ISSN: 2230-9926

Available online at http://www.journalijdr.com

IJDR

International Journal of Development Research

Vol. 12, Issue, 09, pp. 58757-58761, September, 2022

https://doi.org/10.37118/ijdr.25135.09.2022

RESEARCH ARTICLE OPEN ACCESS

MANUFACTURING AND CHARACTERIZATION OF AA3003 ALUMINUM ALLOY POWDERS BY SYNTHESIS OF ELEMENTARY POWDERS BY TECHNIQUES OF HIGH ENERGY BALL MILLING

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ARTICLE INFO

Article History:

Received 09th July, 2022

Received in revised form

17th July, 2022

Accepted 20th August, 2022

Published online 23rd September, 2022

Key Words:

AA3003, aluminum alloy, Powder metallurgy, High energy ball milling.

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ABSTRACT

Manufacturing via powder metallurgy has several advantages such as high composition control, excellent dimensional accuracy, and good surface finish, among others, enabling the processing of different materials, including aluminum alloys, with wide application in diverse sectors of production. The present work aims to analyze the processing of an AA3003 aluminum alloy by the high energy ball milling technique (HEBM) under different production conditions. The particle size distribution of elementary and processed particulate was evaluated by laser diffraction. Elemental powders were processed by HEBM using milling intervals of 30, 60, and 120 minutes. The processed particulate materials were characterized by scanning electron microscopy (SEM), complementing the analysis with energy dispersive spectroscopy (EDS), in addition, the phases present were evaluated by X-ray diffraction (XRD). The characterization of the powders submitted to HEBM revealed that the increase in milling time contributed to the reduction of the average size of the particles, presenting an average diameter close to that verified in the particles of the base elements, Al and Mn.The best results found regarding the manufacture of the AA3003 alloy were found for samples obtained by milling time of 120 minutes, presenting as an effective processing interval to identify characteristic phases of the alloy.

Citation: Moisés Euclides da Silva Junior, Clécio Vicente da Silva, Helen Rodrigues Araújo, Romulo Rocha de Araújo Lima, Yogendra Prasad Yadava and Oscar Olimpio de Araujo Filho, 2022. *"*Manufacturing and characterization of aa3003 aluminum alloy powders by synthesis of elementary powders by techniques of high energy ball milling*", International Journal of Development Research, 12, (09), 58757-58761.*

INTRODUCTION

The combination of properties such as low specific weight, low relative density, ductility, formability, good thermal and electrical conductivity, and corrosion resistance (MEIGNANAMOORTHY e RAVICHANDRAN, 2018; KUMAR e KUMARASWAMIDHAS, 2019), also combining physicochemical characteristics such as corrosion resistance and high electrical/thermal conductivity, in addition to some alloys presenting mechanical resistance superior to structural steels, makes aluminum and its alloys materials of great versatility in engineering applications (SOUSA, DE, 2012; ZARZAR *et al.*, 2021). The use of aluminum and its alloys has experienced constant growth in different sectors of the manufacturing industry, due to the characteristics presented by this metal, in addition to the fact that it can be combined with most engineering materials, forming alloys from these combinations to obtain advantageous properties, presenting itself as the first alternative in terms of material to achieve the requirements of certain applications (TORRALBA *et al.*, 2003; ZARZAR *et al.*, 2021). Another issue worth mentioning is the ability to recycle this metal, as it is reported that around 90% of the aluminum present in vehicles is recovered and recycled, with great environmental and economic advantages (CAPELARI e MAZZAFERRO, 2009; SILVA *et al.*, 2021; ABAL, 2022). Among the classes of aluminum alloys, the 3XXX series formed by the system (Al-Mn) deserves to be highlighted, which has manganese as the main alloying element, which has a maximum percentage of 1.5%. The addition of manganese gives the alloy an increase in mechanical strength, good formability, corrosion resistance, and good performance in welding processes. Widely used in vehicle license plates, beverage cans, and tanks in the chemical industry, among other applications. Alloys of the 3XXX series are not usually heat treatable, which means that the increase in mechanical strength is obtained through cold working (BARBOSA, 2014; SILVA JUNIOR *et al.*, 2021). To manufacture these aluminum alloys by mixing elementary powders, high energy ball milling (HEBM) is a process with great potential to be used on a large scale, as it allows the elaboration of alloys with excellent mechanical properties for applications at room temperature. and temperatures above 150°C, when compared to conventional aluminum-based alloys (COELHO *et al.*, 2003). The initial composition of a mixture of powders, submitted to the HEBM process, can influence the synthesis of the alloy. When the equipment comes into operation, movement occurs promoting impacts between the powder particles, the grinding media, and the container. Initially, mixing occurs and, in parallel, material deformation followed by fracture and cold welding of the powder particles. The activation energy necessary for the occurrence of the formation reaction of new phases is obtained by the release in the collisions between the material and the balls. The reaction rate varies exponentially with the rate of strain accumulation, analogous to what occurs in thermally induced chemical reactions, where the same type of relationship occurs between reaction rate and temperature. This rate varies according to the kinetic energy of the balls. The formation of nanocrystalline particles during milling helps the process, as it minimizes the length of the diffusion pathways. The same occurs as a function of the rate of deformation-induced defects (MURTY e RANGANATHAN, 1998; ALMEIDA *et al.*, 2004).

MATERIALS AND METHODS

The composition's selection of the alloy in manufacture followed the instructions of the ASM Handbook (1992), indicating the appropriate proportion of each element of the alloys to be added, presented in Table 1. After the proper separation of the elemental powders, they were weighed in a balance of semi-analytical precision, model UX420H (SHIMADZU).

Table 1. Composition selected for AA3003 alloy

Aluminum Association	wt%					
	Si	Fe	Cu	Mn	Zn	Al
AA3003	0.60	0.70	0.20	1.50	0.10	96.9

Source: Adapted from ASM Handbook, 1992

Assuring the proper mass quantity of the base elements, the powders were submitted to the high energy ball milling technique (HEBM) using a vibrating ball mill model SPEX, adopting a speed of 720 rpm, performed wet by applying isopropyl alcohol to the milling bath. The balls used in the mill are made of SAE 52100 steel and were applied with a proportion of 7:1, concerning to the total mass added for the composition of the aluminum alloy. The AA3003 aluminum alloy is formed by the Al-Mn system, with manganese (Mn) being the major alloying element. However, these alloys have in their composition concentrations of other alloying elements such as silicon (Si), iron (Fe), copper (Cu), and zinc (Zn). Table 2 shows the concentrations in mass (g) of the chemical elements added to the composition of the aluminum alloy AA3003, in the different intervals of high energy milling. The total mass submitted to milling was 20.000 g, adding 1% process control agent (PCA), as indicated by De Araujo Filho *et al.* (2017), totaling 20.200 g.

Table 2. Mass concentrations of the AA 3003 elementary materials and applied HEM intervals

Aluminum alloy	HEBM time (min)	Composition (g)						
		Si	Fe	Cu	Mn	Zn	Al (1100)	PCA
AA3003	30	0.120	0.140	0.040	0.300	0.060	19.340	0.200
	60							
	120							

Particle size analyzes were performed by laser diffraction, using the Mastersizer 2000 model equipment (MALVERN INSTRUMENTS U.K.) on the pure aluminum AA1100 and Mn powders before being processed by milling, as well as on the particulate material of the alloy processed by HEBM of AA3003. The application of optical microscopy and scanning electron microscopy (SEM) techniques, model MIRA3 (TESCAN), with energy dispersive spectroscopy (EDS) provided the microstructural and chemical characterization of the powders after processing by high energy milling. In addition, RX diffraction was performed using the XRD-700 model diffractometer (SHIMADZU), with Cu Kα (λ = 1.5405 Å), applying acquisition parameters such as sweep angle from θ to 2θ, sweeping from 5º to 120º with a step of 0.02º/s, from which it was possible to determine the XRD spectra of each alloy processed by different milling intervals, with the peaks present in the diffractograms analyzed and indexed through comparison, with the microfiches of diffraction data from the International Center for Diffraction Data (ICDD), using the X'PertHighScore software, thus enabling the identification of the formed phases and laser diffraction for particle size analysis.

RESULTS AND DISCUSSION

Particle size analysis of elementary materials and AA3003 alloy powders processed by HEBM: Through the application of the laser diffraction technique, it was possible to verify the granulometric distribution and the average size of the aluminum and manganese particles, as received, used as the basis for the formation of the AA3003 alloy, as shown in Figure 1.

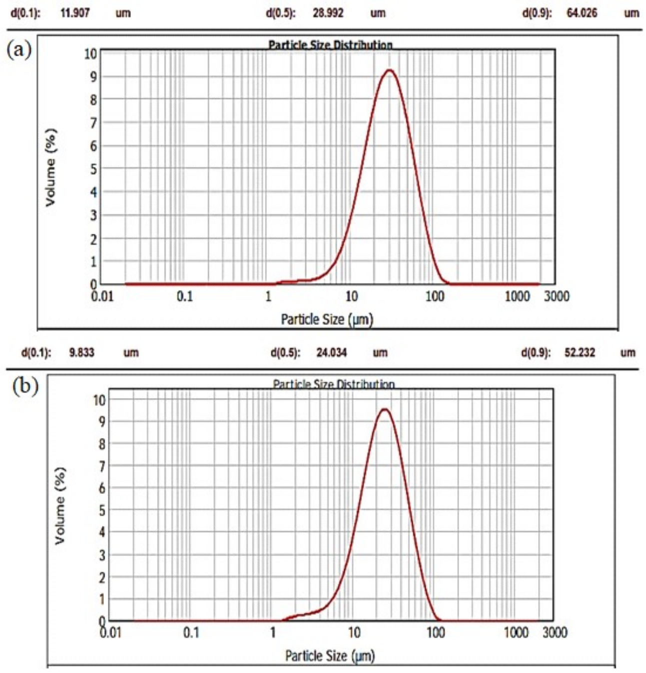

Figure 1. Laser diffraction spectrum of aluminum powder (a) AA1100 and (b) manganese powder

According to results previously presented by Silva Junior (2021), the AA1100 aluminum powder had an average particle diameter of 28.992 µm and for the manganese powder, the results of the granulometric analysis showed an average particle diameter of 24.034 µm. After HEBM processing, at different milling intervals, the particulate materials of the AA3003 alloy were also subjected to laser diffraction (Figure 2), showing the respective granulometric distributions according to the applied milling time. Regarding the granulometric distribution of the analyzed powders, it is possible to observe that in Figure 2a referring to the powder submitted to 30 minutes of HEBM and in Figure 2b referring to the particulate material submitted to 60 minutes of HEBM, the curve shows a unimodal distribution. However, in Figure 2c, which presents the particle size distribution of the powder submitted to 120 minutes of HEBM, it is possible to observe that a small fraction of particles presents a different interval from the main curve, characterizing, thus, a bimodal distribution. This suggests that the cold welding and fracturing of the particles during the milling process had not reached the equilibrium point. The particulate material subjected to milling for 30 min had an average diameter of 50.934 µm, indicating larger particles when compared to the average size of the AA1100 aluminum powder, which suggests that at this milling time the phenomenon of cold welding was predominant. With the increase of the milling time to 60 min, the particle size reduced, presenting an average diameter of 43.491 µm, this reduction is evidence that the fatigue fracture phenomenon begins to overlap the cold welding. Finally, the material submitted to HEBM for 120 minutes, despite exhibiting a bimodal distribution, had an average diameter of 30.904 µm. Through these results, it was possible to analyze that the increase in milling time tends to reduce the size of the processed particles. The particulate material processed for 120min was the one that presented the average particle size closest to the elemental powder of aluminum alloy AA1100.

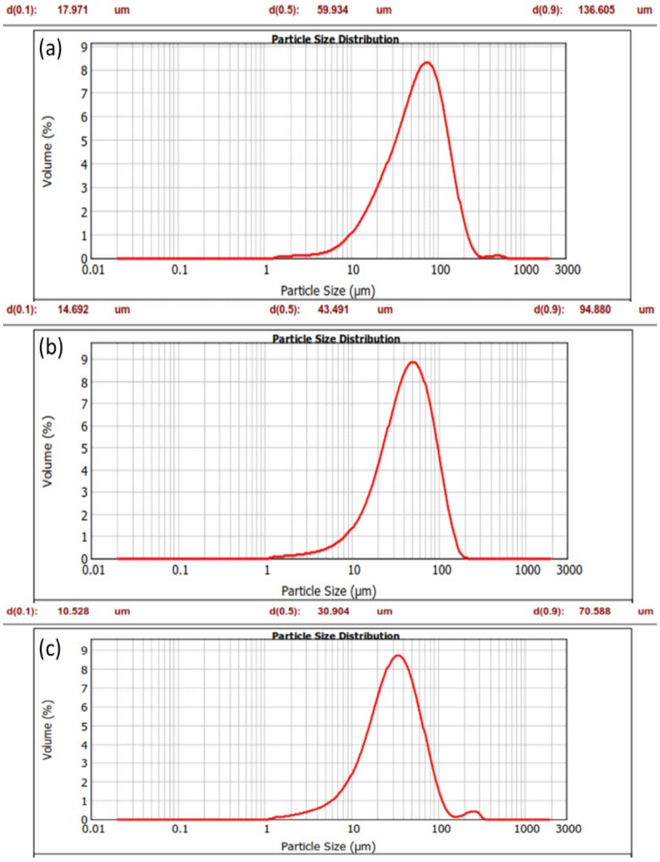

Figure 2. Laser powder diffraction spectrum of AA3003 alloy processed by HEBM for (a) 30 min, (b) 60 min, and (c) 120 min

Morphological and chemical analysis of AA3003 particles processed by HEBM: The evaluation of the particle morphology in those submitted to HEBM was carried out through scanning electron microscopy and X-ray energy dispersive spectroscopy. Figures 3, 4, and 5 are micrographs referring to AA3003 alloy, processed by high energy ball milling at different time intervals.

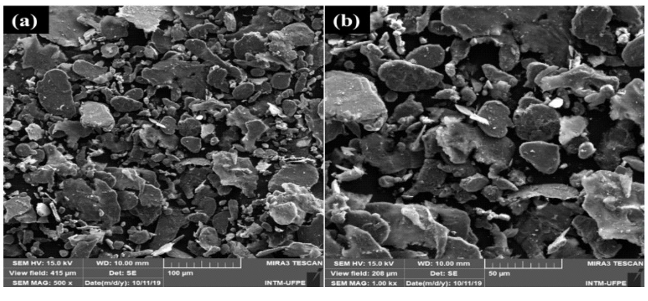

Figure 3. SEM micrograph of AA3003 alloy processed by HEBM for 30 min. (a) 500x magnification; (b) 1000x magnification

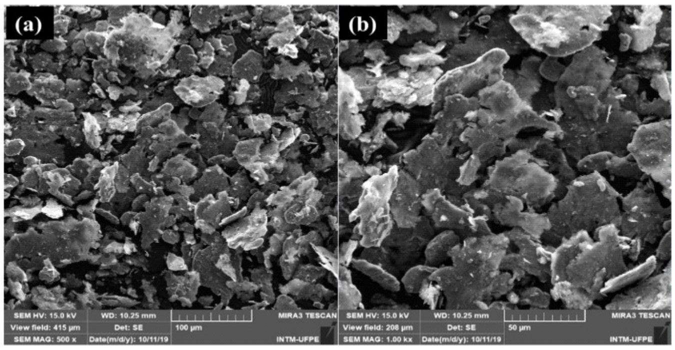

Figure 4. SEM micrograph of AA3003 alloy processed by HEBM for 60 min. (a) 500x magnification; (b) 1000x magnification

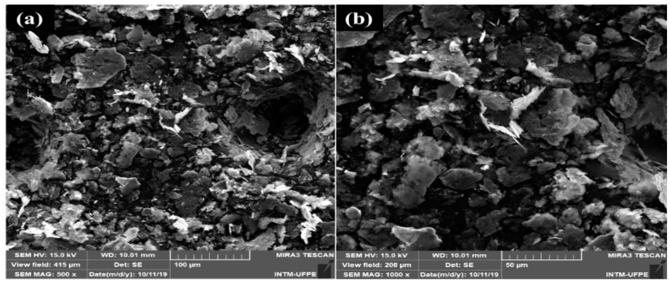

Figure 5. SEM micrograph of AA3003 alloy processed by HEBM for 120 min. (a) 500x magnification; (b) 1000x magnification

From the analysis of the micrographs (Figures 3-5), it was possible to observe that the particles that compose the aluminum alloy AA3003, for all the milling intervals applied, presented irregular and flattened morphology, in agreement with the type of processing (HEBM) (SILVA, 2017). It was also possible to validate the results obtained in the laser diffraction, where it was evidenced that the increase in milling time contributed directly to the reduction in particle size, producing more uniform particulate materials concerning to shape and dimension for longer intervals of milling time. In addition to the microscopy analysis, Figures 6, 7, and 8 present the results obtained by the energy dispersive spectroscopy (EDS) performed, allowing the analytical characterization of the chemical compositions of the AA3003 alloy processed by HEBM at different milling intervals.

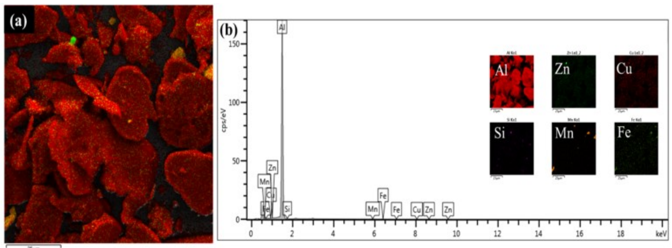

Figure 6. EDS map of the AA3003 alloy powder processed for 30 min, (a) Region analyzed; (b) EDS spectrum

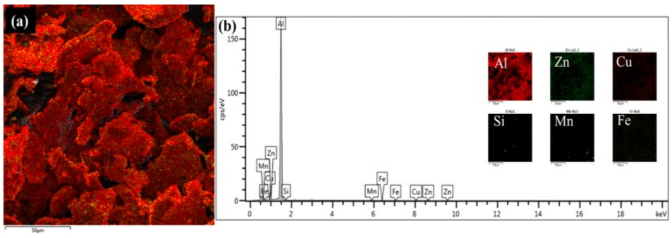

Figure 7. EDS map of the AA3003 alloy powder processed for 60 min, (a) Region analyzed; (b) EDS spectrum

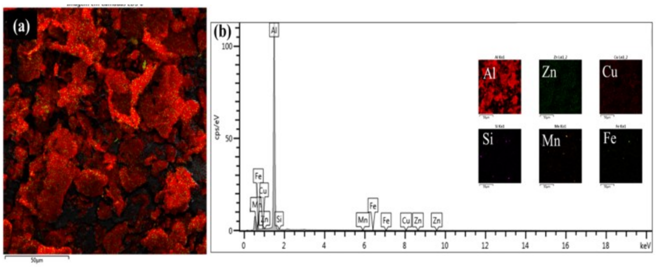

Figure 8. EDS map of the AA3003 alloy powder processed for 120 min, (a) Region analyzed; (b) EDS spectrum

The results obtained through the analysis show that the milling time did not influence the formation of new compounds, since the spectra obtained in the different milling conditions showed only the chemical elements added to the alloy, such as Al, Zn, Cu, Si, Mn, and Fe, as highlighted in Figures 6-8. From the micrographs and spectra related to the different milling intervals, it is plausible to state that the increase in the time used in the HEBM processing provides a greater distribution between the elements that form the AA3003 alloy, as the particles become uniform. It was also possible to verify that there was no contamination of the particulate material since the spectroscopy revealed the presence of only the base materials.

Characterization by X-ray diffraction of AA3003particulate material processed by HEBM: To analyze the constituent phases of the particulate materials of the AA3003 alloy processed by high energy ball milling using the intervals of 30, 60, and 120 min, the diffractograms of the samples produced were obtained, as shown in Figure 9.

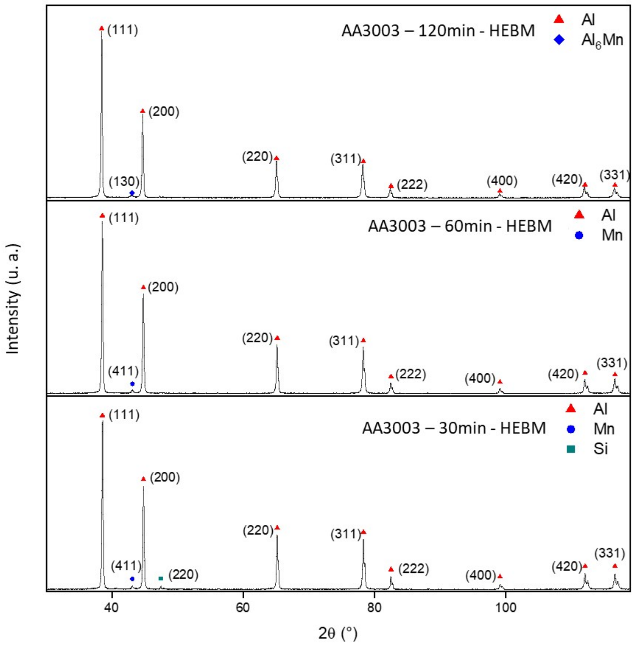

Figure 9. Diffractogram of AA3003 alloy powder, HEBM for 30, 60, and 120 minutes

The diffractograms of the powders processed by HEBM in the intervals of 30 and 60 minutes, presented as phases Al, Mn, and Si, identified by the reference ICDD 03-065-2869, 089-2105, and 01-075-0589microfiches. For particulates processed at 120-minute intervals, the diffractogram revealed the Al phases and the characteristic phase of the AA3XXX series alloys, the orthorhombic phase Al_6Mn, identified by the ICDD reference microfiche 03-065-26 and 00-006-0665, respectively. This presence of the Al_6Mn phase in the samples produced at a milling interval of 120 minutes, corroborates the finding that milling for 30 and 60 minutes does not present satisfactory results for processing the AA3003 alloy by the HEBM technique.

CONCLUSION

The processing of the elemental powders of the chemical elements of the alloy AA3003 carried out via high energy ball milling was efficient in producing powder samples with unimodal distribution, and all powders produced showed irregular and flattened morphology. However, the powder samples processed during the time of 30 and 60 minutes by the HEBM did not obtain satisfactory results about the particle sizes, whose average diameters were larger than the initial aluminum and manganese powders and what are normally used for manufacturing. via powder metallurgy. In these, it was also not possible to identify the presence of the Al_6(Mn, Fe) phase, and such intervals were considered insufficient for the correct consolidation of the AA3003 alloy. For the manufacture of the alloy under study, the milling time of 120 minutes showed satisfactory results, being possible to identify median particles with an approximate size of the elementary aluminum and manganese powders. In the diffractograms of these samples, the Al_6Mn orthorhombic phase was identified, characterizing the production of an AL-Mn alloy, of the AA3XXX series. It is concluded, therefore, that the best results found regarding the manufacture of AA3003 alloy powders through the high energy ball milling technique were found for samples obtained after applying the milling time of 120 minutes, demonstrating an interval effective processing to identify characteristic phases of the alloy under study as well as to find median particle sizes of the expected order of magnitude.

ACKNOWLEDGMENT

The authors wish to thank the Mechanical Engineering Department of the Federal University of Pernambuco (UFPE),the Pos-Graduate Program in Mechanical Engineering (PPGEM) of the UFPE, the Brazilian Institute for Material Joining and Coating Technologies (INTM-UFPE), for providing materials and the infrastructure for this research, the Brazilian Council for Scientific and Technological Development (CNPq), the Superior Education Improvement Coordination (CAPES), and Foundation for the Support of Science and Technology of Pernambuco (FACEPE) for financial support and scholarships for the project.

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