

Characterization of a CuAlBe Alloy with Different Cr Contents

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In this article, the use of chromium (Cr) as a grain refiner for a CuAlBe shape memory alloy is discussed. Alloys with 0.1, 0.2, 0.3, and 0.5 wt.% Cr were characterized by optical microscopy, scanning electron microscope, and x-ray diffraction. Also, the influence of the different percentages of Cr on the grain size and on the mechanical properties was analyzed through macro- and microscopic evaluations and by tensile and hardness tests, respectively. Finally, the phase transformation temperatures of the alloys were determined by thermal analysis using differential scanning calorimetry. The results showed that the higher the Cr content, the greater the grain refinement effect and lower the hardness. In addition, at room temperature the alloys with 0.1 and 0.2 wt.% Cr were austenitic, while the rest were martensitic. The tensile tests showed that the alloy with 0.2 wt.% Cr provided the best strain-stress performance. The conclusion was that the use of 0.2 wt.% Cr as a grain refiner improved the mechanical properties of the CuAlBe alloy; however, the same was not observed for the other Cr contents.

Keywords electron microscopy, grain refinement, mechanical characterization, microanalysis, shape memory alloys, x-ray diffraction

1. Introduction

Alloys with shape memory effect are unconventional functional materials that have a wide range of applications due to their peculiar mechanical properties. Hence, these shape memory alloys (SMAs) have been intensively studied in the Materials Science and Engineering fields, and they have numerous applications; e.g., in thermally activated electrical switches (Ref 1), thermally controlled systems (Ref 2), biomedical applications (Ref 3), and in pipes (Ref 4).

SMAs are materials that have the capacity to return to their pre-deformed shape by heating. This peculiarity has motivated research for materials with improved recovery rates and that are also suitable for applications under high phase transformation temperatures.

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CuAlBe SMAs have raised great interest by presenting a cost-effective advantageous alternative to NiTi alloys. In fact, copper-based alloys with shape memory effect have gained special attention because of their excellent shape memorization ability, pseudoelastic, and strong damping capacity due to the martensitic transformation (Ref 5). The CuAlBe alloy studied in this work has, in particular, several interesting properties, such as superelasticity, strong damping effect, high mechanical strength, resistance to corrosion (Ref 6), low manufacturing costs, as well as suitable for use at low temperatures. This suitability for low temperatures is very attractive, since the introduction of only 0.1 wt.% of beryllium (Be) reduces the phase transformation temperature of the alloy by approximately 100 °C (Ref 7).

Grain refiner elements (inoculants) are frequently added to SMAs to improve their mechanical properties, since these properties are directly related to the material microstructures (Ref 8-10). The grain refining process of metallic materials leads to various benefits, such as an increase in the molten metal fluidity, an improvement of second phase particles and porosity dispersion, an easier surface for machine finishing and improved mechanical properties (Ref 11). Thus, in the inoculation process, particles that act as efficient substrates for the heterogeneous nucleation of grains are added to the molten metal.

The effect of adding different inoculants to SMAs on their microstructural, phase transformation and mechanical properties has been studied by various authors. Montecinos et al. (Ref 10) investigated the grain size dependence of the DO3-18R martensitic transformation start temperature (M_s) and start stress (σ_s) in a Cu-22.66Al-2.98Be (at.%) alloy in a thermodynamic framework. The authors concluded that the M_s decreases and the σ_s increases when the grain size decreases, and that the σ_s /temperature relationship is linear; however, the slope of the relationship depends on the grain size. Moreover, they observed that the zero stress extrapolated temperature decreases with the decrease of the grain size and remains lower than M_s .

Zhang et al. (Ref 12, 13) completed tensile tests to evaluate the mechanical properties of fine- and coarse-grained Cu-11.42Al-0.35Be-0.18B SMAs (wt.%) prepared by equal channel angular processing (ECAP). After eight passes of ECAP and subsequent quenching from 600 °C to room temperature, the mean grain diameter was refined from 227 to 42 μm with purified grain boundaries. The fine-grained alloy exhibited good mechanical properties with a high tensile strength (703 MPa) and featured deeper and closer dimples on its fracture surface. The microcracks were more refined, and the crack extensions along the grain boundaries were improved in the fine-grained alloy. These changes can be attributed to the improvement in the martensite morphology, structural refinement, and grain boundary purification.

Albuquerque et al. (Ref 4) studied the influence of grain size and temperature on the toughness of CuAlBe SMAs with and without NbNi grain refiner elements. The toughness analysis was based on the V-notch Charpy impact test under temperatures of -150, -100, -50, 0, 50, 100, and 150 °C. A statistical analysis of the results led to the conclusion that the toughness of both alloys was influenced by temperature and grain size. The CuAlBe alloy with grain refiners absorbed higher impact energy than the same alloy without grain refiners showing that the refining elements improved the material toughness.

Albuquerque et al. (Ref 14) carried out a nondestructive evaluation of grain size influence on the mechanical properties of a CuAlBe SMA with and without grain refiners. Ultrasonic signal processing, considering only the longitudinal velocity, was used for the nondestructive evaluation. The mechanical tensile testing verified that the addition of grain refiners increases the stress and strain of the alloy. Thus, the modulus of elasticity and consequently the ultrasonic velocity, as well as the stress and strain values of the CuAlBe alloy revealed to be fully dependent on the grain size. Also, ultrasonic analysis showed this alloy to be an excellent sound, vibration, and mechanical wave absorber, presenting a high attenuation coefficient related to the wave scattering through the grains.

Araya et al. (Ref 15) evaluated the properties of a superelastic CuAlBe SMA under cyclic loading to assess its potential for applications in seismic resistant designs. Wires with diameters equal to 0.5 mm, previously heated for different time periods, were tested to study the effect of grain size. The results showed that an increase in grain size led to an increase in the equivalent damping and a reduction in the forward transformation and ultimate stresses.

Adnyana (Ref 16) chose a CuZnAl alloy containing a small quantity of Zr to study the grain size influence on transformation temperatures. Various thermal treatments were performed to obtain a range of grain sizes. All the transformation temperatures shifted to lower values as the grain size decreased, except for the temperature at which no further shape change occurred on cooling. The authors showed that a grain size reduction ranging from 35 to 50 μm caused a reduction of approximately 15 °C in the transformation temperatures. In addition, it was noted that this grain size effect on the transformation temperatures followed the Hall-Petch relationship (Ref 17).

Yang et al. (Ref 18) studied the addition of cerium (Ce) to a CuZnAl alloy as a grain refiner, evaluating its influence on the phase transformations and mechanical properties. They found that the addition of cerium was very effective to reduce the grain size, since the alloy without the refiner elements had a

granulometry greater than 1000 μm, and with the addition of a small amount of Ce, a granulometry value of 30 μm was reached.

Since the grains of CuAlBe alloy are coarse, its use in several industrial applications is often unviable (Ref 4). In this sense, the main goal of this work was to study the improvement of the mechanical properties of the CuAlBe alloy through the refinement of its grains by adding different Cr contents, not yet considered in the literature: 0.1, 0.2, 0.3, and 0.5 wt.% Cr, to its austenitic state.

Optical microscopy was used to analyze the Cr effect on the alloy grain size, and scanning electron microscopy (SEM) and x-ray diffraction (XRD) were used to identify the alloy contents and phases, respectively. Also, to investigate the influence of the different Cr percentages on the mechanical properties of the alloy, tensile and hardness tests were carried out. Finally, to determine the phase transformation temperatures and evaluate the effect of the Cr contents on their values, thermal analytical tests using differential scanning calorimetry (DSC) were performed.

As well as studying the effect of adding a Cr inoculant to improve the mechanical properties of the CuAlBe alloy, a comparison was made between the findings observed here and those using different grain refiner elements described in the literature. As such, this work contributes to enhance the knowledge about this alloy, especially on the effects of adding Cr to its austenitic state.

This article is organized as follows: the next section describes the procedures to produce the alloys in the as-cast condition until the finished machined state, as well as the equipment and techniques used to analyze the effect of the Cr refiner to reduce the grain size of the CuAlBe alloy. In section 3, the findings from the analyses are presented and discussed. Finally, in section 4, the influences and advantages of adding the element Cr to the CuAlBe alloy are enumerated.

2. Materials and Methods

CuAlBeCr and CuAlBe alloys, previously weighed in the relative proportion of the following nominal chemical compositions: Cu-11.8Al-0.6Be-*X*Cr, where *X* = 0.1, 0.2, 0.3, and 0.5 (wt.%), and Cu-11.8Al-0.6Be (wt.%), were prepared. The melting process was carried out using a graphite crucible in an induction furnace at ambient atmosphere. The furnace heating was provided by a generator with an output of 8 kVA manufactured by Politron (Brazil). After the melting process, the alloys were chill cast in rectangular sections of 30 × 4 mm and 40 mm of length. The melting time for each casting was approximately 15 min.

After casting, the alloy ingots were submitted to a homogenizing heat treatment at 850 °C for 12 h in an electric resistance furnace and water-quenched soon after.

After the heat treatment, the ingots were metallographically prepared using alumina for polishing and a watery solution of ferric chloride for chemical etching. Then, the grain size was evaluated by optical microscopy at room temperature. A digital camera attached to the microscope (Axiotech 30 model, from Carl Zeiss, Maple Grove, MN, USA) acquired images of the ingot surfaces. Additionally, an image of each ingot surface was acquired for further macrostructural analysis.

The analysis by SEM permitted the chemical mapping, based on the intensities of the characteristic x-rays by energy dispersive x-ray spectroscopy (EDS), by semi-quantitatively

measuring the component element concentrations in the alloys with Cr inoculant. This analysis was performed in a vacuum, using the LEO 1430 SEM model, from Oxford Instruments (Oxfordshire, UK).

To identify the material phases present in the alloy ingots, the XRD test was performed at room temperature, using a diffractometer (D5000 model, Siemens, Lincoln, UK) with $\text{CuK}\alpha$ radiation equal to 1.54 nm.

The samples for the tensile tests were prepared by melting approximately 2 kg of the alloy in a graphite crucible at ambient atmosphere with inductive heating. The inductive heating was carried out in a furnace with a medium frequency generator from Polatron (Brazil) with a power output of 30 kVA. After melting, the alloys were poured into a steel mold with dimensions of $187 \times 35 \times 40 \text{ mm}^3$, with capacity for six cylindrical samples with a 16 mm diameter and 110 mm length. More detailed information on the preparation of the samples can be found in Ref 4.

The tensile tests were carried out on the 6 ingots of each material using a static and dynamic tensile machine, ServoPulser EHF model from Shimadzu Corporation (Kyoto, Japan), equipped with a 50 kN cell load, heating and cooling chamber. All tests were carried out at a strain rate of $3.0 \times 10^{-4} \text{ s}^{-1}$ at room temperature. The part of the samples between the grips was 40 mm in length.

The mechanical property evaluation of the alloys was complemented with nine hardness measurements on each ingot. This evaluation was done with a RASN-RBD durometer

manufactured by Panambra Técnica Importação e Exportação Ltda (São Paulo, Brazil) and the Rockwell A (HRA) method using a load of 60 kgf, a 120° diamond conical penetrator and a penetration time of 10 s.

The analysis by DSC determined the A_s (starting of the austenite formation), A_f (ending of the austenite formation), M_s (starting of the martensite formation), and M_f (ending of the martensite formation) phase transformation temperatures. The test procedure was performed in an air conditioned environment at a temperature of around 18°C , using a DSC-60 model made by Shimadzu Corporation (Kyoto, Japan), with heating and cooling rates of $10^\circ\text{C}/\text{min}$ between temperatures from -120 to 50°C . All samples used in the DSC test weighed 30 mg and were obtained from the ingots that had been machined and water-quenched at room temperature.

3. Results and Discussion

Images of the ingot surfaces were acquired after the casting, heat treatment, and metallographic processes. Figure 1(a) is a macroscopic image of the CuAlBe alloy, without Cr inoculant, in which the coarse grains can be seen clearly. In contrast, Fig. 1(b) to (e) clearly shows the effect of the addition of Cr as a grain refiner, the higher the Cr content the smaller the grain size.

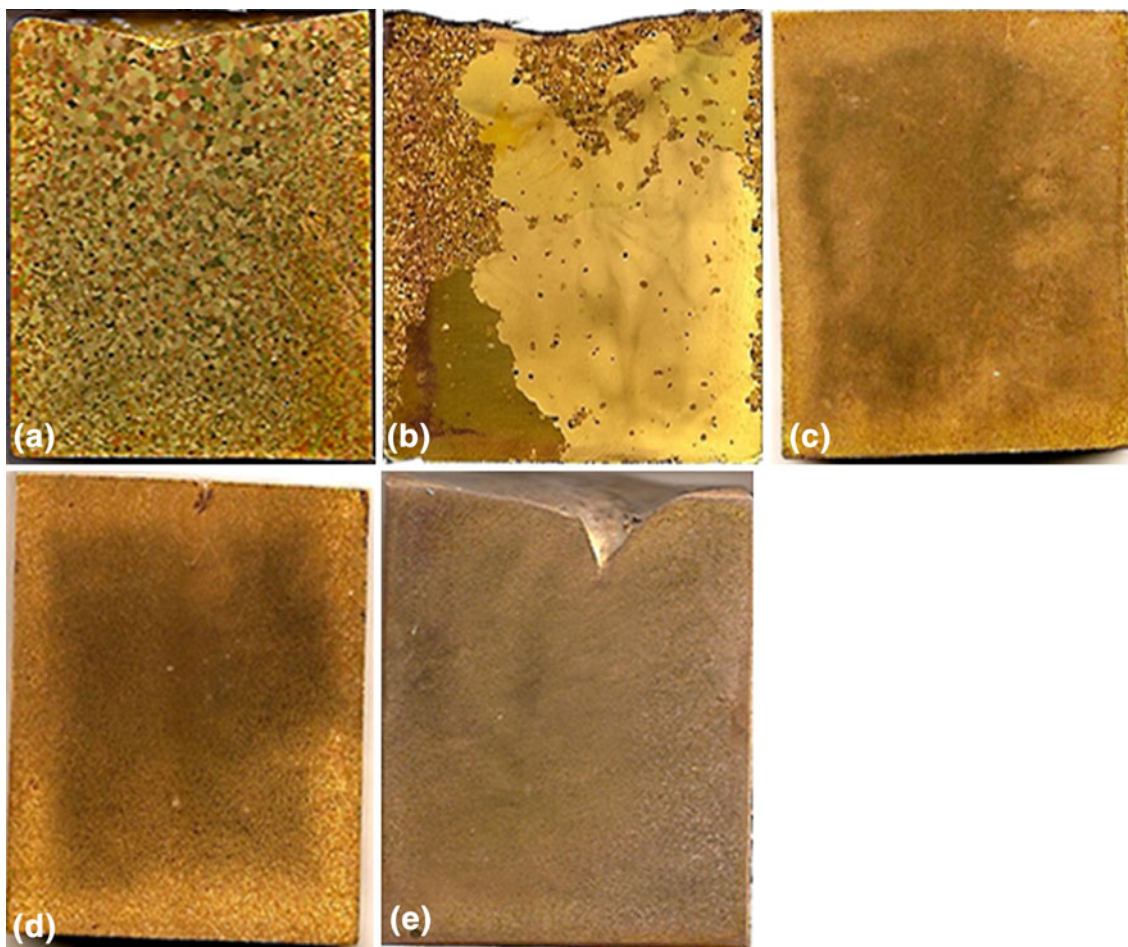


Fig. 1 Macrographs of the CuAlBe alloy: (a) without Cr, and with (b) 0.1, (c) 0.2, (d) 0.3, and (e) 0.5 wt.% Cr

Optical microscopy of the CuAlBe alloy, without grain refiners, but with the same chemical composition and the same manufacturing process followed in this study, gave a mean grain size of 1950 μm in Ref 4. Figure 2(a), which corresponds to the CuAlBe alloy with the addition of 0.1 wt.% Cr, shows a mean grain size of 158 μm , which means a significant reduction in the grain size relative to the alloy without grain

refiners (1950 μm , Ref 4). For the CuAlBe alloys with 0.2, 0.3, and 0.5 wt.% Cr the grain sizes were in the order of 146, 122, and 100 μm , respectively, which are values compatible to those reported in Ref 4 where Nb and Ni were used as the grain refining elements. Therefore, the greater the Cr content in the CuAlBe alloy is, the more significant the grain refining is. The smallest grain size was 100 μm for the 0.5 wt.% Cr alloy,

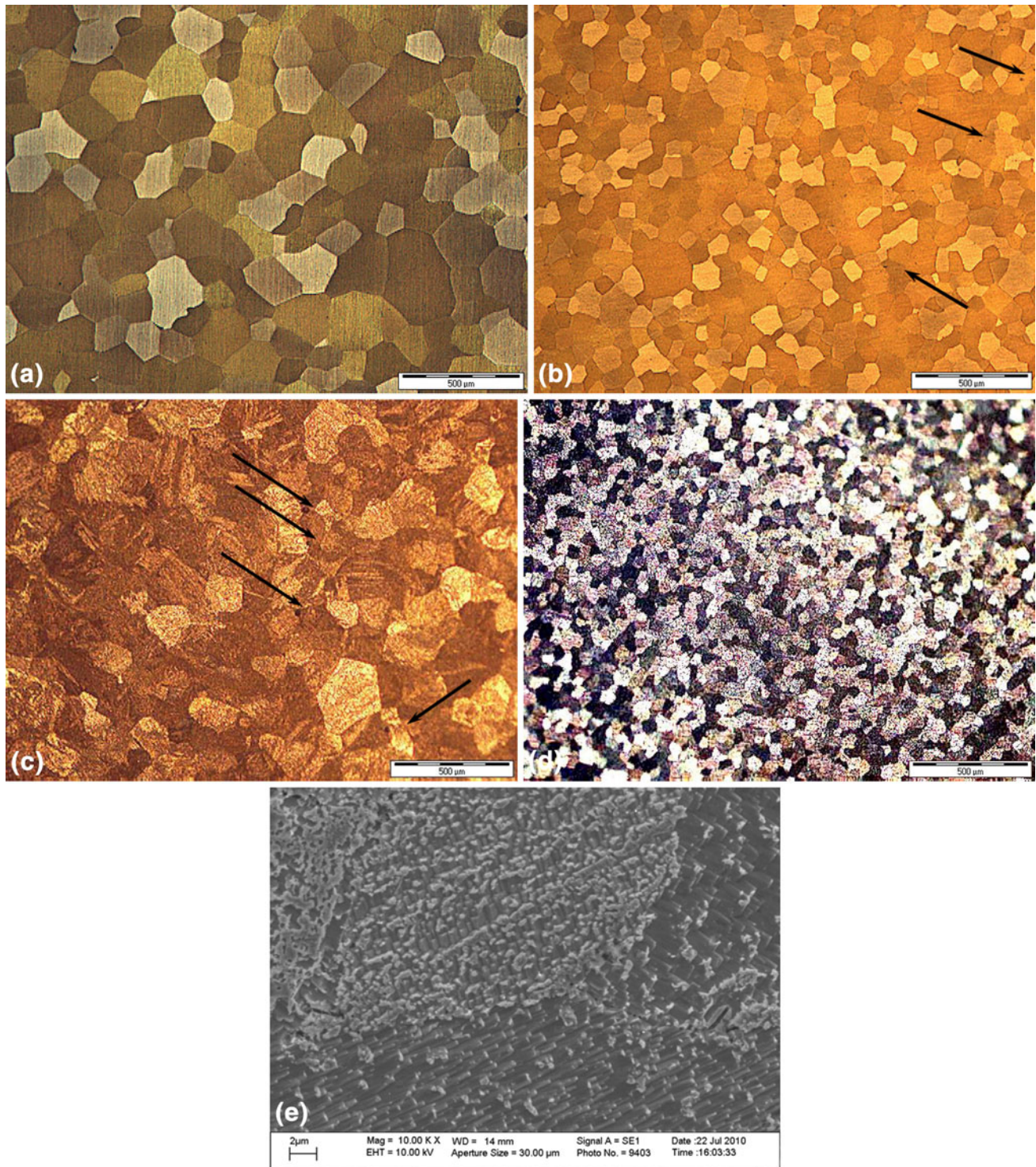


Fig. 2 Optical microscopy of the CuAlBe alloy: with (a) 0.1, (b) 0.2, (c) 0.3, and (d) 0.5 wt.% Cr; (e) SEM of the CuAlBe alloy with 0.5 wt.% Cr

which is approximately equal to the smallest grain size value reported in Ref 4 using Nb and Ni as the grain refiner elements.

The microstructure of the alloy is an aspect of fundamental importance, since it governs the mechanical properties of the alloy, as reported by Albuquerque et al. in Ref 4, 14 when Nb and Ni elements were successfully used as grain refiners of this alloy. Small grain sizes are associated to improved mechanical properties—the effect of embrittling due to coarse grains can be attributed to stress concentration at the ends of the slip bands of grain boundaries. Thus, the larger the grains, the larger the slip bands and, consequently, the higher the stress concentrations (Ref 19).

In Fig. 2(a) to (d), some Cr-rich precipitates, which were later analyzed by EDS, can be seen. At room temperature the CuAlBe alloys with 0.1 and 0.2 wt.% Cr were in the austenite phase, while the CuAlBe alloys with 0.3 and 0.5 wt.% Cr were in the martensite phase. The martensite phase in the alloy with the highest Cr content is seen clearly in the SEM image in Fig. 2(e).

Characteristic mapping by EDS was carried out on the alloys with the Cr refiner element to identify the precipitates

found by optical microscopy in the grain boundaries. The mapping showed a higher Cr content in these precipitates, as can be seen in Fig. 3(a) that presents the mapping obtained from the alloy with 0.5 wt.% Cr which had the greatest quantity of precipitates. If the image of Fig. 3(a) is compared to the image of Fig. 3(b), which refers to the chemical mapping of the Cr element, the dots can be seen to be in identical locations thus concluding that the precipitates were really Cr enriched.

To assess the degree of Cr precipitation, analyses using the secondary electron mode with a 15 kV acceleration voltage were carried out. The secondary electron detector provided images of the polished sample surfaces. Figure 4 shows the increase of the precipitates with the different Cr concentrations. Particularly, for the CuAlBe alloy with 0.5 wt.% Cr, Fig. 4(d) shows a large increase of Cr-rich precipitates. The precipitates are located inside the grains and mainly at the grain boundaries.

Figure 5 shows the four XRD patterns for the CuAlBeCr alloys with the different Cr contents. Comparing the patterns acquired here to the ones found by Rodriguez et al. in Ref 20 for a CuAlBe alloy, one can conclude that the diffraction

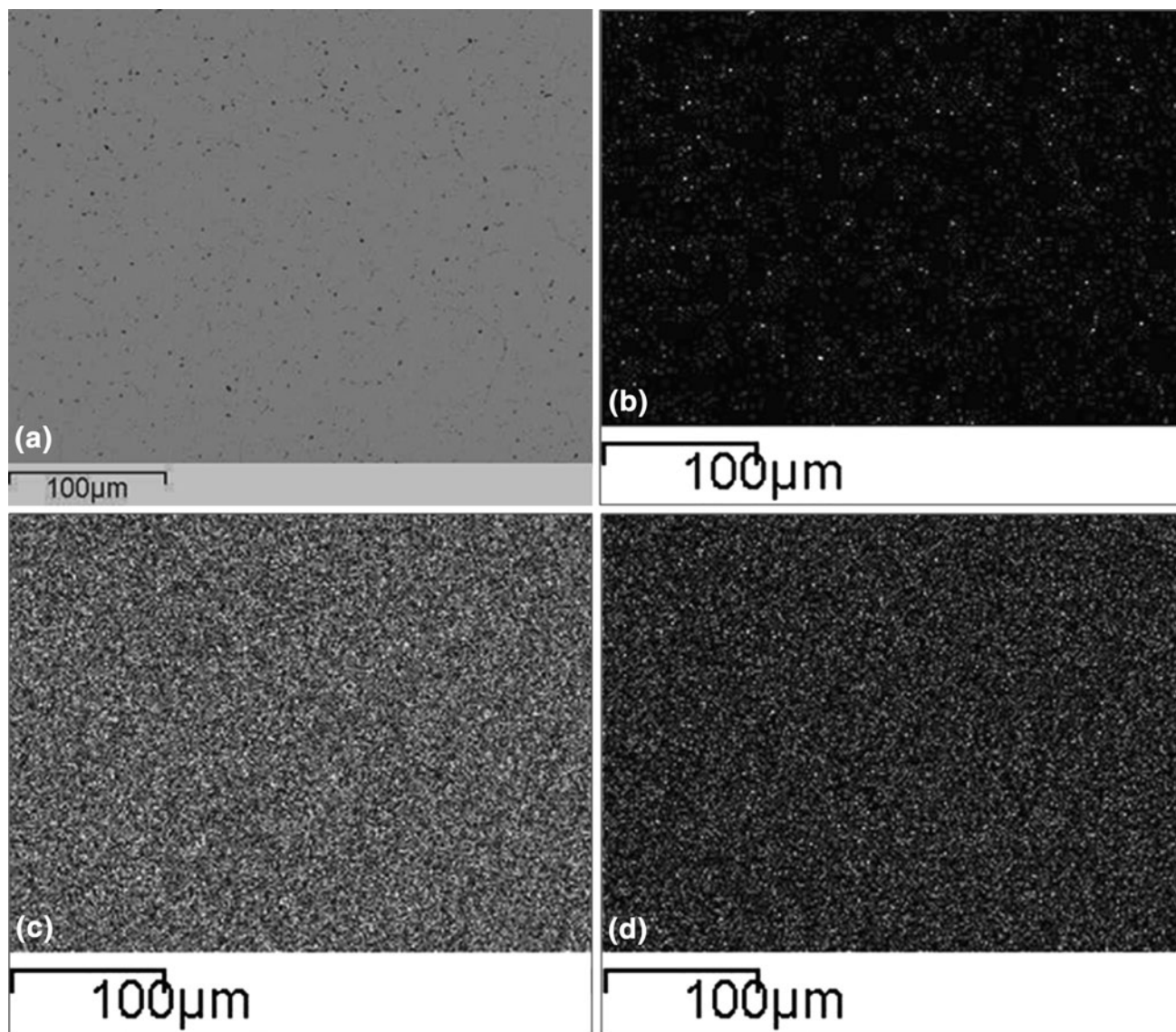


Fig. 3 Chemical mapping of the CuAlBe alloy with 0.5 wt.% Cr for: (a) all elements, (b) Cr, (c) Cu, and (d) Al (obtained by EDS)

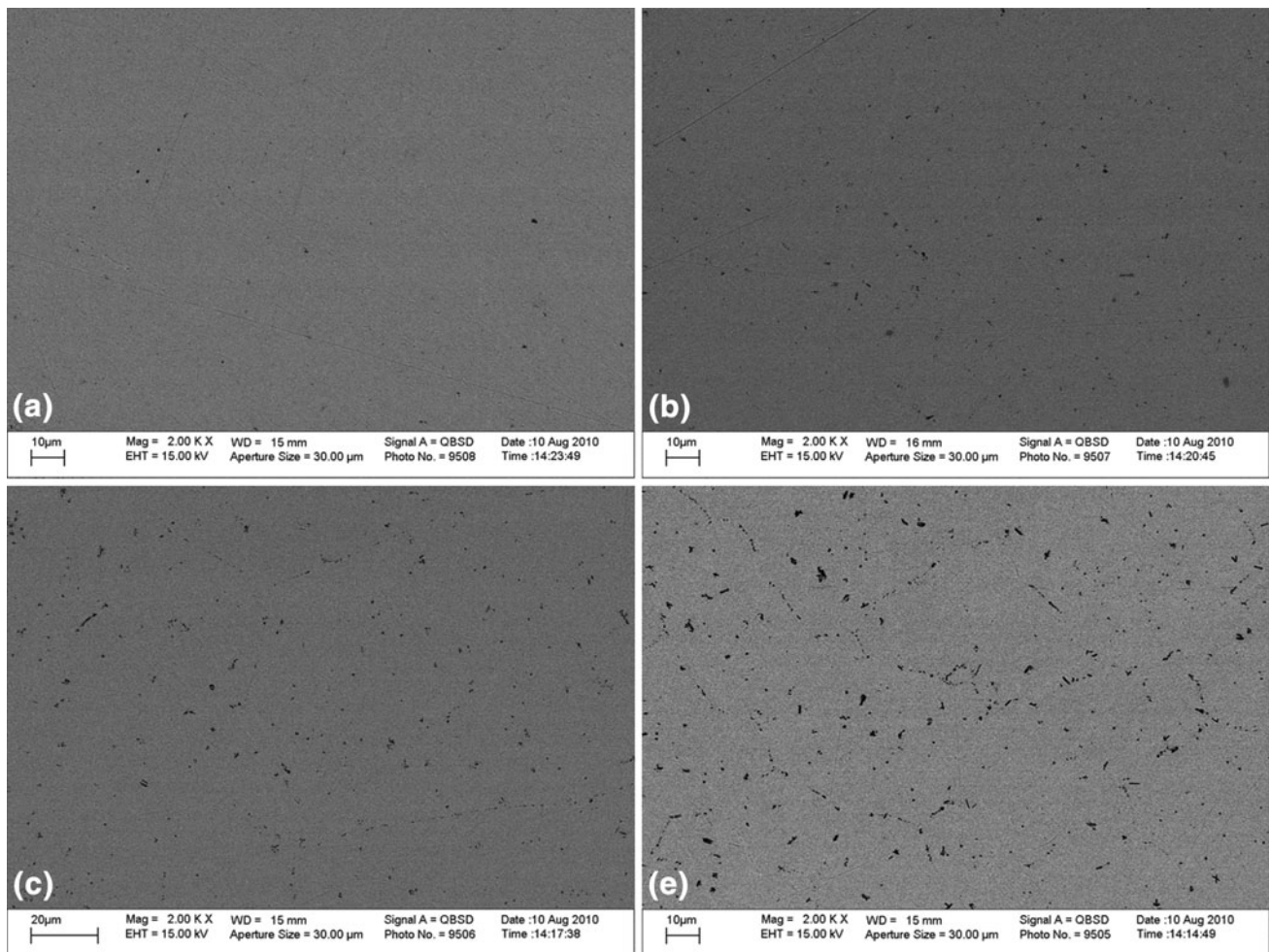


Fig. 4 SEM of the CuAlBe alloy with: (a) 0.1, (b) 0.2, (c) 0.3, and (d) 0.5 wt.% Cr

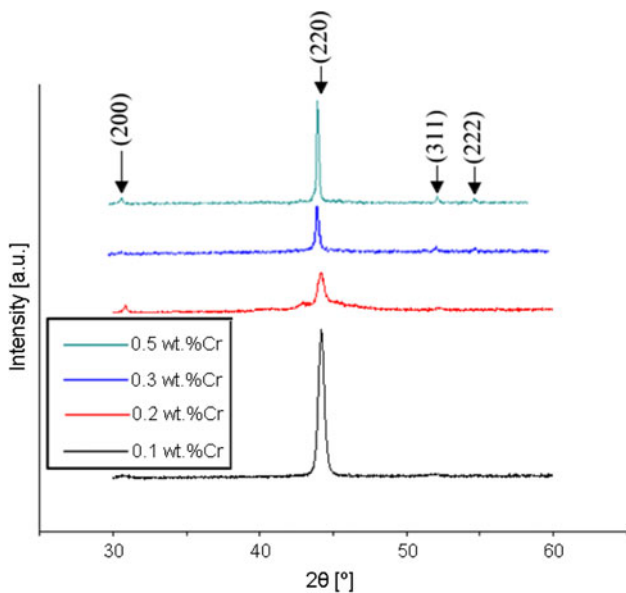


Fig. 5 X-ray diffractograms of the CuAlBe alloy with 0.1, 0.2, 0.3, and 0.5 wt.% Cr

patterns are very similar. Using, as reference, the body-centered cubic lattice with DO3 symmetry, a characteristic of CuAlBe alloys (Ref 21), the indices of these patterns were determined, as well as the lattice parameter of the β phase, $a = 5.79$ nm. In the alloy samples with 0.1, 0.2, and 0.3 wt.% Cr, similar diffracted patterns were recognized.

As mentioned before, the chemical composition of the alloy has a strong influence on the phase transformation temperatures, as can be seen in Fig. 6 that includes the thermograms of the CuAlBe alloys with the addition of 0.1, 0.2, 0.3, and 0.5 wt.% Cr. The phase transformation temperatures of the studied alloys were established by examining the heating and cooling curves.

Table 1 gives the phase transformation temperatures found for the CuAlBeCr alloys, revealing that the addition of Cr alters significantly the austenite \leftrightarrow martensite phase transformation temperatures: the starting and ending transformation temperatures increase as the Cr concentration is augmented. Hence, it was concluded that the addition of Cr affects the transformation temperatures of the CuAlBe alloy. The correlation that can be established between the phase transformation temperatures obtained by DSC and the Cr-enriched precipitates found by optical microscopy (Fig. 2b to d) and further analyzed by SEM

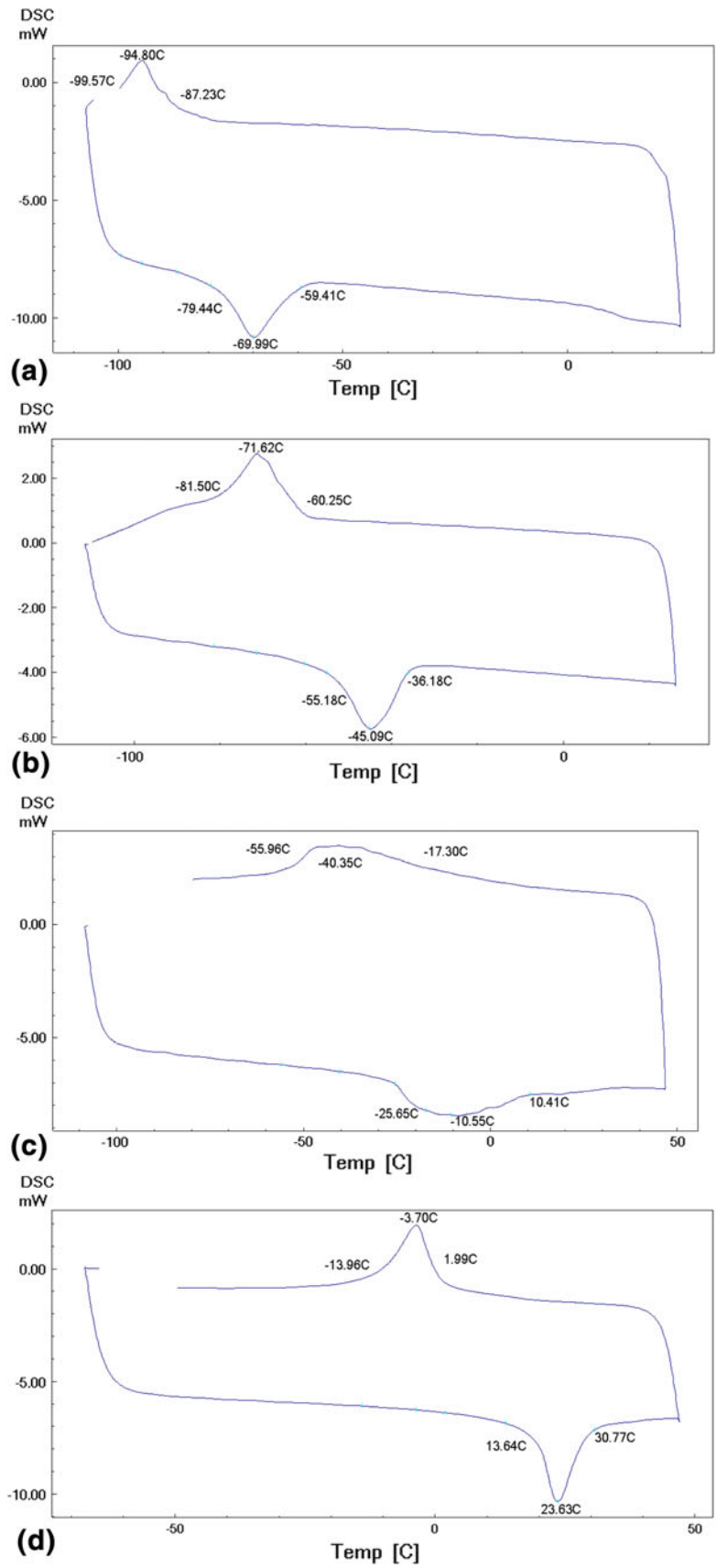


Fig. 6 DSC thermograms of the CuAlBe alloy with: (a) 0.1, (b) 0.2, (c) 0.3, and (d) 0.5 wt.% Cr

(Fig. 3, 4) is that the Cr element entered into solution with the Cu, Al, and Be alloy constituents. This caused the precipitation at the grain boundaries, the removal of the Be element from the alloy matrix, and promoted variations in the phase transformation temperatures.

Figure 7 presents the stress-strain graphic obtained from the static tensile test applied to the CuAlBeCr alloys. As the alloy with 0.1 wt.% Cr, Fig. 7(a), at room temperature was in the austenite phase, the curve is initially characterized by a linear region centered at around 159 MPa (stress) and 0.8% (strain), corresponding to the elastic deformation of the austenite. Then, a transitory curve associated to the starting of the austenite to martensite transformation, induced by stress, is extended to the point of 230 MPa and 1.4%, followed by an approximately linear curve until the rupture of the material around 655 MPa and 9.8%. For the alloy with 0.2 wt.% Cr, Fig. 7(b), the same linear curve found for the previous alloy is observed until

260 MPa, followed by a curvature that represents the starting and ending of the stress-induced martensite transformation. The rupture of this alloy occurred in an apparently brittle mode with mean stress of around 805 MPa and mean deformation superior to 11.0%. That is, this alloy presented rupture values higher than the ones found for the alloy with 0.1 wt.% Cr.

As the studied alloy with 0.3 wt.% Cr consisted of martensite, and the main aim of this work, as already mentioned, was to study the effects of adding Cr to the CuAlBe alloy in its austenite state, the material was analyzed by tensile test at a temperature of 100 °C and a strain rate of $3.0 \times 10^{-4} \text{ s}^{-1}$. In the curve illustrated in Fig. 7(c) for this alloy, the rupture occurred for a mean stress of around 804 MPa and a mean strain of around 10%. As the alloy with 0.5 wt.% Cr was also formed by martensite phase, the tensile test was also performed at 100 °C to ensure the austenitic state. In the stress-strain curve illustrated in Fig. 7(d) for this alloy, a linear region up to approximately 30 MPa followed by a small curvature, which represents the starting and ending of the martensitic phase transformation induced by stress, is observed. The rupture of this occurred at a mean stress of around 744 MPa and a mean strain of around 10%.

The Rockwell A hardness measurement was performed by averaging nine measurements from each CuAlBe alloy with 0.1, 0.2, 0.3, and 0.5 wt.% Cr, shown in Table 2. The mean hardness decreased as the chromium content increased. This behavior may be related to the phases of the alloy at room temperature, since the martensite phase (alloys with 0.3 and 0.5 wt.% Cr) is softer than the austenite phase (alloys with 0.1 and 0.2 wt.% Cr).

Table 1 Phase transformation temperatures of the CuAlBe alloys in function of wt.% Cr

Transformation temperatures, °C	Cr, wt.%			
	0.1	0.2	0.3	0.5
Ms	-87.23	-60.25	-17.30	1.99
Mf	-99.57	-81.50	-55.96	-13.96
As	-79.44	-55.18	-25.65	13.64
Af	-59.41	-36.18	10.41	30.77

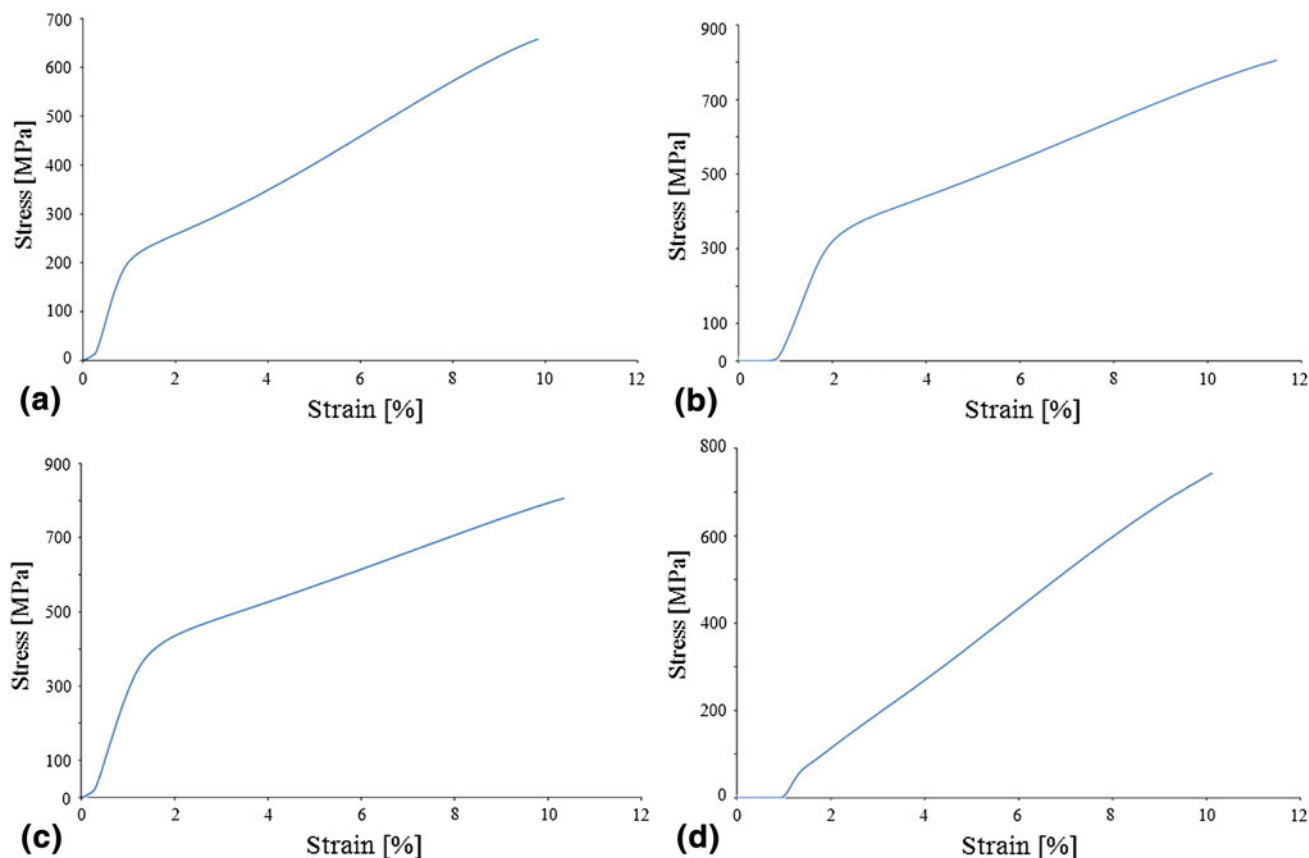


Fig. 7 Stress-strain curves of the CuAlBe alloy with: (a) 0.1, (b) 0.2, (c) 0.3, and (d) 0.5 wt.% Cr

Table 2 Rockwell hardness of the studied CuAlBe alloys

Cr, wt.%	Rockwell hardness, HRA
0.1	64.34 ± 1.10
0.2	62.73 ± 1.05
0.3	61.44 ± 2.11
0.5	56.40 ± 0.47

4. Conclusion

In this study, CuAlBeCr alloys with different Cr contents were prepared at ambient atmosphere and characterized using optical microscopy, SEM, XRD, and DSC. Also, tensile and hardness tests were carried out on the alloy samples. The findings of this work can be summarized as follows:

- (1) The manufacturing and application of the CuAlBeCr alloys are viable at ambient atmosphere.
- (2) The experimental procedures carried out on the CuAlBeCr alloys with 0.1, 0.2, 0.3, and 0.5 wt.% Cr, provided important data of their microstructural characterization. For example, regarding the alloy in its austenite phase (up to 0.2 wt.% Cr content), the metallographic analysis disclosed a grain refinement around 13 times higher than the CuAlBe alloy.
- (3) The DSC analysis carried out on the CuAlBeCr alloys showed that the Ms, Mf, As, and Af values vary significantly with the wt.% Cr content, and that they are greater than the CuAlBe alloy values. This behavior is possibly due to the formation of Cr-rich precipitates and subsequent reduction of Be in the alloy matrixes.
- (4) The behavior assessed by the hardness test on the CuAlBeCr alloy samples with different Cr contents was, in most of the cases, in accordance to the expected behavior for the material phases involved. The highest hardness values were found, in the temperature range in which simultaneous effects of recrystallization and precipitation occurred, due to the intensification of brittle phases.
- (5) As the solidification conditions were practically the same for the CuAlBe and CuAlBeCr alloys, it is possible to conclude that the Cr inoculant had a reducing effect on the grain size and, thus, contributed considerably to diminish the embrittlement of the studied alloys.
- (6) The CuAlBe alloys with 0.3 and 0.5 wt.% Cr showed quite a significant reduction in grain size (grain size around 100 μm). On the other hand, the CuAlBe alloy with 0.2 wt.% Cr revealed higher stress-strain values than the other CuAlBe alloys with Cr. Also the low martensitic transformation temperatures that were observed for this same alloy with 0.2 wt.% Cr, suggests that this alloy is the most suitable for applications that require low transformation temperatures.

In this work, to assess the effect of adding different Cr contents to the CuAlBe alloy in its austenitic state on the mechanical properties, monolithically loads were used in the tensile tests. A new study of this alloy should be made based on data gathered from the dynamic tensile tests as its performance against fatigue could be of great importance in various potential applications.

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