

## Effect on Thermal and Structural Properties of Element Content in CuAlBe Shape Memory Alloys Irradiated with a Constant Gamma Radiation Dose

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**Abstract:** Since gamma radiation is a type of radiation that can change the structural properties of materials, CuAlBe shape memory alloy with two different weight percentages was used in this study. CuAlBe shape memory alloys were irradiated with a constant gamma radiation dose of 40 kGy, and the resulting thermal and structural changes in the alloys were investigated. Changes in enthalpy and in the transformation temperature of the alloys were determined by differential scanning calorimetry (DSC), and thermodynamic parameters of alloy samples were calculated. Microstructural changes were determined by X-ray analysis. Microstructural changes were verified by metallographic observations, and microhardness measurements were taken. The study investigated to what extent the physical parameters of CuAlBe shape memory alloys changed depending on the alloying elements when subjected to a constant irradiation dose.

**Key words:** CuAlBe shape memory alloy, gamma radiation, microhardness.

### Sabit Gama Radyasyon Dozu ile Işınlanan CuAlBe Şekil Hatırlamalı Alaşımlarda Element İçeriğinin Termal ve Yapısal Özellikleri Üzerine Etkisi

**Öz:** Gama radyasyonu malzemelerin yapısal özelliklerini değiştirebilecek nitelikte bir radyasyon çeşidi olduğu için, bu çalışmada iki farklı ağırlık yüzdesine sahip CuAlBe şekil hatırlamalı alaşımı kullanıldı. CuAlBe şekil hatırlamalı alaşımları, 40 kGy sabit gama radyasyon dozu ile ışınlanmış ve bunun sonucunda alaşımlarda meydana gelen termal ve yapısal değişiklikler incelenmiştir. Alaşımların entalpi ve dönüşüm sıcaklığındaki değişimler, diferansiyel taramalı kalorimetri (DSC) ile belirlendi ve alaşım numunelerinin termodinamik parametreleri hesaplandı. Mikroyapısal değişiklikler, X-ışını analizi ile belirlendi. Mikroyapısal değişiklikler metalografik gözlemlerle doğrulandı ve mikrosertlik ölçümleri alındı. Çalışmada CuAlBe şekil hatırlamalı alaşımların sabit ışınlama dozuna maruz bırakıldığında alaşım elementlerine bağlı olarak fiziksel parametrelerinin ne ölçüde değiştiği araştırılmıştır.

**Anahtar kelimeler:** CuAlBe şekil hatırlamalı alaşım, gama radyasyonu, mikrosertlik.

#### 1. Giriş

One of the most obvious effects of irradiation on metals and alloys is the displacement of the atoms that make up the structural components. The materials used in reactor systems are predominantly crystalline alloys. Radiation is simply a form of energy that exists in motion. The types of radiation that can change structural materials are ionizing radiation. By ionizing radiation is meant rays of very small wavelengths or very high frequency and energy sources of radiation. Ionizing radiations consist of neutrons, ions, electrons, and gamma rays. All these forms of radiation have the ability to displace atoms from their lattice regions, which drives changes in metals and alloys [1,2].

In addition, two types of high-energy radiation or beam sources are used on the basis of advanced, innovative, evolutionary nuclear industrial applications. One of them is radioactive isotopes such as Co-60 and Cs-137 that emit gamma radiation, and the other is electron beam accelerators where high-energy electrons are produced. The substance irradiated with these two types of ionizing radiation device or ionizing radiation source, which are completely different from each other, does not turn into radioactive material, radioisotope sources, and radiation sources [3,4].

It is important to have a clear understanding of the effect of radiation on materials in order to take into account the effects of irradiation in material design and to develop materials that are more resistant to radiation. The application of shape memory material technology in the nuclear industry offers new opportunities for safety in this

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industrial sector, such as reducing personal exposure, life cycle cost reduction, and performance improvement [1,5].

Shape memory alloys have the ability to undergo reversible thermoelastic phase transformations from main phase austenite to product phase martensite and from product phase martensite to main phase austenite. This particular behavior accounts for their two main functional properties: superelasticity and the shape memory effect. Shape memory alloys are often used as miscellaneous materials in many applications due to these unique properties and behaviors. With these materials, extraordinary solutions to important engineering problems are made possible, thanks to their unique properties. The fields of application are very diverse and are being expanded continuously. Some include space technology, medicine, robotics and energy engineering among others [6-9].

Among shape memory alloys, Cu-based shape memory alloys, which are used as an alternative to NiTi shape memory alloys, are preferred because of their high electrical and thermal conductivity, good damping and vibration resistance, high transformation temperature properties, and their low cost and ease of manufacture. The Cu-based shape memory alloys can transform from the ordered DO<sub>3</sub> structure to a long-period stacking structure known as 18R. Also, they can achieve shape recovery in the crystallization range known as the  $\beta$ -phase, which contains an irregular body-centered cubic (BCC) structure and can be observed at high temperature during the heating process [10-13].

These properties make Cu-based alloys useful for high temperature applications. In particular, Cu-Al-Ni shape memory alloys exhibit shape memory features around 200 °C. The addition of alloying elements, heat treatment and numerous irradiation types with different parameters can be applied to partially affect the transformation temperatures, phase sequences and morphologies, which improve the performance of the alloy. Gamma irradiation is considered to be the most vital type of exposure because it can pass through protective layers and penetrate materials. Nevertheless, details on the effects of gamma ( $\gamma$ ) irradiation on shape memory alloys are very useful for practical applications in the field of irradiated environments [10].

CuAlBe system has been developed as a shape memory alloy. CuAlBe shape memory alloys are of interest for medium and low-temperature applications. By adding the element beryllium (Be) to the eutectoid CuAl-system, the austenite and martensite phase transformation temperatures decrease without changing the concentration of the eutectoid point. Minute changes in the amount of Be in the CuAlBe shape memory alloys alter the transformation temperatures drastically. Therefore, it enables alloys to be designed over a wide transformation temperature range. Also, it becomes easier to obtain pseudoelastic behavior at room temperature with decreasing conversion temperature. These alloys have a lot of potential for use as passive seismic energy dampers in building structural frames or bridges. [14-16].

In a study by Balo and Eskil, it was observed that a non-monotonic changes in characteristic transformation temperatures with increasing radiation dose in CuAlBe alloy irradiated with different radiation dose, as well as a decrease in crystal size and an increase in microhardness value. The increase in microhardness, ie radiation hardening, is a result of the formation of point defects in the metal structure. Some changes in sample properties were noted with increasing radiation dose [17].

The application of SM material technology in the nuclear industry may offer new opportunities. SMA's have been used as active elements in mechanical devices for monitoring nuclear facilities [18]. SMA technology can be designed to identify specific irradiation areas where applications are practical. Setting critical temperatures has become an important field of study for the use of alloys in targeted technological applications. Adjusting these temperatures to the required values can be accomplished by changing the alloying elements or their composition. It is hoped that the results of our study will shed light on technological applications.

In this study, we focused on the effects on transformation temperatures, thermodynamic parameters, microstructure, and microhardness of CuAlBe shape memory alloys with different weight percentages irradiated with 60 Co gamma radiation source. The study was planned in two phases to observe the effects of irradiation. (1) pre-irradiation measurements and (2) post-irradiation measurements.

In the literature, no study investigating the effect of alloying elements on the physical behavior of Cu-based shape memory alloys irradiated with gamma radiation has been found.

## 2. Experimental

CuAlBe alloys of different weight percentages were obtained from the Tréfinmétaux Research Center in France. Table 1 summarises the compositions of the alloy samples. The parts cut from the alloys were exposed to heat treatment at a temperature in the  $\beta$  phase region. This region was determined from the equilibrium diagrams of CuAlBe. Details of the heat treatment have been provided in previous studies [14,19,20]. In Saraykoy Nuclear Research and Training Center (SANAM), Turkey Atomic Energy Agency was irradiated with 40 kGy dose gamma

radiation some of the heat-treated (homogenous) samples. The rate of the irradiation dose was set at 1273 Gy/hr. The irradiation duration was 31.42 hours, and the irradiation dose was controlled automatically by the irradiation system. Using Perkin Elmer 8000 Differential Scanning Calorimetry (DSC), the transformation temperatures of the unirradiated and irradiated samples were determined. DSC measurements of the unirradiated alloy samples were carried out at a scanning speed heating/cooling of 10°C/min under atmospheric pressure in the appropriate temperature range. X-ray diffraction patterns of the samples were measured with a Bruker AXS D8 Advance Model XRD meter in the range of 30° to 90°. X-ray analyses of the alloy samples were performed using CuK $\alpha$  radiation at room temperature. The wavelength of the X-rays was 1.54060 Å. Optical microscope observations were made following polishing and chemical etching processes that were applied to the alloy samples. Microhardness measurements were then recorded.

**Table 1.** Composition (wt%) of the investigated CuAlBe alloys.

Alloy	Al (wt%)	Be (wt%)
CAB1	11.8	0.47
CAB2	11.6	0.42

### 3. Results and Discussions

#### 3.1. Differential scanning calorimetry measurements

Differential scanning calorimetry (DSC) was used to determine the endothermic and exothermic transformation enthalpies and transformation temperatures of the alloy samples that were unirradiated and irradiated with a constant gamma irradiation dose. The transformation temperatures of the alloys used in this study are given in Table 2. The transformation temperatures of the alloy samples,  $A_s$ ,  $A_f$ ,  $M_s$  and  $M_f$  were determined using the tangent method. Here, the subindexes A austenite, M martensite, s, and f indicate the starting and ending temperatures [21,22]. The samples put in the DSC for calorimetric analysis were obtained at a 10°C/min heating–cooling rate in under atmospheric pressure atmosphere. The DSC curves for the CAB1 alloy samples were obtained by increasing the temperature from –40°C to 30°C and back to –40°C over a certain period of time. The DSC curves for the CAB2 alloy samples were obtained by increasing the temperature from 30°C to 110°C and back to 30°C over a certain period of time. Considering the results obtained, it was observed that phase transformation temperatures can change depending on the 40 kGy constant irradiation dose level applied to the alloy samples. An increase in transformation temperatures was observed of the samples irradiated with constant 40 kGy gamma irradiation.

The DSC curves of the unirradiated and irradiated samples of the CAB1 and CAB2 alloys are given in Fig. 1 and Fig. 2, respectively. As seen in Table 1, when the Al weight percent was decreased from 11.8% to 11.6% and the Be percent was decreased from 0.57 to 0.47 in the CAB2 alloy, the transformation temperatures increased. Transformation temperatures were observed to increase to positive temperature values. The thermodynamic parameters of the alloys used in this study are given in Table 3. The decrease in Al and Be percentages also increased the equilibrium temperature,  $T_0$ .

The equilibrium temperature,  $T_0$ , is defined as the critical temperature at which the Gibbs free energies of the austenite and martensite phases are equal. It is used to calculate the entropy values,  $\Delta S_{M \rightarrow A}$  in reverse transformations. The area under the endothermic and exothermic peaks in the DSC curves gives the enthalpy values,  $\Delta H_{M \rightarrow A}$  and  $\Delta H_{A \rightarrow M}$ . To compute the values of  $T_0$  parameter the following relations were used [20,23-27].

$$\Delta S_{M \rightarrow A} = \Delta H_{M \rightarrow A} / T_0 \quad (1)$$

$$T_0 = (A_f + M_s) / 2 \quad (2)$$

The supercooling  $T_0 - M_s$  represents the hysteresis in the transformation, which is characterized by the driving force for the nucleation of martensite,  $\Delta G^{A \rightarrow M}$  as,

$$\Delta G^{A \rightarrow M}(M_s) = \Delta G^{M \rightarrow A}(T_0) - \Delta G^{M \rightarrow A}(M_s) = -(T_0 - M_s) \Delta S^{M \rightarrow A} \quad (3)$$

The elastic energy  $G_E$  stored in the self-accommodated martensitic variations is related to the difference  $M_f - M_s$  by

$$\Delta G_E = \Delta G^{A \rightarrow M}(M_s) - \Delta G^{A \rightarrow M}(M_f) = (M_s - M_f)\Delta S^{M \rightarrow A} \quad (4)$$

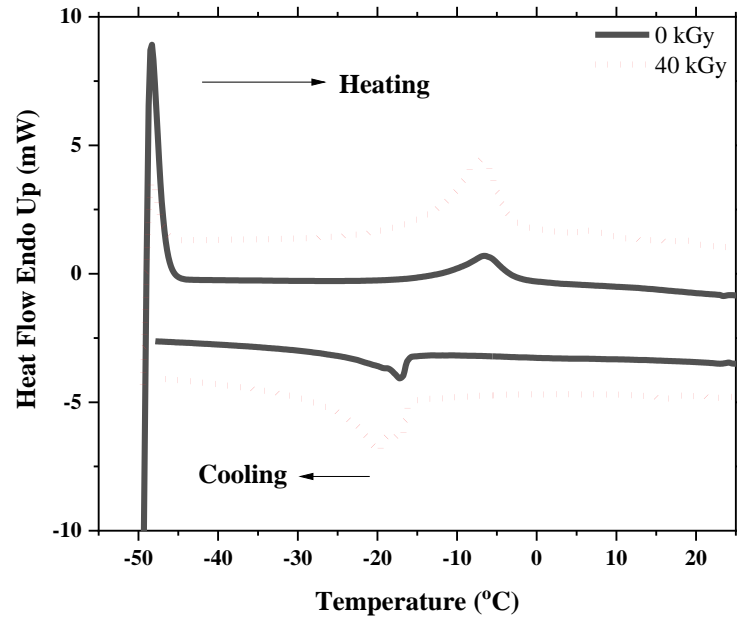
Entropy is the state of disorder. In martensite  $\rightarrow$  austenite ( $M \rightarrow A$ ) transformations of the unirradiated and irradiated alloy samples were changed entropy values. The variation of irregularity (entropy) in the unirradiated and irradiated alloy samples showed that the alloys did not remain stable. Entropy increased in the irradiated CAB1 alloy and decreased in the CAB2 alloy. At the same time, the entropy of the irradiated CAB2 sample is smaller than the entropy values of the unirradiated CAB1, irradiated CAB1, and unirradiated CAB2 samples. In other words, the irradiated CAB2 sample has a more stable structure. Similar changes are also observed in the elastic energy. The elastic energy of the alloy sample with a small entropy is also small. Elastic energy varies inversely with Gibbs free energy.

**Table 2.** Characteristic transformation temperatures unirradiated and irradiated CuAlBe shape memory alloy samples at a 10°C/min heating-cooling rate.

Dose (kGy)	Alloys	$M_s$ (°C)	$M_f$ (°C)	$A_s$ (°C)	$A_f$ (°C)
0	CAB1	-16.02	-20.79	-12.88	-1.73
40	CAB1	-15.92	-24.91	-13.59	-2.73
0	CAB2	65.66	57.23	74.28	91.23
40	CAB2	68.34	62.63	73.49	93.39

**Table 3.** Thermodynamic parameters of unirradiated and irradiated CuAlBe shape memory alloy samples at a 10°C/min heating-cooling rate.

Dose (kGy)	Alloys	$T_0$ (K)	$\Delta H_{M \rightarrow A}$ (J/kg)	$\Delta H_{A \rightarrow M}$ (J/kg)	$\Delta S_{M \rightarrow A}$ (J/kgK)	$\Delta G_{A \rightarrow M}$ (J/kg)	$\Delta G_E$ (J/kg)
0	CAB1	264.12	4464.7	-2411.1	16.90	-120.66	80.61
40	CAB1	263.67	5656.3	-4190.5	21.45	-141.35	192.83
0	CAB2	351.45	7871.4	-4549.7	22.39	-286.36	188.74
40	CAB2	353.86	4708.7	-3791.7	13.30	-166.51	75.94



**Fig. 1.** DSC curves of the CAB1 alloy sample at a) unirradiated and b) irradiated.

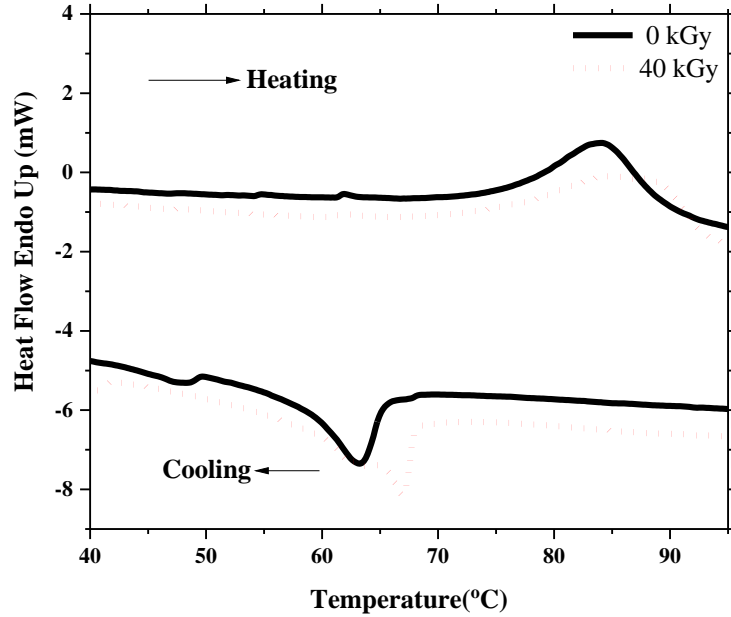


Fig. 2. DSC curves of the CAB2 alloy sample at a) unirradiated and b) irradiated.

### 3.2. X-ray diffraction (XRD) results

X-ray patterns were used to analyze the structures of samples unirradiated and irradiated with 40 kGy gamma radiation. X-ray analyses of CuAlBe shape memory alloy samples were performed at room temperature with CuK $\alpha$  radiation. The XRD patterns of the CAB1 alloy samples that were unirradiated and irradiated with 40 kGy radiation dose are given in Fig. 3a and 3b. In Fig. 3a, the basic peak (220) and (400) (331) peaks of the austenite structure were observed. The peak (220) is at maximum intensity. In addition, the peaks of the martensite structure (122) and very weakly, (320) were observed. In Fig. 3b, the  $\beta$  peak (331) and (511) are very weak, and  $\beta'$  peak at maximum intensity (320) is observed. Diffraction patterns of the CAB2 alloy samples are given in Fig. 4a and 4b. They only show the peaks belonging to the martensite structure. Peak intensities changed with the application of the irradiation at 40 kGy [28-30]. Unirradiated CAB1 alloy exhibited peaks of the martensite phase as well as austenite phase, depending on the cooling rate. According to the phase diagram given in the literature, the CuAl alloy of near eutectoid composition (Cu-11.6-11.8wt. %Al) tends to form an austenite phase above 560°C [15,28].

The crystallite size for the alloy samples were calculated using the Debye Scherrer equation [31-33]:

$$D = \frac{0.9\lambda}{FWHM \cos\theta} \quad (3)$$

where D is crystallite size,  $\lambda$  is the X-ray wavelength, *FWHM* is the full width at half the maximum peak and  $\theta$  is the Bragg angle.

Crystallite sizes for the alloy samples are given in Table 4. As seen from this table, the crystallite size increased when irradiation was applied at the dose of 40 kGy to the CAB1 alloy. The CAB2 alloy shrank following the 40 Gy irradiation dose and produced the smallest crystallite size.

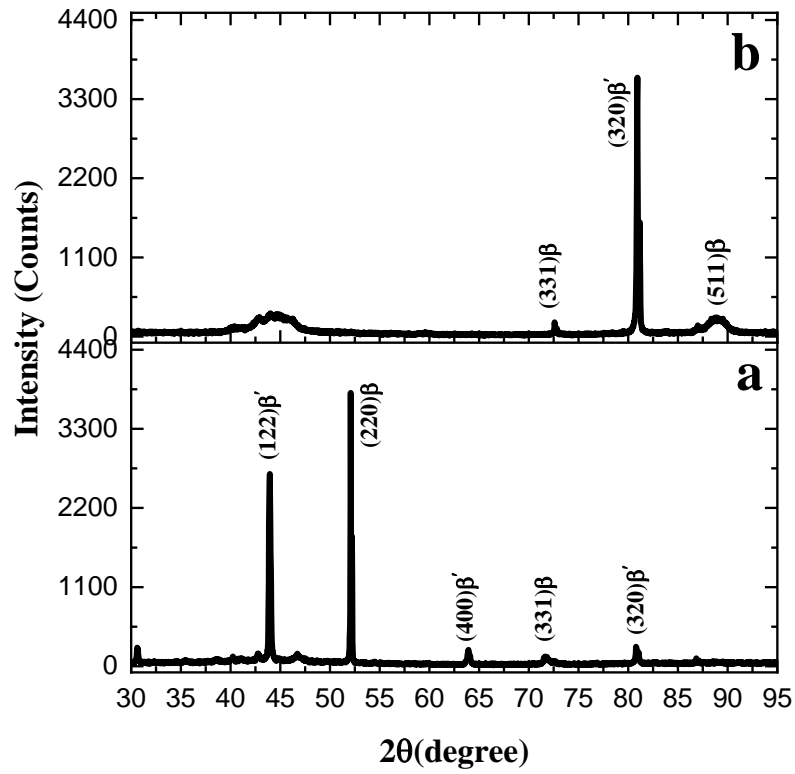


Fig. 3. X-ray diffraction patterns of CAB1 alloy sample a) unirradiated b) irradiated.

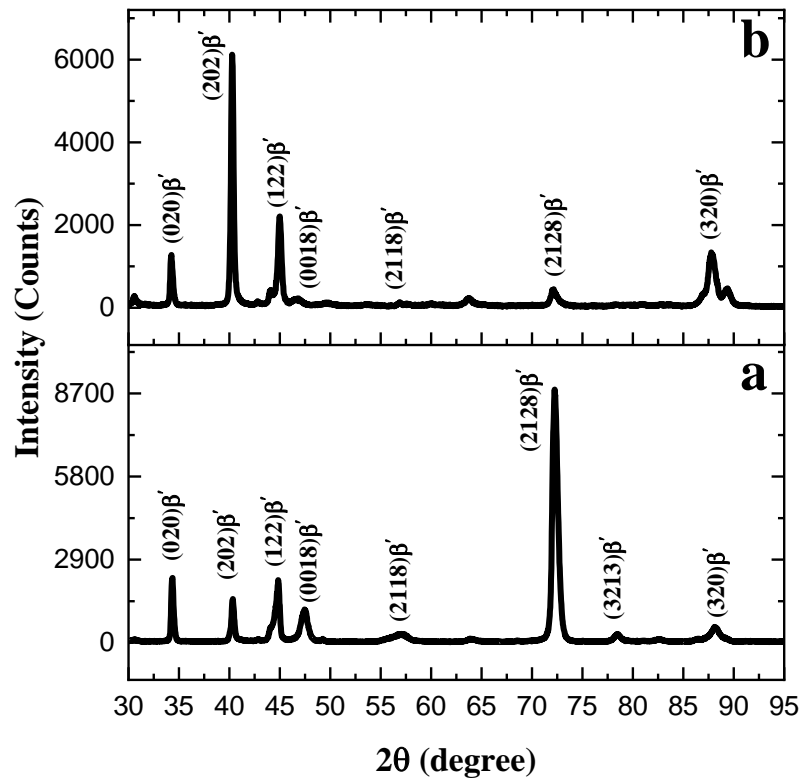


Fig. 4. X-ray diffraction patterns of CAB2 alloy sample a) unirradiated b) irradiated.

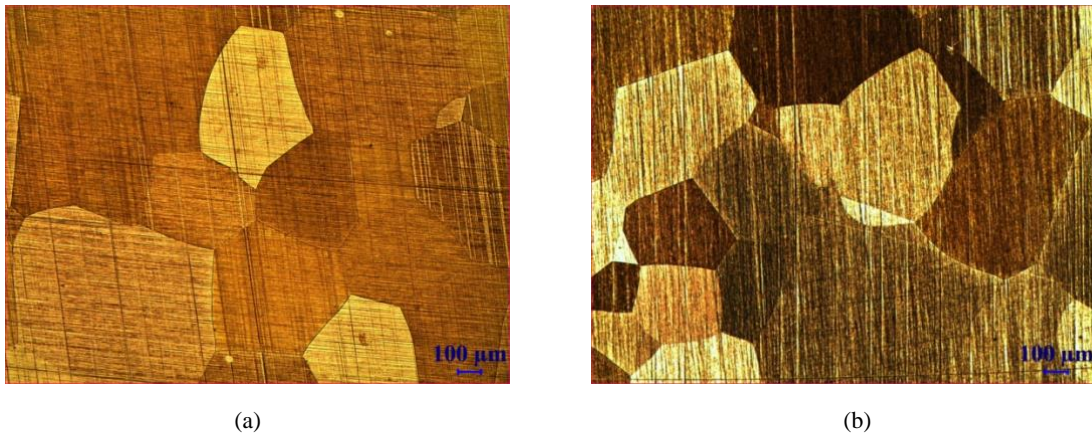
### 3.3. Metallographic observations and microhardness results

Optical images of the CAB1 alloy sample are given in Fig. 5a and 5b. As can be seen from the images, the alloy has a polycrystalline structure. Although the alloy seems to exhibit austenite structure at room temperature, there are still untransformed martensite areas [34,35]. More grains are seen in the surface photograph of the sample irradiated with 40 kGy radiation dose at the same magnification and the amount of martensite has increased.

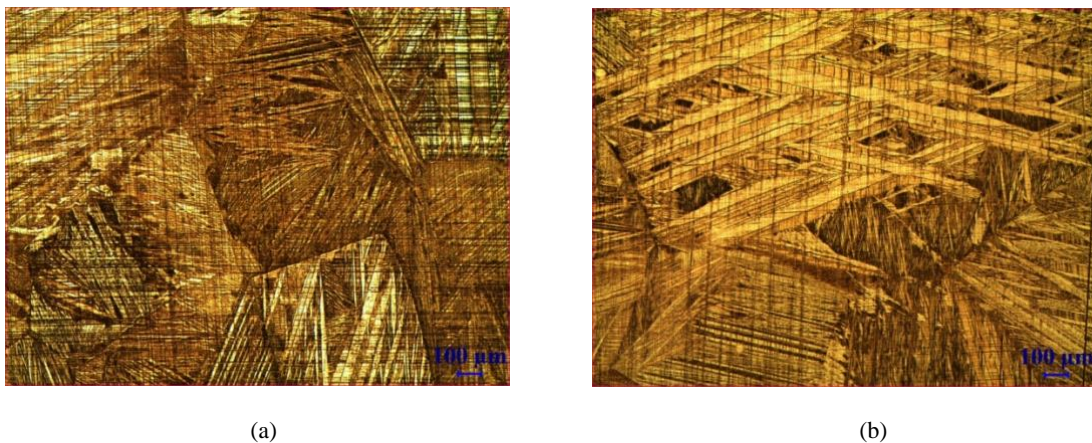
As can be seen from the optical images of the CAB2 alloy sample, the structure turned into a martensite structure with the decrease of Al and Be elements in the alloy. The grain boundaries are distinct within the structure, and the sizes of the grains are different from each other. The structure is dominated by V-type and zigzag-like martensites (Fig. 6a) [36]. With the irradiation dose of 40 kGy, the grains seem to have grown. Fewer grain boundaries were observed. V-type and needle-like martensites are present in the structure (Fig. 6b).

The Cu-based alloy samples were subjected to a 300-gram force (gf) load for 10 seconds to measure their microhardness. Average microhardness values are given in Table 4. Comparing the microhardness values of CAB1 alloy sample when it was heat-treated and treated with 40 kGy irradiation, the microhardness of the sample treated with radiation was observed to be lower. The microhardness value of the CAB2 alloy sample a very small decreased with irradiation at the dose of 40 kGy.

The results showed that small changes in the weight percentages of alloying elements and the irradiation dose affected the structure, grain size, and microhardness values.



**Fig. 5.** Optical images of CAB1 alloy sample a) unirradiated b) irradiated.



**Fig. 6.** Optical images of CAB2 alloy sample a) unirradiated b) irradiated.

**Table 4.** Average Vickers hardness and crystal size of CuAlBe shape memory alloys.

Dose (kGy)	Alloys	Average HV <sub>0.3</sub> Measurements	Crystallite Size (nm)
0	CAB1	265.4	94.4710
40	CAB1	246.8	111.5525
0	CAB2	201.6	64.0801
40	CAB2	199.6	30.0718

#### 4. Conclusions

Cu-based shape memory alloys are highly sensitive to alloying elements. In the CuAlBe shape memory alloys, the transformation temperatures ( $A_s$ ,  $A_f$ ,  $M_s$ , and  $M_f$ ), increased with the decrease of Be and Al content. In the CuAlBe alloys used in this study, the structure was completely transformed into martensite at room temperature by decreasing the Be content to 0.42. This result was verified by, the austenite→martensite and the martensite→austenite transformation temperatures obtained from DSC measurements of the alloy sample. X-ray diffraction measurements and optical microscope observations also support this result [37,38]. Comparing the DSC analysis of CuAlBe shape memory alloy samples treated with an irradiation dose of 40 kGy, the transformation temperatures changed and the temperature difference  $A_f-M_s$  decreased. It was also observed that the thermodynamic parameters  $\Delta H_{M\rightarrow A}$  and  $\Delta H_{A\rightarrow M}$ ,  $\Delta S_{M\rightarrow A}$  changed. With entropy, the elastic energy and average stiffness of samples can be controlled. As the entropy increases, the elastic energy of the sample increases, and the average microhardness decreases.

From the XRD analysis results, the shape memory alloy CAB1 has a two-phase structure (austenite, martensite), while the CAB2 shape memory alloy has only the martensite phase structure. Although it looks like there is only austenite at a first glance in the optical image of the CAB1 alloy sample, needle-like martensites are also present in the structure.

The crystallite size calculated according to the XRD analysis results of unirradiated CuAlBe alloys decreased with the decrease in Al and Be contents. The crystallite size of the unirradiated CAB1 alloy sample was 94.4710 nm, while the crystallite size of the unirradiated CAB2 alloy sample was 64.0801 nm. While the crystallite size of the CAB1 alloy irradiated with a radiation dose of 40 kGy increased, a very small decrease in the crystallite size of the CAB2 alloy was observed.

Microhardness is inversely proportional to the grain size, decreasing with an increase in grain size. Of the alloy samples irradiated with a 40 kGy dose, the microhardness values of the CAB1 alloy decreased, while the microhardness of the CAB2 alloy a small decreased.

In conclusion, we have evaluated that in CuAlBe shape memory alloys, both the weight percentages of the elements and atomic effects due to the irradiation affected transformation temperatures, thermodynamic parameters, and the microstructure.

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