | Bal |
| Al | 13 | 13 | 13 | 13 |
| Ni | 4 | 4 | 4 | 4 |
| Cr | ---- | 0.3 | 0.6 | 0.9 |



Figure(1).Microstructures for alloy(Cu-13%Al-4%Ni): a-sintered alloy not etched 40x,b-sintered alloy etched40x ,c-quenched alloy and etched 200x

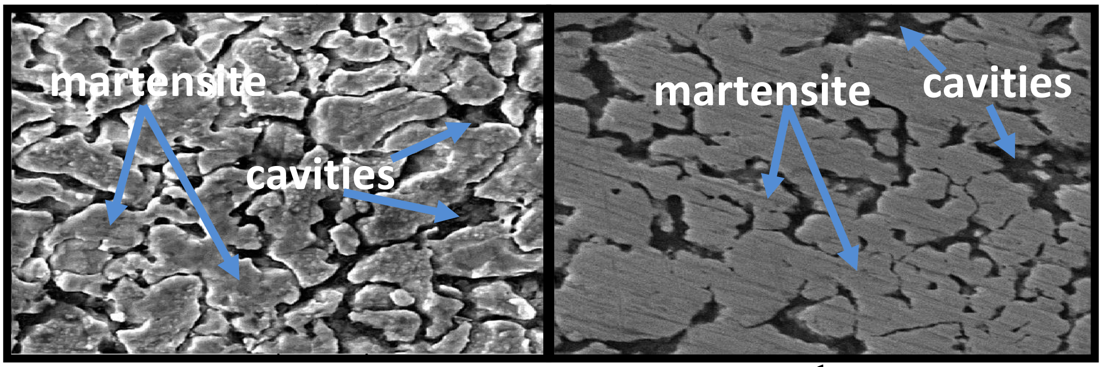
b




a b c

Figure(2).Microstructures for alloy(Cu-13%Al-4%Ni-0.9Cr): a-sintered alloy not etched40x b-sintered alloy etched10x ,c-quenched alloy etched 200x

Figure(3).SEM instrument.




Figure(4). SEM image for etched in direct quenching state for:
a- alloy (Cu-13%Al-4%Ni) b- alloy (Cu-13%Al-4%Ni-0.9Cr)

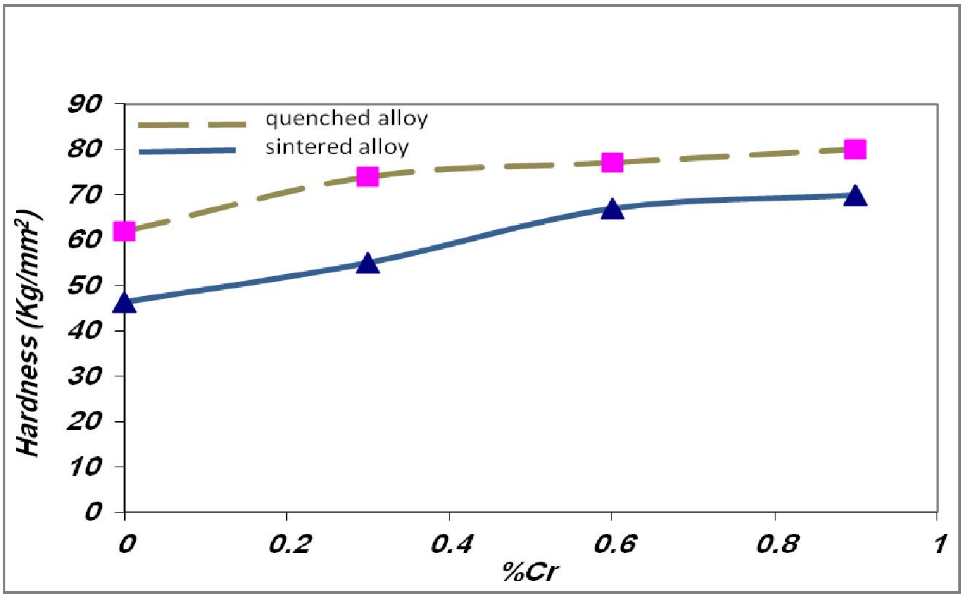



Figure (5). Hardness-%Cr relation for sintered and quenched copper base shape memory alloy.

Table(3): porosity values

| Alloy | Porosity % |
|---|---|
| Alloy 1 | 33% |
| Alloy 2 | 27% |
| Alloy 3 | 22.8% |
| Alloy 4 | 22% |

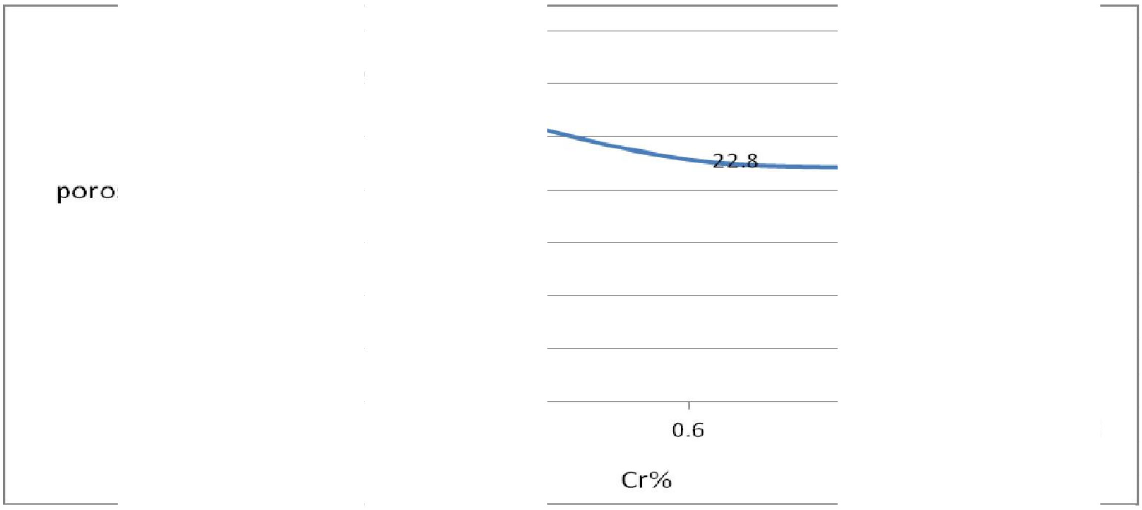



Figure (6) porosity - %Cr relation

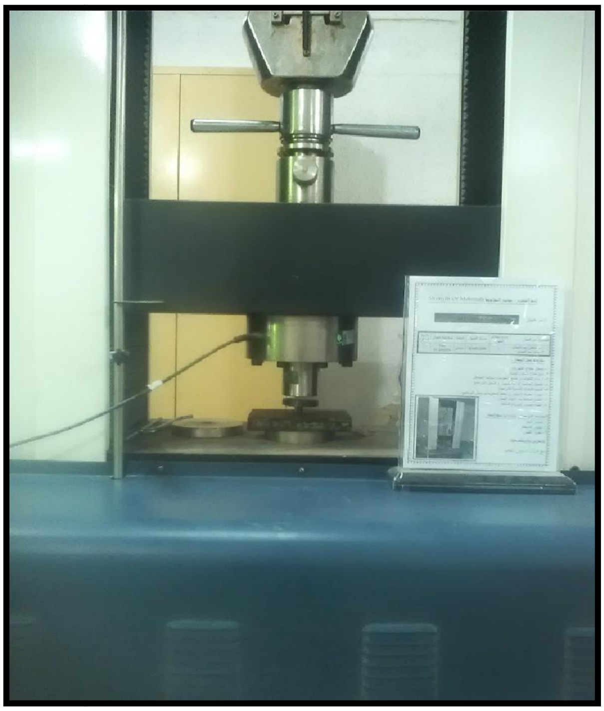
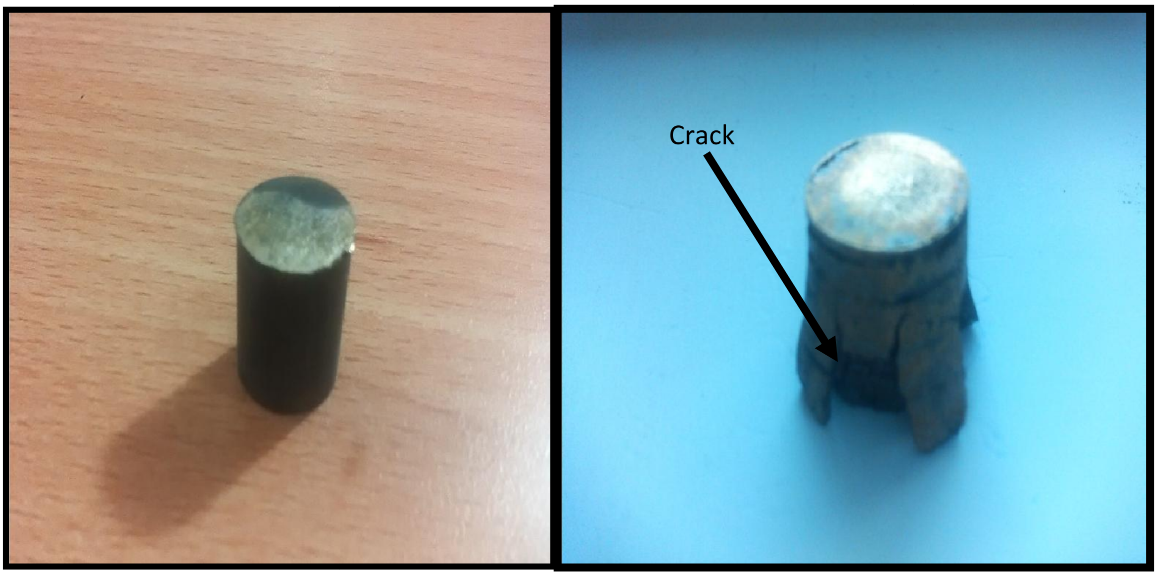


Figure (7). Universal Testing Machine




a b

Figure(8). Compacted Specimen :a-before compression test , b-after compression test (failed specimen)

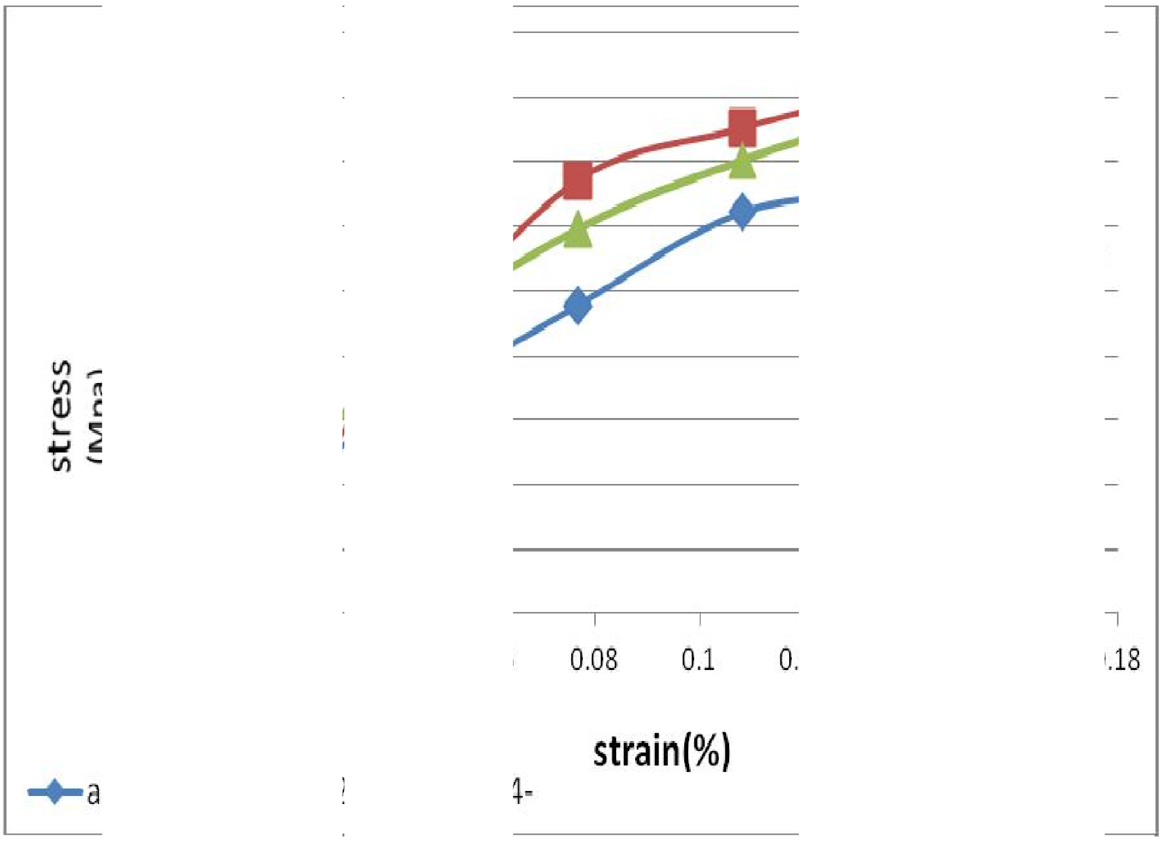



Figure (9) Compression stress-strain curve of alloy (Cu-13Al-4%Ni),Alloy (Cu-13Al-4%Ni-0.3Cr) and Alloy (Cu-13Al-4%Ni-0.9Cr)

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