

Influence of the Cryogenic Treatment in Micro Structure, hardness and Micro hardness of the Brass 60-40 and Bronze SAE 64

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Abstract— At the end of the 60's with the beginning of the space missions it was observed, that once the airships were sent into space and when they entered at the atmosphere, they presented an increase at the wear resistance on the structure of the space shuttles. It was concluded that the low temperature would have influenced in such changes. Over the year's liquid nitrogen, gaseous nitrogen, helium and dry ice were used on metals and tool that were submitted to high wear and fatigue periods in order to increase the useful life of the material, this procedure was called cryogenic treatment. Once it was identified that low temperatures generated improvements to certain materials as steels, aluminum and some a ceramic materials and the problem emerged to establish the effect of this phenomena in materials as brass and bronze, so it was take the decision to determine the influence of the low temperatures in this materials. What microstructural changes and mechanical behavior could the cooper alloys suffer as the brass 60-40 and bronze SAE 64? Taking into account the time spent in the steel a pilot test was done to evaluate the effect in the variation of grain size and micro hardness to the state of delivering of material through ANNOVA method determined the amount of tubes to study per each material and time. In this case, the effect in the microstructure is evaluated by metallographic and the effect in the mechanic behavior will be evaluated with micro hardness and hardness testing. Were spent four brass tubes were used per each time of exposition where 24, 48, 72 and 120 hours respectively and bronze's tubes used were there per each time of exposition likewise to brass. Finally it was observed a microstructural change and variations of mechanical properties where it is possible to conclude that to longer exposure of cryogenic treatment the resistance of the material increases. With an improvement in it is microstructure showing a grain with major dimensional stability.

Keywords— space missions, microstructural changes, ANNOVA, SAE 64.

I. INTRODUCTION

The cryogenic treatment has given to know for generating improvements in his properties, being his main objective reduce costs and increase performance of the material. In United States studied the effect of the cryogenic treatment in the microstructure of the pure copper and compared the effect to environment temperature, found a reduction of 60% of the size of grain in the polycrystalline microstructure compared with the environment temperature [4]. In Canada studied steel AISI D2, subjected to austenización, temperate, and cryogenic treatments, analyzed his microstructure with optics and electronic microscopy, the mechanical properties with hardness and the wear of the proofs. It showed greater hardness and resistance to the wear with the cryogenic treatment. This attributed to the transformation of some austenite retained in martensite and the strengthening of the matrix by the refinement of the grouping atom of carbon [8].

These two studies show the profits that generate the cryogenic treatments in the materials, by which arises the study and analysis of the influence of the cryogenic treatment in the microstructure, hardness and micro hardness in the brass 60-40 and bronzes SAE 64, materials employees in the manufacture of pieces subjected to the wear and fatigue, that although they have answered well to the industry demands, have been left backwards, because of this the treatment could generate an increase in his resistance to the wear and to the fatigue, causing an elongation in the useful life of the material. The most important variable in this treatment is the time, which we go to establish according to previous studies of other materials, in some cases suggest an hour by each inch of transversal section [10]. The application of the cryogenic treatments shows advances in comparison with the conventional thermal treatments like the increase the strength of the material in front of distinct efforts, with an improvement in his microstructure showing a grain of greater dimensional stability, which sees reflected in the material with a better resistance to the wear and a relief in the residual tensions of the microstructure.

II. METHOD

2.1 Type of study

For the work was realized a study of experimental type given that the test tubes were exposed to the cryogenic treatment, manipulating the time of exhibition. It determined that it would be an investigation of exploratory type, due to the fact that the influence of the cryogenic treatment in Brass 60/40 and Bronze SAE 64 has not been worked before, equally treats of a scientific investigation of experimental development because is headed to the improvement of the already existent materials from the analysis of the proofs realized. Finally the variable studied was the influence of the time in the properties of the material, by means of the analysis of Variance (ANNOVA) what allowed determined the quantity of test tubes for obtain a coefficient of confidence of the results of 95%.

2.2 Participating

It was realized an analysis of state of delivery of the material evaluating the size of grain, hardness and micro hardness of the material obtained.

TABLE 1
SPECIMEN RESULTS PATTERN

Brass 60/40	Grain Size	Hardness (Rockwell C)	Micro Hardness (Vickers)
Media	1685,3 μm^2	35,05HV200/15	118,435
Standard Deviation	2,6527	1,5198	5,63479
SAE 64	Grain Size	Hardness (Rockwell C)	Micro Hardness (Vickers)
Media	1685,3 μm^2	22,405HV200/15	95,9105,435
Standard Deviation	2,6527	2,9676	3,66808

Source: Author of the Project: 2015

It was realized a pilot proof subjecting 4 test tubes of each material to the cryogenic treatment, each one was subjected to a time of cryonics following the procedure established in the Metal Handbook where subject the test tubes from environment temperature (20 °C) until the temperature of the liquid nitrogen (-196 °C) , sustaining the test tubes to this temperature during 24, 48, 72 and 120 hours, after each one of these times leave them go back to environment temperature of gradual way, they were took 10 proofs of size of grain and 20 of micro hardness to each one of the test tubes subjecting these to a statistical analysis with which obtained the standard deviation and the average of each one.

TABLE 2
TEST RESULTS PILOT

Brass	24 hrs.	48 hrs.	72 hrs.	120 hrs.
Grain Size G	1055,85 μm^2	845,7 μm^2	870,6 μm^2	905,05 μm^2
	7	7	7	7
Micro Hardness Vickers	116,21	119,0705HV 200/15	106,98	126,77
	5,85	12,5797	7,26	7,49
SAE 64	24 hrs.	48 hrs.	72 hrs.	120 hrs.
Grain Size G	1156,85 μm^2	669,81 μm^2	562,50 μm^2	1143,3 μm^2
	7	8	8	7
Micro Hardness Vickers	108,081	92,723505HV 200/15	109,456	108,515
	10,3343	6,1885	11,48084	7,49

Source: Author of the project: 2015

By the method of ANNOVA, determined that to obtain a 95% of reliability had to analyze 3 test tubes of bronze and 4 of brass, by each time established of cryonics. The dimensions of the test tubes were of 1" of Diameter X 1" of long as it shows in the figure 1.



FIGURE 1: TEST TUBES ANALYZED THE TEST TUBE OF THE LEFT IS OF BRASS 60-40 AND THE TEST TUBE OF THE RIGHT IS BRONZE SAE 64. AUTHORS 2015



FIGURE 2: MATERIAL IN STATE OF DELIVERY, IN THE STUDY, WAS EMPLOYED TEST TUBES OF 1" X 1 1/2". AUTHORS 2015

2.3 Materials and Instruments

To realize the proofs of state of delivery of the figure 2. And pilot proof was employed 2 cm of each material, analyzed by means of a conventional microscopy through an optical microscope, by means of which obtained the size of grain (μm^2), of equal way was realized the essay of hardness Vickers by means of a micro durometer analyses the superficial hardness of the materials with greater precision, at the same time evaluates the hardness of the material in an Essay Rockwell, the hardness measures in function of the difference between the depths of penetration in two stages. Finally the cryogenic treatments realized by means of a cryogenic thermos that contains liquid nitrogen which finds to -196°C , this generates a thermal crash in the material.

2.4 Procedure

It was realizes an analysis of state of delivery of the material, realizes an analysis micro structural by means of his size of grain as it shows in the figures 4 and Figure 5, and mechanic with proofs of micro hardness and hardness.

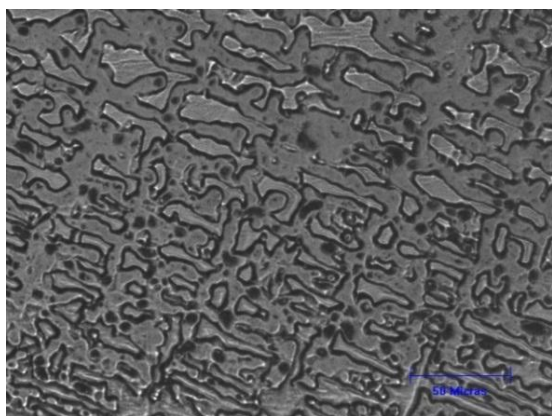


FIGURE 3 BRONZE SAE 64 500 X

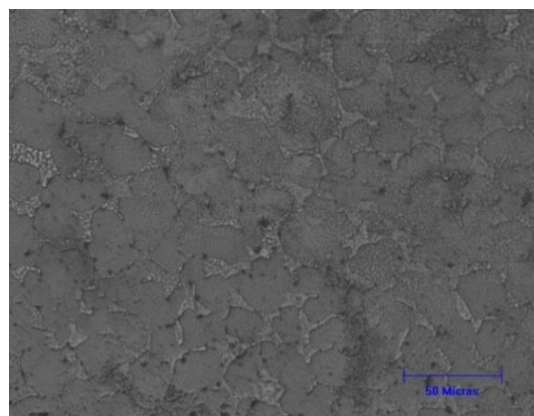


FIGURE 4 BRASS 60-40 500 X

FIGURES 3-4: ANALYSIS OF THE TEST TUBES IN STATE OF DELIVERY BY MEANS OF MICROSCOPY OPTICAL CONVENTIONAL TO 500 X. AUTHOR OF THE PROJECT 2015.

Taking into account that did not find antecedents referents to the times of cryogenisation of alloys of copper realizes a pilot proof with the most used times in the studies realized with diverse materials to determine a rank of time in which it causes effects. The times that established were 24 hrs. 48 hrs. 72 hrs. and 120 hrs. subjecting 1 test tube of each material to each time of cryogenisation, showed in the Figure 5. Photos of Submission to Cryogenic. The thermos in which they realized the proofs of cryogenic shows in the Figure 6. Thermos Cryogenic., with base to the results obtained in the pilot proof and the statistical analysis ANNOVA subject the test tubes to cryogenic.



(A)



(B)

FIGURE 5: CHARACTERISTICS RECEIVED BY THE TEST TUBES AFTER 48 HOURS OF CRYOGENIC TREATMENT. (A) TEST TUBE OF BRONZE SAE 64 AFTER 1 MIN TO GO OUT OF THE THERMO OF CRYOGENIC. (B) TEST TUBE OF BRONZE BRASS 60-40 AFTER 1 MIN TO GO OUT OF THE THERMO OF CRYOGENIC. AUTHOR OF THE PROJECT 2015.



FIGURE 6: CRYOGENIC THERMO WITH CAPACITY OF 35 LS. AUTHOR OF THE PROJECT 2015.

It evaluates the effect of the treatment by means of an attack micro structural to observe the variation of the size of grain for this analysis as it shows in the Figure 7., to attain this shine is necessary to realize a preparation with sandpapers, when the polishing of the test tube find in the same direction, rotate 90°, repeats the process from the calibrate 80 until 1500 followed of the cloth (Colombian School Of Engineers, 2011) [5].



FIGURE 7: POLISHED TEST TUBE MIRROR SHINE BY MEANS OF SANDPAPERS FROM THE 80 UNTIL THE 1500 FOLLOWED OF CLOTH WITH OXIDE OF CHROMIUM. AUTHOR OF THE PROJECT 2015.

It proceeds to realize the chemical attack according to the norm ASTM E-407 May 2011 [11]. Later it realizes a quick wash to avoid the oxidation, obtaining the images of the figure 9. Metallographies of the brass 60-40 to 500X where it shows 2 phases, the clear is the matrix α and the dark is phase β .

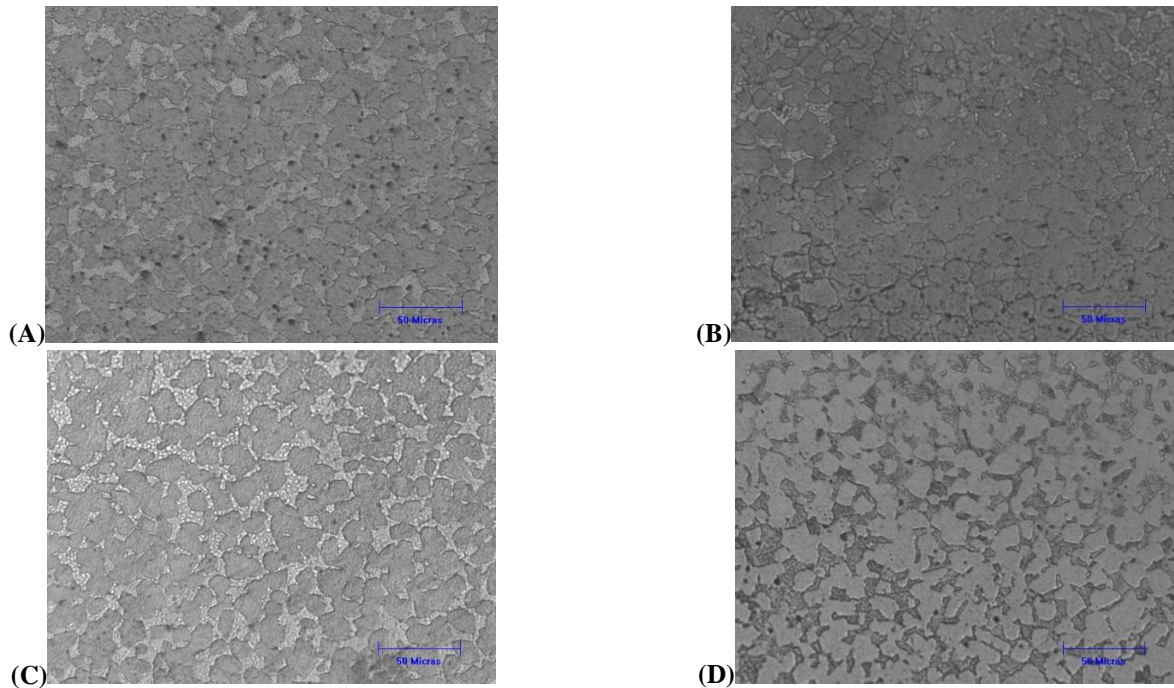


FIGURE 8: COMPARISON OF THE TEST TUBES OF BRASS 60-40 ATTACKED WITH 2G K₂Cr₂O₇ + 8mL H₂SO₄ + 4DROOP HCL+100mL TOILET ACCORDING TO THE NORM ASTM AND-407 MAY 2011 DURING 10 SECONDS. (A) TEST TUBE OF BRASS 60-40 SUBJECTED TO 24 HOURS OF CRYOGENIC TREATMENT. (B) TEST TUBE OF BRASS 60-40 SUBJECTED TO 48 HOURS OF CRYOGENIC TREATMENT. (C) TEST TUBE OF BRASS 60-40 SUBJECTED TO 72 HOURS OF CRYOGENIC TREATMENT. (D) TEST TUBE OF BRASS 60-40 SUBJECTED TO 120 HOURS OF CRYOGENIC TREATMENT. AUTHOR OF THE PROJECT 2016.

On the other hand in the figure 9. Metallography of bronze SAE 64 to 500X can observe after being attacked a matrix α and a phase dendritic δ .

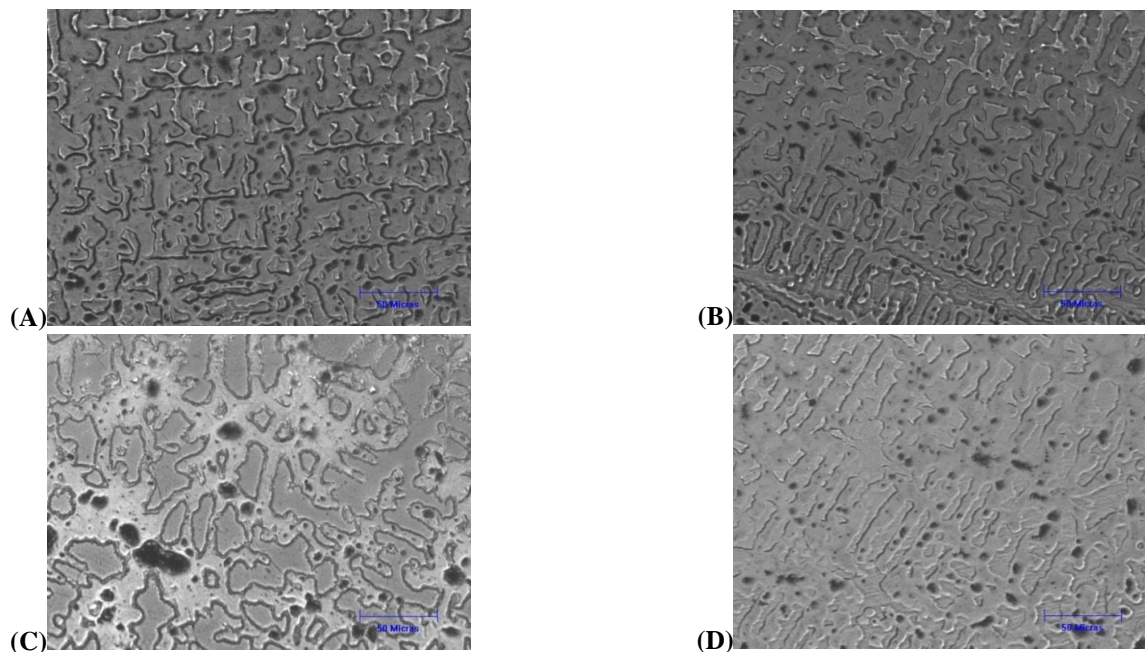


FIGURE 9: COMPARISONS OF THE TEST TUBES OF BRONZE SAE 64 ATTACKED WITH 2 g FeCl₃ + 5 mL HCl + 30 mL toilet + 60 mL ETHANOL OR METHANOL ACCORDING TO THE NORM ASTM E-407 MAY 2011. (A) TEST TUBE OF BRONZE SAE 64 SUBJECTED TO 24 HOURS OF CRYOGENIC TREATMENT. (B) TEST TUBE OF BRONZE SAE 64 SUBJECTED TO 48 HOURS OF CRYOGENIC TREATMENT. (C) TEST TUBE OF BRONZE SAE 64 SUBJECTED TO 72 HOURS OF CRYOGENIC TREATMENT. (D) TEST TUBE OF BRONZE SAE 64 SUBJECTED TO 120 HOURS OF CRYOGENIC TREATMENT. AUTHOR OF THE PROJECT 2016

III. RESULTS

3.1 Brass 60-40

- **Grain Size:** The results obtained in the proofs are the following:

TABLE 3
COMPARISON OF GRAIN SIZE OF BRASS 60-40

Analysis	Pattern	24 hrs.	48 hrs.	72 hrs.	120 hrs.
Media (μm^2)	1685,3	905,7125	729,55	398,7875	2240,4
G	6,5	7	7,5	8,5	6

Source: Author of the project.

Evidence that the Figure 11. Metallographies of brass 60-40 500X, that is a brass alpha-beta these are resistant to the traction and with greater percentage of zinc are more economic. But they are more susceptible to the corrosion by descinification [2].

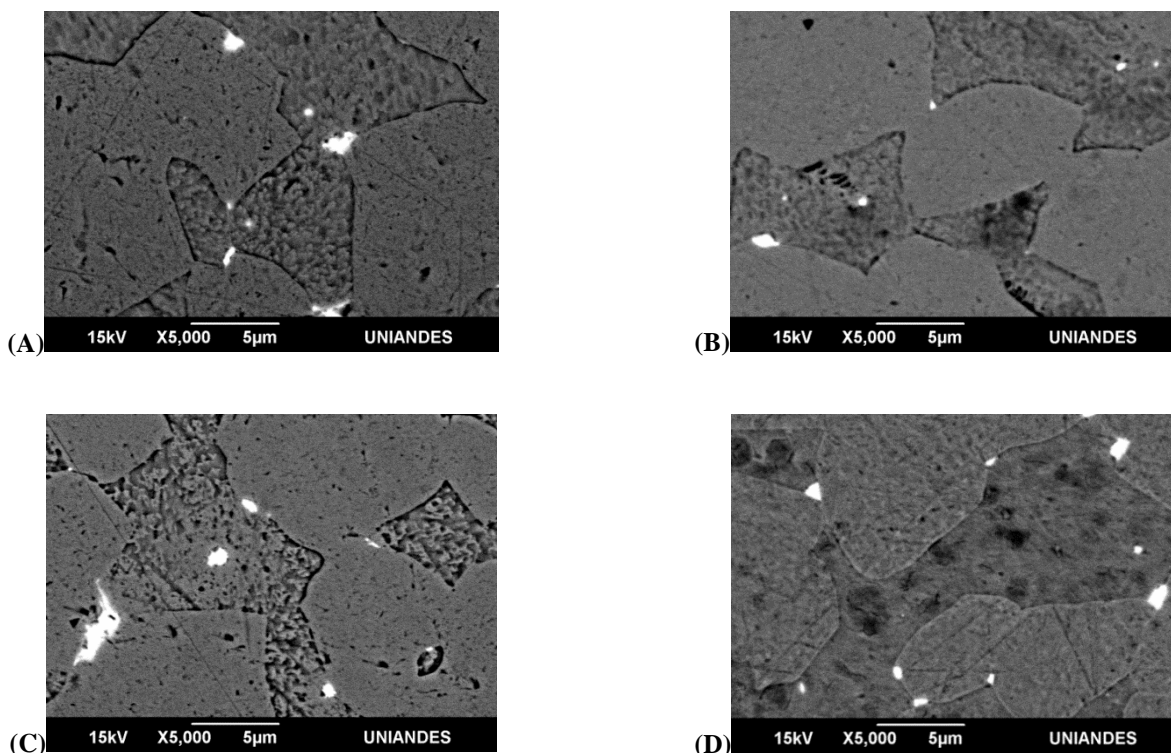


FIGURE 10: COMPARISONS OF THE TEST TUBES BRASS 60-40 IN THE PROOF OF SEM TO 5000X. (A) TEST TUBE OF BRASS 60-40 SUBJECTED TO 24 HOURS OF CRYOGENIC TREATMENT. (B) TEST TUBES OF BRASS 60-40 SUBJECTED TO 48 HOURS OF CRYOGENIC TREATMENT. (C) TEST TUBES OF BRASS 60-40 SUBJECTED TO 72 HOURS OF CRYOGENIC TREATMENT. (D) TEST TUBES OF BRASS 60-40 SUBJECTED TO 120 HOURS OF CRYOGENIC TREATMENT. LABORATORY OF MICROSCOPY ANDES UNIVERSITY. JULY 2016.

It realizes an analysis EDS. to analyze the white points that observe in SEM as it shows in the figure and determines that they are precipitated of copper and lead, this can be true because according to the professor PERO [7], this is a variation of metal Muntz call "Naval brass". This lead increases the machinability of the material, generates a no porous microstructure and appears like small particles in the limits of grain [1].

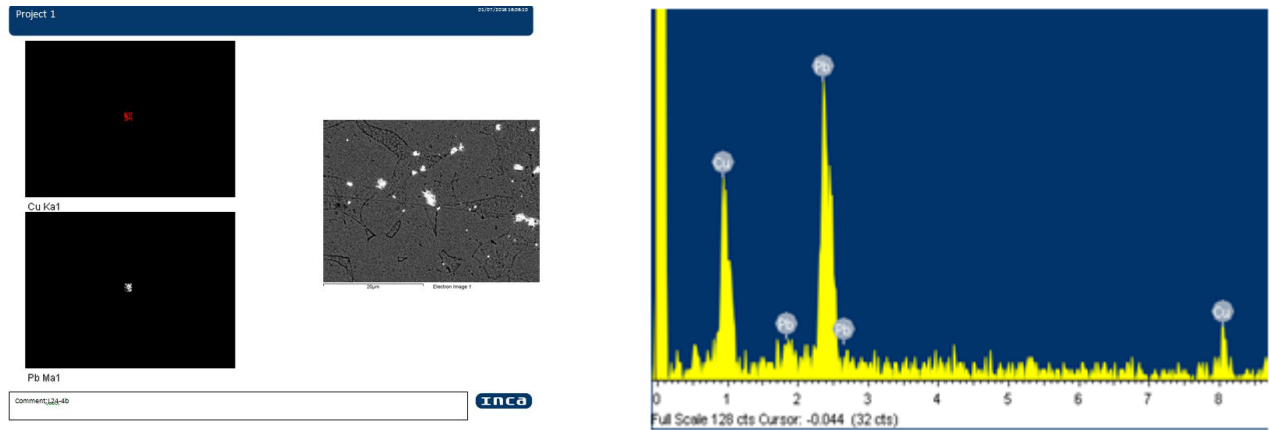


FIGURE 11. IN THE IMAGE OF THE LEFT IDENTIFY THE PRECIPITATED ANALYZED THAT WAS ANALYZED AND IN THE IMAGE OF THE RIGHT OF THE COMPONENTS THAT CONTAINS THE PRECIPITATED. LABORATORY OF MICROSCOPY ANDES UNIVERSITY. JULY 2016.

- Hardness and Micro hardness**

TABLE 4
COMPARISON OF PROOF OF MICRO HARDNESS OF BRASS 60-40

Analysis	Pattern	24 hrs.	48 hrs.	72 hrs.	120hrs.
Media	118,4	132,4075	139,13125	133,58	134,9325
Standard Deviation	5,63479	4,9280333	9,30937453	8,2544564	6,54626092

Source: Author of the project.

TABLE 5
COMPARISON OF PROOF OF HARDNESS OF BRASS 60-40

Analysis	Pattern	24 hrs.	48 hrs.	72 hrs.	120 hrs.
Media	35,05	37,765	42,33	41,42	42,12
Standard Deviation	1,51986	8,26509942	5,00179968	2,04499389	1,4131525

Source: Author of the project

Analyzing the results obtained with the cryogenic treatment in the times of proof in the brass can establish that the size of grain goes back finer to measure that increases the time of cryogenic, whereas the micro hardness and hardness observe an increase until the 120 hrs.; It is important to point out that the variation of the micro hardness is not significant in comparison to the size of grain.

3.2 Bronze SAE 64

- Grain size:** The results obtained in the proofs are the following:

TABLE 6
COMPARISON OF GRAIN SIZE OF BRONZE SAE 64

Analysis	Pattern	24 hrs.	48 hrs.	72 hrs.	120 hrs.
Media (μm^2)	1685,3	1443,11111	1751,2	1086,45	880,3
G	6,5	6,5	6,5	7	7,5

Source: Author of the project.

It shows in the figure 13. Metallography of bronze SAE 64, this material finds in $(\alpha + \delta)$. The eutectoid $(\alpha + \delta)$ It appears associated with copper phosphide Cu_3P ; particles very hard and that increase the resistance to the wear of the constituent $(\alpha + \delta)$ [6].

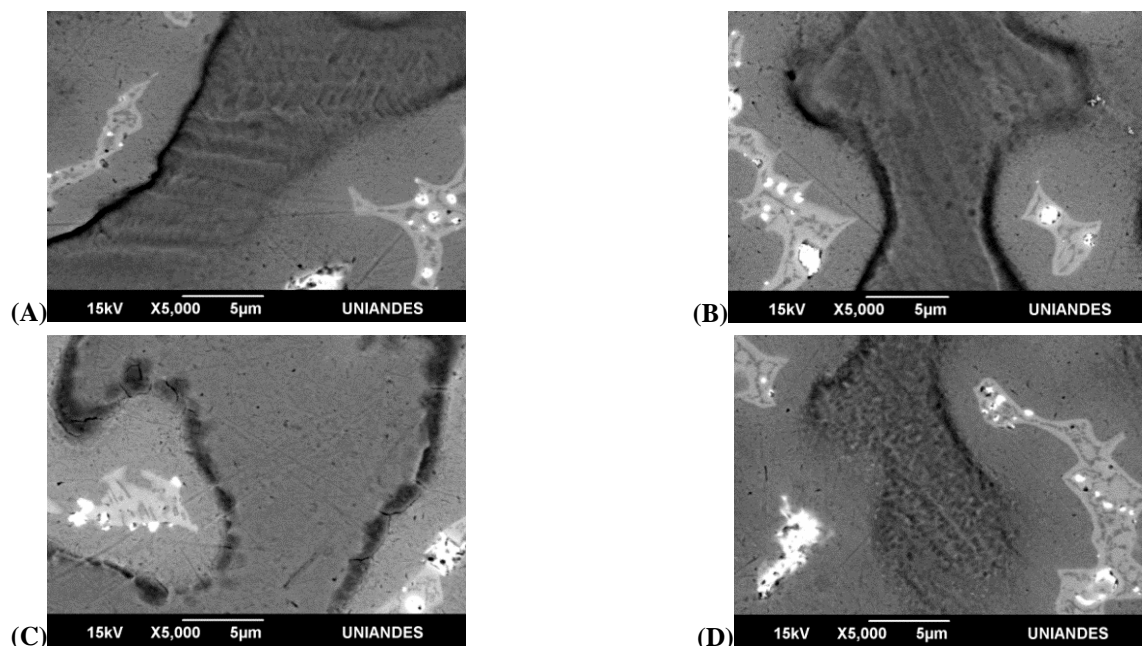


FIGURE 12: COMPARISONS OF THE TEST TUBES OF BRONZE SAE 64 IN THE PROOF OF SEM TO 5000X. (A) TEST TUBE OF BRONZE SAE 64 SUBJECTED TO 24 HOURS OF CRYOGENIC TREATMENT. (B) TEST TUBE OF BRONZE SAE 64 SUBJECTED TO 48 HOURS OF CRYOGENIC TREATMENT. (C) TEST TUBE OF BRONZE SAE 64 SUBJECTED TO 72 HOURS OF CRYOGENIC TREATMENT. (D) TEST TUBE OF BRONZE SAE 64 SUBJECTED TO 120 HOURS OF CRYOGENIC TREATMENT. LABORATORY OF MICROSCOPY UNIVERSITY OF ANDES. JULIO 2016

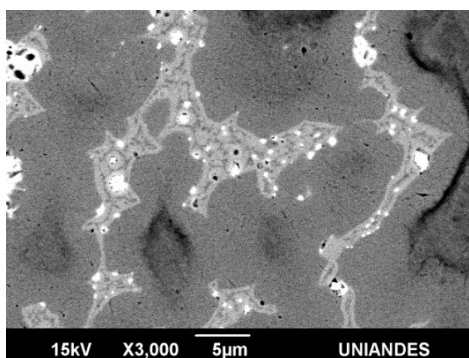


FIGURE 13: PRECIPITATED OF THE TEST TUBE OF BRONZE SAE 64 TO 24 HOURS OF CRYOGENIC. PROOF OF SEM TO 300X IN THE LABORATORY OF MICROSCOPY OF ANDES UNIVERSITY. 2016.

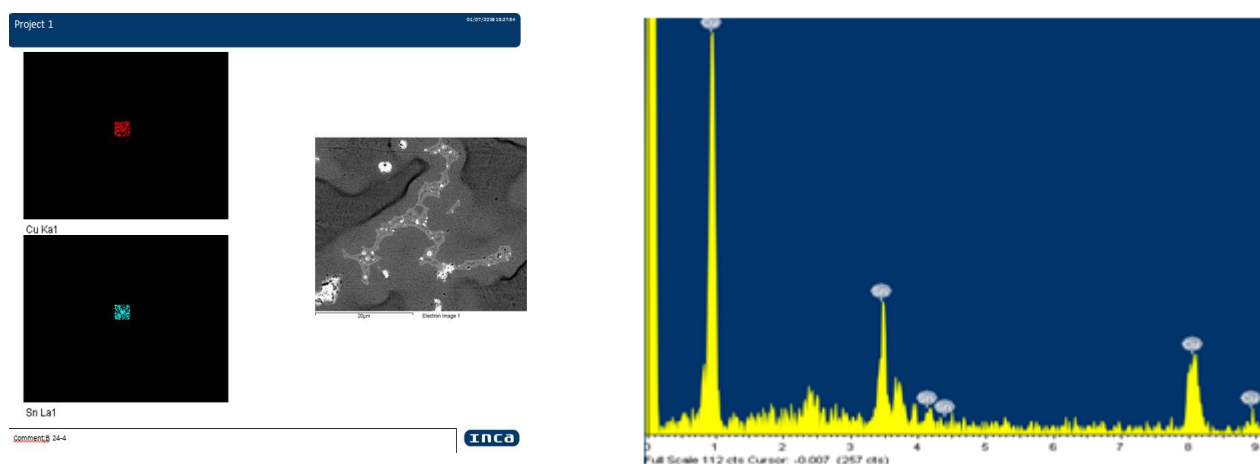


FIGURE 14: ANALYSIS EDS TO THE PRECIPITATED OF THE TEST TUBE OF BRONZE SAE 64 TO 24 HOURS OF CRYOGENIC. PROOF OF SEM TO 300X IN THE LABORATORY OF MICROSCOPY OF ANDES UNIVERSITY. 2016

It was evidenced that they present odd samples in the test tubes of Bronze SAE 64 as it shows in the Figure 14. Precipitated of the test tubes of Bronze SAE 64 to 24 hours of cryogenic, realizes an analysis EDS to determine the composition. It concludes that they are precipitated of Cu Sn according to the figure 14.

TABLE 7
COMPARISON OF PROOF OF MICRO HARDNESS BRONZE SAE 64

<i>Analysis</i>	<i>Pattern</i>	<i>24 hrs.</i>	<i>48 hrs.</i>	<i>72 hrs.</i>	<i>120 hrs.</i>
<i>Media</i>	95,91	111,8375	117,665	141,1	155,880833
<i>Standard Deviation</i>	3,66	6,04576911	6,80572603	4,85215904	11,6353094

Source: Author of the project

TABLE 8
COMPARISON OF PROOF OF HARDNESS BRONZE SAE 64

<i>Analysis</i>	<i>Pattern</i>	<i>24 hrs.</i>	<i>48 hrs.</i>	<i>72 hrs.</i>	<i>120 hrs.</i>
<i>Media</i>	22,4	25,76	25,36	33,59	35,92
<i>Standard Deviation</i>	2,967659	3,1445644	3,14456447	3,64916168	3,03625709

Source: Author of the project

Analyzing the results of the bronze SAE 64 we can establish that the size of grain goes back finer to measure that increases the time of cryogenic until 72 hours and in 120 hours goes back to increase the size of grain almost until in size of grain obtained in 24 hours, whereas the micro hardness observe a variation increasing to the 24 hours and descending again to the 48 hours, increases between 48 hours and 72 hours, and sustains until the 120 hours, the variation of the micro hardness is very uncertain because of the few antecedents.

With the refinement of grain we can conclude that the material suffers a hardening by reduction of size of grain, because a material with grain finer is harder and resistant that one of thick grain, since has greater number of edges of grain for the same volume that the test tube pattern.

IV. DISCUSSIONS AND CONCLUSIONS

1. The cryogenic treatment effected to the Brass 60-40 and Bronze SAE 64 does not contribute any significant improvement regarding the hardness and micro hardness. Although according to the company CRYOBEST (CRYOBEST INTERNATIONAL, 2010) [3] obtains an increase in the resistance to the wear, resistance to the corrosion and increase in the useful life. It suggests realize proofs of tenacity, resistance to the abrasive wear, and compare the results with a back treatment of a tempering to low temperature and analyze again the hardness and in the resistance to the adhesive wear.
2. To simple sight we can observe a more organized micro structure with a finer grain in the material, that generates a greater dimensional stability and elimination of tensions, doing the material more tough, additional can check that it is a treatment that affects all mass of the material and is not superficial.
3. In the brass we can observe that between more times of cryogenic it increases the apparition of lead in the limits of grain of the material. The presence of the lead in the brass provides a lubricant effect.
4. In the Bronze SAE the particles precipitated act like obstacles of the movement of the displacements and, like this, reinforce the alloy treated thermally. The alloy results reinforced by means of this restriction of the movement of the displacements during the deformation. It will take place the aging to environment temperature, which will give like result an increase in the resistance and in the hardness.

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