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INFLUENCE OF ACETONE AND METHANOL PROCESS CONTROL AGENTS IN THE SIZE AND MORPHOLOGY OF THE PARTICLES IN THE HIGH ENERGY MILLING OF THE Nb-25%Al ALLOY

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Abstract. The objective of this paper is to study the size and morphology of the particles before and after high energy milling of the elementary Nb and Al powders, with and without the use of Process Controlling Agents (PCA), through the mechanical alloying process (MA). The mixture of powders with chemical composition of Nb-25%Al underwent milling in a high energy planetary mill with controlled atmosphere using argon gas for 10, 30, 40 and 50h. In order to analyze the best milling condition for the use of PCA, samples were prepared without PCA, 2% by mass of acetone and 2% by mass of methanol as PCA. Heat treatment was carried out at 850°C for 10h in the samples with 50h of milling. Scanning Electron Microscopy (SEM) was used for microstructural analysis, for the analysis of the distribution and the size of the particles was used sedimentography.

Keywords: Mechanical Alloying, High Energy Milling, Particle Sizes, Particle Morphologies.

1. INTRODUCTION

The production of the Nb-Al alloy by conventional methods, as by casting, is difficult because of the large difference between the melting temperatures of the two metals (Al: 660.4°C; Nb: 2468°C) (Zhang et al., 2016; Callister Jr. and Rethwisch, 2007). Therefore, the use of powder metallurgy in the preparation of these alloys is more advantageous and simple by providing phase stabilization at temperatures much lower than the melting temperature of the niobium (Karpov et al., 2011). For this, the MA is used, which is a high energy milling powder processing technique, which allows the production of homogeneous materials from mixtures of elementary powders, involving repeated welding, fracture and resoliding of powder particles, which may give rise to the solid crystalline, almost crystalline and amorphous solution phase (Suryanarayana, 2001; Peng et al., 1996; Naghiha et al., 2017).

In MA the PCA's are used, which are mostly organic materials in the solid, liquid or gaseous state (Suryanarayana, 2001). The use of PCA in ductile-ductile processes has as its main function the prevention of cold welding (Khayati and Janghorban, 2012). Depending on the nature and the purity of the powder, the presence of PCA may influence the milling time, the final particle size, the presence of hard phases after sintering or even porosity of the workpiece (Suryanarayana, 2001). The literature on the milling process of Nb-Al presents little variety of PCA's applied to the MA process in planetary mill.

2. MATERIALS AND METHODS

The Nb used in this work was obtained by the HDH (hydrogenation-dehydrogenation) process with purity > 99.9% and a particle size of 2 to 10µm produced at the Lorena School of Engineering - USP. Al obtained by atomization was

donated by ALCOA, has a purity of 99% and a particle size of 3 to 10 μm . The powders were mixed with stoichiometric composition of Nb-25%Al.

For the milling of the precursor powders, a high energy planetary mill of the brand NOAH, model NQM-2, with atmosphere controlled by Argon, stainless steel balls with diameter of 10, 11 and 14 mm was used. The mass/ball ratio was 1:10 and the rotational speed of 350 rpm, the total milling time was 50h. To avoid cold welding 2% by mass of PCA's, methanol and acetone were used. Samples were drawn at 10, 30, 40 and 50h for characterization, with 1% of PCA being added at each withdrawal because they were volatile. The pressing was performed on a uniaxial press with a pressure of 260 MPa, the press die used was 12 mm in diameter.

To study the morphology, structural changes in the particles and to determine the structure and size of the crystallites of the powders obtained after high energy grinding, an Scanning Electron Microscope (SEM) was used in the secondary electron mode. To evaluate the particle size and distribution, the Microtrac granulometer and model S3500 were used.

3. RESULTS AND DISCUSSION

3.1 Particle size analysis

At the graph of average particle size as a function of milling time, shown in Figure 1, it is observed that for the pure Nb-Al sample, without PCA presence, the particle size has grown up with 30 hours of milling, characterizing the stage of plastic deformation and the occurring of cold welding process. Right after, what is seen is an abrupt reduction of particle size, indicating the occurrence of hardening due to plastic deformation and consequently fractures of the sample particles, resulting in an average particle size of 70 μm . The cold welding was facilitated in this sample due to the absence of PCA.

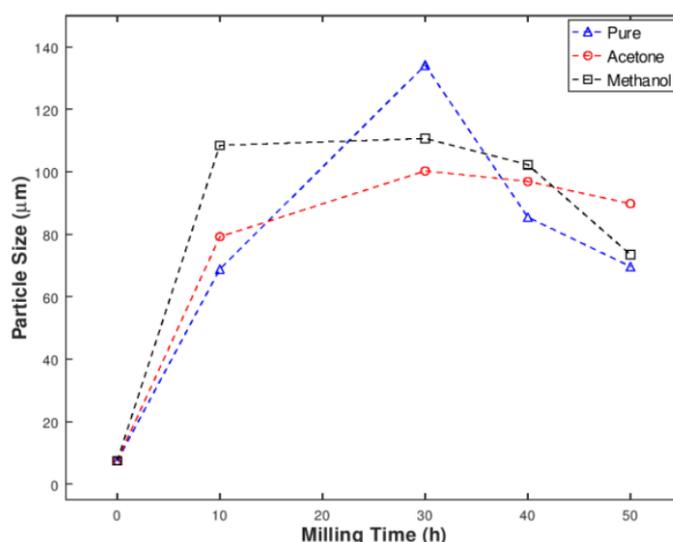


Figure 1. Average particle size as a function of milling time for Nb-25%Al composition.

In the sample milled with 2% of acetone, it is noticed the gradual raise of particle size until 30 hours of milling, it is also duo to cold welding. After this, the particles size keeps going lower until it reaches 90 μm . Showing the effects of hardening and fracturing.

The sample with 2% addition of methanol showed less effects of cold welding. This PCA was able to act as a coating for the particle surface, avoiding the metal-metal contact and consequently the cold welding and generating good particle fracturing. Due to the lower surface energy, less milling time is required to obtain small particle size. The Average Particle size for this sample was 73 μm .

Between the observed situations, that one with acetone as PCA was the one that showed to be the most effective, it is because this was the process with less cold welding effects, characterizing good particle coating effect. The sample with methanol was the most aggressive process until 10 hours milling, occurring plastic deformation, agglomeration, cold welding and hardening. Because of the high energy levels stored with 30 to 50 milling hours, the particles start to fracture, heading to a smaller particle size that the acetone sample.

The pure sample (with no PCA) shows large particle size until 30 hours milling, the responsible for this is agglomeration and cold welding. After 30 hours, the effects of hardening cause the start of fracturing process.

3.2 Particle morphology analysis

The SEM micrograph exhibited in Figure 2 shows the morphology of particles of the pure Nb-Al (with no PCA) sample. In Figure 2a, with milling time of 10 hours, it is observed many particles with irregular shapes and 70 μm size.

In Figure 2b, with 30 hours milling, the frequent impact of the Nb-Al particles leads to cold welding and hardening. In Figure 2c, with 50 hours milling time, the particles start going smaller, it occurs due to the big levels of contact energy between particles and milling balls, which causes an intensification in the hardening and particles fracture.

With the decrease of particles size there is a bigger electrostatic attraction between them, leading to particle agglomeration. Samples without PCA are slim and tends to round shape, this morphology indicates the occurrence of cold welding followed by hardening and fracturing.

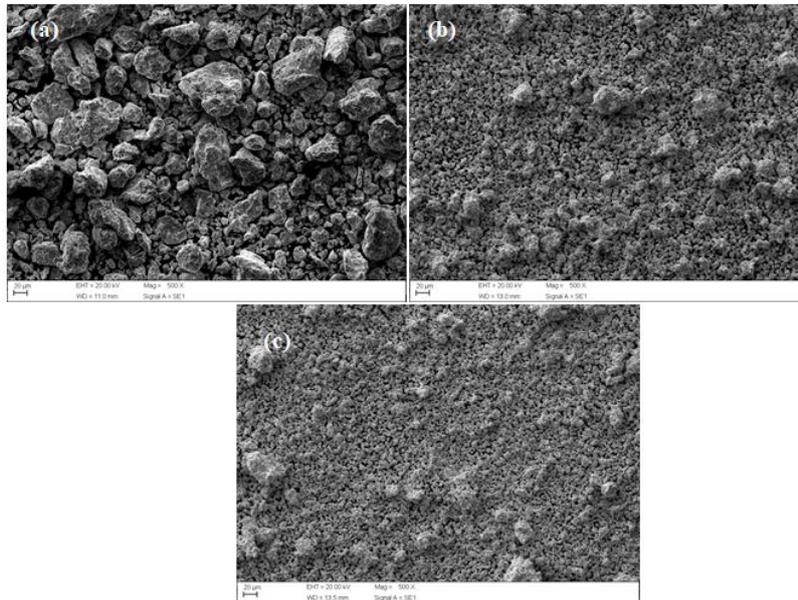


Figure 2. Micrograph for sample of Nb-25%Al without addition of PCA: (a) 10h of milling, (b) 30h of milling and (c) 50h of milling.

The SEM micrograph of Exhibited in Figure 3 shows the morphology of the sample with acetone addition of 2% in weight, with the intent of the acetone act as a PCA. In Figure 3a, with 10 hours milling time, it is observed a smaller particle if compared with the no PCA sample, it shows that acetone was able to coat the particles surface properly, reducing cold welding. In Figures 3b and 3c, with milling time of 30 and 50 hours, respectively, it is noticed that the particles acquired irregular shapes due the adhesion of acetone in the particle surface, restraining the contact between particles and avoiding cold welding.

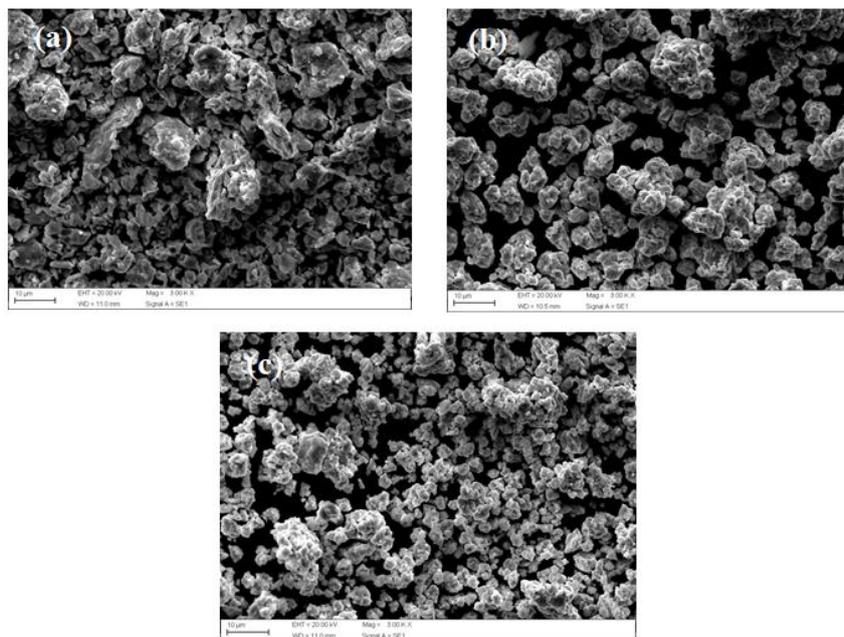


Figure 3. Micrograph for sample of Nb-25%Al with addition of 1% mass of acetone as PCA: (a) 10h of milling, (b) 30h of milling and (c) 50h of milling.

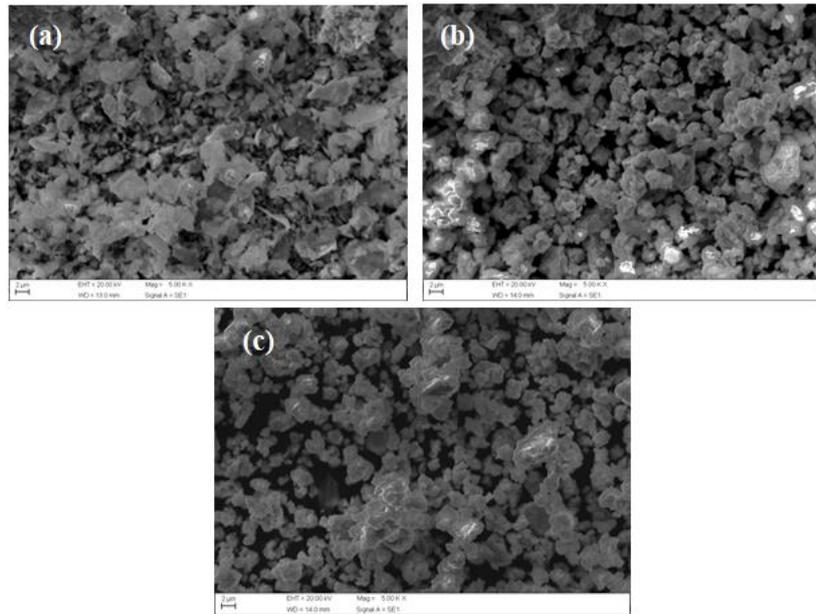


Figure 4. Micrograph for sample of Nb-25%Al with addition of 1% mass of methanol as PCA: (a) 10h of milling, (b) 30h of milling and (c) 50h of milling.

The SEM micrograph in figure 4 exhibits the morphology of the sample with 2% weight addition of methanol as PCA. The Figure 4a shows the 10 hour milling sample, occurred a severe size raise of the particles. Methanol showed up as an efficient suppressor of cold welding, that's because particles with 10 hour milling had intense plastic deformation and sheared into flake shapes. In Figure 4b, with 30 hours milling time, particles tended to get smaller with milling time, as a result of hardening and fracturing. In figure 4c, with 50 hours milling, it is observed that particles had almost spherical shape.

3.3 Particle analysis after heat treatment

Figure 5 shows the SEM micrograph of thermally treated samples. It is observed better density in samples with PCA. The sample with methanol addition had better result, once it presented good particle homogeneity and spherical shape, this factors would lead to a final product with lower porosity after bulking and sintering. The sample without PCA addition had also spherical shape, but without satisfactory density and homogeneity.

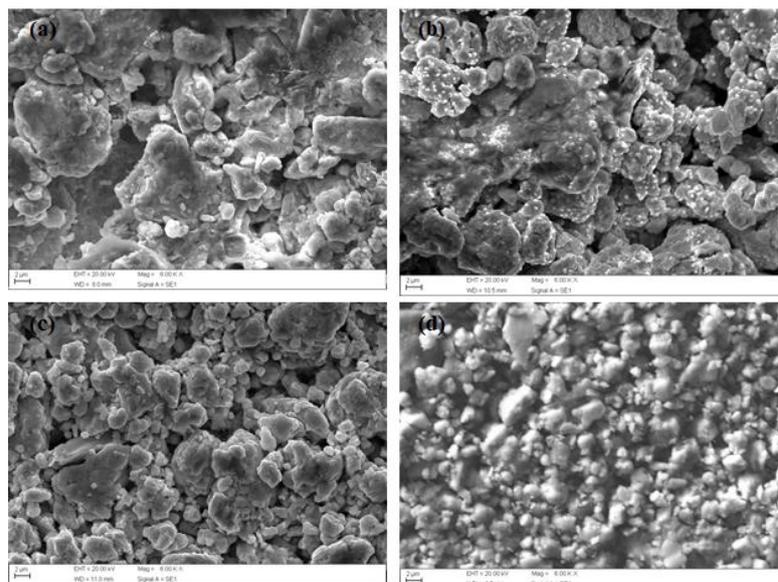


Figure 5. Micrograph of the thermally treated samples: (a) manually mixed, (b) pure, (c) acetone and (d) methanol.

4. CONCLUSIONS

It was observed that in the sample without the use of PCA cold welding was facilitated, reaching the largest particle size with 30 hours of grinding, between the samples. However, due to the intense impact between grinding particles and spheres, they are hardened and fractured, leaving the smallest particle size at the end of grinding. Samples without PCA were found to be thin, spherically shaped, which actually indicates cold welding followed by hardening and fracture.

Using acetone it was noted that the particles acquire irregular shapes due to the adhesion of the acetone molecule on the surface of the particles preventing contact between them, ie avoiding cold welding. Methanol was efficient in suppressing cold welding, as the 10-hour grinding particles deformed intensely and sheared into flakes.

After heat treatment, it was observed that PCA's exert a great influence on the density of the samples. It is noted that samples with better densification contained PCA's. The methanol sample obtained better results because it had a more homogeneous particle distribution and because they are in spherical shape the contact area is larger, thus reducing the amount of pores. The pure sample even with spherical shape did not obtain as satisfactory density as the samples with addition of PCA, because its particles were not evenly distributed.

5. ACKNOWLEDGEMENTS

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6. REFERENCES

- Callister Jr., W. D. and Rethwisch, D. G.. *Materials science and engineering: An introduction*. 8. ed. New York: John wiley & sons, 2007.
- Karpov, M, I, Khorzov, V. P., Prokhorov, D. V., Vnukov, V.I., Kiiko, V. M., Tolstun, A. N., Kolobov, Yu. R., Golosov, E. V. *Structure and Properties of Nb–Al Alloys Prepared by Powder Metallurgy*. Russian metallurgy (Metally). 2011, 251-255, 2011.
- Khayati, G. R., Janghorban, K. An investigation on the application of process control agents in the preparation and consolidation behavior of nanocrystalline silver by mechanochemical method. *Advanced Powder Technology*, 23, 808–813, 2012.
- Naghiha, H. et al. Amorphization and nanocrystalline Nb₃Al intermetallic formation during mechanical alloying and subsequent annealing. *Advanced Powder Technology*, v. 28, n. 2, p. 340-345, 2017.
- Peng, Z., Suryanarayana, C.; Froes, F. H.. Mechanical alloying of Nb-Al powders. *Metallurgical and Materials Transactions A*, v. 27, n. 1, p. 41-48, 1996.
- Suryanarayana, C. Mechanical alloying and milling. *Progress in Materials Science*, 46, 1-184, 2001.
- Zhang, Y, Lin, W.J., Xu, L.Y., Yang, D.W., Chen, Y.L., Li, P.Y., Pan, X.F., Yan, G., Zhao.Y. Fabrication of Nb₃Al superconductor by the optimized mechanical alloying method with low temperature. *Physica C: Superconductivity and its applications*, Austrália, 530, 38-41, 2016.

7. RESPONSIBILITY NOTICE

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