Original Research Article

Evaluation of Nb-Ni Influence on the Mechanical Behavior in a Cu-Al-Be Shape Memory Alloy

José Joelson de Melo Santiago[1*], Carlos Cassio de Alcântara[2], Enilson de Sousa Costa[2], Ieverton Caiandre Andrade Brito[3]

(Corresponding author: e-mail: Joelson.santiago@hotmail.com)
[1]Federal University of Campina Grande, Campina Grande/PB, 58429-140, Brazil
[2]Federal Rural University of the Semi-Arid, Caraúbas, RN, 59780-000, Brazil
[3] Federal University of Paraíba, Department of Mechanical Engineering, João Pessoa/PB, 58059-900, Brazil

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ABSTRACT

Aims: the objective was to investigate the modifications induced by grain refiners on microstructural and mechanical behaviour of Cu-Al-Be shape memory alloy
Study design: The experiment was conducted in a completely randomized design.
Place and Duration of Study: the experiment was carried out at the Laboratory of Rapid Solidification of the Center Technology - CT, Federal University of Paraíba – UFPB, João Pessoa Campus, Paraíba, Brazil, between October 2017 and December 2018.
Methodology: The alloys were prepared by induction melting and hot rolled into strips of 1.0 mm thickness at room temperature without protective atmosphere, followed of heat treatments. Subsequently the microscope analysis, differential scanning calorimetry (DSC), and mechanical tests were carried out.
Results: The shape memory alloys produced present phase transformations corresponding to the superelastic effect (SE). Grain size reduced considerably with increases content of Nb-Ni. Additionally the mechanical tensile testing and hardness tests verified that the addition of Nb-Ni increases the stress of the alloy.
Conclusion: The manufactured of Cu-Al-Be alloys by induction melting and hot rolled without protective atmosphere is viable. The microstructure analysis shows the grain refinement in Cu-Al-Be alloys containing 1.0wt% and 1.5wt% of Nb-Ni alloy with considerable reduction in grain size. The reduction in the grain size shows the improvement in the hardness and mechanical tensile properties.

Keywords: Shape Memory Alloys; Cu-Al-Be; Grain Refiners; Nb-Ni; Mechanical Strength.

1. INTRODUCTION

Shape memory Alloys (SMAs) were discovered by Arne Ölander, but the importance of these materials came to the forefront, when researches revealed shape memory effect (SME) in Nickel-Titanium (Ni-Ti) alloys [1]. Since then, due the functional properties of SMA, many applications were proposed in different fields such as biomedical [2-4], civil engineering [5,6], automotive [7] and aerospace [8]. SMAs are smart materials which can recover their original shape after large strains (~8%) by heating or removing mechanical load. It occurs because of reversible martensitic transformation (MT) [9,10]. SMAs can present Shape Memory Effect (SME) or Pseudoelastic Effect (PE). Shape Memory Effect was described as ability to return to a predetermined shape on heating above specific temperature, on the other hand Pseudoelastic Effect was described as an ability to recover large deformations after removing mechanical load that caused deformation [11].

Several alloys exhibit functional properties associated with phase transformation, but only alloys such as nickel-titanium (Ni-Ti) and copper (Cu) based alloys have been extensively studied. Researches show that Ni-Ti SMA parts with shape from elementary products such as wires [12], coil springs [13] and Belleville washers [14] have exceptional mechanical properties, however the Ni-Ti SMA parts have high cost to process. On the other hand, copper based SMA have excellent cost-benefit ratio to process and show excellent ductility [15], however the industrial application is limited due to low plastic property, difficulty for machining, and short fatigue life [1,16].

The main Cu-based SMAs are derived from the copper-aluminium (Cu-Al) binary system, in which the stabilization of β-phase at lower temperatures is crucial to improve their thermo-mechanical properties. The β-phase stabilization is achieved through heat treatments and the addition of an element such as manganese (Mn), nickel (Ni) and beryllium (Be) have been used [17]. The addition of small amounts of Be cause to a sharp decrease in the martensitic transformation temperature in Cu-Al alloys close to the eutectoid composition [18].

Among differents Cu-based shape memory alloys, the Cu–Al–Be exhibits a technological interest, due their properties such as mechanical damping capability [19,20], high mechanical strength [21] and resistance to corrosion [22,23]. This alloy has been considered for several applications such as petroleum industries [21,24] and design of seismic resistant structures, due to damping or internal friction, resulting in a significant quantity energy absorbed [25].

The high energy absorption capability and consequently the recoverable strain of Cu-Al-Be depends on the grain size, therefore the effect of grain refiners on the mechanical properties, microstructure and phase transformations have been extensively studied in Cu-Al-Be SMA [26,27]. Current researches have shown that grain refiners can improve mechanical properties of Cu-Al-Be, Cu-Al-Mn and Cu-Al-Zn SMA by means of yield strength and structural optimize. [28-30]. Albuquerque[31] shows that the addition of small amounts (about 0,5% wt%) of Nb element in Cu-Al-Be alloy, cause grain refinement on the order of 19 times in relation to the alloy without Nb. Cândido[32] verified that the presence of up to 0.2% of Cr in similar alloy, provides a significant reduction of the average grain size without causing phase changes in the microstructure.

In this context, its relevant evaluate the behaviour of Cu-Al-Be with addition of small amounts of grain refiners, thus this research studies the modifications induced by grain refiners on microstructural and mechanical behaviour of Cu-Al-Be shape memory alloy strips manufactured by induction melting. A Cu-11.8%Al-0.58%Be shape memory alloy containing a small quantity of Nb-Ni was chosen as a model alloy for the study of grain-size effects on mechanical behavior following previously studies that show the high properties of this alloy [26,31,33].

2. MATERIAL AND METHODS

For the present study. three different Cu-Al-Be-Nb-Ni alloys were prepared by induction melting without protective atmosphere. Pure metals were used: Cu (99.9%). Al (99.9%). Cu-4%Be master alloy and Nb-35%Ni master alloy (wt%). The alloys ingot's nominal composition given in Table1.

Table 1 Percentage Composition of Cu-Al-Be-Nb-Ni Alloy

Alloy ID	**Chemical nominal composition (wt%)**				
	Cu	Al	Be	Nb	Ni
$Alloy_1$	87.12	11.8	0.58	0.32	0.18
$Alloy_2$	86.62	11.8	0.58	0.65	0.35
$Alloy_3$	86.12	11.8	0.58	0.97	0.53

The alloys were cast in graphite crucible in inductive heating in an 8 KVA high frequency furnace followed of ingots manufacture using rectangular molds. Subsequently, the ingots produced were heat treated by homogenized at 850 ºC for 12 hours in an electrical resistance furnace. Thereafter, the ingots were heated to 850°C at each step of hot rolling and cut into strips of 100x10x1 [mm]. The strips were heat treated at 850ºC for 1 hour followed by water quenching at 25ºC to obtain the shape memory effect.

Samples used for metallographic examinations were mechanically ground polished and chemically etched with ferric chloride ($FeCl_3$). The microstructural characterization was analyzed by optical microscopy (OM). Mean grain sizes were determined by grain boundary intersection count method in which the determination of the number of times a test line cuts across or is tangent to grain boundaries.

The transformation temperatures (TTs) were determined by differential scanning calorimetry (DSC) using Shimadzu DSC-60 calorimeter machine. DSC measurements were performed in argon atmosphere through one heating/cooling cycle from -120ºC to 60 ºC with heating/cooling rates 10ºC.min^{-1}. The TTs were determined by drawing tangent lines to the beginning and end regions of the transformation and baseline of the heating and cooling curves.

The tensile tests were carried out at room temperature (about 25 ºC). with maximum applied strains of 6%. using the Shimadzu static-dynamic Servo pulser EHF machine. equipped with a 50kN load cell. The hardness tests were carried out with maximum load of 100 kgf for 10 sec.

3. RESULTS AND DISCUSSION

3.1. Grain Refinement

The grain size for each sample was acquired by optical microscopy. Figure 1 shows the microstructures of Cu-Al-Be-Nb-Ni SMAs. The images present typical optical micrograph of the austenite phase (Fig. 1a. Fig. 1b. Fig. 1c). The samples preparation process (hot roller process) induced the phase transformation. So. it is possible to visualize the martensite phase in hot rolled samples without heat treatment Figure 1d. Grains in the ingots were roughly equiaxed, therefore with similar size in the longitudinal and transversal faces. It is also possible to verify the presence of precipitates characterized by dark spots.

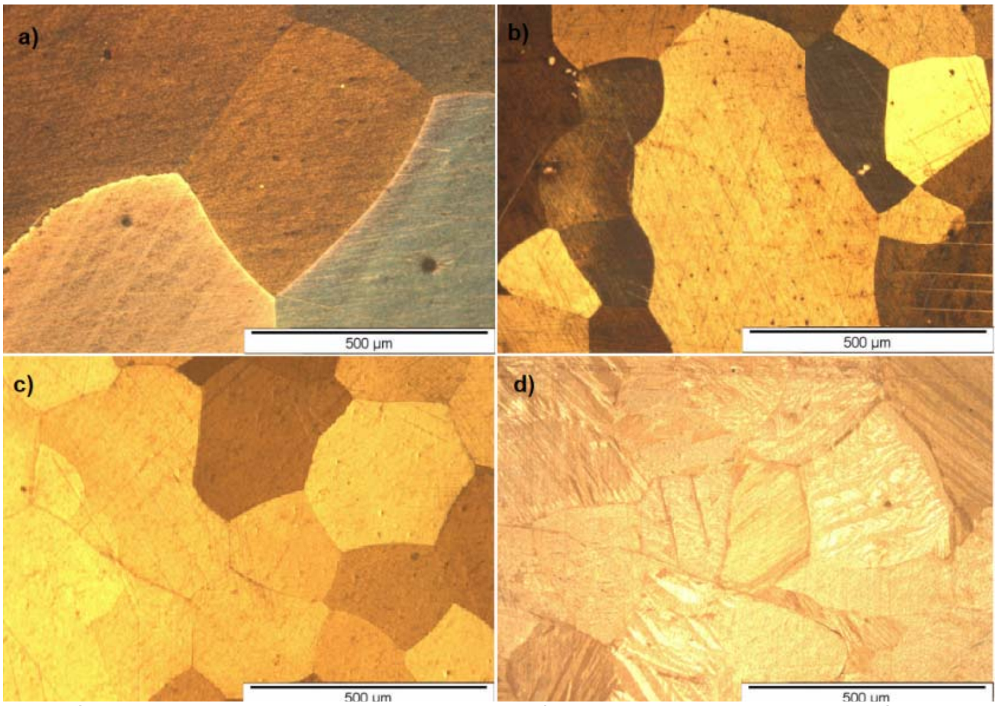

Figure 1 - Optical micrograph illustrating the presence of austenite and martensite in the Cu-Al-Be-Nb-Ni SMA: (a) Alloy$_1$. (b) Alloy$_2$. (c) Alloy$_3$. (d) Alloy$_3$ hot rolled.

The average grain sizes determined by arithmetic mean are shown in Figure 2 for each alloy. As general behavior, it can be observed that the grain size decrease considerably with the increase of Nb-Ni content.

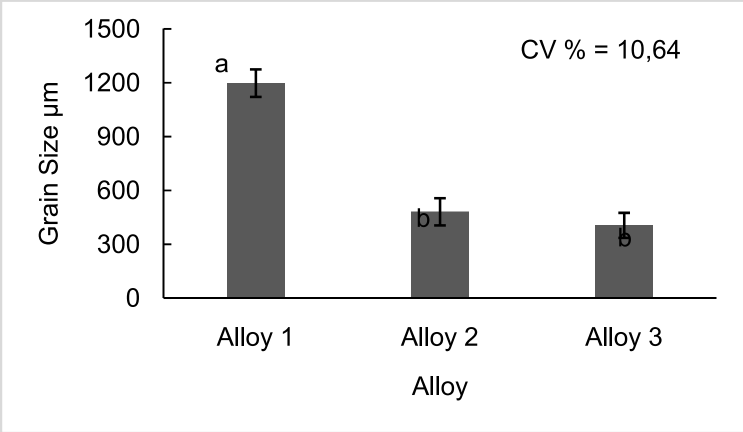

Figure 2 - The average grain sizes.

As can be seen in Figure 2, the obtained average grain size is 1200µm 469µm and 394µm for $Alloy_1$ $Alloy_2$ and $Alloy_3$. respectively. It is worth mentioning that grain refinement of Nb in Cu-Al-Be SMAs have also been reported acts such as strengthening mechanism [33,34]. The grain size refinement is influenced of fine Nb-rich precipitates that form nucleation sites during the solidification. Furthermore. grain growth is inhibited due Nb precipitates that appear by the pinning mechanism [35].

3.2. Thermal Characterization

After the microstructural evaluation the samples underwent DSC to determine the critical phase transformation temperatures without applied load. The samples used in these analyses were heat treated in the same way of the SMA strips produced. In Table 2 is listed the average of transformation temperatures for each alloy. The phase transformation temperatures were defined as: Martensite Finish Temperature (M_f), Martensite Start Temperature (M_s), Austenite Start Temperature (A_s) and Austenite Finish Temperature (A_f)

Table 2 - Critical temperatures obtained from DSC for the Cu-Al-Be-Nb-Ni SMA.

Alloy ID	**Temperatures [°C]**			
	M_f	M_s	A_s	A_f
$Alloy_1$	-22.5	19.9	2.2	27.7
$Alloy_2$	-23.7	19.7	5.6	33.5
$Alloy_3$	-27.8	15.5	2.6	19.4

Thermal characterization indicates superelastic behavior at room temperature (about 25ºC. superior to M_s). From Table 2 the austenite temperature intervals span between 2.2ºC to 33.5ºC. Figure 3 shows typical curves resulted from DSC tests for one heating/cooling cycle. In this case. the results are presented only for $Alloy_2$, because all the alloys present similar curves. It was possible to confirm phase transformations throughout the presence of two peaks that characterize the transformations zone.

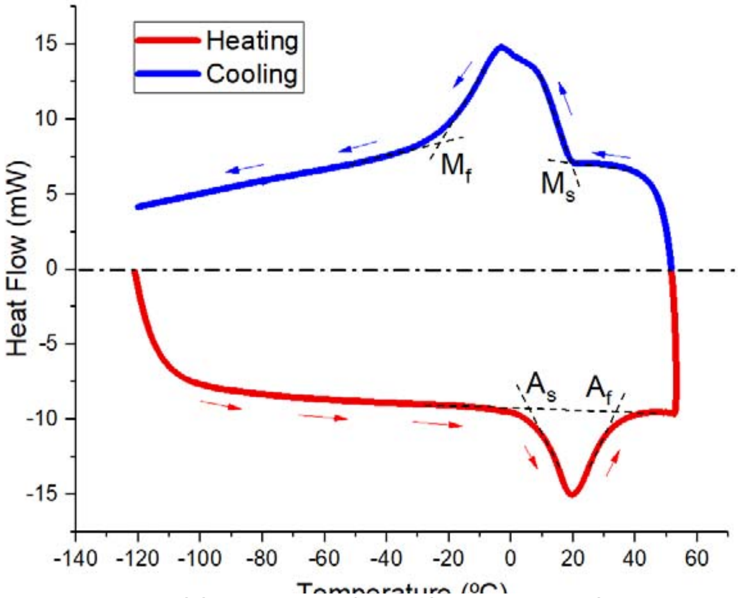

Figure 3 -DSC thermogram for the Cu-Al-Be-Nb-Ni SMA.

3.3. Mechanical properties

As previously discussed, the grain size is an important parameter in polycrystalline specimens. It was observed that as the grain size decreases. the Rockwell hardness increases. The variation of Rockwell A hardness of the samples produced is shown in Figure 4. The averages of values were 71 RCA. 72 RCA and 76 RCA for Alloy$_1$. Alloy$_2$ and Alloy$_3$. respectively. This increase of hardness observed with the addition of Nb. It is believed that this increase is due to the Nb-rich precipitates increase the rigidity of the material.

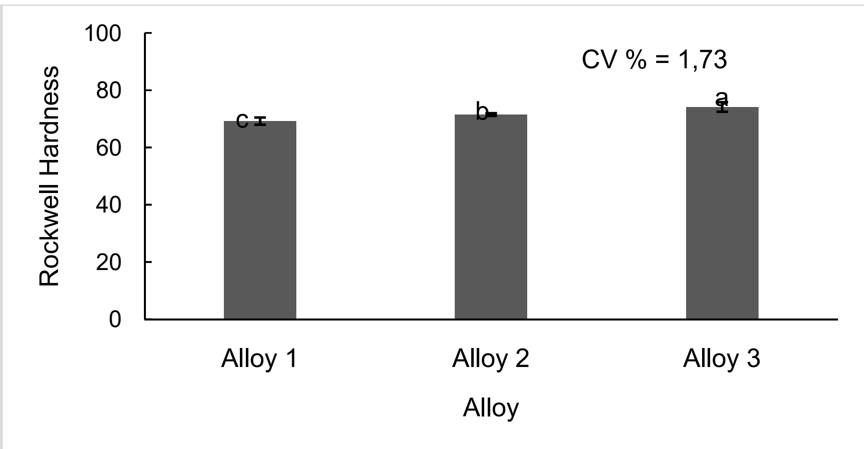

Figure 4 - Rockwell hardness for the Cu-Al-Be-Nb-Ni SMA.

The samples were always heated at temperature higher than A_f and cooling down to the room temperature before tensile tests. Figure 5 shows stress-strain curves obtained at room temperature (~25ºC). On loading, the austenite elastically deforms in initial linear part followed pseudoelastic slope when starts the typical martensite induced transformation.

The conventional tensile test carried out show that the addition of Nb-Ni increases the ultimate strength of the Cu-Al-Be alloy. The maximum strain to the rupture and the rupture stress were 250.7 MPa and 6.2% for $Alloy_1$. 283.2 MPa and 7.1% for $Alloy_2$ and 529.2 MPa and 6.5% for $Alloy_3$. As the grain size decreases the stress corresponding to the end of the initial linear part will referred as the martensite-start stress (σ_{ms}) increases, following a Hall–Petch relation type in β Cu-Al-Be [36,37]. This behavior has been found in other shape memory alloys [26,34,37]. To compare the results between the alloys produced, the σ_{ms} were 44.1MPa, 53.4 MPa and 103.4MPa for $Alloy_1$. $Alloy_2$ and $Alloy_3$, respectively. These results indicate that the effect of grain size on σ_{ms} for Cu–Al–Be is stronger than for the other alloys [34].

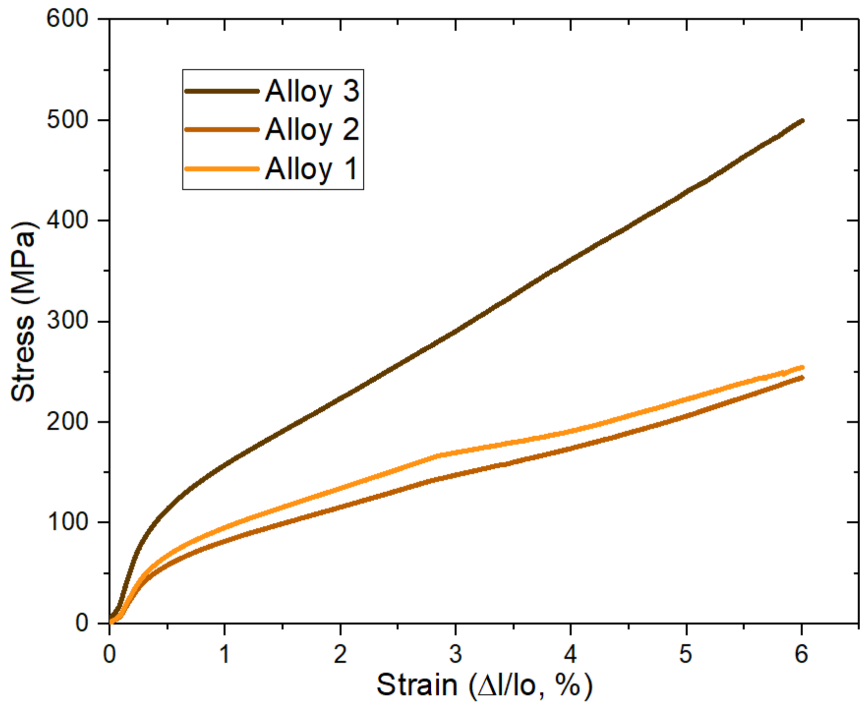

Figure 5 - Typical stress-strain curve to Cu-Al-Be-Nb-Ni alloy T=298K.

It was not possible to distinguish the plastic strain of the martensite that should precede the rupture. The average strength values were 243.2MPa. 255.3MPa and 501.9MPa for $Alloy_1$. $Alloy_2$ and $Alloy_3$. respectively in the maximum strain (6%). The grain refiners seem to have a reducing effect on the ductility of Alloy, as well show in other studies [26,34], so the strength increases considerably to submit the material to the same elongation, this is in accordance with the stiffness values shown previously.

It is known that the mechanical strength increases as larger the volumetric fraction of precipitates and the smaller their dimensions [38]. Orovan's theory explain that the mechanical resistance increases by precipitation, when very small precipitates and a large volumetric fraction are added to the metal matrix. The precipitates work as barriers to the movement of the dislocations increases mechanical resistance [39].

4. CONCLUSION

In this study, the effect of grain size on the mechanical behavior of Cu-Al-Be shape memory alloy has been evaluated and the following conclusions can be drawn:

1. Increasing the amount of Nb-Ni leads to grain reduce considerably (about four times lower) comparing $Alloy_1$ and $Alloy_3$. Grain refinement is considered effective in alloys with a percentage of Nb-Ni greater than 1%. The grain size effects not only on the start critical parameters but on the entire transformation behavior.
2. Cu-Al-Be-Nb-Ni SMA strips manufactured by hot rolled present fully martensitic microstructure. Also, after the heat treatment all the alloys present an austenitic microstructure and the superelastic effect. According with the DSC curves the difference in chemical composition is not enough to change the critical temperatures. The TTs show austenite phase appears in SMA produced at room temperature without loading.
3. Decreasing the grain size improve the mechanical strength values, which increases as grain size is reduced. This mechanical strength-grain size dependence has been found to be governed by the Hall-Perch relationship.
4. The mechanical presence of Nb precipitates presence. increase the rigidity of the material in accordance of showed in literature.

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Ethical declaration and consent are not applicable

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