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Examining the effets of niobium pentoxide on the physical and mechanical properties of an aluminum alloy produced by powder metallurgy

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ABSTRACT

This study aims to perform various analyses to characterize the physical (linear shrinkage, mass variation, and porosity) and mechanical (hardness) properties of the aluminum alloy, considering the influence of niobium pentoxide through powder metallurgy. The results revealed a lack of uniformity in the linear shrinkage data. Additionally, it was observed that increasing proportions of niobium pentoxide led to higher percentages of mass variation and porosity, indicating the presence of internally generated gases not entirely eliminated during sintering. Regarding hardness, there was a significant increase, going from an average of 29.31 HRA in the alloy without Nb_2O_5 to 82.40 HRA in the mixture with 50% Nb_2O_5, an overall increase of approximately 280%. Therefore, this study confirms that adding different amounts of niobium pentoxide to an aluminum alloy can improve specific properties, such as hardness. However, it is important to consider the implications for other properties, such as porosity, linear shrinkage, and mass variation, depending on the desired application.

Keywords: *niobium pentoxide.powder metallurgy.aluminum alloy.hardness*

INTRODUCTION

Aluminum is a non-ferrous metal abundant in the Earth's crust and ranks as one of the most widely sold and consumed materials globally, with Brazil being the sixth-largest producer of this metal. Its attractive properties, such as strength, lightweight, and electrical conductivity, make it valuable in various applications, ranging from aeronautical engineering to shipbuilding, bridges, and automobiles. Additionally, aluminum has a relatively low melting point, high resistance to oxidation, superior thermal conductivity compared to steel, high reflectivity, and is non-magnetic. Notably, aluminum can be infinitely recycled without a loss in its physicochemical properties.
Aluminum alloys are formed when alloying elements are added to an aluminum matrix to enhance mechanical strength without compromising other properties. Elements like copper, silicon, tin, and titanium can be incorporated to impart specific properties. Niobium, a transition metal, is one such element with diverse applications, from bridge structures to aircraft turbines, rockets, and space probes. Niobium is known for its high thermal and electrical conductivity,

malleability, ductility, and resistance to corrosion, heat, and wear, improving the properties of metal alloys.
This study aims to blend aluminum alloy powders with niobium pentoxide in different weight proportions (5%, 10%, and 50%) using powder metallurgy techniques. The objective is to characterize the mixtures in terms of linear shrinkage, mass variation, porosity and hardness, to assess the influence of niobium pentoxide on the overall properties of the aluminum alloy.

MATERIALS AND METHODS

To manufacture the alloys developed in this study, two types of metal powders were utilized: powder of an aluminum alloy and niobium pentoxide powder (Nb2O5). Three types of test specimens (TS) were produced: from aluminum alloy powder, niobium pentoxide powder, and a combination of both, with varying percentages of niobium pentoxide (5%, 10%, and 50% by weight). The compaction of these powders was carried out to form green compacts. This involves depositing the powders into a mold (or a die) and applying a specific compressive force. In the present study, a cylindrical metallic mold with a single movable punch was employed. This mold produces test specimens with a fixed diameter of 25.40 mm, as illustrated in Figure 1.

Fig. 1 - Metal mold used to compact powders. (Author, 2023)

The equipment used for applying loads to the mold is a universal testing machine by Emic with a maximum load capacity of 300 kN. The chosen compression pressure for the niobium pentoxide test specimens was 222 MPa, which corresponds to approximately 112 kN when using a cylindrical mold with a diameter of 25.40 mm [1]. The Table 1 below provides details of all the loads adopted in the production of the various test specimens manufactured.

Table 1: Compaction loads of the manufactured specimens. (Author, 2023)

Material	100% Al	100% Nb_2O_5	5%Nb_2O_5	10%Nb_2O_5	50%Nb_2O_5
Applied Load (kN)	100	112	120	120	120

For the sintering of the samples, the equipment used in this work is an electric muffle furnace, Linn Elektro Therm model, which reaches a maximum temperature of 1300°C. It does not have a controlled atmosphere or controlled cooling, and therefore, the samples were cooled within the muffle itself. The Equation (1) below was used to determine the sintering temperature.

$$sintering\ temperature = \frac{\%Alloy * Alloy\ temperature + \%Nb_2O_5 * Nb_2O_5 temperature}{100\%} \quad (1)$$

Table 2: Sintering time and temperature. (Author, 2023)

Material	100% Al	100% Nb2O5	5%Nb2O5	10%Nb2O5	50%Nb2O5
sintering time (min)	40	240	120	120	240
sintering temperature (°C)	625	1200	654	682	862

Table 2 indicates all the time and temperature values used in the samples. To conduct the linear shrinkage test, the manufactured test specimens were measured in two distinct situations. The first measurement was taken after the powder compaction process, obtaining the dimensions of the still green compacts. The second measurement was conducted after the sintering of the samples. Subsequently, Equation (2), based on ABNT NBR 9623 [2] , was employed.

$$\%LS = \frac{L_S - L_C}{L_C} \qquad (2)$$

Here, %LS represents the linear shrinkage percentage, L_S is the length of the sample after sintering, and L_C is the length of the sample after compaction (green compact). The dimension analyzed in the test specimens was the height.
Mass variation (Δm) is a property similar to linear shrinkage. The difference lies in the fact that this time, variations in mass occurring in the test specimens will be evaluated. These variations are generated by the chemical reactions during the sintering step of the samples and can be determined using Equation (3), as presented below [3].

$$\Delta m(\%) = \frac{m_S - m_c}{m_C}.100 \qquad (3)$$

Here, Δm represents the mass variation percentage, m_S is the mass of the sample after sintering, and m_C is the mass of the sample after compaction (green compact). To determine these values, the precision balance used in the powder weighing step was reused.
To determine the porosity, it is necessary to know the percentage of pores within a structure to assess its level of densification and, thus, analyze the influence of these pores on the general properties of the analyzed material. One way to calculate the percentage of pores in a test specimen is through Equation (4).

$$porosity(\%) = 1 - \frac{d_m}{d_t}.100 \qquad (4)$$

Here, $porosity(\%)$ represents the percentage of pores in the sample, dm is the measured density of the sample after sintering, and dt is the theoretical density provided by the companies that supplied the powders.
Mechanical characterization was performed by conducting hardness tests on the samples. The test conducted was of the Rockwell type. The testing machine used in this study was a benchtop analog Rockwell hardness tester. It allows for the use of both 1/16-inch steel ball and diamond penetrators, resulting in values on the Rockwell B (HRB) and Rockwell C (HRC) scales, respectively

RESULTS AND DISCUSSION

In Table 3, the %LS is presented, determined using the equation outlined in the previous section, along with the mean and standard deviation of each specimen. Analyzing the results for the test specimens with 100% Al and 5% Nb_2O_5, it can be observed that the height of most test specimens experienced a slight increase after sintering, indicating linear expansion, and %LS exhibited positive values. This may suggest that the samples were not fully sintered, and, consequently, the gases generated within them were not entirely eliminated.
Conversely, for the test specimens with 50% Nb_2O_5, there was a considerable increase in the heights of the test specimens, with an average variation of 4.36%. Similar to the previous analysis, this positive variation in %LS indicates incomplete sintering, implying that gases resulting from internal chemical reactions were not completely eliminated. Examining the results for 10% Nb_2O_5 and 100% Nb_2O_5, it becomes evident that the height of the samples exhibited a slight reduction after sintering (%LS with negative values), signifying that the samples underwent successful sintering, and the gases generated within them were effectively eliminated.

Table 3 - Linear shrinkage calculated for each composition (Author, 2023)

specimen	100% Al	100% Nb_2O_5	5%Nb_2O_5	10%Nb_2O_5	50%Nb_2O_5
S1	0.21	-2.20	-1.02	-0.78	4.44
S2	0.32	-1.76	0.32	0.00	4.56
S3	0.55	-1.36	0.72	-0.49	4.07
$\overline{x}$	0.36	-1.77	0.01	-0.42	4.36
σ	0.20	0.42	0.91	0.39	0.25

In Table 4, the mass variation percentage ($\Delta m(\%)$) is presented, determined using the equation outlined in the previous section, along with the mean and standard deviation of each specimen. Analyzing the results from 100% Al to the 50% Nb_2O_5 addition, it is evident that all samples exhibited an increase in mass, with very low standard deviations (values below 0.14%). Concerning the average mass variation, the values ranged from 6.094% in samples without the addition of niobium pentoxide to 17.069% in samples with 50% (in % by weight) of Nb_2O_5. Hence, there is an almost uniform increase in the samples, proportionate to the percentages of Nb_2O_5. This increase can be attributed to the formation of oxides during sintering.
Regarding the sample containing 100% Nb_2O_5, it is noticeable that the samples showed negative mass variation, with an average loss of -0.123%. This mass loss is linked to the non-sintering of the test specimens, as the powder in the samples continued to detach even after sintering. Thus, handling the samples resulted in mass losses, which subsequently affected the mass variation results.

Table 4 - Mass variation calculated for each composition (Author, 2023)

specimen	100% Al	100% Nb_2O_5	5%Nb_2O_5	10%Nb_2O_5	50%Nb_2O_5

S1	6.184	-0.121	6,512	7.361	17.183
S2	6.157	-0.107	6.563	7.239	16.951
S3	5.940	-0.140	6.465	7.358	17.074
$\bar{x}$	6.09	-0.12	6.51	7.32	17.07
σ	0.13	0.02	0.05	0.07	0.12

Regarding porosity and considering the dimensions of the test specimens after sintering (height, diameter, and mass), measured density was calculated using the relationship: d = m/V. The theoretical density for niobium pentoxide samples is 4.60 g/cm³. For aluminum alloy samples, a proportion between the percentages and densities of the pure elements was used to define the theoretical density of the alloy, as per Equation (5).

$$theoretical\ density = \frac{\%Alloy * Alloy\ density + \%Nb_2O_5 * Nb_2O_5\ density}{100\%} \quad (5)$$

The measured density of the test specimens followed a similar methodology as that applied to niobium pentoxide samples. Thus, theoretical densities of 5.56 g/cm³, 5.51 g/cm³, and 5.11 g/cm³ were adopted for mixtures with 5%, 10%, and 50% Nb_2O_5, respectively.
In Table 5, the porosity calculated is presented, determined using the equation outlined in the previous section, along with the mean and standard deviation of each specimen. It is noticeable that the compaction pressure, temperature, and sintering time conditions used in the sample manufacturing allowed for a considerable degree of particle cohesion, as the maximum average %porosity reached was 36.27% (for the Nb_2O_5 samples). Furthermore, all analyzed samples exhibited a certain uniformity, as the obtained standard deviations were all below 1%. Additionally, it can be stated that the higher the percentage of niobium pentoxide in the samples, the greater the existing %porosity. This may be associated with incomplete sintering of the test specimens, as the sintering temperatures adopted for the mixtures were much lower than the sintering temperature of pure niobium pentoxide.

Table 5 - Porosity calculated for each composition (Author, 2023)

specimen	100% Al	100% Nb_2O_5	5%Nb_2O_5	10%Nb_2O_5	50%Nb_2O_5
S1	2.69	36.82	4.30	6.67	26.40
S2	1.68	35.77	3.90	5.89	25.77
S3	1.15	36.22	4.74	5.94	26.47
$\bar{x}$	1.84	36.27	4.31	6.17	26.21
σ	0.78	0.53	0.42	0.43	0.39

For the samples composed solely of aluminum alloy, ten indentations were made in different regions of the test specimen to determine their average hardness, using the Rockwell B scale (HRB). In the Alloy-5%Nb_2O_5 mixture, the Rockwell B scale was also employed, with ten indentations performed in different areas of the sample. Comparing the data, it can be affirmed

that the addition of 5% Nb_2O_5 to an aluminum alloy matrix significantly increases the sample's hardness, as the average hardness increased by approximately 190%, going from 36.52 HRB for the sample made solely of aluminum alloy to 69.66 HRB for the sample with 5% Nb_2O_5.
As for the Alloy-10%Nb_2O_5 and Alloy-50%Nb_2O_5 mixtures, the Rockwell C scale was used, as the values exceeded the Rockwell B scale limit. Additionally, eight indentations were made in different areas of the sample. Comparing the data obtained for the samples with 10% and 50% Nb_2O_5, it can be stated that the hardness increased by approximately 124%, going from 50.08 HRC for Alloy-10%Nb_2O_5 to 62.20 HRC for Alloy-50%Nb_2O_5. However, in terms of percentages, this increase was slightly less than what was observed when comparing the sample composed solely of aluminum alloy and the one with 5% Nb_2O_5.
Under the testing conditions used, it was not possible to obtain hardness values for the niobium pentoxide samples because they exhibited high fragility, leading to their fracture during indentation. To conduct a comparative analysis of the average hardness values obtained for the aluminum alloy samples, Alloy-5%Nb_2O_5, Alloy-10%Nb_2O_5, and Alloy-50%Nb_2O_5, it is necessary to convert all values to a single scale, as a direct comparison between HRB and HRC scales is not feasible. To do so, the ASTM E140 - 2007 standard was utilized, which provides conversion tables between different hardness scales. After evaluating the conversion tables within the standard and taking into account the values obtained in the tests, it was decided to convert all values to the Rockwell A scale (HRA), which encompasses all the values obtained in the tests. Given this, Table 6, containing both the actual values and the converted values, was constructed.
It is evident that the increase in the percentage of niobium pentoxide in the mixture led to a consequent increase in hardness values, ranging from a minimum of 29.31 HRA for the sample composed solely of aluminum alloy to a maximum of 82.40 HRA for the sample with 50% Nb_2O_5. Nevertheless, the standard deviations found were significant, with the largest being approximately 19.70%. Despite this, it can be affirmed that niobium pentoxide enhances the hardness of the aluminum alloy.

Table 6 - converted hardness(Author, 2023)

Alloy	$\overline{x}$	σ	$\overline{x}$ (converted)	σ (converted)
100%Al	36,52 HRB	5,70 HRB	29,31 HRA	5,05 HRA
5%Nb_2O_5	69,66 HRB	9,81 HRB	44,13 HRA	8,70 HRA
10%Nb_2O_5	50,08 HRC	2,75 HRB	75,93 HRA	8,31 HRA
50%Nb_2O_5	62,20 HRC	2,14 HRB	82,40 HRA	6.47 HRA

CONCLUSIONS

The linear shrinkage results displayed no consistent trend, making it challenging to assess the precise influence of niobium pentoxide on the characteristics of this test. It would be necessary to examine a broader range of percentages, between 5% and 50%, to identify any patterns and, thus, determine the influence of niobium pentoxide on the aluminum alloy.

The mass variation and porosity results exhibited similar trends, where the percentage of Nb_2O_5 in the mixture directly influenced the outcomes. As the concentration of Nb_2O_5 increases in the mixture, both mass variation and porosity increase. The gradual increase in mass variation, going from an average of 6.513% in the mixture with 5% Nb_2O_5 to 17.069% in the mixture with 50%, indicates that higher concentrations of this element in the mixture lead to the formation of oxides during sintering, consequently increasing the mass of the test specimens.

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