

Original Research Article

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

ABSTRACT

Aims: the objective was to investigate the mechanical behavior and grain size of a Cu-11.8%Al-0.58%Be shape memory alloy containing 0.5wt%, 1.0wt% and 1.5wt% of Nb-Ni alloy master.

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 2017.

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), tensile test and hardness test 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) 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). SMAs can present Shape Memory Effect (SME) or Pseudoelastic Effect (PE) [1,3].

Several alloys exhibit functional properties associated with phase transformation, but only some alloys such as nickel-titanium (Ni-Ti) and copper (Cu) based alloys have been extensively studied. Ni-Ti alloys have exceptional mechanical properties, however they have high cost and are difficult to process. On the other hand, copper based SMA have excellent cost-benefit ratio to process, however the industrial application is limited due to low plastic property, difficulty for machining, and short fatigue life [4,5].

The main Cu-based SMAs are derived coming from the copper-aluminium (Cu-Al) binary system [6], in which to improve their thermo-mechanical properties is crucial to stabilize β -phase at lower temperatures, thus heat treatments

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and the addition of a third element, such as [manganese \(Mn\)](#), [nickel \(Ni\)](#) and [beryllium \(Be\)](#) have been used. Especially the addition of small amounts of Be [are required, tofor](#) allowing good thermal stability in a broad range of transformation temperatures. [6,7].

Cu–Al–Be SMAs present interesting properties such as mechanical damping capability, high mechanical strength and resistance to corrosion [8,9]. This alloy has been considered for several applications, such as petroleum industries [10,11] and design of seismic resistant structures, due to damping or internal friction, [resulting in aed-of](#) significant quantity energy absorbed [12].

The high strain 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 studied in Cu-Al-Be SMA [9,14]. Current research has 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. [15-18].

In this study, the microstructure and mechanical properties of Cu-Al-Be shape memory alloy strips manufactured by induction melting and hot rolled are analyzed based on the induced microstructural modifications by grain refiners.

2. MATERIAL AND METHODS

For the present study, three different Cu-Al-Be-Nb-Ni alloys were prepared by induction melting without protective atmosphere. The nominal compositions were Cu-11.8Al-0.58Be-0.33Nb-0.18Ni, Cu-11.8Al-0.58Be-0.65Nb-0.35Ni and Cu-11.8Al-0.58Be-0.98Nb-0.53Ni (wt%). [These were referred to as called](#) Alloy₁, Alloy₂, and Alloy₃, respectively. Pure metals were used: Cu (99.9%), Al (99.9%), Cu-4%Be master alloy and Nb-35%Ni master alloy (wt%). The Figure 1 resumes the steps of the experimental work performed.

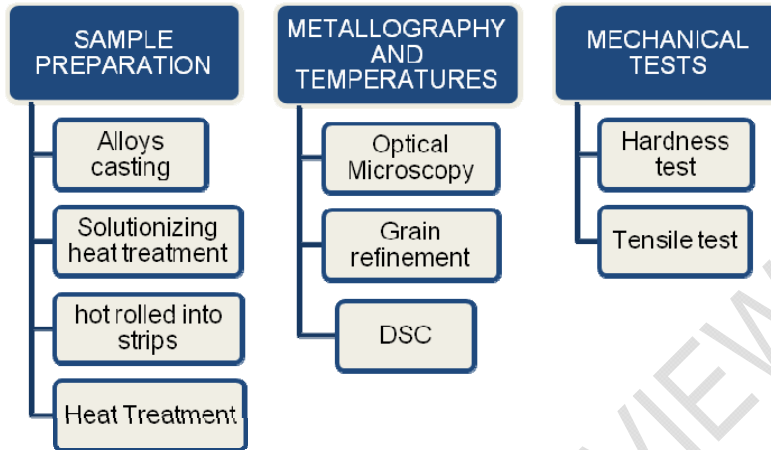


Figure 1 - Workflow diagram.

For each nominal composition was prepared about 0.5kg of the alloy in inductive heating in an 8 KVA high frequency furnace. The alloys were cast in graphite crucible and spills into rectangular steel molds with 22x35x100mm dimensions. Subsequently, the ingots were homogenized at 850 °C for 12 hours in an electrical resistance furnace. The ingots were hot rolled and cut off into 100x10x1[mm] strips. The ingots were hot rolled into a thickness of 1mm and cut off into 100x10x1mm strips. Following this, Later the strips were heat treated at 850°C for 1 hour; the treatment was followed by water quenching at 25°C to obtain the shape memory effect.

Samples used for metallographic examinations were mechanically ground, inded, polished and chemically etched with ferric chloride (FeCl₃). The microstructural characterization was analyzed by microscopy optical. Mean grain sizes were determined by the intercept line method from micrographs obtained of the β phase austenite at room temperature.

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 -120°C to 60 °C with heating/cooling rates 10°C.min⁻¹.

The tensile tests were carried out at room temperature (about 25 °C), with maximum applied strains of 6.0%, 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 gram for 10 sec.

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3. RESULTS AND DISCUSSION

3.1. Grain Refinement

The grain size for samples of different compositions was acquired by optical microscopy. Figure 2 shows the microstructures of Cu-Al-Be-Nb-Ni SMA.

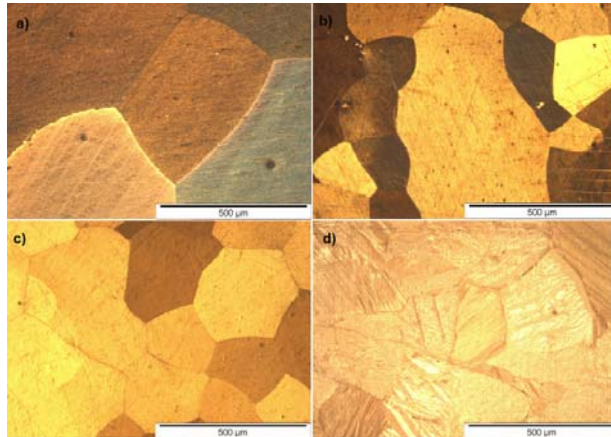


Figure 2 - Optical micrograph illustrating the presence of austenite and martensite in the Cu-Al-Be-Nb-Ni SMA: (a) Alloy₁. (b) Alloy₂. (c) Alloy₃. (d) Alloy₃ hot rolled.

The images present typical optical micrograph of the austenite phase (Fig. 2a. Fig. 2b. Fig. 2c). 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 2d. Grains in the ingots were roughly equiaxed, therefore with similar size in the longitudinal and transversal faces.

The average grain sizes, determined as the arithmetic mean, are shown in Figure 3 for every alloy. As general behavior, it can be observed that the grain size decreases considerably with the increase of Nb-Ni content. The average grain size is 1200 μ m, 469 μ m and 394 μ m for Alloy₁, Alloy₂ and Alloy₃, respectively. It is worth mentioning that grain refinement of Nb in Cu-Al-Be SMA has also been reported acts such as strengthening mechanism [19-20].

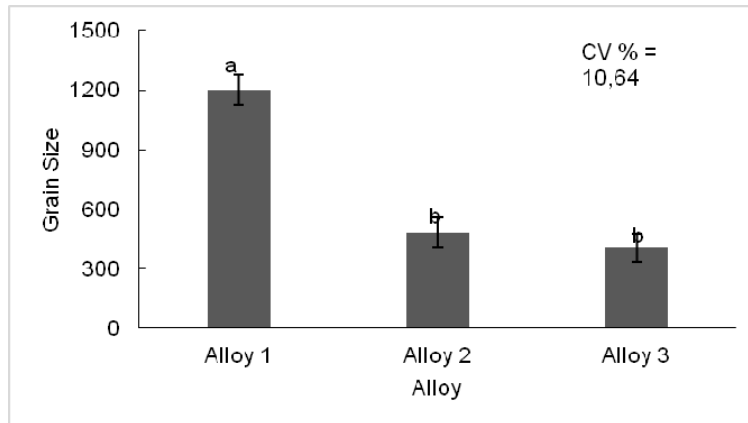


Figure 3 - The average grain sizes.

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 appear by the pinning mechanism [21].

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 this analyze were heat treated in the same way of the SMA strips produced. In Table 1 is listed the average of transformation temperatures for each alloy.

Table 1 - 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	2.9	2.6	15.5

Thermal characterization indicates superelastic behavior at room temperature (about 25°C, superior to M_s). From Table 1 the austenite temperature

intervals span between 2.2°C to 33.5°C. Only the values of A_f and M_s temperatures of Alloy₃ are different considerably.

Figure 4 shows typical curves resulted from DSC tests for one heating/cooling cycle. In this case, the results are presented only for Alloy₃. It was possible to confirm phase transformations throughout the presence of two peaks that characterize the transformations zone.

UNDER PEER REVIEW

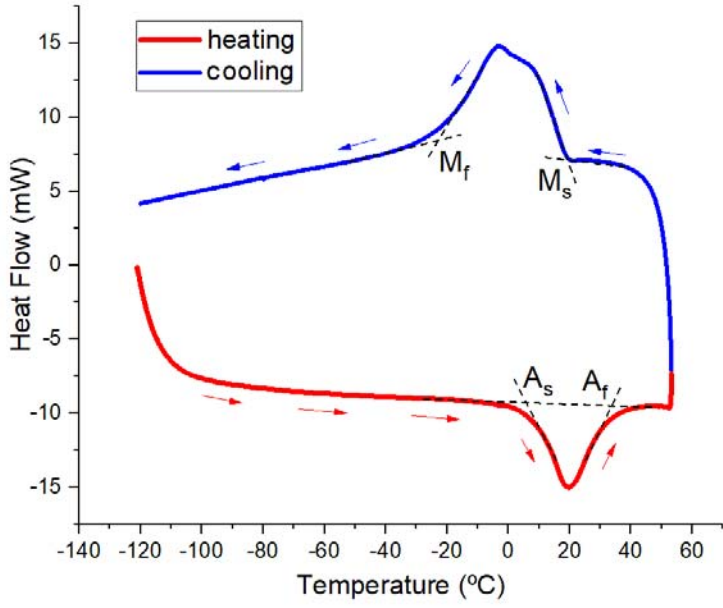
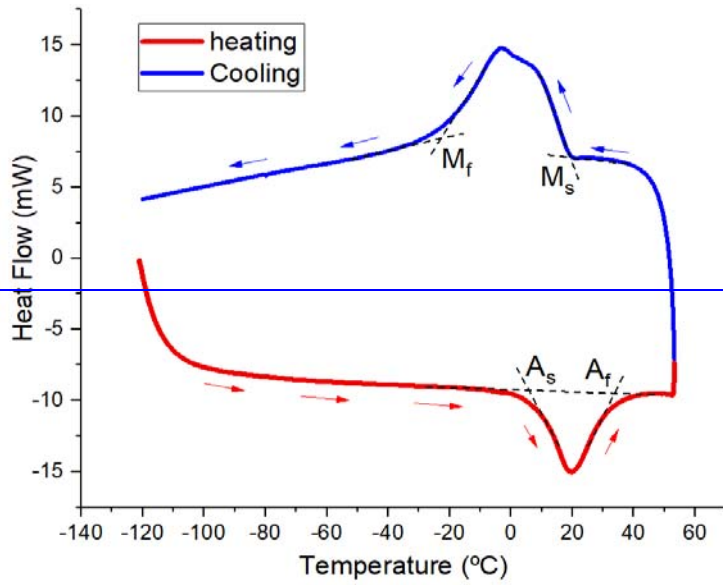


Figure 4 - DSC thermogram for the Cu-Al-Be-Nb-Ni SMA.

3.3. **Hardness Tests**

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 5. The averages of values were 71 RCA, 72 RCA and 76 RCA for Alloy₁, Alloy₂ and Alloy₃, 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.

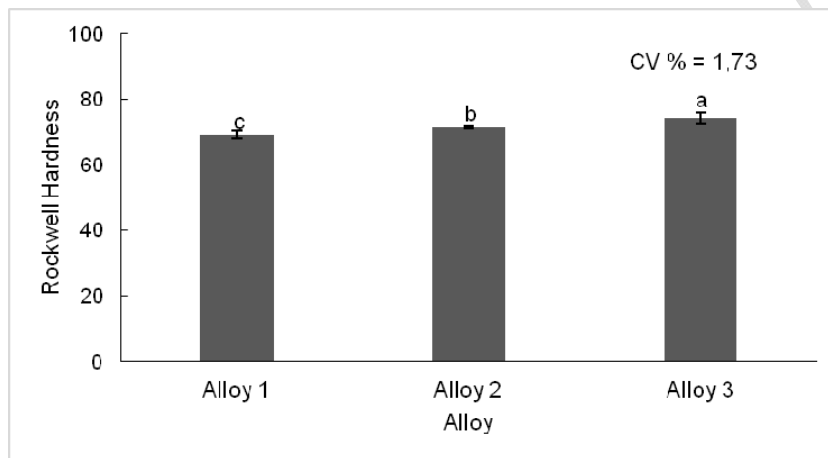


Figure 5 - Rockwell hardness for the Cu-Al-Be-Nb-Ni SMA.

3.4. Tensile Tests

The samples were always heated at temperature higher than A_f than and cooling down to the room temperature before tensile tests. Figure 6 shows the typical stress-strain curve. As the grain size decreases, the critical applied stress to start the martensitic transformation increases, following a Hall-Petch relation type in β CuAlBe [22,23]. The maximum strain to the rupture and the rupture stress were 210.7 MPa and 6.01% for Alloy₁, 283.2 MPa and 7.1% for Alloy₂ and 529.2 MPa and 6.48% for Alloy₃.

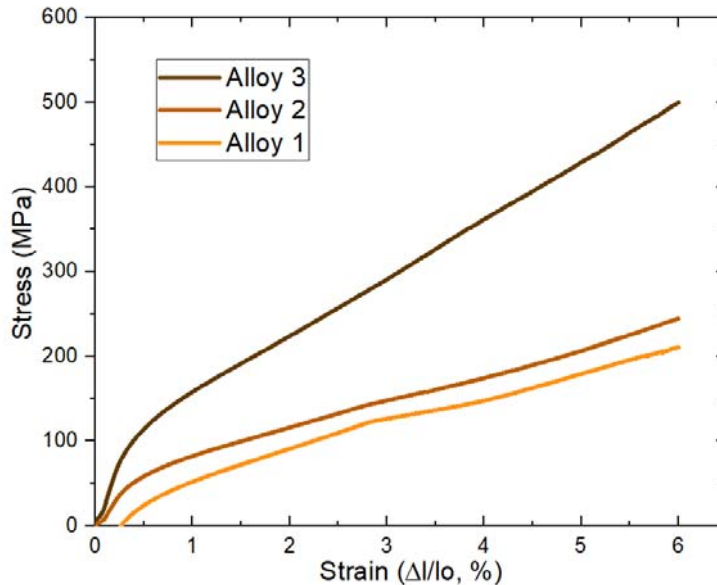


Figure 6 - Typical stress-strain curve to Cu-Al-Be-Nb-Ni alloy T=298K.

The conventional mechanical tensile testing carried out show that the addition of Nb-Ni increases the ultimate strength of the Cu-Al-Be alloy. The austenite elastically deforms in initial linear part followed pseudoelastic slope when starts the typical martensite induced transformation. It was not possible to distinguish the plastic strain of the martensite that should precede the rupture. The average strength values were 144.2 MPa, 255.3 MPa and 501.9 MPa for Alloy₁, Alloy₂ and Alloy₃, respectively in the maximum strain (6%).

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4. CONCLUSION

The microstructure analysis shows the grain refinement in the alloy. Grain size reduced considerably with increases content of Nb. Cu-Al-Be-Nb-Ni SMA strips manufactured by hot rolled present fully martensitic microstructure; also, after the heat treatment the austenitic phase was identified. The DSC curves exhibits that the A_f and M_s temperatures decreases considerably in Alloy₃. The reduction in the grain size shows the improvement in the hardness. The mechanical tensile testing that was carried out showed that the addition of Nb increases the mechanical resistance of the Cu-Al-Be alloy.

REFERENCE

1. K. Otsuka, C.M. Wayman (Eds.). Shape Memory Materials. Cambridge University Press. Cambridge. 1998.

2. Lagoudas. D. Shape Memory Alloys: Modeling and Engineering Applications: Springer Science+Business Media. LLC. 2008.
3. Rao. A. Sirivasa. AR. Reddy. JN. Design of Shape Memory Alloy (SMA) Actuators. Springer International Publishing. 2015.
4. Dasgupta RA look into Cu-based shape memory alloys: Present scenario and future prospects. Journal of Materials Research. p.1681-1698. 2014.
5. Jani. JM. et al. A review of shape memory alloy research. applications and opportunities. Materials & Design. 2014; 56: 1078-1113.
6. Ferreño. IL. et al. Thermal treatments and transformation behavior of Cu–Al–Be shape memory alloys. Journal Of Alloys And Compounds. [s.l.]. v. 577. p.463-467. nov. 2013. Elsevier BV.
7. Belkahla, S., Zuñiga, HF, Guenin, G.. Elaboration and characterization of new low temperature shape memory Cu–Al–Be alloys. Materials Science And Engineering: A; 1993; 169(1-2): 119-124.
8. Oliveira. J.P. et al. Laser welding of Cu-Al-Be shape memory alloys: Microstructure and mechanical properties. Materials & Design. 2018; 148: 145-152.
9. Albuquerque. Victor Hugo C. de et al. Evaluation of grain refiners influence on the mechanical properties in a CuAlBe shape memory alloy by ultrasonic and mechanical tensile testing. Materials & Design. 2010; 31(7): 3275-3281.
10. Oliveira. DF et al. Mechanical Strength Evaluation of a CuAlBe Shape Memory Alloy under Different Thermal Conditions. Materials Science Forum. 2010. 643: 105-111.
11. Oliveira. DF et al. Assessment of Pipe Coupling by Using the Recovery of Stress-Induced Martensites in Superelastic Cu-11.8Al-0.6Be-0.5Nb Alloy. Journal Of Materials Engineering And Performance. 2017; 26(5): 2264-2270.
12. Qiu. CX, Zhu. S. Characterization of cyclic properties of superelastic monocrystalline Cu–Al–Be SMA wires for seismic applications. Construction And Building Materials. 2014, 72: 219-230.
13. Shivasiddaramaiah. AG. et al. Evaluation of shape memory effect and damping characteristics of Cu–Al–Be–Mn shape memory alloys. Perspectives In Science. 2016, 8: 244-246.
14. Montecinos. S.; Cuniberti. A.; Romero. R. Effect of grain size on the stress–temperature relationship in a β CuAlBe shape memory alloy. Intermetallics. 2011, 19(1): 35-38.
15. Prashantha. S.; Mallikarjun. U.S.; Shashidhara. S.M. Preparation and Characterization of Cu-Al-Be Shape Memory Alloys with Cr as Grain Refining Additive. Applied Mechanics and Materials 2014, 592-594: 700-704.
16. Zhang. Ping et al. Effect of grain refinement on the mechanical properties of Cu–Al–Be–B shape memory alloy. Materials & Design. 2011, 32(1): 348-352.

17. Yang, J. et al. Effects of grain refinement on the structure and properties of a CuAlMn shape memory alloy. *Materials Science And Engineering: A*. 2016, 664: 215-220.
18. Stošić, Z. et al. Effects of Composition and Thermal Treatment of Cu-Al-Zn Alloys with Low Content of Al on their Shape-memory Properties. *Materials Research*. 2017, 20(5): 1425-1431.
19. Montecinos, S. et al. Grain size evolution in Cu-based shape memory alloys. *Journal Of Materials Science*. 2015, 50(11): 3994-4002.
20. Albuquerque, V.H.C. de et al. Grain size and temperature influence on the toughness of a CuAlBe shape memory alloy. *Materials Science And Engineering: A*. 2010, 528(1): 459-466.
21. KHAN, A.Q.; BRABERS, M.; DELAEY, L. The Hall-Petch relationship in copper-based martensites. *Materials Science And Engineering*, 1974, 15(2-3): 263-274.
22. Montecinos, S.; Cuniberti, A.; Sepúlveda, A.. Grain size and pseudoelastic behaviour of a Cu-Al-Be alloy. *Materials Characterization*, 2008, 59(2): 117-123.
23. Paradkar, A.G. et al. On the validity of Hall-Petch equation for single-phase β Ti-Al-Nb alloys undergoing stress-induced martensitic transformation. *Materials Science And Engineering: A*, 2009, 520(1-2): 168-17.