Influence of microstructure on the Young's modulus in a Cu-2Be (wt%) alloy

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ABSTRACT

The influence of microstructure on the Young's modulus and hardness in a Cu-2Be (wt%) alloy was analyzed. The material was homogenized at 1113 K and submitted to thermal aging at different times and temperatures between 540 and 680 K. The improvement of the mechanical properties of the material, with an increase of the hardness and Young's modulus is mainly due to the formation of metastable phases in the α matrix. For temperatures up to 623 K, the formation of γ'' precipitates produces an increase of the hardness in an early stage. At 676 K, the maximum hardness is reached for 0.75 h due to the presence of γ' phase, and further aging times induce a softening of the material.

The Young's modulus curves present a similar behavior respect to the hardness curves. However, no significant variation of the modulus was found for aging longer than 1 h at 676 K. Then, only the size and shape and not the change in the orientation of the precipitates would have influence on the value of the modulus. A micromechanical model was applied, and the volume fraction curves of precipitates were estimated from E modulus. For the aging temperature of 676 K and times longer than 1 h, the modulus is almost constant because there is no increase in the fraction of precipitates and their maximum volume fraction is reached.

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1. Introduction

Copper beryllium alloy (Cu-Be) with contents of 1.8—2.0 wt% of beryllium is a high strength alloy with non-magnetic, non-sparking qualities and good corrosion resistance. These characteristics allow its use in different specialized applications like non-sparking tools, blow molds and plastic injection molding, springs with complex shapes and sensors. The promising characteristics of Cu-Be alloys are mainly based on the improvement of their strength and hardness by aging treatments from the Cu solid solution (α phase), which induce the formation of precipitates [1,2].

The sequence of precipitation in high strength Cu-Be alloys has been extensively studied, and it is recognized the formation of the following metastable phases prior to the equilibrium γ phase:

Guinier-Preston zones (G.P. zones), γ'' phase and γ' phase [3,4]. Rioja et al. [3] also reported the formation of beryllium equiaxed clusters prior to the G.P. zones. The Guinier-Preston zones are coherent monolayers of beryllium atoms formed on the {100} planes respect to the α matrix [3,4]. With the increase of the aging time, γ'' precipitates are formed from the piling up of G.P. zones [4]. Zhou et al. [1] studied a Cu-0.23Be-0.84Co alloy and found that the morphology of γ'' phase is spherical and its size increases with the aging time. Then, they change the shape to ellipsoids and transform to γ' phase with the habit plane {112}$_\alpha$ [1,3]. After that, the γ' precipitates continue growing and the habit plane changes to {113}$_\alpha$ [3]. For longer aging times, the formation of the equilibrium γ phase occurs, having a B2 structure and a lattice parameter of 2.7 Å [3,5]. Rioja et al. [3] found that the sequence of precipitation and the resulting properties of the material depend strongly on the quenching method.

The physical properties of the material are determined by the microstructure of the sample and the precipitates present. Several studies about the different mechanical properties that are strongly affected by the precipitation of metastable phases in Cu-Be alloys

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have been reported. Guoliang et al. [2] studied the strengthening behavior of a Cu-2Be (wt%) alloy aged at 593 K, and they suggested that the Orowan mechanism would be the leading strengthening mechanism. Tang et al. [6] also studied the strengthening behavior of a Cu-1.9Be (wt%) alloy submitted to different thermal treatments. They analyzed the influence of the nature of the precipitates and established a yield stress model to explore the properties of the material. Yagmur [7] studied the effect of the microstructure on internal friction of an aged Cu-1.92Be (wt%) alloy, and found that the specimens with G.P. zones have low internal friction. Era et al. [8] studied the spontaneous warping deflection of a Cu-1.9Be (wt%) alloy initially submitted to an external load and aged at different temperatures.

For technological applications, the Young's modulus (E) is a very important mechanical property. Small variations of the modulus can be of great importance, especially for applications as sensors or springs. Yagmur [7] determined the Young's modulus of a Cu-1.92Be (wt%) alloy aged for 2 and 6 h at 588 K, and found a difference of around 2 GPa between both samples. To our knowledge, no systematic study has been reported with respect to the variation of the modulus for Cu-Be alloys submitted to aging treatments.

The impulse excitation technique (IET) is a dynamic technique which allows to determine the Young's modulus of a material in a non-destructive and easy way. This technique has gained importance lately because it is easy to implement, which permits measurements at industrial level. In a recent work [9], the variation of the modulus E in CuAlBe samples with complex microstructures was determined by IET and analyzed.

In this study, the influence of microstructure on the Young's modulus in a Cu-2Be (wt%) alloy was analyzed. The material was aged at different times and temperatures between 540 and 680 K. The hardness curves were obtained as a function of time for different temperatures, and calorimetric measurements were made searching for identification of phases present. The volume fraction of precipitates was estimated using the variation of modulus with the aging treatments.

2. Material and methods

The alloy studied, of composition Cu-2Be (wt%) with some impurity elements (Ni, Co and Si, with contents $\leq 0.5\%$), was obtained from Roberto Cordes S.A., as polycrystalline 6 and 10 mm diameter cylindrical bars. The UNS number for this material is C 17200. Samples of the alloy were heat treated at 1113 K, followed by quenching into water at room temperature (~293 K). Then, they were aged at different temperatures in the range of 540−680 K for times between 15 min and 6 h.

For observations by optical microscopy, samples were electropolished in a saturated solution of chromium trioxide in phosphoric acid at around 4 V, and suspended for 5 s in a ferric chloride solution. The micrographs were obtained using a Reichert N384-180 microscope with an image acquirer Sony.

A calorimetric analysis of the samples was carried out in a TA Instrument Q20 calorimeter. Calorimetric specimens of $3.5 \times 3.5 \times 1$ mm^3 were cut before the heat treatments, using an Isomet Low Speed Saw with a diamond disc.

Vicker hardness was carried out using a Mitutoyo MVK-H11. For hardness measurements disks of 1.5 mm in thickness were obtained from the 10 mm diameter bars. Previously to the measurements, the aged samples were smoothed with 600 and 1000 grit emery paper and a final diamond polished (3 μm size) was carried out.

IET measurements were performed in a specifically developed device, selecting longitudinal mode. More details of the device are given in Ref. [9]. The vibration excitation was done using the impact of a small ball at one end of the sample under test. A commercial microphone, a system of amplification and an oscilloscope were used to monitor and record the signal in a personal computer. Applying Fourier analysis the fundamental frequency (f) was determined and the E modulus was calculated using equation (1), valid for cylindrical samples with length L and diameter D. In the mentioned equation, n is the vibration mode, m is the mass of the sample and K_n is a correction factor, that was estimated using the approximation described in Ref. [9].

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

The sample used for IET measurements was a 6 mm diameter bar with a length of 132.5 mm. The specimen was measured after been heated at 1113 K for 3.5 h and after been aged. To evaluate microstructural changes at room temperature, a second measurement was performed 24 h later. However, no changes were observed in the value of E. Prior to the next measurement at a different time, sample was homogenized for 10 min at 1113 K. For each experimental value, at least 10 measurements were done, and an average was obtained.

3. Results

In Fig. 1(a) and (b) the micrographs of a Cu-Be sample from the 6 mm diameter bar after a thermal treatment at 1113 K for 10 min and 3.5 h, respectively, are shown. The samples are in α phase, which corresponds to a fcc (Cu) solid solution. In Fig. 1, some
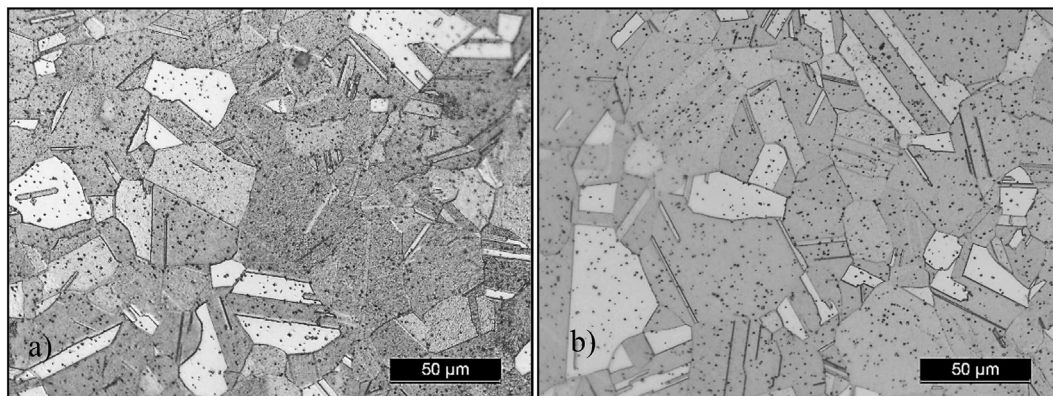

Fig. 1. Micrographs of a Cu-Be sample from the 6 mm diameter bar after a thermal treatment at 1113 K for 10 min (a), and 3.5 h (b).

annealing twins are observed in the grains. The grain size of the samples are 27 ± 0.8 μm and 35 ± 1.4 μm, for the times of 10 min and 3.5 h at 1113 K, respectively. It is expectable that the sample treated for 3.5 h has reached the largest grain size, and from this time it will remain stable [10]. Similar grain sizes were obtained for the 10 mm diameter bar.

The Vicker hardness of the specimen in α phase (previously submitted to a thermal treatment of 10 min at 1113 K) and after different aging times (t_{aging}) and aging temperatures (T_{aging}) is presented in Fig. 2. The measurements were performed under different loads: 50, 100 and 300 g. The variation of the hardness with the applied load for a t_{aging} of 4 h at different aging temperatures is presented in Fig. 2(a). As is expected, higher values of hardness are obtained under lower loads [11,12]. However, the variation of the hardness with the temperature presents the same behavior for each load. The hardness curves with t_{aging} for different T_{aging} under a load of 300 g are presented in Fig. 2(b). After the thermal treatment at 1113 K, samples in α phase exhibit a low hardness of 98 HV. For T_{aging} of 540, 580 and 623 K, the hardness increases with the aging time, however, the maximum hardness is not reached after 5 h, indicating that those samples are in an early

stage of aging. As the temperature increases, the hardness curves reach higher values as is observed in Fig. 2(b).

For T_{aging} of 676 K, the hardness presents a maximum at 0.75 h, followed by a slow decrease. According to Era et al. [8], the decrease in hardness corresponds to a later stage of the aging, and in our work it is only reached for a T_{aging} of 676 K.

In order to analyze the effect of the previous thermal treatment on the grain size of the samples, and its possible influence on the hardness, a Vickers hardness curve was also obtained for a sample heated for 2.5 h at 1113 K, and aged at 676 K for different times. No significant influence on the hardness curve was found when the homogenization time was increase from 10 min to 2.5 h.

The variation of the Young's modulus with the aging time for different T_{aging} in a sample previously heated during 3 h at 1113 K is depicted in Fig. 3. It is noted that the Young's modulus increases with t_{aging} for all the temperatures. Results in Fig. 3 show that higher values of E are obtained for higher temperatures. A maximum value is reached at 676 K for t_{aging} of 1 h, and then, the modulus remains almost constant. For the other temperatures, the modulus does not reach a maximum up to 4 h of t_{aging} at 540 and 580 K or 6 h of t_{aging} at 623 K. Is clearly observed that the behavior of the Young's modulus with the aging is similar to the behavior observed in the hardness curves, and can be assigned to the precipitation process developed during aging.

In order to obtain microstructural information of samples homogenized at 1113 K (as-quenched specimen) and after different treatments, differential scanning calorimetry (DSC) curves were obtained for the as-quenched specimen (5 K/min) and the samples aged at 580 and 676 K for 0.75 and 5 h (10 K/min). The results are presented in Fig. 4. In the curve (a), three exothermic reactions at 520, 660 and 705 K are observed, denominated A, B and C. According to previous studies [13,14], the reaction A can be attributed to the formation of G.P. zones and the precipitation of γ″ phase, reaction B is associated with the formation of γ′ phase and reaction C can be attributed to the precipitation of γ phase. This fact corroborates that the as-quenched specimen corresponds to a solid solution. In the curve (b) for the sample aged for 0.75 h at 580 K, the reactions B and C also appear, and two endothermic reactions are observed, reaction D at 627 K and a slight reaction E at 560 K. Reaction E can be attributed to the dissolution of G.P. zones and
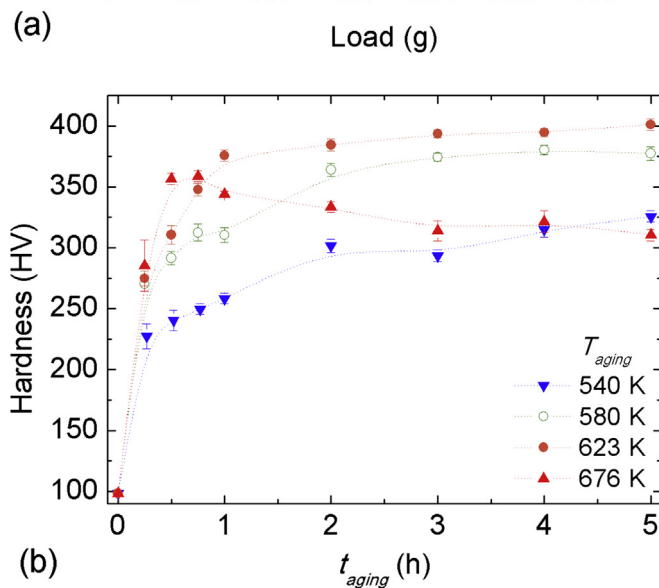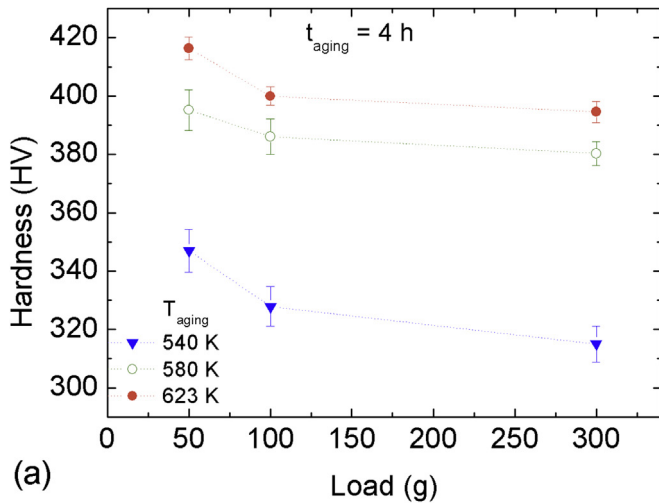

(a)

(b)

Fig. 2. (a) The variation of hardness with the applied load for different T_{aging} and t_{aging} = 4 h. (b) Hardness as a function of t_{aging} for different T_{aging} under a load of 300 g. The dotted lines are for reference.
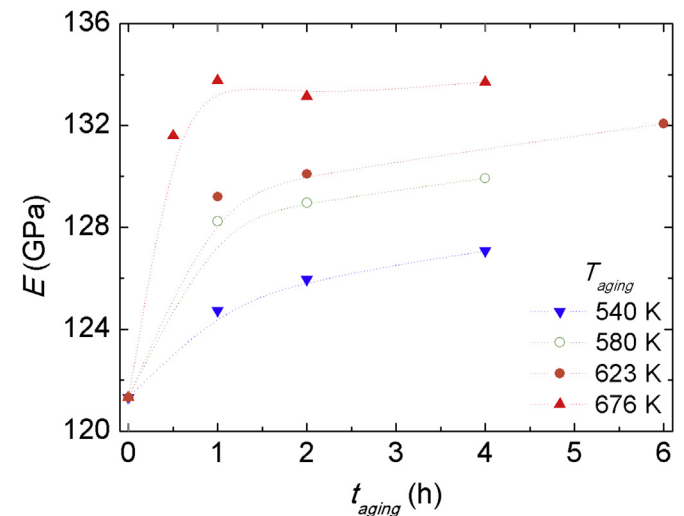

Fig. 3. Variation of the modulus E with t_{aging} for different T_{aging} for samples previously homogenized at 1113 K and aged at different times and temperatures. The dotted lines are for reference.
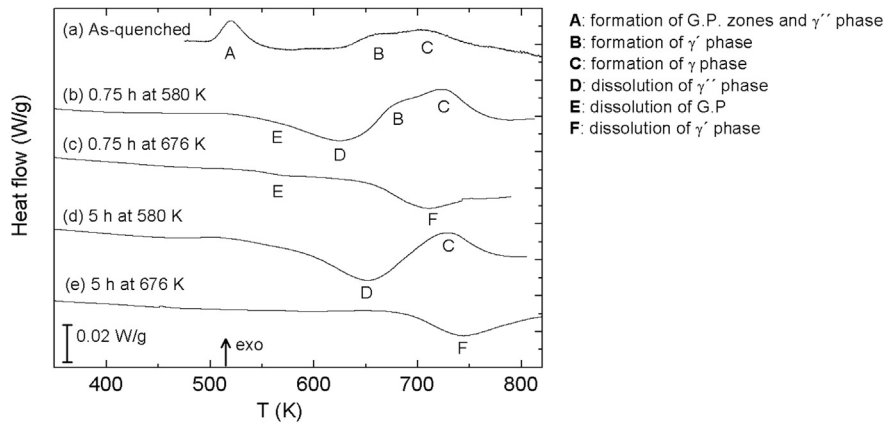
Fig. 4. DSC curves for the samples homogenized at 1113 K (as-quenched specimen) and after different treatments.

reaction D to the dissolution of γ'' phase [6,14]. In the curve (c), for the sample aged for 0.75 h at 676 K, the endothermic reaction E at 574 K is observed, besides of a reaction at 707 K named F. The reaction F would correspond to the dissolution of γ' phase [14]. In the curve (d) for the sample aged for 5 h at 580 K, the reactions D and C are clearly observed. The reaction D is detected at 652 K, temperature higher than that observed in the curve (b). The presence of γ'' phase in the sample aged for 5 h at 580 K is in agreement with the hardness curve, due to the maximum hardness is not reach yet. In the curve (e), for the sample aged for 5 h at 676 K, only the reaction F is observed at 744 K. It is important to note that the temperatures of dissolution determined for the metastable phases are lower than the reversion temperatures reported by Chakrabarti et al. [4], obtained by electrical resistivity, hardness methods and TEM microscopy. That difference has been reported by other authors [14].

4. Discussion

The aging treatments in the range of temperature of 540–676 K induce the formation of precipitates in Cu-Be alloys. The precipitation sequence has been extensively studied and it has been summarized as: α solid solution → G.P. zone → γ'' phase → γ' phase → γ phase [3,13]. The variation of hardness in samples in α phase aged for different times (Fig. 2(b)) is mainly due to the formation of metastable phases, which harden the material.

According to the hardness curve presented in Fig. 2(b), the hardness increases during the early stage. The DSC results for a sample aged at 580 K for 0.75 h (curve b in Fig. 4) indicates the presence of a small amount of G.P. zones and γ'' phase. Some authors have also found those phases in Cu-Be samples aged at around 580 K for less than two hours [14–16]. The DSC results for a sample aged at 580 K for 5 h (curve d in Fig. 4) indicate the presence of γ'' phase, but there is a shift in the temperature of dissolution. This shift has been previously reported by Yamamoto et al. [14], and has been attributed to the gradual change of the crystal structure of the γ'' precipitates. The precipitates would grow and change the shape from spheres to ellipsoids or plates [1,16]. However, these changes are produce in an early stage in where γ' phase has not yet been formed, and the maximum hardness has not been reached. Therefore, the hardness increases due to the formation of G.P. zones and γ'' precipitates. This fact has been previously reported by some authors [1,3,8].

At 676 K, the maximum hardness is reached for 0.75 h, as is shown in Fig. 2(b). The calorimetric results indicate the presence of γ' phase at this aging time. It has been reported that the

precipitation of γ' produces the most effective hardening of the alloy among the precipitates [1,3,8]. However, further aging times induce a softening of the material. Gröger et al. [15] associated the coarsening of the γ' particles with the decrease of the hardness. Yagmur et al. [16] studied the behavior of a Cu-1.9Be (wt%) alloy aged at 593 K and reported that in the softening stage occurs an increase of the misfit angle between the γ' precipitates and the matrix and the elongation of the particles. After 5 h at 676 K, only the dissolution of γ' phase is observed in calorimetric curves, but a shift to higher temperatures is observed. That shift could be associated to the change in the precipitates that induce the softening of the material. It has been reported that γ' phase changes continuously to the coarse and spherical γ phase as aging time increases [1,14]. So, it could be difficult to differentiate the dissolution reaction of both phases in the calorimetric curves [14].

In the Young's modulus curves (Fig. 3), the same stages found in the hardness curves are observed. The largest modulus is accomplished at the highest hardness for each temperature. The modulus E would be strongly affected by the fraction, morphology, size and distribution of the precipitates. During the early stage, the formation of fine sphere-shaped γ'' precipitates and their further growth produce the increase of E. As the aging temperature increases, higher values of the modulus are obtained. At 676 K the highest modulus is obtained after 1 h aging, when the precipitates are mainly γ' phase. It has been reported that the morphology of the sphere-shaped γ'' precipitates transforms to ellipsoid or plate shape γ' phase [1]. Yagmur [7] reported higher values of the modulus E for a Cu-1.92Be (wt%) sample with γ' precipitates respect to a specimen with G.P. zones, both aged at 588 K for 6 and 2 h, respectively. That difference was attributed to the shape and orientation of the γ' phase [7].

After the highest hardness value, a softening of the material is observed due to the coarsening of the γ' phase, and the inconsistency of the orientation weaken the jamming effect on the dislocations [1]. For aging longer than 1 h at 676 K, the Young's modulus remains almost constant. Therefore, the change of the orientation would not have a significant influence on the value of the Young's modulus.

A complete analysis of the behavior of the modulus E observed in the experimental results is complex. But with reasonable assumptions an interpretation of the variation of E with t_{aging} can be given. It will be assumed that the different precipitates have the same elastic constants, i.e. γ' and γ'' have the same E modulus, although they can have different size, shape and orientation. In the last part of the E versus time curve for aging at 676 K, a change in orientation of γ' phase is supposed, but no changes in E are

observed. Based on this fact, the changes in the orientation of the precipitates respect to the matrix will not be considered as causing the variations of E, and only the size and shape of precipitates will be taken into account. The size modifies the fraction of precipitates in the matrix, while the shape changes of spheres to disks, depending on the stage of precipitation. In this sense, a micromechanical model will be applied considering sphere or disk type precipitates. Although a variety of works studying the influence of inclusions of a different phase in a matrix are been reported since decades ago, for the sake of simplicity the approach presented by Benveniste [17] will be used in this work. In this approach, two different geometries are studied, spheres and disks, as isotropic inclusions in a matrix. The anisotropy ratio can be obtained from the elastic constants c_{11}, c_{12} and c_{44} [18]. Using the ratios $c_{12}/c_{11} \approx 0.5$ and c_{44}/c_{11} between 0.3 and 0.5 [5], the estimated anisotropy ratio for Cu-Be precipitates is near to 1. In the present work, it will be also considered the behavior of the matrix as the average of all grains, which corresponds to the sample in α phase without aging.

To our knowledge, the elastic constants of the precipitates γ'' and γ' of Cu-Be alloys have not been reported. In the literature, the modulus E measured by nanoindentation in hard γ_2 precipitates in CuAlBe alloys is approximately twice the modulus of the matrix [19]. Based on this fact, it will be used a value for the bulk modulus of the precipitates (k_p) as twice the bulk modulus of the matrix without precipitates (k_m). From the modulus E of the matrix for the studied alloy without aging ($E_m = 121.3$ GPa), reported in Fig. 3, k_m can be obtained using the following general relationship [18]:

$$E = 3\,k(1 - 2\nu) \tag{2}$$

where ν is the Poisson ratio and will be assumed $\nu = 0.35$, for both the matrix and the precipitates. Then, the following values of the bulk modulus: $k_m = 134.8$ GPa and $k_p = 260.0$ GPa, will be used. In this way, the volume fraction of precipitates (φ_p) can be estimated using the equation (3) given in ref. [17], which considers a material with spherical inclusions:

$$\frac{k - k_m}{k_p - k_m} = \frac{\phi_p}{1 + (1 - \phi_p)\frac{k_p - k_m}{k_m + \frac{4}{3}G_m}} \tag{3}$$

where G_m is the shear modulus of the matrix. The results are shown in Fig. 5 as a function of t_{aging} at different T_{aging}.

It can be observed that the maximum φ_p obtained is near to 0.17, which is the maximum fraction expected from the phase diagram according to Tang et al. [6]. Then, the assumed value of 260 GPa for k_p can be considered as a feasible value. An approach considering disks as isotropic inclusions, given in Ref. [17], was also used. As an example, for a T_{aging} of 540 K, differences in the fraction of precipitates are lower than 10% between the model that assumes spheres respect to the model that assumes disks. The value of E assuming a model of disks is lower than the value assuming spheres. Then, the increment of the fraction of precipitates is considered as the main contribution to the variation of the modulus E, while their change of shape would only slightly contribute to this change.

For T_{aging} of 676 K, Fig. 3 depicts that for times longer than 1 h, E is almost constant. Based on the previous analysis, it can be concluded that there is no increase in the fraction of precipitates for times longer than 1 h (Fig. 5), and the maximum φ_p is reached at this time. Fig. 5 resembles Fig. 3 supporting the analysis exposed.
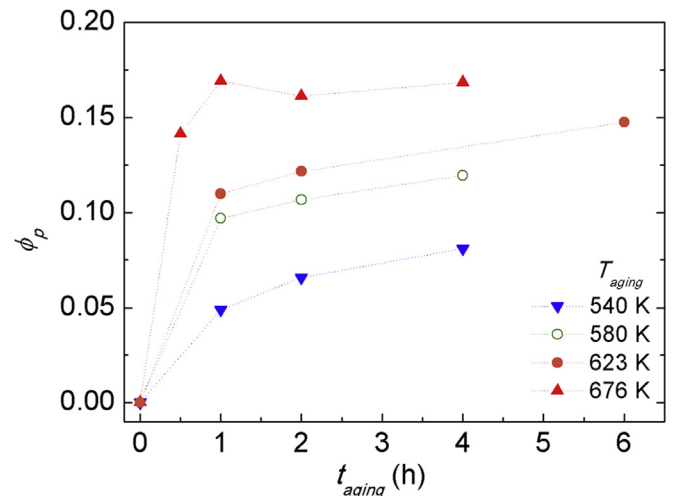

Fig. 5. Variation of the fraction φ_p with t_{aging} for different T_{aging}. φ_p was estimated using equation (3) and considering a material with spherical inclusions. See text. The dotted lines are for reference.

5. Conclusions

Determination of hardness, Young's modulus and calorimetric measurements were performed in a Cu-2Be (wt%) alloy, submitted to heat treatment at 1113 K and thermal aging at different times and temperatures in the range of 540—680 K. The influence of the microstructure on hardness and Young's modulus was analyzed.

It was corroborated that the variation in hardness is mainly due to the formation of metastable phases in the α matrix. For aging temperatures of 540, 580 and 623 K, the hardness increases mainly due to the formation of γ'' precipitates and their subsequent gradual change. At 676 K, the maximum hardness is reached for 0.75 h due to the presence of γ' phase, and further aging times induce a softening of the material.

The Young's modulus curves present a similar behavior respect to the hardness curves. However, no significant variation of the modulus was found for aging longer than 1 h at 676 K. Then, the changes in the orientation of the γ' precipitates respect to the matrix would not cause variations of the modulus, while the size and shape of the precipitates would influence on the value of the Young's modulus. The size modifies the fraction of precipitates in the matrix, while the shape changes of spheres to disks, depending on the stage of precipitation. A micromechanical model was applied, and the volume fraction curves of precipitates were estimated from E values considering sphere type particles. The change of shape from sphere to disk would only produce differences lower than 10% in the fraction of precipitates. The increment of the fraction of precipitates is considered as the main contribution to the variation of the modulus E. For the aging temperature of 676 K and times longer than 1 h, the modulus is almost constant because there is no increase in the fraction of precipitates and their maximum volume fraction is reached.

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