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Determination of the Young's modulus in CuAlBe shape memory alloys with different microstructures by impulse excitation technique

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ABSTRACT

The Young's modulus (E) was determined in samples of CuAlBe shape memory alloys with selected grain sizes using the non-destructive technique of impulse excitation. The variation of E with the quenching temperature reached after a slow cooling from 1073 K was also studied and the microstructure of quenched samples was characterized by optical microscopy. A strong dependence of E with the grain size was found and a comparison of the obtained results with values reported in the literature was done. The behavior of E with the quenching temperature was analyzed considering the formation of γ_2 and α' precipitates, the presence of martensite in the β matrix, the reordering process and the vacancy concentration. The impulse excitation experimental device was specifically developed and mounted. An evaluation of its performance was made by means of measurements of the modulus E in samples of materials commonly used (commercial aluminum and copper) and using different vibration modes. The obtained results evidence the potentialities of the impulse excitation technique for the determination of the modulus E in alloys with a complex microstructure, which allows to characterize the behavior of E with the quenching temperature in the alloy studied.

1. Introduction

The proper determination of the elastic constants of a material is of great significance from a scientific point of view because it allows to analyze microstructural parameters of a solid. In technological applications this is of paramount importance because it establishes the input for structural calculus in engineering design. The measurement of the elastic constants can be done by different experimental methods, and the most commonly applied are classified as static or dynamic. The static techniques, in their different forms (e.g. tensile, compressive, and flexural), are probably the most frequently used in the academic and technological field, and their use has been documented since decades ago. An advantage of the static techniques is that they usually allow to obtain a complete characterization of the mechanical behavior of the samples. However, it is required specific equipment and the use of extensometers is necessary for the correct determination of the strain. The instrumented indentation can be considered as a static technique and has the advantage that it requires a reduced volume of material for the study of mechanical properties.

On the other hand, the dynamic techniques have great advantages in other aspects that make them very attractive. For example, in the resonance techniques, a sample vibrates in its characteristic frequencies as a response to an excitation. Knowing the characteristic frequencies and using the dimensions and mass of the sample, the elastic constants can be calculated. The excitation can be produced by an impact and the vibration produced can be detected using an inexpensive microphone. The impulse excitation technique or IET has gained importance in the last years for the determination of elastic constants, basically the Young's modulus (E). This technique is not destructive, enables fast measurements, from the experimental point of view is easy to implement, and some authors attribute it a greater accuracy respect to some static techniques [1]. These characteristics determine that IET becomes a convenient method for characterization and testing of materials during the fabrication process in the industry. In general the flexural mode with rectangular cross-section bars is the most used. In this mode IET has been used by some of the authors to determine the variation of the modulus E with the filler content in epoxy matrix composites, and the variation of E with

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water absorption in a commercial polyamide [2,3]. On the other hand, for example, it is reported in the bibliography that IET was used to study ceramics at high temperatures [4], nevertheless, this technique has not been extensively used for metallic alloys.

β CuAlBe alloys can exhibit shape memory properties and pseudoelastic behavior at room temperature [5–7], which makes highly promising the use of these alloys as passive dampers [8–10]. Some authors have estimated values of E in CuAlBe alloys in β phase [11–16] and with martensitic microstructure [16], using different techniques. However, most of them are destructive techniques or do not allow estimate an accurate and reliable value of E.

In the present work the Young's modulus of CuAlBe alloys is measured by means of the impulse excitation technique under different experimental conditions. Because of the different microstructures obtained submitting the CuAlBe samples to specific thermal treatments, an analysis of the effect of the grain size and thermal treatments on the modulus was also done. The Young's modulus values determined by IET were compared with the values obtained by other techniques and with those reported in the literature.

1.1. IET technique

For the determination of the modulus E by means of the IET technique, the equations given by Spinner et al. [17] were used. For the case of flexural vibration in rectangular cross-section bars, E was obtained from Eq. (1):

$$E = 0.94642\,\frac{mL^3f^2}{wt^3}T_1 \tag{1}$$

where m is the mass, L is the length, w is the width, and t is the thickness of the sample, f is the flexural fundamental frequency, and T_1 is a correction factor that can be estimated using the Eq. (2):

$$T_1 = 1 + 6.585\,\left(1 + 0.0752\,\mu + 0.8109\,\mu^2\right)\left(\frac{t}{L}\right)^2 - 0.868\,\left(\frac{t}{L}\right)^4 - \frac{8.340\,\left(1 + 0.2023\mu + 2.173\,\mu^2\right)\left(\frac{t}{L}\right)^4}{1 + 6.338\,\left(1 + 0.1408\,\mu + 1.536\,\mu^2\right)\left(\frac{t}{L}\right)^2} \tag{2}$$

where μ is the Poisson's ratio.

For the case of flexural vibration in cylindrical cross-section bars, E is obtained from Eq. (3) for the fundamental mode:

$$E = 1.261886\,\frac{4\,m\,L^3\,f^2}{\pi\,D^4}T_2 \tag{3}$$

where D is the diameter of the bar, and T_2 is a correction factor.

The Eqs. (1) and (3) are expressed as a function of the mass and the dimensions of the sample, in contrast to the original equations of Spinner et al. [17], where they are expressed as a function of the density. In Ref. [17], the correction factor T_2 is given for several values of D/L and μ. For simplicity, a value of $\mu = 0.35$ was assumed for all samples [14,18] and T_2 was estimated fitting the reported values with a polynomial function of second grade ($R^2 > 0.999$):

$$T_2 = 0.98524 + 0.34144\quad D/L + 3.90672\,\left(D/L\right)^2 \tag{4}$$

For the case of longitudinal vibration in cylindrical cross-section bars, E was obtained by:

$$E = \frac{1}{K_n}\frac{4\,m}{\pi\,D^2L}\left(\frac{2L}{n}\right)^2 f^2 \tag{5}$$

where n is the vibration mode, and K_n is a correction factor.

There are different approximations for determining K_n. In this work, the numerical value of K_n was obtained by fitting the reported data to a polynomial function of fifth grade with $\mu = 0.35$ (Eq. 6). In this case the fit is a function of D/λ, being λ the wavelength or $2\,L/n$.

$$K_n = 0.99999 + 0.00370\;D\,n/(2\,L) - 0.68940\;\left(D\,n/(2\,L)\right)^2 + 0.65597\;\left(D\,n/(2\,L)\right)^3 - 2.70303\;\left(D\;n/(2\;L)\right)^4 + 2.31795\;\left(D\;n/(2\;L)\right)^5 \tag{6}$$

2. Experimental procedure

The Cu-11.41Al-0.50Be (A1) and Cu-11.40Al-0.55Be (A2) (wt%) polycrystalline alloys under study were provided by Trefimetaux S. A. as 3.6 and 15.1 mm diameter extruded bars, respectively. The grain sizes in the as-received samples were 0.07 mm (A1) and 0.25 mm (A2), both are in β phase at room temperature. It was also observed that the grains are equiaxial on longitudinal and transversal faces. The grain size of the specimens was increased by means of thermal treatments in a resistive furnace at 1073 K for different times and quenched in water at room temperature. The average grain size was determined from micrographs obtained at room temperature, measuring the area of each grain by the software Image Tool 3.0, and an equivalent diameter (d) was estimated considering them with circular shape.

To study the influence of different thermal treatments on the Young's modulus of CuAlBe alloys, a sample of A2 of rectangular section of $4.2 \times 2.3\ mm^2$ and 43.3 mm of length was cut using an Isomet Low Speed Saw with a diamond disc. Then, it was homogenized in a resistive furnace at 1073 K (in the β field) and quenched in water at room temperature. The sample with a grain size around 1.7 mm was submitted to a continuous cooling from 1073 K to different quenching temperatures, T_q, at a cooling rate ~1.5 K/min, and then it was quenched in water at room temperature. After the thermal treatments, the modulus E was measured by IET. On the same sample and before a following treatment, a homogenization at 1073 K for at least 10 min was made.

To analyze the microstructure of the samples, specimens were observed by optical microscopy using a Reichert N384-180 microscope with an image acquirer Sony. Samples were previously electropolished in a saturated solution of chromium trioxide in phosphoric acid at around 4 V. To reveal microstructural details, specimens were also suspended for a few minutes in a solution of ferric chloride.

A device for IET measurements was specifically developed to measure the modulus E. In this work, different vibration modes were employed, due to that the studied samples have diverse shapes and sections. In the case of flexural vibration, the sample was placed on two sharp edges that are located at a distance of 0.224 of the total length of the sample from one end, see the scheme in Fig. 1. The impact of a small polymer ball on the center of the sample was used to excite the vibration of the bar. For the case of longitudinal vibration, the sample is supported from the center with a wire coiled, and the ball impacts at one end. When the sample is impacted by the ball, the frequencies that are not in resonance are attenuated. To detect the acoustic signal produced by the vibrations of the sample, a commercial microphone was used. The output electrical signal of the microphone was amplified and monitored by means of an oscilloscope and recorded using a personal computer. The frequency spectrum was obtained using Fourier analysis, and the fundamental resonance frequency (f) was determined from this spectrum. For each sample, an average of 10 frequency measurements were used. Samples of β CuAlBe with

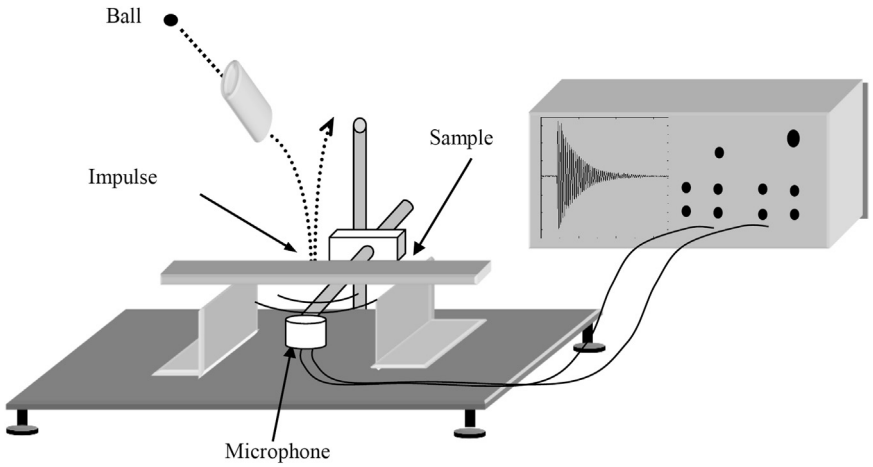

Fig. 1. Schematic experimental setup of the IET device developed. The ball impacts on the sample, which vibrates. The vibration is detected by a microphone, and the signal is observed and analyzed in an oscilloscope.

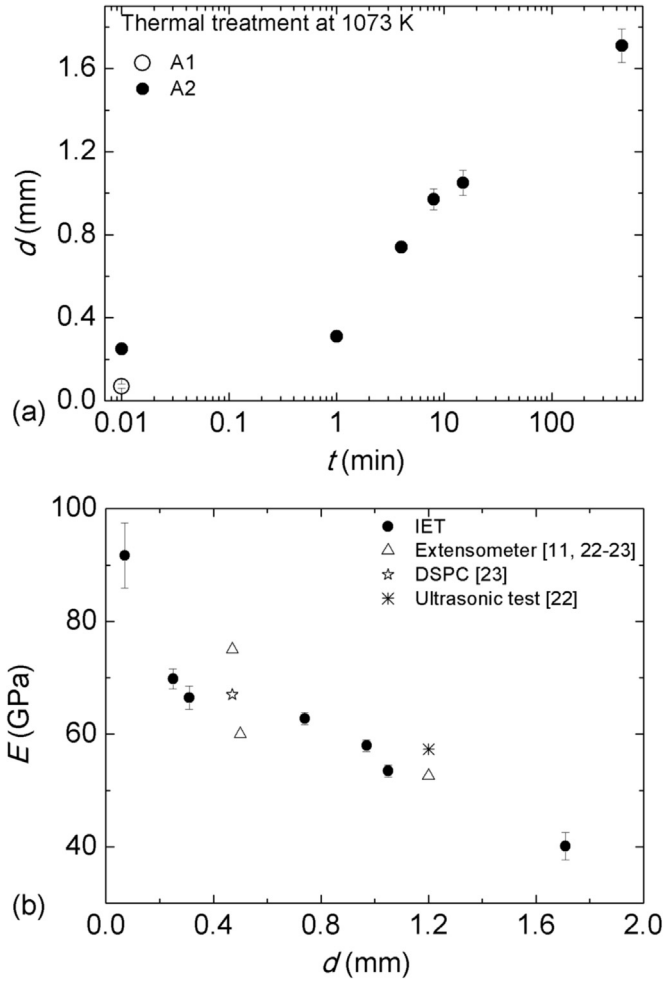

Fig. 2. (a) Values of d obtained from micrographs of sample A2 quenched as a function of the time (t) at 1073 K. The time t=0.01 min corresponds to the as received samples. (b) Variation of the modulus E as a function of d for β CuAlBe samples obtained by different experimental techniques.

different geometry and grain size were measured by IET as detailed below (D=diameter of the bar, L=length):

- A1: D=3.6 mm; L=90.2 mm, as received.
- A2a: D=15.1 mm; L=106.2 mm, as received.
- A2b: D=5.1 mm; L=103.4 mm, mechanized from a bar similar to A2a and homogenized for 1 min at 1073 K.
- Square cross section bar mechanized from a bar similar to A2a. This sample was submitted to different thermal treatments to study their influence on the modulus E.

3. Results

3.1. Evaluation of the IET device performance

To evaluate the performance of the IET device, a commercial aluminum sample (99.8%) with square cross section bar was measured [3]. An average Young's modulus of E=68.5 ± 1.4 GPa was obtained, in agreement with the values reported in the literature [19]. On the other hand, a sample of electrolytic commercial copper was measured in both flexural and longitudinal modes in a cylindrical sample, and in flexural mode in a square cross section bar mechanized from a cylindrical sample. The values of E obtained were 129.0 ± 1 GPa, 128.5 ± 0.4 GPa, and 127 ± 3 GPa, respectively, in agreement with the reported values [19].

The modulus E was measured by IET in the sample A2a using the two methods explained in Section 1.1: flexural and longitudinal. Considering the experimental error, the fundamental frequencies gave the same value of E by both methods: 70.3 ± 2.5 GPa (flexural) and 69.3 ± 1.1 GPa (longitudinal). These results indicate that the determination of E using the fundamental frequency by either methods gives the same information, therefore, hereafter we will use indistinctly the values obtained by both measurements.

A comparison of E obtained by IET and by static tests was carried out. Differences of less than 20% were found between the values of E of the same CuAlBe sample measured using an extensometer and by the IET technique. That differences are expected considering for example Radovic et al. [1], who reported a higher modulus E obtained from measurements of static tests (four point bending) respect to that obtained by IET in aluminum and steel samples.

3.2. Grain size influence

The grain growth curve for A2 sample submitted to a thermal treatment at 1073 K for several times and then quenched at room temperature was obtained. In Fig. 2a the value of d as a function of the time (t) at 1073 K is shown. The as-received alloys are also

included in the figure, with a time $t=0.01$ min. In order to analyze the influence of the grain size on E, samples of CuAlBe with different average grain size were measured by IET. Two alloys with different compositions, A1 and A2, were used to extend the range of grain sizes analyzed. As was previously indicated, both alloys are in β phase at room temperature and differ by only 0.05 wt% of Be. No significant differences are expected in the value of E due to the composition. It was corroborated in a previous work [15], in which E estimated by instrumented indentation in the same β CuAlBe alloys did not show differences. However, due to that the as-received samples have different dimensions, the grain size reached during the manufacturing process was different: 0.07 mm (A1) and 0.25 mm (A2). The variation of the modulus E measured by IET with the average grain size for β CuAlBe samples is presented in Fig. 2b. As was indicated, the first point ($d=0.07$ mm) was obtained using A1, and the other points using A2. Each value corresponds to the average between those obtained by the flexural and longitudinal method, using the fundamental frequency. The modulus of the as received alloys is also included in Fig. 2b and corresponds to 91.7 ± 5.8 GPa and 69.8 ± 1.8 GPa for A1 and A2, respectively. It can be observed that the modulus E decreases for higher grain sizes up to $E \approx 40$ GPa for d of 1.7 mm. The modulus E obtained from stress-strain curves in β CuAlBe well oriented single crystals (with a high Schmid factor) by tensile tests using an extensometer, presents values in the range of 16–23 GPa [7,20,21]. These values are in agreement with the results shown in Fig. 2b.

In order to compare, values of E reported in the literature obtained in β CuAlBe polycrystalline alloys with a similar composition by different experimental techniques, are also presented in Fig. 2b. They include mechanical tensile tests using an extensometer [11,22,23] and a digital speckle pattern correlation with laser (DSPC) [23], and other non-destructive techniques as ultrasonic tests [22]. A good agreement is observed between the values obtained by IET and the other techniques. The values obtained from mechanical tests from literature were measured at room temperature, and at different speeds. Casciati et al. [11] found that the value of E does not vary with the temperature, in the range of 303–343 K, or with the test speed, in the range of 0.0001–0.1 s^{-1}.

On the other hand, some authors have reported values measured by different experimental techniques in β CuAlBe alloys with the addition of refiners [22]. Those values were not included in Fig. 2b, because the addition of refiners vary the composition of the alloy and therefore could modify the obtained modulus.

The elastic modulus can also be estimated from load-displacement curves obtained by instrumented indentation and using the Oliver and Pharr method [24]. In a previous work, a value of $E=74 \pm 6$ GPa was obtained for a β CuAlBe alloy with a similar composition and using a Poisson ratio of 0.35 [15]. However, it is worth noting that this estimation does not strictly correspond to the modulus of β phase, because the estimation was done using the unloading curve, and some fraction of martensite would be present in the sample when the unloading begin.

Gédouin et al. [16] and Saint-Sulpice et al. [12] studied the mechanical properties under tensile tests of β CuAlBe polycrystalline wires of 1 and 1.4 mm diameter, respectively, but the grain size of the wires was not reported. However, according to the fabrication processes of the wires, it is expectable that they present a fine grain size. Both works reported a value of $E=75$ GPa using an extensometer, which would correspond to a sample with small grain size in Fig. 2b.

3.3. Influence of thermal treatments

The square section bar of A2 with a large grain size was cooled slowly from 1073 K to different temperatures T_q and then quenched in water at room temperature. After each treatment the sample was measured by IET. The variation of the modulus E with T_q is presented in Fig. 3a. As T_q decreases, the modulus increases sharply to a maximum of 61 GPa at 822 K, and then E decreases. The microstructure obtained at each temperature was microscopically analyzed, and the phases present are shown in Fig. 4. At 1073 K the sample is in β field [29,30]. At 858 K, γ_2 precipitates are formed, with an increase of their volume fraction at 847 K (Fig. 3b), from 2.3% to 35.6%. For lower temperatures, besides of γ_2 precipitates, the presence of 18 R martensite needles in the β matrix is observed at room temperature. At 697 K and lower T_q, the microstructure corresponds to a matrix of β phase with precipitates γ_2 and α′. The transformations during continuous cooling at slow rate in CuAlBe shape memory alloys have been previously studied in detail [30]. The sequence of precipitation observed in the present work is in agreement with the previous studies.

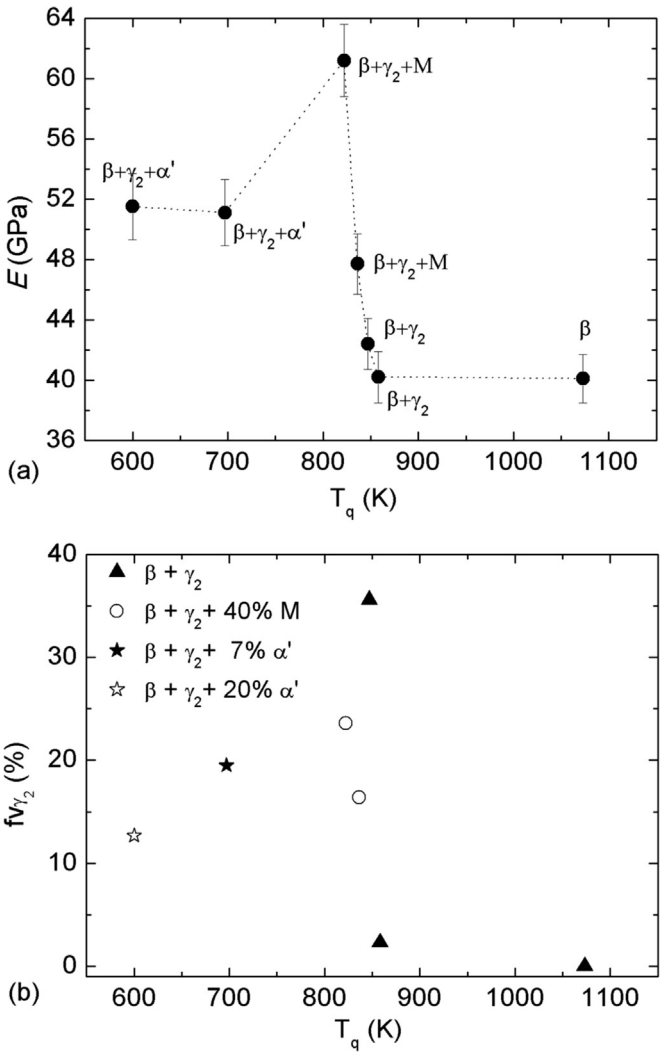

Fig. 3. (a) Variation of E measured by IET as a function of T_q. The phases present at each temperature are indicated: β phase, 18 R martensite (M), α′, and γ_2 precipitates. (b) Volume fraction of γ_2 for microstructures obtained at different T_q.

With the aim to analyze the effect of the microstructure on the variation of E with the quenching temperature, the volume fraction of each phase was quantified from the micrographs. The volume fraction was calculated as the relative area occupied by the phase with respect to the total area in at least three different zones of the sample [30,31]. The variation of the volume fraction of γ_2 precipitates ($fv\gamma_2$) with the quenching temperature is shown in Fig. 3b. As the temperature T_q decreases, the volume fraction of γ_2

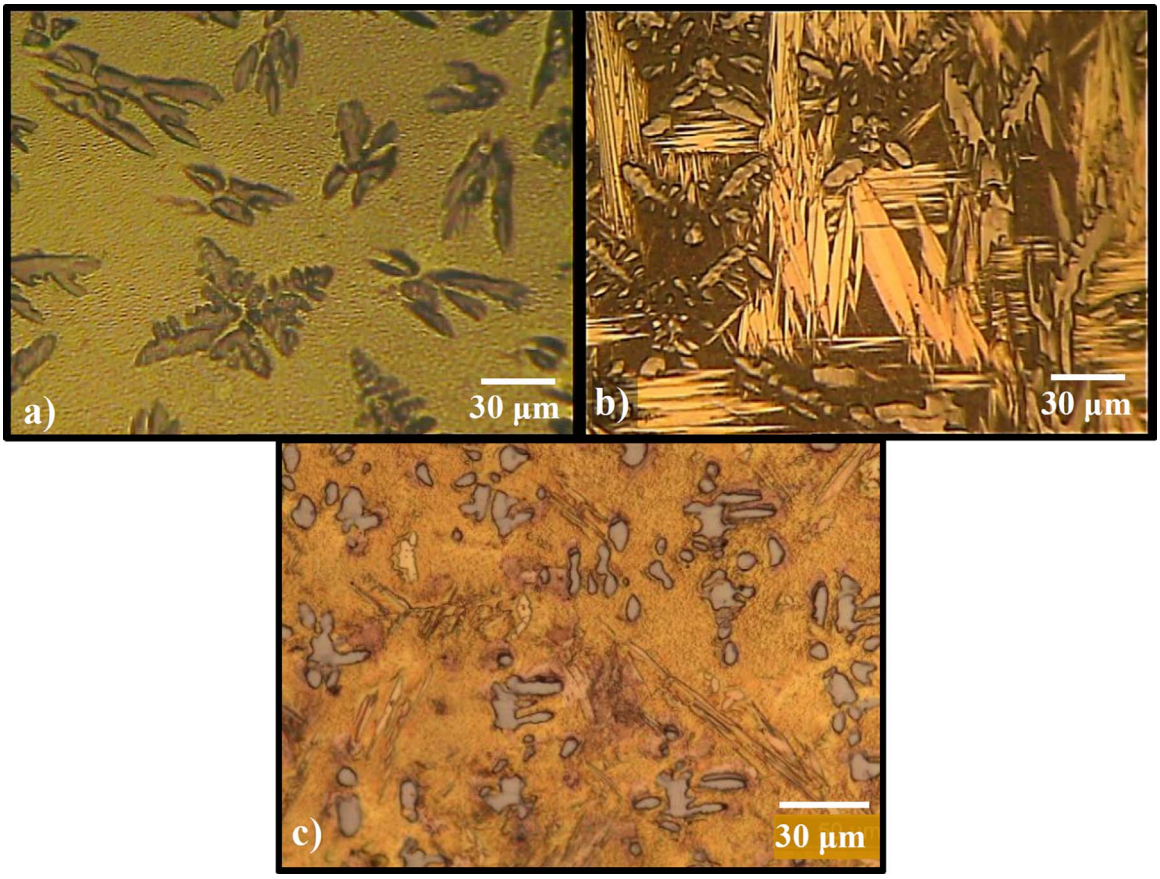

Fig. 4. Micrographs of the samples with different quenching temperature: 847 K (a), 822 K (b), and 697 K (c).

phase increases up to a maximum at ~820 K and then decreases for lower temperatures. This behavior has been previously reported [30] and is similar to that shown for the modulus E in Fig. 3a.

4. Discussion

The modulus E of a polycrystalline material could be considered as an average of the elastic properties of all grains. It is not simple to obtain this average, and different approaches have been used, in particular lower and upper bounds can be estimated using the Voigt and Reuss models, which assume randomly oriented grains [25,26]. However, when the number of grains is not high, an important dispersion in the average value of E is expected. Several authors have studied this problem using numerical simulations: Nygårds [27] for example, analyzed the number of grains necessary to get a representative result in materials with cubic symmetry and reported a dependence with the anisotropy of the material. The anisotropy can be calculated from elastic constants and in this alloy is relatively high: 13.2 as reported by Rios-Jara et al. [28]. For a high anisotropic material a higher number of grains would be necessary.

The number of grains in a specimen can be estimated from the ratio between the volume of the sample and the average volume of the grains. If we consider the sample A2, in which the grain has grown up to $d=1.05$ mm (see Fig. 2b), and assuming spherical grains, it would be expected that there are around 3500 grains in the sample. Then, the number of grains seems to be high enough to obtain representative values of E, and the decrease of E for higher grain sizes would not be only associated to a low number of grains in the sample. In this sense, it would be expected preferential crystallographic orientations in samples with fewer grains.

For the samples with fine grain size, where a minor influence of preferential crystallographic orientations would be expected, a comparison of the values of E measured by IET with those expected using the Voigt and Reuss models [25,26] was performed. From these values an average was calculated following the Hill approach [25]. The values obtained were 154.9 GPa using the Voigt model, 46 GPa using the Reuss model, and 103.2 GPa for the Hill average; the latter value overestimates by 12% the experimental measurement.

To analyze the variation of the modulus E with the quenching temperature, the relationship between E measured by IET and the microstructure of the samples is presented in Fig. 5. In the samples with the microstructure ($\beta+\gamma_2$), the matrix corresponds to β phase with the presence of γ_2 precipitates, which are rich in aluminum [30,32]. In a previous work on CuAlBe alloys with the same microstructure, the modulus E of each phase was measured by instrumented indentation [15]. A value of $E=148\pm18$ GPa was reported for γ_2 precipitates, twice than that obtained for the β matrix. If we consider that the modulus of the sample corresponds to the weighted average of each phase, E would increase by about 35% for the highest volume fraction of precipitates in Fig. 3b. However, the measured E increases up to 43 GPa, that is only 7.5% (Fig. 5). This result indicates that the presence of γ_2 precipitates can produce only a slight increase of the modulus.

If we consider the samples with γ_2 precipitates and $\approx$40% of martensite phase at room temperature, E increases with the volume fraction of γ_2 phase, but higher values are obtained respect to the samples without martensite (Fig. 5). It is known that the martensite phase has a modulus E lower than that of the β phase. This fact can be corroborated during a pseudoelastic cycle, where

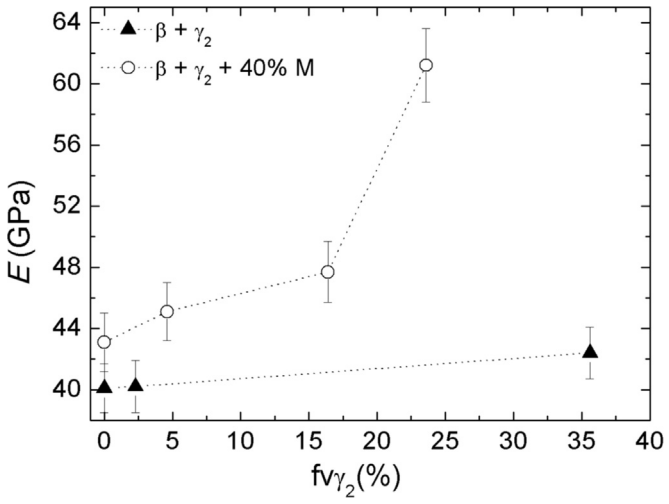

Fig. 5. E as a function of $fv_{\gamma 2}$ for samples without martensite and with 40% martensite.

the slope of the loading curve decreases when the transformation of β to stress-induced martensite begins [6]. Some authors have assumed that the modulus of the stress-induced martensite and thermally induced martensite are equal [16]. Measurements carried out on CuAlBe polycrystalline alloys by mechanical tests with extensometer have reported values of 49 GPa for a mixture of phases (β+ stress-induced martensite) [23], and 35 GPa for a thermally induced martensite sample [16], with a modulus of 75 GPa for β phase. These previous results would suggest that the presence of martensite on samples (β+γ_2) decreases the modulus E. Therefore, we can assume that the presence of martensite phase has not influence on the increase of the modulus.

Other possible contribution to the variation of E with T_q is the influence of the vacancies retained in quenching. Romero et al. [33] studied the concentration and nature of the defects introduced during quenching at different T_q by positron annihilation lifetime spectroscopy and calorimetric measurements. They reported a maximum concentration of retained single vacancies after quenching at around 775 K. For higher and lower T_q values the vacancy concentration decreases. The authors proposed that the quenched-in vacancies would affect the effective relative change of beryllium concentration, which produce a shift of the transformation temperatures to higher values [30,33]. It is important to consider that the disorder-order transition of β phase, from A2 to DO_3 structure, occurs at around 800 K [30,34]. The reordering process takes place during the quench, and it may have some influence on the variation of the modulus E with the quenching temperature. Some authors have indicated that the high concentration and mobility of vacancies favor the reordering of the β phase [33].

All the analyzed contributions must be taken into account to understand the variation of the modulus E with the quenching temperature: the microstructure, the vacancy concentration and the reordering process. It is also important to consider that each of these issues would have some influence over the others.

5. Conclusions

The Young's modulus was determined in CuAlBe shape memory alloys using the non-destructive impulse excitation technique in samples with different microstructures. The technique was evaluated in samples of materials commonly used (commercial aluminum and copper) and in CuAlBe using different vibration modes. The obtained results are in agreement with those reported in the literature. The fundamental frequencies gave the same value of the modulus by both vibration modes.

The modulus was determined in β CuAlBe samples with different grain sizes, and a strong dependence of E with the grain size was found. The modulus decreases for higher grain sizes up to $E \approx 40$ GPa for d of 1.7 mm, which could be associated to preferential crystallographic orientations in the samples with fewer grains. The values measured by IET are in agreement with those reported in the literature and obtained by other experimental techniques. For the samples with fine grain size, a comparison of the values of E measured by IET and those expected using the Voigt and Reuss models was performed. An average using the Hill approach was calculated, which overestimates by 12% the experimental measurement.

The variation of E with a thermal treatment consisting of a slow cooling from 1073 K to different quenching temperatures was studied. As T_q decreases, the modulus increases sharply to a maximum of 61 GPa at 822 K, and then E decreases. The obtained microstructure was characterized by optical microscopy, and quantified by an area analysis. The formation of γ_2 and α′ precipitates and the presence of martensite in a β matrix was observed. The behavior of E with the quenching temperature was analyzed considering the microstructure, the reordering process and the vacancy concentration. All these contributions must be taken into account to understand the variation of the modulus E with the quenching temperature.

The obtained results evidence the potentialities of the impulse excitation technique in the measurement of the modulus E in metallic samples. By using this technique, the variation of the modulus with the grain size, and with the quenching temperature was analyzed in CuAlBe shape memory alloys. This technique is non-destructive and permit to carry out measurements in samples with different dimensions in a fast and low cost way.

Acknowledgments

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