REPUBLIC OF TURKEY
FIRAT UNIVERSITY
THE GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCE

FABRICATION OF Cu-BASED HIGH-TEMPERATURE SHAPE MEMORY ALLOYS AND INVESTIGATION OF THEIR PHYSICAL PROPERTIES

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Master Thesis
Department: Physics
Program: Solid State Physics
Supervisor: Assis. Prof. Dr. Fethi DAĞDELEN
JANUARY-2017

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Abstract

In this study, High Temperature Shape Memory Alloys CuAlCr and CuAlCrNi that have drawn attention recently due to their technological applications were investigated. These alloys were produced by melting method in an arc-melter furnace. The phase transformation and microstructure properties of produced high-temperature shape-memory alloys were studied by means of Differential Scanning Calorimeter (DSC), X-ray diffraction (XRD), Optical microscopy (OM), Scanning electron microscopy (SEM) and Vickers microhardness measurements at different heat treatment. According to DSC measurement, all of alloys exhibited high temperature shape memory effects. Crystal structure of alloys measurement by means of X ray diffractometer. Two martensite phases named as tin and thick plate can be seen in XRD results. At the end of experimental measurements, it was observed that the addition of nickel decreases the transformation temperature of CuAlCr and also change the crystal structure. Optical micrographic shows that there are a lot of grain and precipitates in the structure of alloys. Also, it is seen in SEM micrographic that the grain border is clearly visible.

Keywords: *Shape memory alloy, transformation temperature, microstructure.*

Özet

Cu-Bazlı Yüksek Sıcaklık Şekil Hatırlamalı Alaşımların Üretimi Ve Fiziksel Özelliklerinin Incelenmesi

Bu çalışmada, son zamanlarda teknolojik uygulamalarda dikkat çeken Yüksek Sıcaklık Şekil Hatırlamalı Alaşım (High Temperature Shape Memory Alloys) olan CuAlCr ve CuAlCrNi alaşımlarının fiziksel özellikleri araştırıldı. Alaşımlar ark-ergitme yöntemi ile üretildi. Faz dönüşüm sıcaklıkları DSC (Differential Scanning Calorimeter), kristal yapı tayini XRD ölçümleriyle (X-ray diffraction), yüzey morfolojisi OM (Optical Microscopy) ve SEM (Scanning Electron Microscopy), mikro sertlik özellikleri ise Vickers mikro-sertlik belirleme yöntemiyle belirlendi. DSC sonuçları sonucunda numunelerin yüksek sıcaklık şekil hatırlama özelliğine sahip olduğu, ve ayrıca CuAlCr alaşımına Ni katılınca oluşan CuAlCr Ni alaşımının faz dönüşüm sıcaklıklarının düştüğü gözlendi. X-ışını sonuçlarında alaşımlarda iki martensit faz gözlendi. Optik mikroskobi sonuçlarında yapı içinde birçok grain ve çökeltiye rastlandı. SEM ölçümlerinde ise, grain sınırlarının belirgin olduğu gözlendi.

Anahtar kelimeler: *Yüksek Sıcaklık Şekil Hatırlamalı Alaşım, Dönüşüm Sıcaklığı, mikroyapı.*

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Mohammed Al-DALAWI

Elazig-2017

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ABBREVIATION

SMAs : Shape Memory Alloys
SME : Shape Memory Effect
SE : Superelastisity
TTs : Transformation Temperatures
HTSMAs : High Temperature Shape Memory Alloys
SIM : Stress Induced Martensite
As : Austenite Start
Af : Austenite Final
T_o : Equilibrium Transformation Temperature
Ms : Martensite Start
Mf : Martensite Final
DSC : Differential Scanning Calorimeter
XRD : X-Ray Diffraction
OM : Optic Microscopy
SEM : Scanning Electron Microscopy
EDX : Energy-Dispersive X-ray

1. INTRODUCTION

The study of the shape memory alloy (SMAs) in the past few years on a wide scale as a result of two of the unique characteristics of its kind, namely superelasticity (SE) and shape memory effect (SME) which are closely linked to the reversible martensitic transformation [1]. The shape memory effect (SME) is recovering from large strain as a result of martensitic transformation upon heating at austenite finish (A_f) temperature. The low-temperature phase is called martensite and the high temperature phase is it referred to as austenite. Another unique feature, superelasticity (SE), has been observed in the SMAs. SE is related to large nonlinear recoverable strain upon loading and unloading at higher temperatures [2-4].

Ni–Ti-based alloys having excellent SME and SE properties, sufficient strength and ductility are considered the most important shape memory alloys [3-5]. Recently another Cu-based shape memory alloys such as Cu–Al–Ni, Cu–Zn–Al and Cu–Al–Mn alloys have gained the attention due to lower cost and better SME and SE properties compared to Ni–Ti-based and Fe-based shape memory alloys [1,6,7]. However, an important challenge which limits the application of these SMAs is that martensitic transformation temperatures are lower than 120^{o}C. Therefore, various engineering applications demand the development of high-temperature shape memory alloys which can function over 120^{o}C.

In addition, there are many control and actuation-type applications for materials exhibiting the shape memory effect at higher temperatures. High-temperature shape memory alloys (HTSMAs) could be used in the aeronautic, automotive, power generation and chemical processing industries. While specific applications have been identified based on some form of an HTSMA, no suitable materials have been developed. As it is common in the materials field the development of applications for advanced materials is slightly ahead of the materials development itself, and such is the case for the development of HTSMAs.

Yet many of the alloy are investigated and development systems as HTSMAs. For example, Ni-Ti-Zr/Hf, Zr-Ru-Ta/Nb, Ni-Ti-Pd/Pt, Ni-Al, Ni-Mn alloys [8,9]. However, there are some practical problems that remain without solution in this alloys. For example, consider Ni-Al alloy is unstable as well as the Ni–Mn and Ni–Ti–Zr alloys are very fragile for the actual output. Also, the high cost of Pt/Pd leads to obstruction Ni–Ti–Pd/Pt applications. They show good SMEs and high martensitic TTs. So, it was the research interest in low-cost and new HTSMAs [10].

2. SHAPE MEMORY ALLOYS

2.1. History of Shape Memory Alloy

Shape memory effect was first Discovered by Ölander with ‘rubber like effect’ study in 1932 and Greninger and Mooradian’s ‘Bronz (Copper-Zinc) alloys’ study in 1938. After the years, Chang and Read first used shape recovery term (1952). The importance of shape memory alloys (SMA) was not clearly recognized until discover of NiTi shape memory alloy by William Beuhler and Frederick Wang in 1962. NiTi alloys know as NiTiNOL which derived from Composition element and Naval Ordnance Laboratory. Since then, there is great demand for shape memory effect of alloys [11].

In 1970s, scientists became aware of potential uses areas of shape memory alloys and the production of SMAs increased. Sawyer used NiTi alloy for moving artificial heart in 1971. In 1982, Sharp used shape memory alloy as actuator for furnace [12].

Since discovery of NiTi shape memory alloy, the demand for shape memory alloys for engineering and other technical applications have been increasing in commercial field as automotive, aerospace, actuator, biomedical and robotics [13].

2.2. Definition of Shape Memory Alloys

One of the classes of the smart material is shape memory alloy which can recover original shape when they are heated above a certain temperature. There are two phases in shape memory alloys with different crystal structures [14, 15]. One is called as high temperature phase ‘austenite’ the other is low temperature phase ‘martensite’.

2.3. Austenite Phase and Martensite Phase

The properties of shape memory alloys are connected to martensite transformation. Martensitic transformation is diffusion less phase transition. The diffusion less phase transition means that atoms of solids move cooperatively. The parent phase is high temperature phase as austenite, the other phase is low temperature phase as martensite.

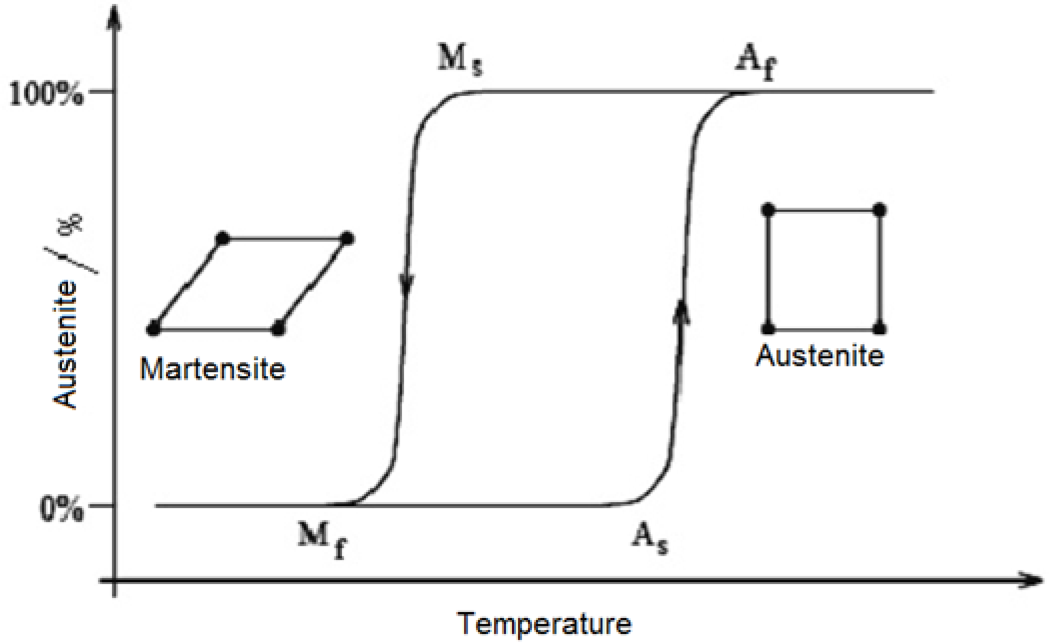


Figure 2.1. Phases and hysteresis of shape memory alloys

Transformation defined as common temperatures. At low temperature, shape memory alloys are martensite was with %100 percentage, by racing temperature martensite phase of SMA return to austenite phase in Fig (2.1). Shape changes of alloys by temperature can be seen in Fig (2.2).

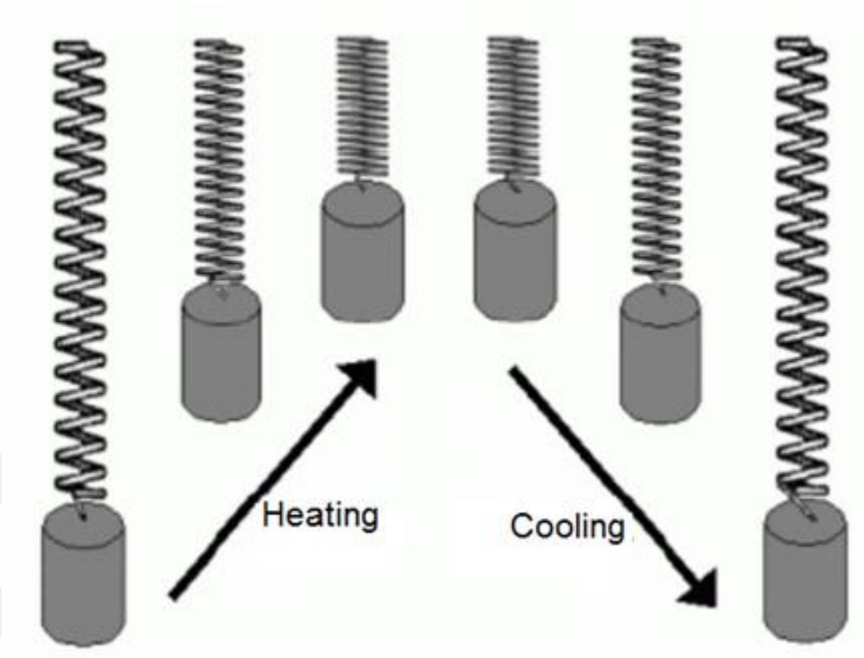


Figure 2.2. Relationships between Shape memory alloys and Heat

2.4. Shape Memory Effect

Shape memory effect is based on martensitic-austenite, austenite-martensitic transformation after applied heat or stress [16].

2.4.1. One - Way Shape Memory Effect

One - way shape memory effect can be seen in Figure (2.3) and microscopic one - way shape memory effect is seen in Fig (2.3).

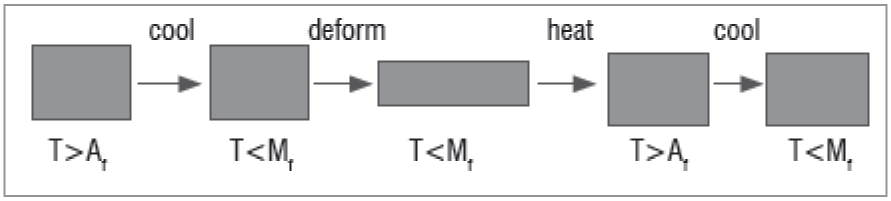


Figure 2.3. One - way shape memory effect

In one - way shape memory effect, there is once shape remembered in alloys [16]. After applied external force on shape memory alloys, alloys return original shape when heating.

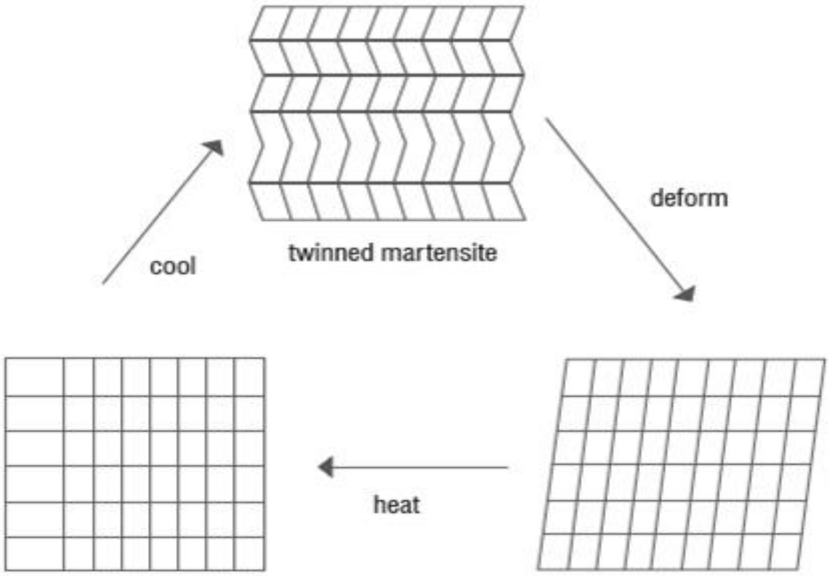


Figure 2.4. Microscopic image of one - way shape memory effect

2.4.2. Two - Way Shape Memory Effect

Oppose to one-way shape memory effect, two-way shape memory effect can be processed to remember both heating and cooling of alloys as seen in Fig (2.5) [16].

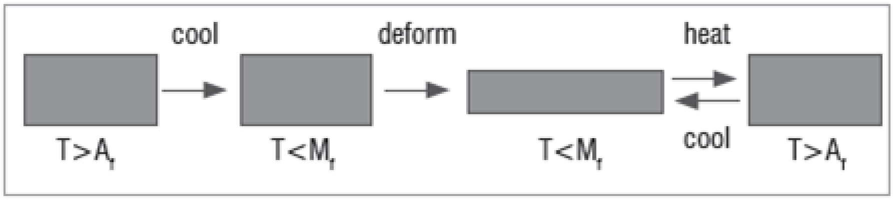


Figure 2.5. Two-way shape memory effect

2.5. Superelasticity

Shape memory alloy display superelasticity. Superelasticity is a mechanical form of this material, when a shape memory alloy trained above their transformation temperature, this effect is observed.

The mechanical properties of these material is different above the temperature range spanning their transformation. Shape memory alloy exist a martensitic at the low temperature and is deformed by applied force. Later, material exhibits shape memory by heating. The material exhibits austenite phase, this phase isn't easily deformed.

When SMA is tested above its TT (transformation temperature) to austenite, the applied force transforms the austenite phase to martensitic phase. The material exhibits increasing force at constant applied force. Substantial deformation occurs for a relatively applied force stress. If the force is removed, the martensitic phase reverts to austenite phase. This effect is known as superelasticity or pseudoelasticity.

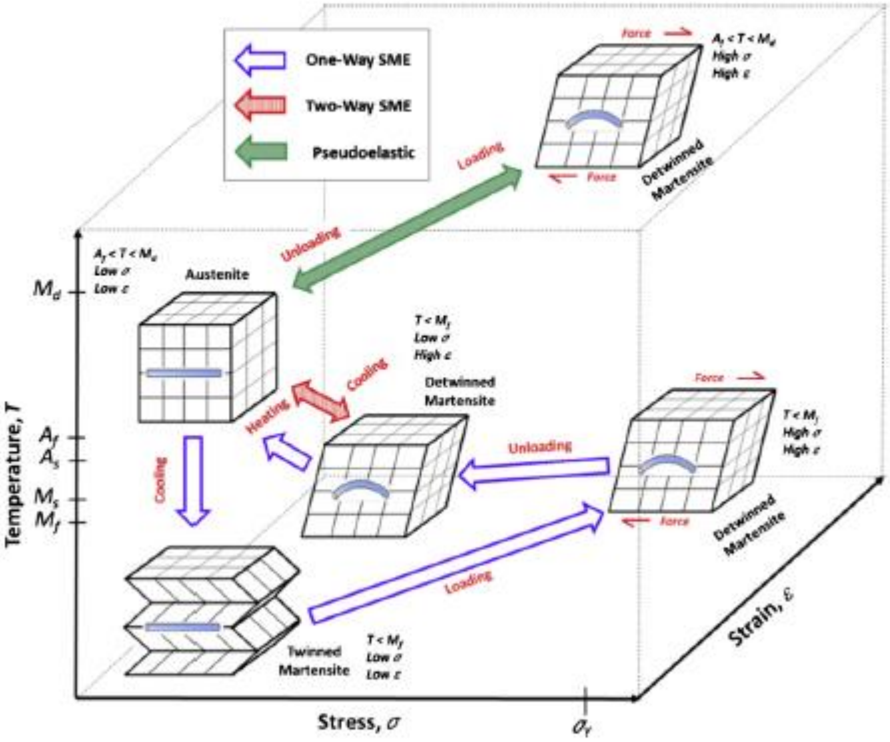


Figure 2.6. Shape Memory alloy phases and Crystal structure

3. HIGH TEMPERATURE SHAPE MEMORY ALLOYS (HTSMA)

3.1. High Temperature Shape Memory Alloys

Current practical employ of shape memory alloys (SMAs) is finite to below 100°C which is the limit for the transformation temperatures of the more commercially successful SMAs such as NiTi and Cu-based alloys. High-temperature SMAs (HTSMAs) can be classified as SMAs having M_s of above 100°C [17-19]. Since below this temperature the diffusion process is difficult to take place, reversibility of the transformation is easy [18]. However, at high temperatures diffusion controlled mechanisms are activated easily and the changeability of the martensitic transformation is difficult.

Some of the challenges for HTSMAs are listed as; stabilization of the martensite at high temperatures before reverse transformation occurs, decomposition of the martensite or the parent phase, recrystallization, creep, oxidation etc. One of the main problem for HTSMAs is low critical stress for sliding at high temperatures. The negative slope is shown in the Fig (3.1) demonstrates how the critical stress for sliding reduces upon temperature increase. At high temperatures slip occurs more easily than stress induced martensite (SIM). In this regard, HTSMAs has many obstacles to perform like functionally stable and reliable low temperature SMAs, such as NiTi [18,19].

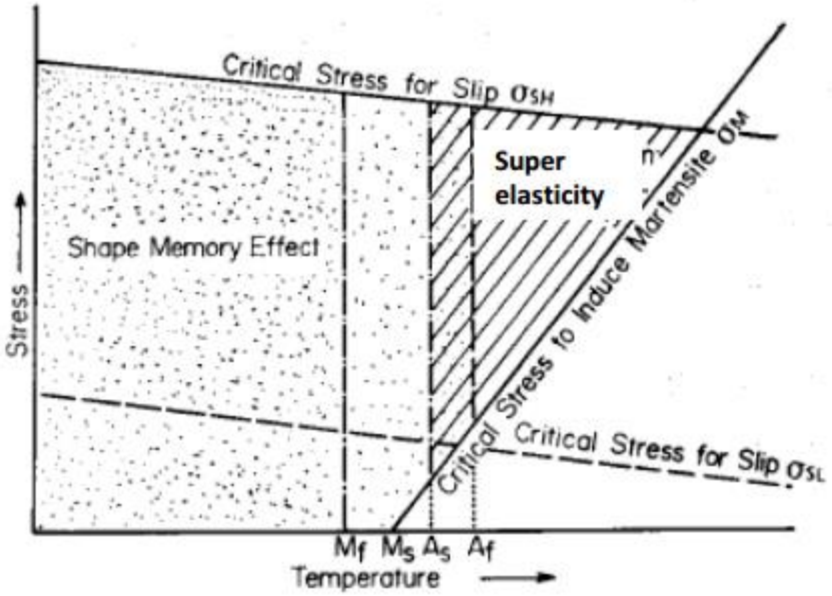


Figure 3.1. Schematic diagram showing the regions of the shape memory effect and superelasticity in the temperature-stress coordinates

Recently, automotive, aerospace, and public useful industries have become interested in high temperature SMAs (HTSMAs) with transformation temperatures among 100°C and 250°C [17,18]. A few of the known "HTSMAs" with transformation temperatures in this field includes Ni-Ti-X, where X is Pd, Au, Pt, Hf and Zr, Cu-Al-Ni (Zn), Ni -Mn-Ga and Ni-Al alloys. Among Ni-Ti-X alloys, (Ni-Ti-Pd), (Ni-Ti-Pt), (Ni-Ti-Au) alloys are most promising HTSMAs by virtue of their relatively perfect workability, low transformation hysteresis and dimensional stability, however, they are so expensive because of Pt, Pd, and Au additions [19-21]. Also, NiAl alloys have poor low temperature ductility and breaking toughness [22]. In addition, Ni_5Al_3 phase fashioning at high temperatures prevents martensite reversibility [22,23]. Cu-based alloys be affected from chemical and thermal stabilities because of the tendency for compositional decomposition at high temperatures [24,25]. Ni-Mn-Ga alloys are susceptible to intergranular break. NiTi (Hf, Zr) alloys show high transformation hysteresis, thermal cyclic degradation and dimensional instability [19,26,27].

Thermal stability upon cycling through reversible martensitic transformation either thermally or thermo-mechanically is the one of the critical features that HTSMAs should possess because it is utilized in practical applications. Therefore, there is a pressing necessity for the evolution of relatively inexpensive HTSMAs with sensible thermal and chemical stability. In addition, HTSMAs must have functional stability and have more mechanical properties. Dislocation and cereal boundary mediated plasticity is the most basic problem for HTSMAs that shows itself as the large irrecoverable strain in the SME and as the few of SE.

3.2. Fe - Based High Temperature Shape Memory Alloys

In view of the fact that prices one of the key considerations for applications, the inexpensive iron based shape-memory alloys have attracted large attention in latest years [28,29]. Of particular benefit are (Fe - Mn - Si), (Fe - Ni -Mn) and (Fe - Ni - C). These alloys show perfect or almost perfect SME after complex thermo-mechanical remedy, because of stress-induced martensite transformation and the reversion, generally, only one way SME of several percent is realizable because the fcc austenite to the ‘bct’ or ‘hcp’ martensite transformations in the alloys is not thermo-elastic. In other interesting alloys, Fe-Ni Co-Ti, the transformation from fcc austenite to bct martensite is thermo-elastic and a very big recovery stress (>1GPa) cannot be attained, with a ratio low thermal hysteresis (20-40K) and transformation temperatures close to ambient temperatures. Recently, the Fe-Pt and Fe-Pd alloys, which has been discussed previously, mostly as an experiment materials, have got more attention again because of the martensite transformations or improve variables martensite in the alloys can be affected by magnetic fields and that is why it can develop materials of shape memory magnetic [30].

“HTSMA” is the alloy of which the revers transformation ‘A_s’ begin only above 120˚C in stress-free condition after any thermo-mechanical remedy. More of the (Fe-Mn-Si) based alloys are in agreement with the given definition. in spite of the fact that those alloys do not display a thermoelastic martensite transformation, a technique of the shape memory effect and the method to optimize the thermo-mechanical remedy for optimal performance.

[31,32] Those alloys, usually alloyed with Cr, Ni, or Co to make it stainless, also have finite practical behavior: no significant two-way memory effect neither reasonable pseudoelasticity has been seen [33].

3.3. NiTi-X -Based High Temperature Shape Memory Alloys

At the point when nickel in Ti-Ni SMAs is substituted by palladium, platinum and gold components by up to 50 at % and titanium in Ti-Ni is substituted by hafnium and zirconium by up to 20at %, the martensite transformation temperatures can be expanded to as high as 873K while the fundamental shape-memory performance as yet exists [34,35].

These composites are promising shape-memory alloys for applications at higher temperatures (393K), in spite of their high cost. the Ni-Ti-Hf alloys are most appealing, and therefore have been the most investigated in the most recent couple of years. The alloys have enough of ductility and their shape memory ability (recoverable elongation) is near to the binary Ti-Ni alloys. In spite of, a methodical investigation of the phase stability of the alloys at high temperatures has not yet been undertaken. In some components, like Ti-Ni-Au, it showed it that at high temperatures the triple alloys are also apt to the ageing effects, like the martensite stabilization phenomenon [36].

Ti-Ni-Pd HTSMAs It has got to consideration the most comprehensive throughout the years. the first concentrate was centre on making better their high-temperature shape memory behaviour, but as of late, the focus has shifted to develop their work output, as well as improving the dimensional and microstructural stability. In this framework, transformation temperatures can be modified by exchange nickel with palladium. If the focus of titanium is kept constant at nearly 50 at-%, the relationship among the relative concentration of nickel and palladium and transformation temperatures is parabolic. A minimum in transformation temperatures happens at around 10 at-% Pd [37-39]. In composition with palladium concentrations bigger than the concentration of palladium in this rule, exchange nickel with palladium cause higher transformation temperature to almost 15˚C/at-% [38,40,41]. Similar to the Ti–Ni–Pd system, exchange nickel with platinum increases the transformation temperatures, In spite of the Ti–Ni–Pd system, exchange nickel with platinum raise the transformation temperatures but only after a threshold value of nearly 10–15 at-% Pt is reaching. At ≤ 10 at. % Pt, the martensite shape is ‘B19’(monoclinic) [42].

Also, the transformation temperature is not relatively affected to put the content or reduce slightly with a minimum observed in 5-10% Pt atoms. However, at larger amounts of Pt, at minimum 16 at-% or more, the martensite formed is B19 (orthorhombic) [42,43]. Also, transformation temperatures linearly rise with Pt concentration till transformation temperatures close to 1000°C are reached for the bilateral Ti-Pt alloy. Lastly, in antithesis to the Ti-Ni-Pd system, the transformation temperatures for (Ni,Pt) wealthy compositions do not land sharply with deviations from stoichiometry and a (Ni,Pt)3 Ti2 precipitate phase easily happen in alloys slow cooled from higher temperatures [44].

3.4. Cu- Based High Temperature Shape Memory Alloys

Copper from the more commercial materials considered at present which is used widely in shape memory alloys. Despite the fact that their mechanical properties and shape memory are the second rates compared to those of Ni–Ti alloys in polycrystalline shape, low-cost of copper SMAs allows them to be competitive in several applications. There are two types of binary alloy systems in copper: Cu-Zn and Cu–Al it is considered the most suitable candidate for use in high-temperature shape memory applications, not just because of the higher transformation temperatures but its microstructural stability better than the Cu-Zn counterpart [45].

Of the composites, the ternary alloy which extensively studied, which can get commercially are (Cu-Zn-Al) and (Cu-Al-Ni). It also includes commercial alloys Cu-Al-Be and Cu-Al-Mn alloys. However, the applications of these alloys is limited largely for two reasons:(1) big grains are the reason of the weak ductility and workability in the polycrystalline alloys, The high elasticity, and the precipitation of crisp second- phase particles; and (2) the metastability of both the parent (B2, D0 & or L2) and martensite (9R or 18R) stages in the alloys, which result in complex ageing effects and thus an undesirable dependability of the performance of the alloys [46,47].

As the (Cu-Al-Ni) alloy has a thermal stability and high temperatures very better compared with (Cu-Zn-Al) alloys, so it can become a good candidate for high-temperature shape memory alloy a process, if only their poor processability can be enhance. Lately, some attempt has been made to realize this goal. Accordingly, a few of quaternary and pentatonic alloys based on (Cu-Al-Ni), such as (Cu-Al-Ni-Mn) and (Cu-Al-Mn-Ti) (B) which has a M_s dot higher than 423K, have been developed. Really, the workability of the (Cu-Al-Ni) alloys can be altogether improved by adding little amounts of alloying ingredient to the alloys, and cold-drawn wires can be produced [48,49]. Nevertheless, the thermal stability of the alloys remains a significant point. Both martensite stabilization and parent-phase ageing effects were observed at temperatures high than 393K [48-50].

The transformation temperature of (Cu-Al-Ni) can be different by change the aluminum or nickel content. Changing the aluminum installation between 14 at. % and 14.5 at. % can change the M_s temperature from -140˚C to 100˚C. The relative change in transformation temperature is not considerable, and the hysteresis remains constant to some extent. Since this alloy is more difficult to produce, manganese is regularly added to enhance its ductility and titanium is added to refine its grains. However, the baseline limitation of the (Cu-Al-Ni) system is the reduced ductility due to inter-granular cracking [51].

The principle issue of polycrystalline (Cu-Al-Ni) alloys, however, is their crispness. These ternary alloys are very crisp due to the great average grain size, excellent elastic anisotropy and separation of impurities towards grain border [52]. To improve the ductility can be added some elements, for example Ti, B, Zr can be added in composition with a suitable thermos-mechanical treatment, resulting in grain refinement [53].

3.5. Application of High Temperature Shape Memory Alloy

SMAs is an exceptional range of substance that can produce a very high change recoverable shape changes, stresses, and work outputs as a result of a reversible martensitic phase conversion. Due to their remarkable properties, which can be used for a variety of application, such as actuation, vibration damping, and noise reduction. SMAs have permeated into the mainstream of many industries, particularly in the biomedical, automotive, energy, and aerospace field [54].

That most of the expected applications in high-temperature used on SMAs as a solid-state engine. This is because the SMAs can a reply to the change in temperature (incentive) with mechanical strain, it is easy to imagine how the shape memory alloys can be a sight as the engine or solid-state drives. SMAs Components has a high energy density greater than the pneumatic motors or direct current motors also have the same performance of hydraulic motors while weigh less and especially maintaining the compact footprint much more [55,56].

Aviation and space have been suggested for some other applications for HTSMAs such as engines to deploy satellites structures in space, and screens to protect the satellite facilities of the fracture, and release mechanisms during the firing of missile [57]. All of this needs to high transformation temperatures. Many users have been suggested for HTSMA including fuel management applications in the car’s engine and motor control tools. [58].

Self-expanding stents are another significant application that is applied to maintain the inner diameter of a blood vessel. In fact, these devices are used in some case in order to support any tubular transit like the esophagus and bile duct [59], and blood vessels like femoral arteries, iliac, coronary, aorta, and carotid. [60]. In this kind of application, a cylindrical scaffold with shape memory Fig (3.2) is put, such as, inside a blood vessel through a catheter. At first, this scaffold is pre-compressed in its martensitic condition. Because the scaffold is heated due to the body heat, it tends to regain its original shape, expanding same. This device can be used not just in the angioplasty procedure, in order to block another obstruction of a vessel, however also in the remedy of aneurysms for the support of a weak vessel [60].

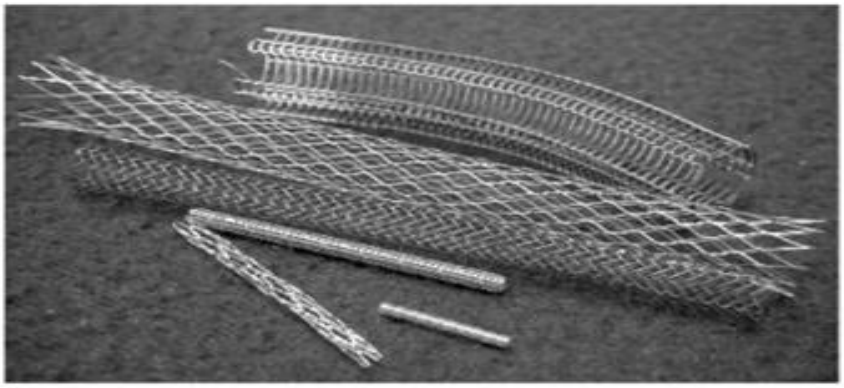


Figure 3.2. Shape memory self-expanding stents.

There are a wide number of orthopedic applications in shape memory alloys. such as the spinal vertebra spacer that can be seen in Fig (3.3). This injection interval between two vertebrae assures the local strengthening of the vertebrae of the spine, preventing any movement painful during the healing process. The use of a shape memory spacer allows the application to load the level regardless of the status of the patient, which maintains a certain degree of movement [61].

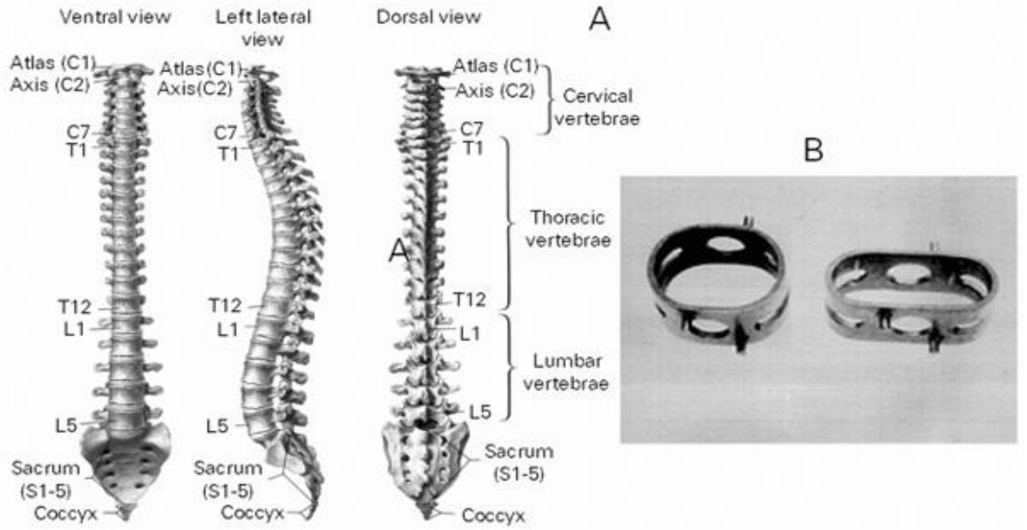


Figure 3.3. Spinal vertebrae (A) and shape memory spacers (B) in the martensitic state (left) and in the original shape (right).

4. RESULTS AND DISCUSITION

4.1. Experimental Procedures

In this study, we use some Cu-based shape memory alloys. $Cu_{85}Al_{12}Cr_3$ and $Cu_{83}Al_{12}Cr_3Ni_2$ alloys used. We carry out investigation of these two types alloys in this study by Differential Scanning Calorimeter (DSC), Optic Microscopy (OM), X-ray diffraction (XRD), Hardness measurement and Scanning Electron Microscopy (SEM).

Table 4.1. The weight and atomic presentences of fabricated samples

		Cu	**Al**	**Cr**	**Ni**
Sample 1	**wt%**	85	12	3	-
	at.%	73.8	24.44	3.16	-
Sample 2	**wt%**	83	12	3	2
	at.%	71	23.36	3.11	1.86

Firstly, the alloys were prepared using arc-melting process. Different annealing temperature do it for these two alloys. They were put in the furnace at 930^{o}C for 12 hours then following by water quenching to obtain an order metastable β-phase. Later, this process has been re for same alloys at 700^{o}C and 800^{o}C temperature for 1 hour. We prepare about 50 mg for DSC measurement from ingot sample. Austenite and martensite phase transformation temperature determined by means of DSC technique between 100-500^{o}C at the heating rate of 20^{o}C in nitrogen atmosphere. The microstructure was observed by optical microscopy (OM). Sample for microscopic observation were mechanically polished and chemically etched in a solution of (10ml Hcl + 25ml $FeCl_3$ + 48ml CH_3OH) for 17 sec.

The crystal structure of alloys was determined by XRD measurement. At room temperature, all measurement was done between 20^o to 80^o. Vickers hardness test set with 0.5kg for 10 secs was performed to measure the hardness of these alloys. Microstructural and elemental analysis measurements were made at room temperature by SEM-EDX measurement.

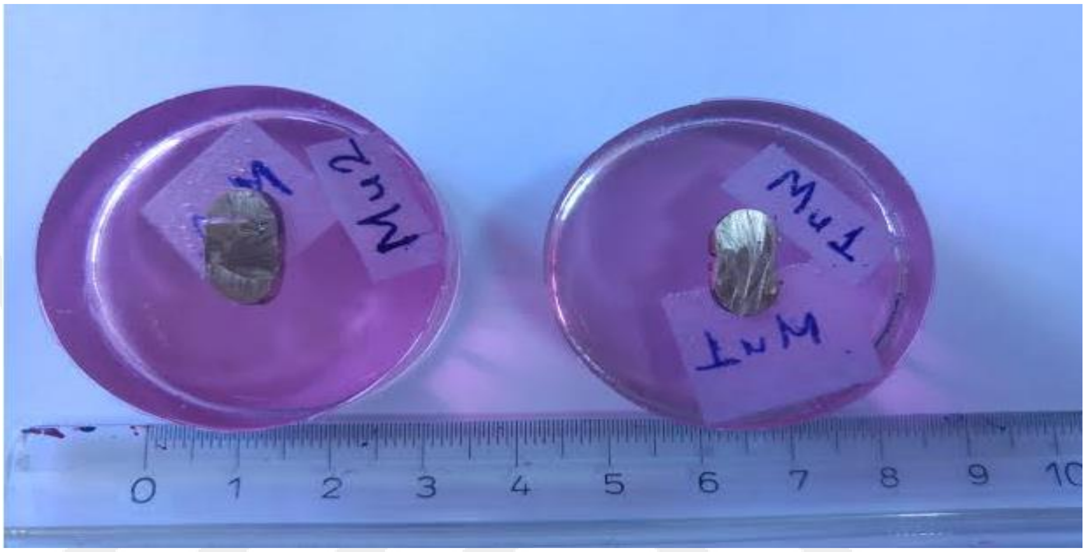


Figure 4.1. The alloys embedded to polyester resins

4.2. DSC Measurement Result

The DSC curves of $Cu_{85}Al_{12}Cr_3$ and $Cu_{83}Al_{12}Cr_3Ni_2$ wt% alloys, which exposed to three different heat treatment temperature, obtained. The Austenite and martensite phase transformation temperatures were determined by means of DSC between 100-500°C at the heating/cooling rate of 20°C in nitrogen atmosphere.

First heat treatment temperature is 930°C until 12 hours for CuAlCr alloy. At this temperature, austenite and martensite transformation of CuAlCr observed that can be seen in Fig (4.2). CuAlCr alloy has austenite start temperature A_s = 302.1°C, austenite finish temperature A_f = 399.4°C and martensite start temperature M_s = 243.7°C, martensite finish temperature M_f = 187.9°C as show in Table 4.2.

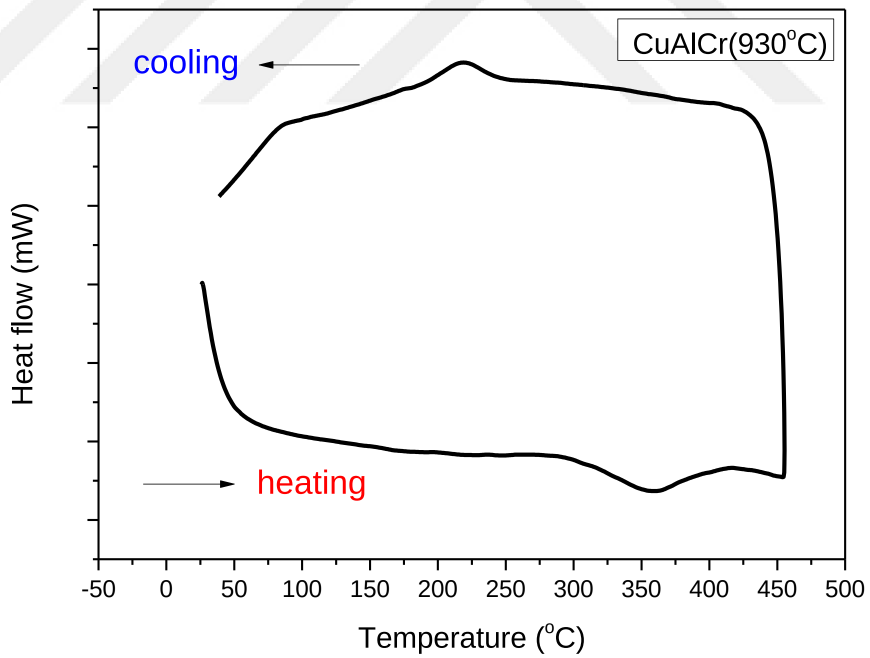


Figure 4.2. DSC curve heat treatment temperature of CuAlCr

Table 4.2. Transformation temperatures and enthalpy results of sample

Sample	As (°C)	Af (°C)	T_0(°C) (Salzbrenner and Cohen)	Ms(°C)	Mf(°C)	ΔH(mj/mg)
CuAlCr (930°C)	302.1	399.4	272.9	243.7	187.9	3.35
CuAlCrNi (930°C)	256.3	299.1	232.9	209.5	148.2	2.80
CuAlCr (700°C)	312.1	399.9	287.65	263.2	207	3.31
CuAlCr (800°C)	301.8	385.2	314.1	326.4	215.5	6.515
CuAlCrNi (700°C)	-	-	-	-	-	-
CuAlCrNi (800°C)	214.1	280.3	218.8	223.5	147.8	1.915

Second heat treatment temperature is 930°C until 12 hours for CuAlCrNi alloy. Also at this temperature, austenite and martensite transformation of CuAlCrNi observed that can be seen in Fig (4.3). CuAlCrNi alloy has austenite start temperature A_s = 256.3°C, austenite finish temperature A_f = 299.1°C and martensite start temperature M_s = 209.5°C, martensite finish temperature M_s = 148.1°C as show in Table 4.2.

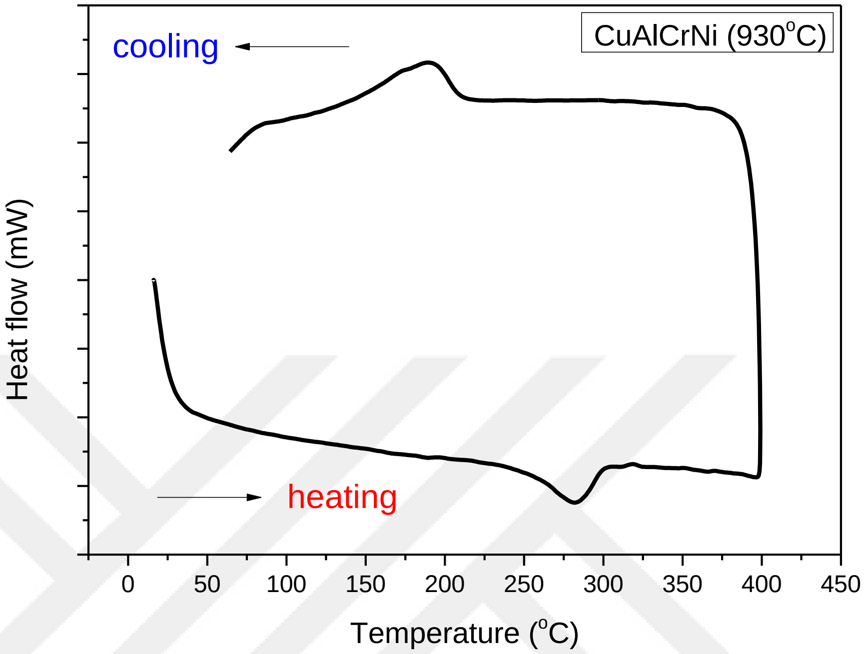


Figure 4.3. DSC curve heat treatment temperature of CuAlCrNi

The characteristics of DSC curves show that the transformation temperature decrease in the CuAlCrNi alloy. This is because of the addition of (Ni), which has reduced the proportion of transformation temperature. But the peaks in the CuAlCrNi become bigger than CuAlCr as show in the Fig (4.4).

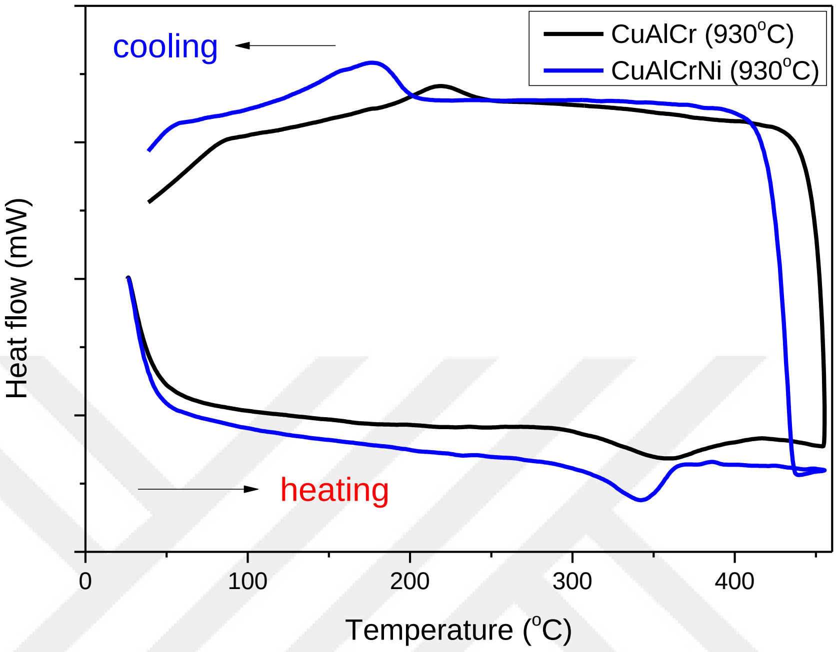


Figure 4.4. The comparison of DSC curves of CuAlCr and CuAlCrNi

CuAlCr alloy carried out heat treatment temperature 700°C for 1 hour austenite and martensite transformation of CuAlCr observed that can be seen in Fig (4.5). CuAlCr alloy has austenite start temperature As = 312.1°C, austenite finish temperature Af = 399.9°C, and martensite start temperature Ms = 263.2°C, martensite finish temperature Mf = 207°C as show in Table 4.2.

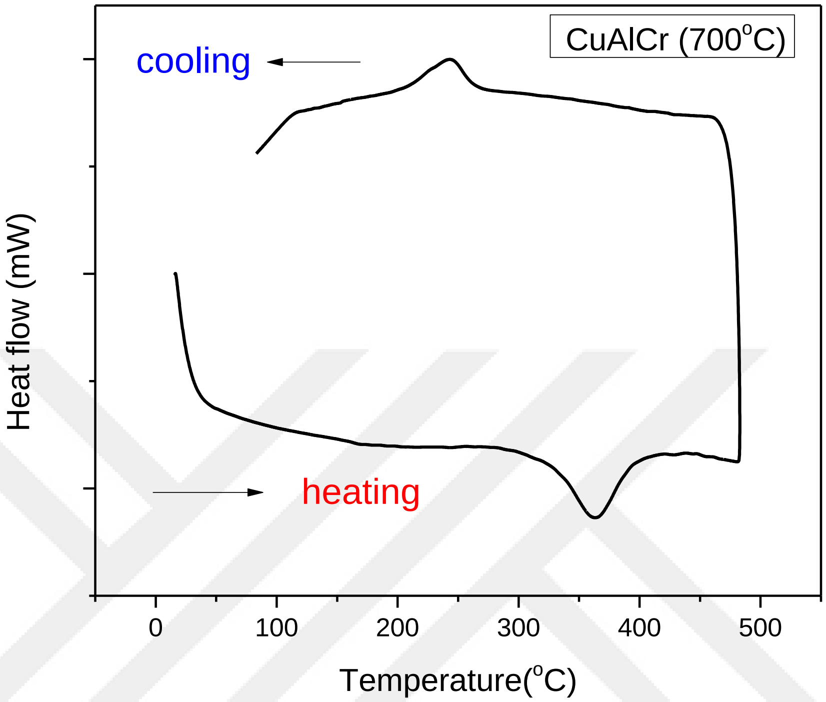


Figure 4.5. DSC curves heat treatment temperature at (700°C) of CuAlCr

The situation has differed for heat treatment temperature 700°C until 1 hour for CuAlCrNi alloy. Sample cycled two time but didn't show any transformation temperature that can be seen in Fig (4.6). The reason can be explained as microstructure and crystal structure of alloy. There could be two phases as austenite and martensite. The martensite phase in alloy little than austenite phase. So, the martensite transformation cannot be occurred.

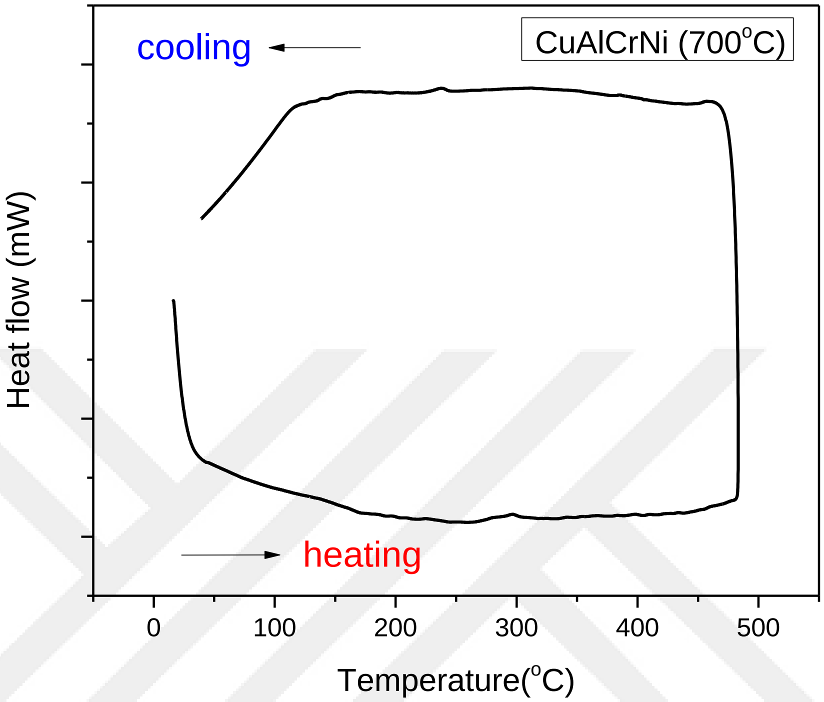


Figure 4.6. DSC curve heat treatment temperature at (700°C) of CuAlCrNi

CuAlCr alloy shows high temperature shape memory behaviors at heat treatment temperature 700°C. But CuAlCrNi didn't show any austenite and martensite transformation at this heat treatment that can be seen in Fig (4.7).

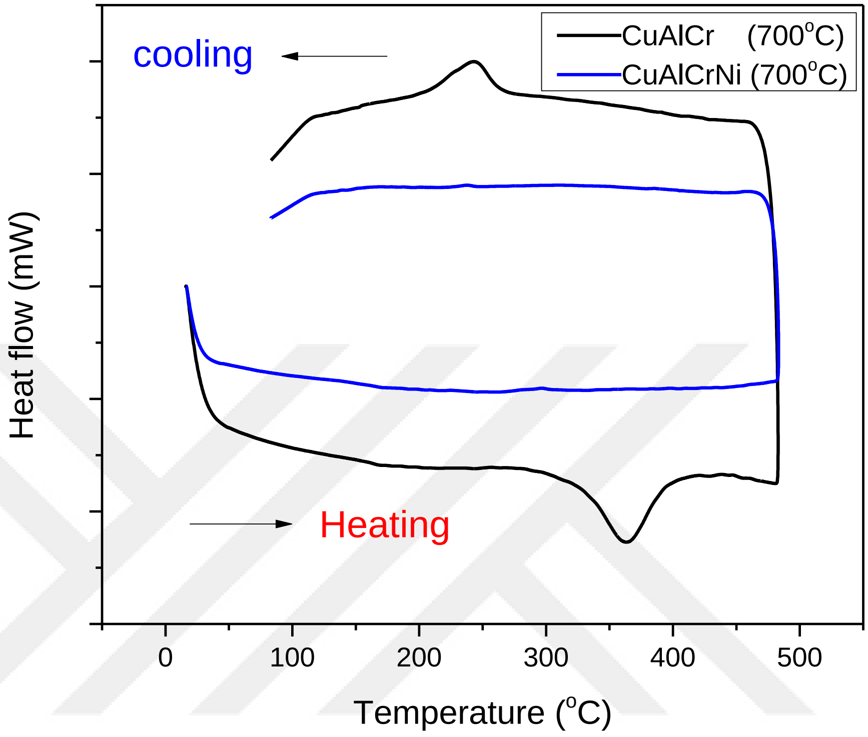


Figure 4.7. The comparison of DSC curves at (700^{o}C) of CuAlCr and CuAlCrNi

Other heat treatment temperature is 800^{o}C until 1 hour for CuAlCr alloy. Also at this temperature CuAlCr work as a high temperature shape memory alloys, austenite and martensite transformation of CuAlCr observed that can be seen in Fig (4.8). CuAlCr alloy has austenite start temperature A_s = 301.8^{o}C, austenite finish temperature A_f = 385.2^{o}C and martensite start temperature M_s = 326.4^{o}C, martensite finish temperature M_f = 215.5^{o}C as show in Table 4.2.

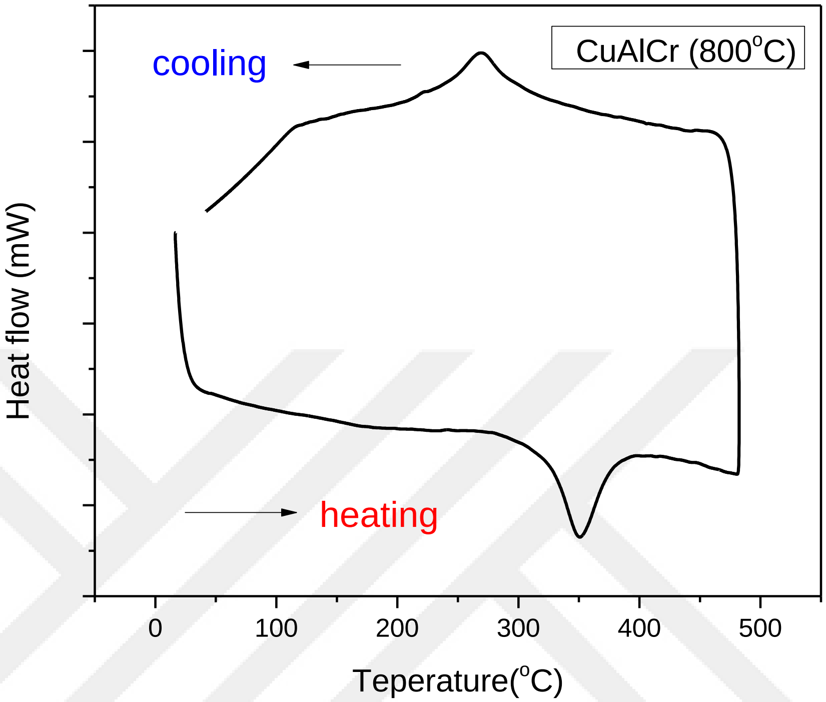


Figure 4.8. DSC curve heat treatment temperature at (800°C) of CuAlCr

Fig (4.9) shows the DSC curves of 1 hour heat treatment temperature at 800°C for CuAlCrNi alloy. As can be seen in this figure, heat treatment decrease transformation temperature of CuAlCrNi SMA. CuAlCrNi alloy has austenite start temperature A_s = 214.1°C, austenite finish temperature A_f = 280.3°C and martensite start temperature M_s = 223.5°C, martensite finish temperature M_s = 147.8°C as show in Table 4.2.

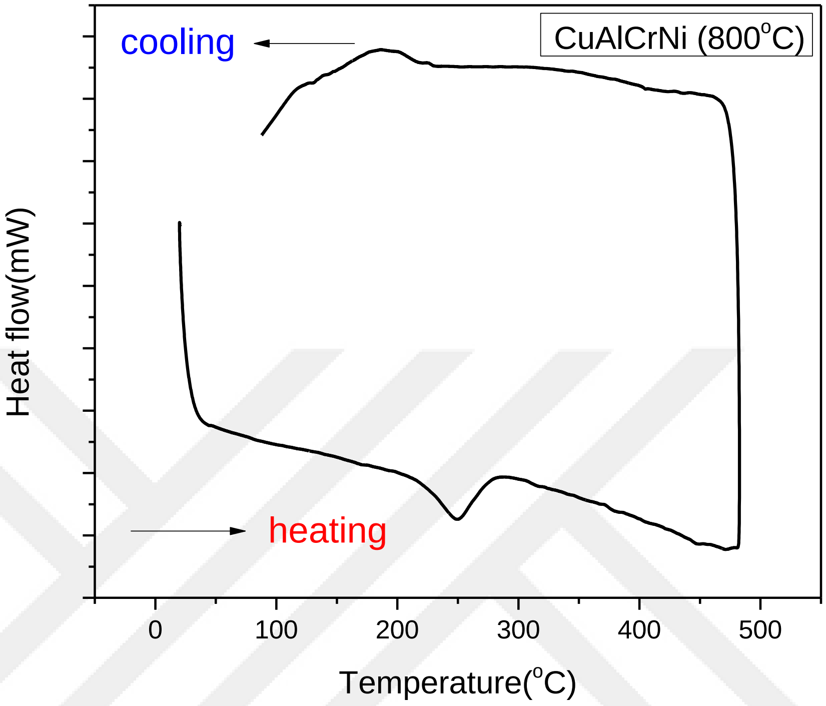


Figure 4.9. DSC curve heat treatment temperature at (800°C) of CuAlCrNi

The DSC curves were compared of CuAlCr and CuAlCrNi shape memory alloys, in Fig (4.10). After heat treatment at 800°C, it seen that the transformation temperature was decreased. Also, it can be said that the transformation temperature is decreased for CuAlCr alloys because of addition of (Ni)

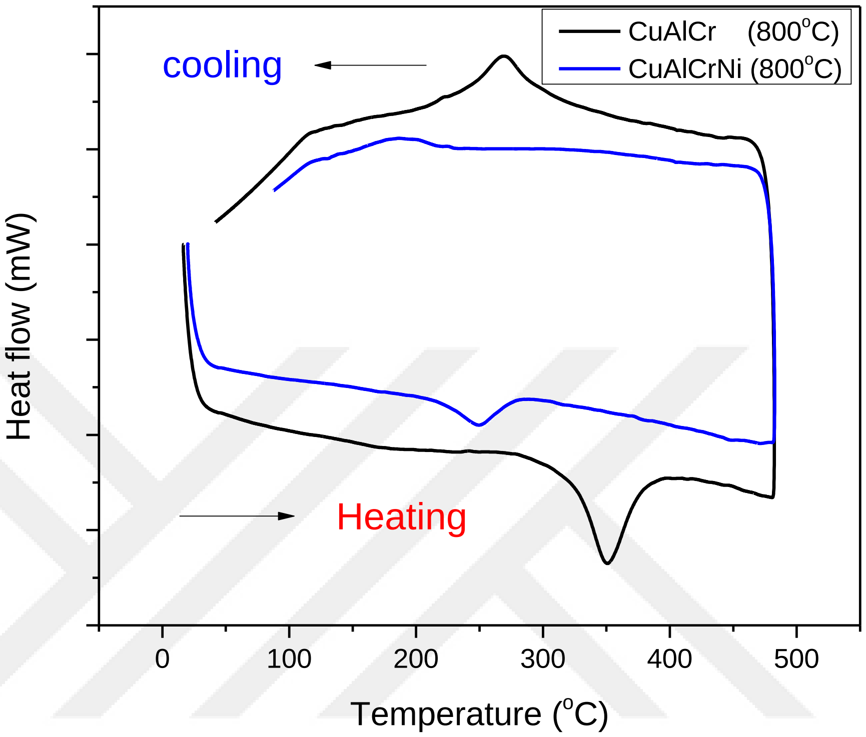


Figure 4.10. DSC curves results of CuAlCr and CuAlCrNi at 800°C

Thermal instability has been observed in some as-cast and heat treated $Cu_{85}Al_{12}Cr_3$ and $Cu_{83}Al_{12}Cr_3Ni_2$. When the transformation temperatures and transformation enthalpies change in each cycle notable, it is called unstable.

4.3. X-ray Measurement Result

X-ray measurement of CuAlCr and CuAlCrNi shape memory alloys were made at room temperature at the scanning rate of 20°-80°. The X-ray diffractograms for all alloys were indexed by literature [10].

Fig (4.11a) and (4.11b) show that X-ray diffractograms of CuAlCr and CuAlCrNi alloys which homogenized at 930°C for 12 hours. It can be seen that there are two phases in both of alloys. These phases names are γ'_1 and β'_1 phase. Both of phases are martensite and γ'_1 martensite phase is different from β'_1 phase. Because, martensite plates of γ'_1 is very thin when compared with β'_1 martensite phase. X-ray diffractograms of all alloy (homogenized at 930°C) almost same but CuAlCr alloy have new peak at the 80°. For CuAlCr alloy, intensity values are higher than CuAlCrNi alloy.

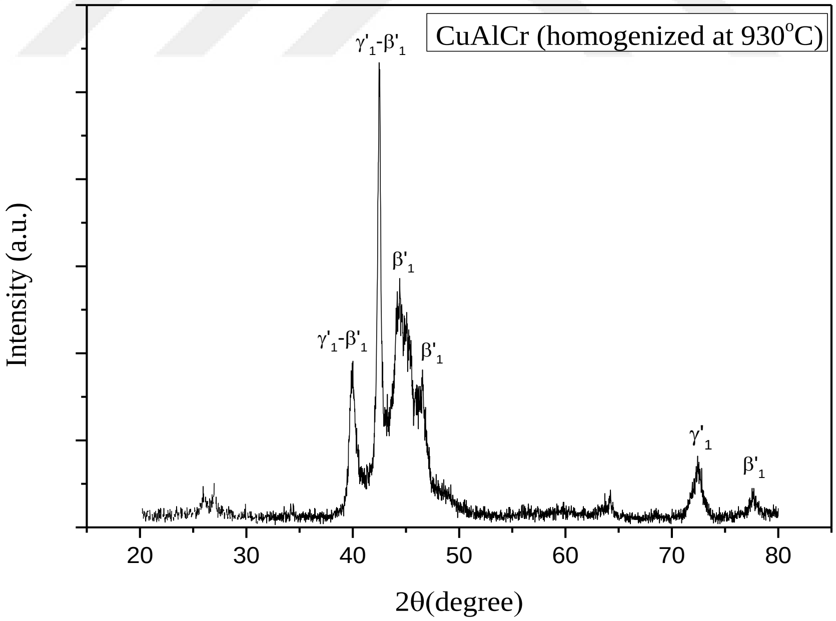


Figure 4.11a. XRD diffractogram of CuAlCr shape memory alloy

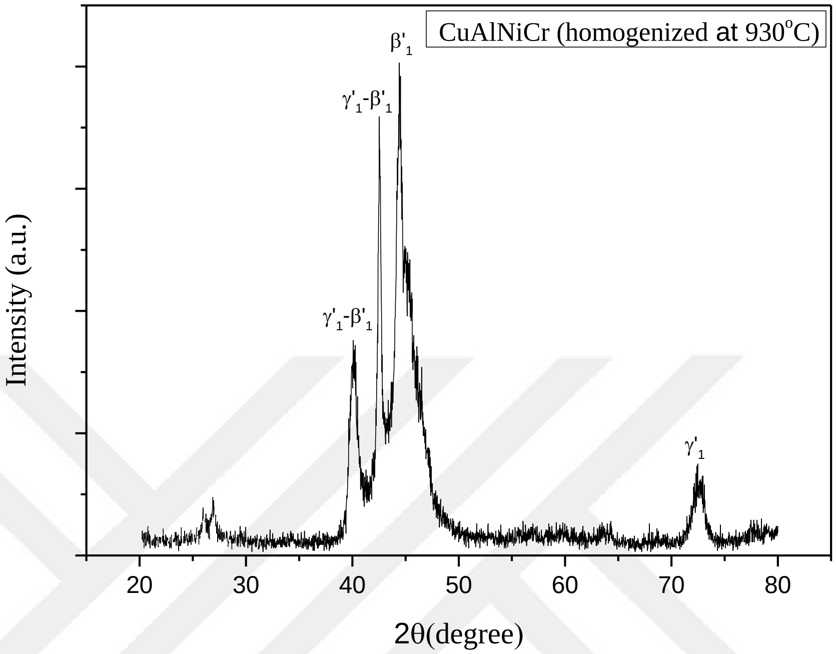


Figure 4.11b. XRD diffractogram of CuAlCrNi shape memory alloy

Fig (4.12a) and (4.12b) show x-ray analysis result of CuAlCr and CuAlCrNi shape memory alloys which exposed heat treatment at 700°C. There are two martensite phases named as γ'_1 and β'_1, too. If these X-ray diffractograms compared first group of alloys (Homogenized at 930°C for 12 hours) intensity values of alloys increased and all peaks of alloys between 40° and 50° became clear visible.

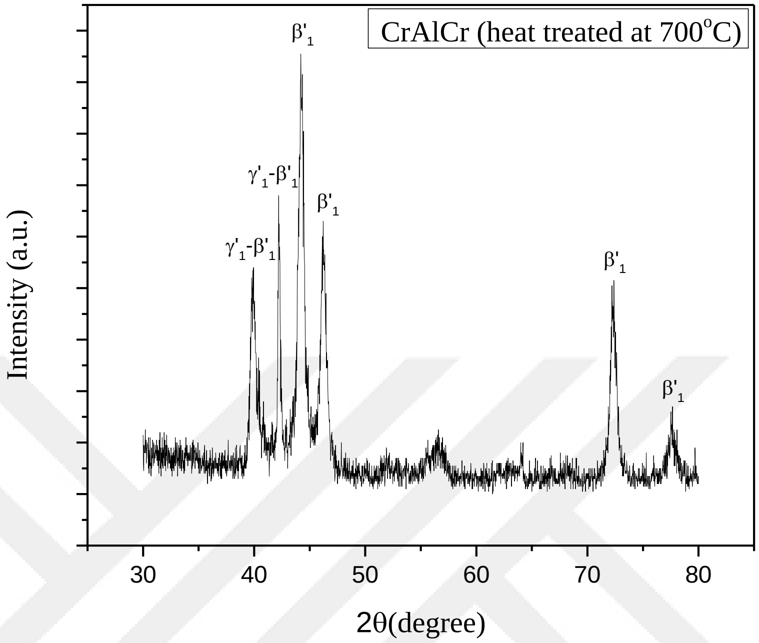


Figure 4.12a. XRD diffractogram of CuAlCr shape memory alloy heat treated at 700ºC

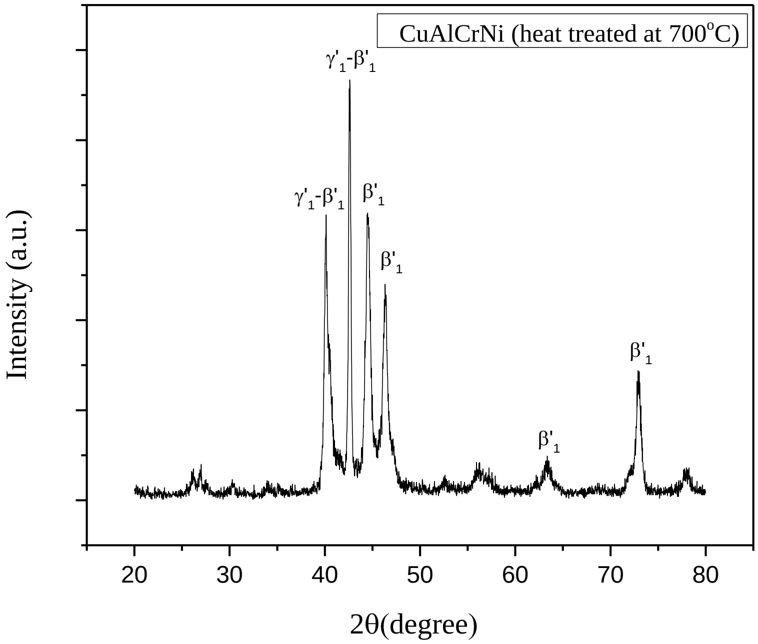


Figure 4.12b. XRD diffractogram of CuAlCrNi shape memory alloy heat treated at 700ºC

X-ray diffractograms of third group of alloys, which exposed to 800°C for an hour, can be seen in Fig (4.13a) and (4.13b). According to figures, 800°C heat treatment temperature was not change martensite phases in CuAlCr and CuAlCrNi alloys. There are thin and thick martensite phase in alloys. Intensity of alloys had highest value in this heat-treated temperature and peaks are very clear visible.

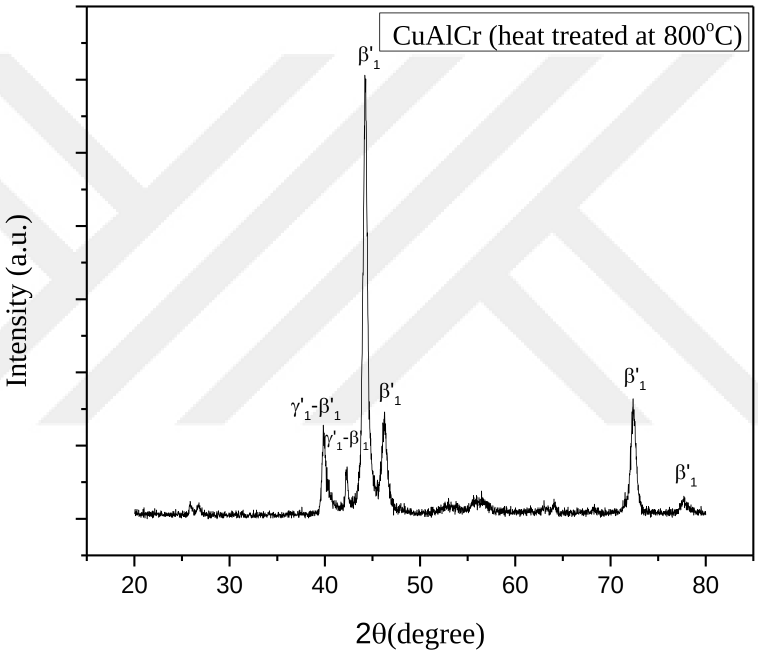


Figure 4.13a. XRD diffractogram of CuAlCr shape memory alloy heat treated at 800°C

According to XRD measurements, there group of CuAlCr and CuAlCrNi Shape memory alloys compared each other. Homogenized alloys peaks were not clear. When the alloys exposed heat treatment at 700°C and 800°C for an hour, peaks intensity was increased and distortion of X-ray value disappear and peaks seen clearly.

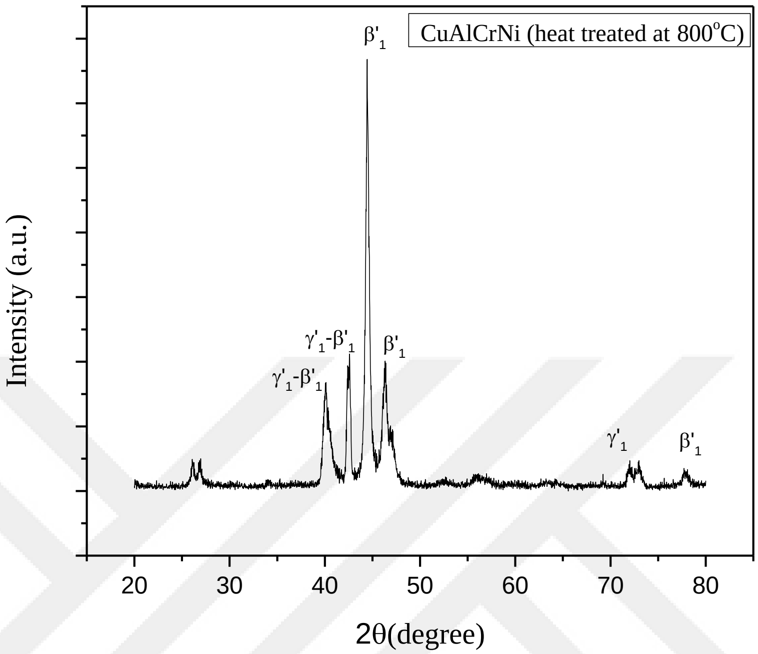
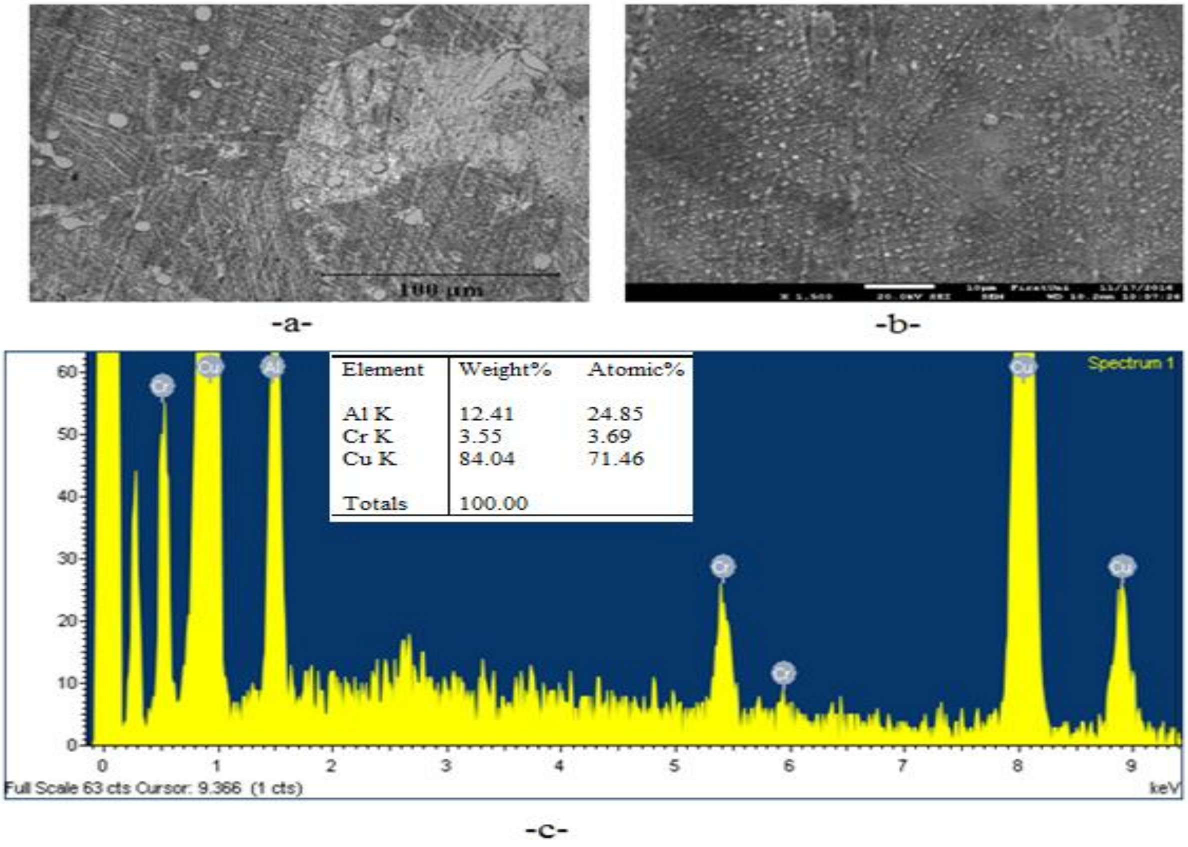


Figure 4.13b. XRD diffractogram of CuAlCrNi shape memory alloy heat treated at 800ºC

4.4 Optical and Scanning Electron Microscopy Measurement Result

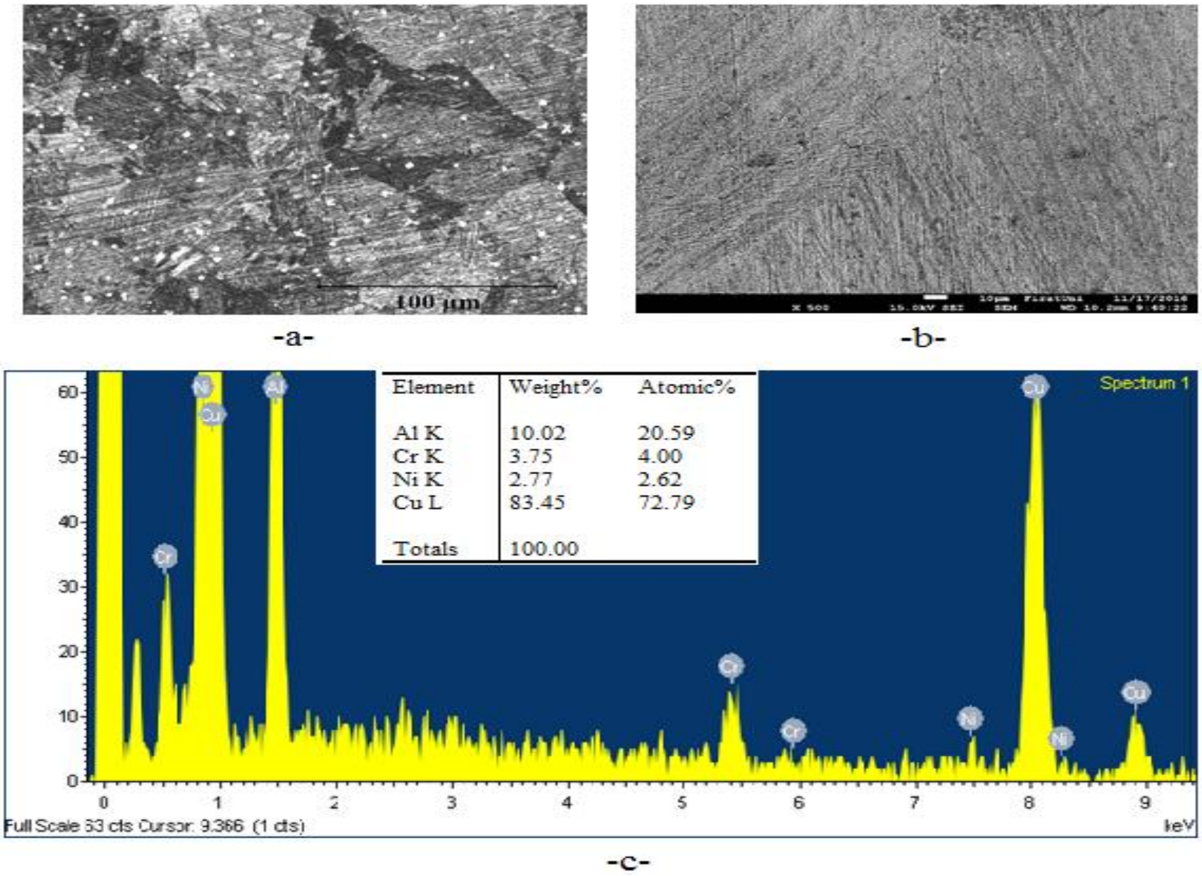
The microstructure of CuAlCr shape memory alloy quenched at 930ºC temperature is shown in Fig (4.14). As can be seen that in optical microscopy Fig (4.14a), there are a lot of grain in the structure, and these grains have martensite plate. It is also seen that there are many precipitates in the structure. These precipitates are Cr precipitates according to EDX analyses. It is seen in SEM microscopy that the grain border is clearly visible. Also, SEM microscopy have martensite plates. Additionally, as can be seen in the EDX result, the matrix of CuAlCr alloy contains 71.46%at Cu, 24.85%at Al, and 3.69%at Cr.



Element	Weight%	Atomic%
Al K	12.41	24.85
Cr K	3.55	3.69
Cu K	84.04	71.46
Totals	100.00	

Figure 4.14. Optical and Scanning Electron Microscopy result of CuAlCr at 930ºC (a) Optical microscopy (b) SEM microscopy (c) EDX analysis.

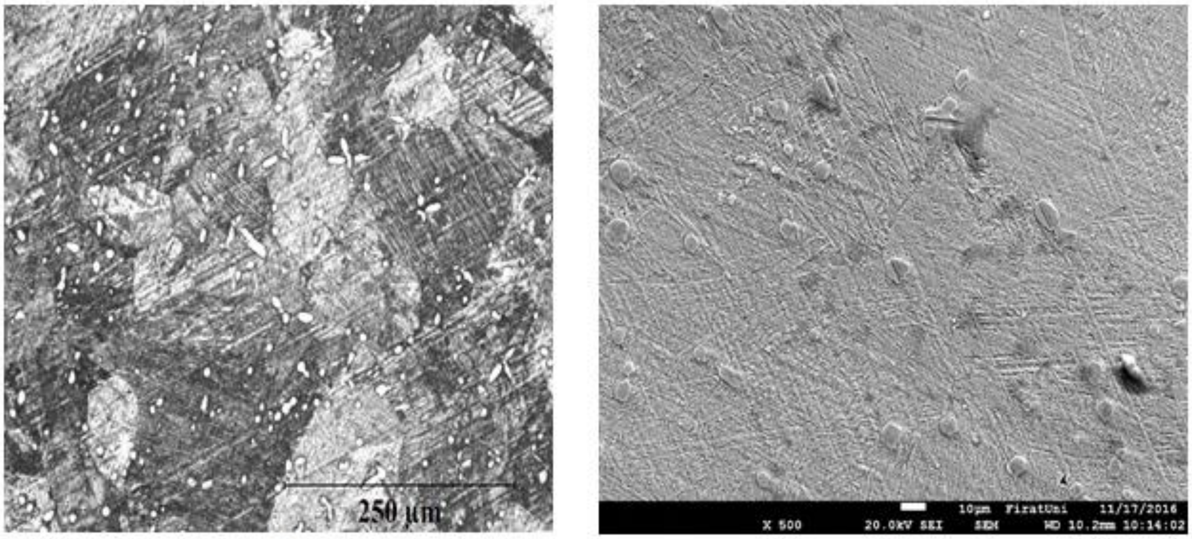
The microstructure of CuAlCrNi shape memory alloy quenched at 930°C temperature is shown in Fig (4.15). Optical microscopy shows that there are a lot of grain and precipitates in the structure more than CuAlCr alloy and this is due to addition of (Ni) that can be seen in Fig (4.15a), and these grains has a martensite plates. And these precipitates are Cr precipitates according to EDX analyses. It is seen in SEM microscopy that the grain border is clearly visible. Also, SEM microscopy has martensite plates as seen in Fig (4.15b). Additionally, as can be seen in the EDX result, the matrix of CuAlCrNi alloy contains 72.79%at Cu, 20.59%at Al, 4.00%at Cr and 2.62%at.



Element	Weight%	Atomic%
Al K	10.02	20.59
Cr K	3.75	4.00
Ni K	2.77	2.62
Cu L	83.45	72.79
Totals	100.00	

Figure 4.15. Optical and Scanning Electron Microscopy result of CuAlCrNi at 930°C (a) Optical microscopy (b) SEM microscopy (c) EDX analysis

Figure (4.16) and (4.17) show microstructure of CuAlCr and CuAlCrNi shape memory alloy quenched at 700ºC temperature. Optical microscopy shows that there are a lot of grains and precipitates in the both alloys in the structure as seen in Fig (4.16a) and (4.17a), but CuAlCrNi has a big grain compare with CuAlCr and a small proportion of precipitate. These grains have a martensite plates as a parallels palate. And these participates are Cr participates according to EDX analyses. It is seen in SEM microscopy that the grain border is clearly visible. Also, SEM microscopy has martensite plates as seen in Fig (4.16b) and (4.17b).



-a- -b-

Figure 4.16. Optical and Scanning Electron Microscopy result of CuAlCr at 700ºC (a) Optical microscopy (b) SEM microscopy

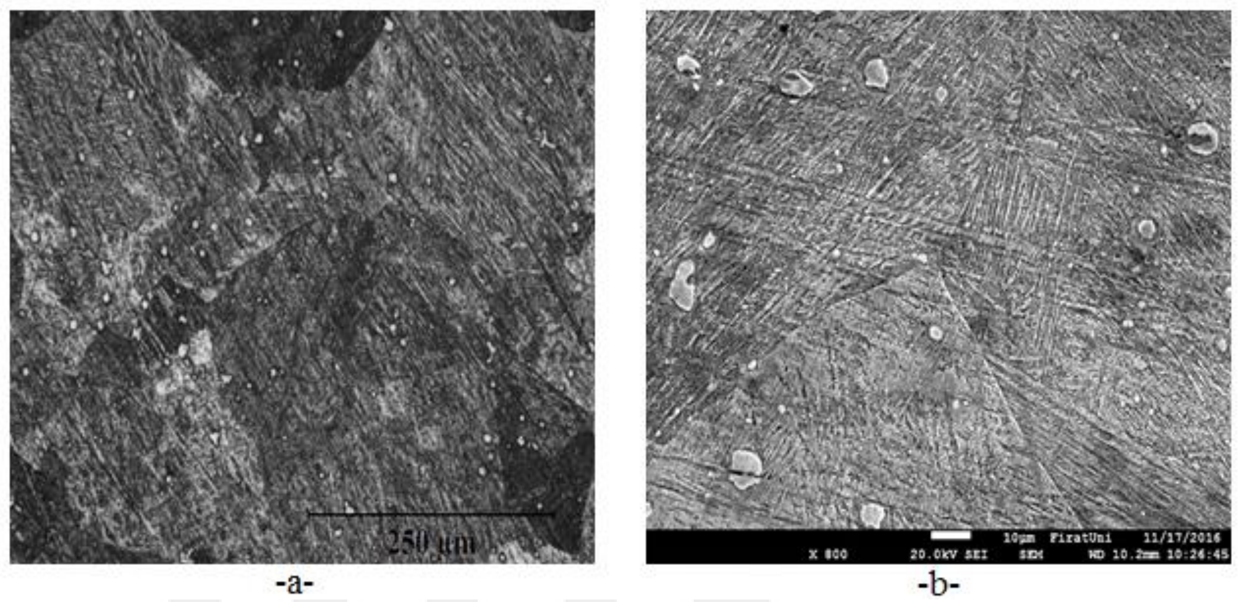
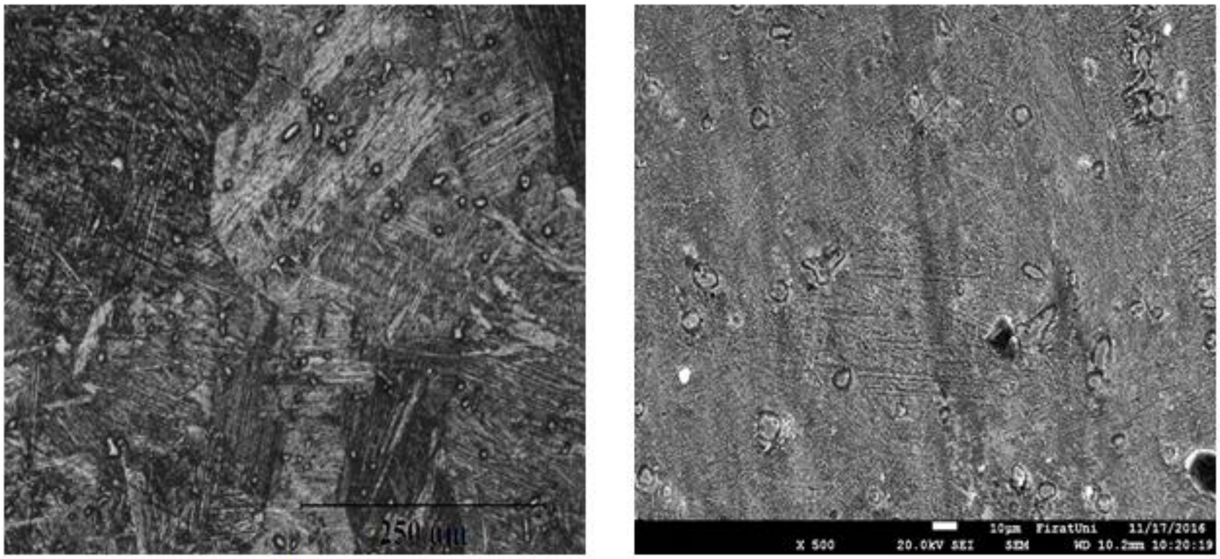


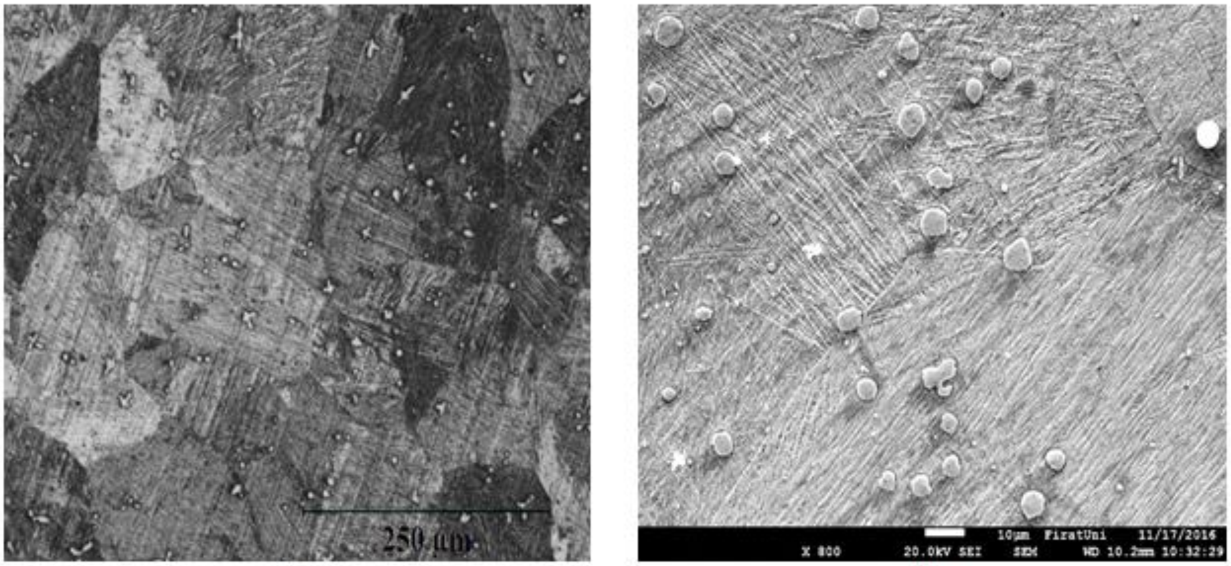
Figure 4.17. Optical and Scanning Electron Microscopy result of CuAlCrNi at $700^{o}C$ (a) Optical microscopy (b) SEM microscopy

Microstructure of of third group of alloys CuAlCr and CuAlCrNi, which exposed to $800^{o}C$ temperature that can be seen in Fig (4.18) and (4.19). optical microscopy shows that there are a lot of grains and precipitates in the both alloys in the structure as seen in figure (4.18a) and (4.19a) These grains have a martensite plates. And these participates are Cr participates according to EDX analyses. It is seen in SEM microscopy that the grain border is clearly visible. Also, SEM micrmisgscopy has martensite plates as seen in Fig (4.18b) and (4.19b).



-a- -b-

Figure 4.18. Optical and Scanning Electron Microscopy result of CuAlCr at 800ºC (a) Optical microscopy (b) SEM microscopy



-a- -b-

Figure 4.19. Optical and Scanning Electron Microscopy result of CuAlCrNi at 800ºC (a) Optical microscopy (b) SEM microscopy

4.5. Hardness Measurement Results

Vickers hardness test set with 0.5kg for 10sec was performed to measure the hardness of these alloys are given in Table 4.3.

First hardness test at heat treatment temperature 930^{o}C show that CuAlCr alloy have 289 HV. But the hardness value decrease for CuAlCrNi alloy, its 263 HV in the same heat treatment that can be seen in Table 4.3.

Second group of alloys, which exposed to 700^{o}C for an hour, can be seen in Table 4.3. CuAlCr alloy have 235.8 HV. Also, the hardness value decrease to 230.6 HV for CuAlCrNi alloy in the same heat treatment.

Last heat treatment of alloys, which exposed to 800^{o}C for an hour, can be seen in Table 4.3. CuAlCr alloy have 261.2 HV. Also, the hardness value decrease to 229.4 HV for CuAlCrNi alloy in the same heat treatment. In all heat treatment, we note that the addition of (Ni) decrease hardness value of CuAlCr.

Table 4.3. Hardness value of CuAlCr and CuAlCrNi at different heat treatment

Sample	Hardness (HV)
CuAlCr (930^{o}C)	289
CuAlCrNi (930^{o}C)	263
CuAlCr (700^{o}C)	235.8
CuAlCrNi (700^{o}C)	230.6
CuAlCr (800^{o}C)	261.2
CuAlCrNi (800^{o}C)	229.4

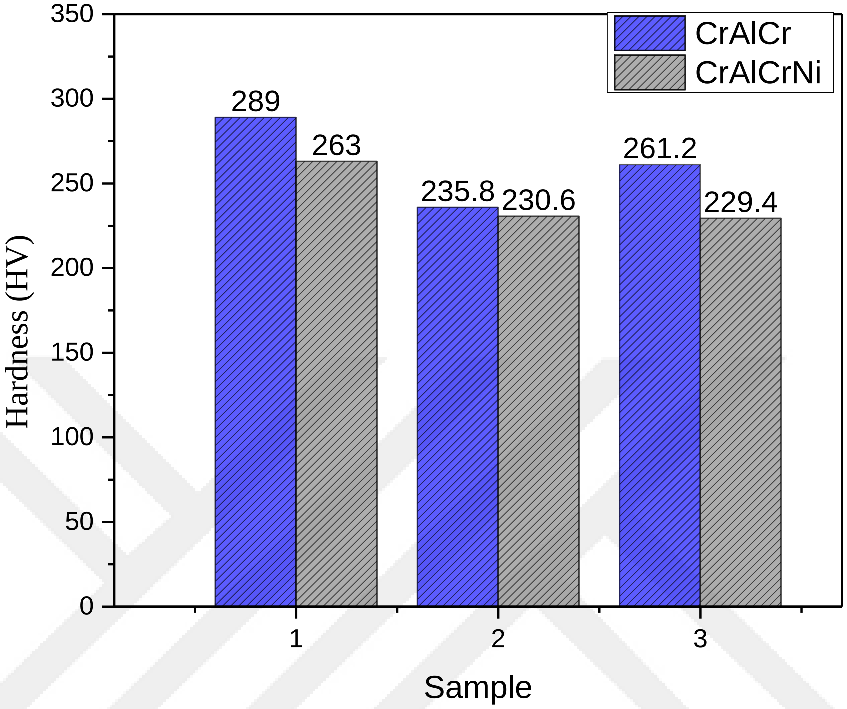


Figure 4.20. The hardness result of CuAlCr and CuAlCrNi SMAs (Sample 1; main Sample, Sample 2; heated treatment at 700ºC, Sample 3; heated treatment at 800ºC)

5. CONCLUSION

CuAlCr and CuAlCrNi high temperature shape memory alloys were produced by arc-melter furnace. For the homogenization, all of alloys subjected to long time heat treatment at 930^{o}C. Then, alloys cut and divided two part. One part alloys subjected to 700^{o}C for one hour and quenching into ice brined water. Other parts of alloys subjected to 800^{o}C for one hour and quenching into ice brined water. These three type of alloys properties were examined by DSC, XRD, optical microscope and SEM-EDX measurement. The results can be explained as follow:

- In DSC (differential Scanning Calorimeter) measurement, CuAlCr and CuAlCrNi alloys show high temperature shape memory effect. Heat treat effect changed transformation temperature of alloys expect for CuAlCrNi HTSMA for 700^{o}C. The reason of no transformation can be explained as high volume austenite phase.
- XRD measurement of all alloys were measured at room temperature. Alloys exhibited to two martensite phases. One of thin plate martensite named as γ'_1 phase other is named as β'_1 phases with thick martensite plate. By applying heat treatment at 700^{o}C and 800^{o}C, the intensity value of xrd increased and there was a lot of peaks.
- In optical image and SEM image, it can be seen that there are thin and thick martensite plate and addition of this there are precipitates. In long term homogenization at 930^{o}C, CuAlCr and CuAlCrNi can be compared each other. The grain size of CuAlCr alloys bigger than CuAlCrNi and the precipitates number of CuAlCrNi more than CuAlCr alloys. According to EDX measurement, content of precipitates is Cr element.
- According to hardness measurements, by heat treatment hardness value of CuAlCr and CuAlCrNi shape memory alloys decreased. In CuAlCrNi the hardness values are almost same for two heat treatment temperature. In CuAlCr alloys, At 700^{o}C heat treatment temperature, hardness value smaller than at 800^{o}C heat treatment temperature.

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