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Heat Treatment Effect on Microstructural, Mechanical and Tribological Properties of Nickel Aluminium Bronze Alloy Manufactured by Laser Powder-Bed Fusion Technique

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Abstract

High density nickel-aluminium bronze alloy ($Cu_gAl_4Fe_3Ni$) was manufactured using Laser Powder-Bed Fusion Technique (L-PBF), it was investigated regarding the effect of different heat treatment conditions on its mechanical, microstructural and tribological behaviour. Correlations between the microstructures generated (k phases) and the behaviour observed were established. Regardless the heat treatment applied (annealing, tempering, quenching and tempering), friction coefficient, wear loss and hardness have been shown to decrease with increasing heat treatment temperature, while tensile strength and the elongation improved compared to the as-built sample. On the other hand, correlations using increased precipitates content resulting from different heat treatments confirmed the improvement of the material's mechanical properties at the expense of the tribological ones. A possible interpretation of this results maybe the role of precipitates in impeding dislocations motion leading to increased shear forces, thus deteriorating the embeddability of the soft α phase along with detachment of the hard κ phases allowing a three body abrasive wear to occur. However, in a process similar to strain hardening, hardness and tensile strength are shown to improve with increased precipitation.

Keywords: Additive manufacturing • Copper alloy • Nickel Aluminum Bronze (NAB) • Tribology • Friction • Wear • Laser Powder-Bed Fusion (L-PBF)

Introduction

Nickel aluminium bronze is a category of aluminium bronze alloys consisting of aluminium as the important alloying element ranging from 8% to 12%, iron and nickel with percentages from 3% to 6% each, manganese is added with a small amount to improve cast ability. Despite their percentages difference, this category of alloys shows similar properties. Many industries such as marine applications, electrical contacts, landing gears and bearings make use of this alloy owing to its high corrosion and wear resistance, high mechanical properties and lightweight material compared to other alloys.

In our previous study [1], we successfully manufactured the alloy whose chemical composition is shown in Table 1 using L-PBF technique, results revealed higher mechanical and tribological properties compared to other manufacturing techniques in the as-built condition. The microstructure of Nickel Aluminium Bronze (NAB) may show various phases depending on the manufacturing technique and the cooling rates applied, besides the martensitic β phase, the high temperature β phase and the lamellar phase, k phases also may be generated, these phases are different in shape, chemical composition and properties [2-5]. Generally, there are 4 kinds of -phases as shown in Figures 1a and 1b along with their formation temperatures.

 k_1 -phase forms when iron content is higher than 5 wt.% [6,7], coarse and globular in shape (Figure 1), rosette form, they precipitate at higher temperature before precipitation of α phase, when they form; they give rise to phase upon cooling. Having ordered bcc structure, they can precipitate as Fe₃Al or FeAl. At 900°C precipitation of k_{11} phase (Fe₃Al) begins initially in the phase and develop in phase, they are found at the boundary of β/α phases along with k_{111} -phase which starts precipitating between 840°C and 600°C. k_{111} are a nickel-rich particles (NiAl) and lamellar or globular in shape depending on solidification conditions, increasing nickel and

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aluminium content or decreasing iron content favor their formation. They are reputed for improving the proof strength of the alloy. $k_{1\nu}$ -phase is a finely divided iron-rich particles in the phases (Fe₃Al with bcc structure), they can appear if the cooling rate is sufficiently slow [8], the strength and hardness of the alloy improve by their precipitation [9-11].

Microstructural analysis showed that upon rapid cooling during L-PBF process, freezing of the microstructure occurs yielding mainly phase and some lamellar phase. Knowing that nickel aluminium bronze can generate various phases during solidification capable of further improving the properties of the alloy, we aimed to apply heat treatments in order to enhance the alloy's performance. In the following, some specific heat treatments will be applied aiming to yield -phases, we will conduct a full investigation regarding the effect of every phase on the mechanical and tribological properties of the alloy. Some studies [9] showed that phases do not have the same improvement behavior on the alloy properties, if mechanical properties are enhanced, negative effect is observed on tribological ones and vice versa, also some phases have higher improvement effect than others as high lighted in Nascimento et al. study [10]. We endeavour to obtain the best combination of phases allowing improvement of both mechanical and tribological properties, taking as a reference, the combination of properties obtained in the as built condition.

Materials and Methods

Experimental

We aim by this study to disclose the effect of different microstructures on the behaviour of nickel aluminium bronze alloy in order to define the best treatment regarding mechanical and tribological properties, appropriate heat treatments were performed for specific microstructures generation as shown in Table 2.

After each heat treatment, microstructural analysis was performed using FE-SEM (FE-SEM Supra40, Carl Zeiss, Germany) after etching with a solution composed of 5g of Fe₃Cl, 10 ml of HCl and 100 ml of distilled water with a reaction time of 20 seconds. The same characterization strategy is followed as adopted for our previous investigation [1] after every heat treatment using the samples shown in Figures 2a and 2b tensile testing, bulk material hardness measurement, friction tests, wear measurements, microstructural analysis based on X-Ray diffraction and FE-SEM. Comparison between the as-built samples and the heat treated Table 1. Chemical composition of Ni-Al-Bronze alloy used in this study.

Al-Bronze	Mass%
Al	9.56
Fe	4.2
Ni	3.39
Zn	0.28
Р	0.02
Pb	0.012
Sn	0.082
0	0.09
Mn	0.843
Cu	balance



Figure 1. a) Schematisation of different NAB phases; b) Temperature formation and precipitation of NAB phases during cooling.

Table 2. Heat treatments conditions applied.

Heat treatment	Temperature (°C)	Time at temperature (h)
Tempering (air cooling)	600	1
Tempering (air cooling)	700	2
Quenching+tempering	850	1
	720	5
Annealing (furnace cooling)	930	1/2
Annealing (furnace cooling)	980	2
Annealing (furnace cooling)	980 to 600	2
+tempering	600	



Figure 2. Samples used for the investigation: a) for tensile testing; b) for friction tests (cylinder on disc configuration).

specimens is considered to evaluate the effect of heat treatment on the alloy's behaviour.

Results

Microstructural analysis

Heat treatments are applied to nickel aluminium bronze alloys in order to eliminate the corrosion prone eutectoid phase leading to degradation of mechanical properties [5], in addition to precipitation of the hard kappa phases. Based on a very interesting model proposed by Pisarek [11] for a cast Ni-Al bronze (Cu-11Al-6Ni-5Fe), in which he unraveled the crystallization and phase transformation temperatures of different micro constituents using TDA (Thermal and Derivative Analysis), we, hereby, propose some heat treatments to nucleate different phases in order to evaluate their effect on mechanical and tribological properties of our additively manufactured alloy. According to the model, precipitation of phases is ranged.

We also considered fast and slow cooling rates as elucidated in Table 2.

Tempering at 600°C yield a rich microstructure of needle-like $\alpha + \kappa_{III}$ as shown in Figures 3a-3f, increasing the temperature from 600°C to 700°C along with time at temperature (2 hours) has a coarsening effect on the phase, which explains the drop in tensile strength and hardness as well as the friction coefficient. Fast cooling (air cooling) promotes particles formation, depending on temperature and time at temperature, these particles may be coarse or fine. Finer particles improve mechanical properties at the expense of tribological ones. Quenching and fast cooling yield and particles, lower friction coefficient and wear loss are observed compared to previous heat treatments. Upon slow cooling (Furnace cooling), particles form leading to further improvement of tribological properties. Globular are also observed



Figure 3. FE-SEM micrographs of etched samples upon various heat treatments: a) tempered at 600°C at-1 h; b) Tempered at 700°C at-2 h; c) Quenched from 850°C-1 h, Tempered at 720°C-5 h; d) Furnace cooled from 930°C-1/2 h; e) Furnace cooled from 980°C-2 h; f) Furnace cooled from 980°C-2 h; d) Furnace cooled from 930°C-1/2 h; e) Furnace cooled from 980°C-2 h; f) Furnace cooled fr

owing to the slow cooling rates as confirmed by Gaoyong et al. [12].

The solubility of iron in phase is exceeded at 850°C [4] (860°C for Anantapong et al. [7]), in the case of slow cooling (furnace cooling heat treatments), formation of and takes place at high temperatures while upon rapid cooling (tempering) forms, however, quenching at 850°C followed by tempering at 720°C does not generate precipitates, we can deduce that at high temperatures and slow cooling rates, the spherical precipitates form while formation of the lamellar precipitates form upon rapid cooling from lower temperatures. Through his investigation, Jahanfrooz [4] concluded that the higher the temperature at which the phase forms, the higher its iron content, this elucidates the absence of upon tempering at 600°C and 700°C. increasing the heat treatment temperature to 850°C and 930°C increases the solubility of iron in leading to precipitationAccording to Pisarek [11], upon cooling the solubility of Al in β phase increases leading to diffusion of Al in the grains and dealuminizing the boundary, thus transforming it to the high excess of Al in phase is resent to β phase by diffusion and locates in the front of its crystallized grains, upon cooling the α phase (Nirich) nucleates and grows as (NiAl). This transformation is situated between 660°C and 860°C, while for Anantapong it is observed at 800°C [7].

Several EDS analysis were performed for each heat treatment on the precipitated phases aiming to reveal their chemical composition, Table 3 summarizes the averaged results for each phase (considering all heat treatments applied). Culpan [5] showed through analyzing intensively k phases (20 to 30 times) that chemical composition of a phase may change from sample to another and even within the same sample, thus, elucidating the fact that these phases can exist within a large range of chemical composition even if they have precipitated at the same temperature from the matrix.

Tribiological characterization

To elucidate the effect of heat treatments applied and the resulting microstructures on tribological properties of Ni-Al bronze, lubricated friction tests were conducted under the same conditions used for the asbuilt sample (50 N, 50 Hz, 1000 μm sliding distance, 80°C and 1 hour test duration. Further details may be found in [1]), using cylinder on disc configuration. The friction coefficient evolutions are depicted in Figures 4a-4f and the mean friction coefficient in Table 4, for the first three heat treatments (tempering and quenching-tempering), fluctuant evolutions and high friction coefficients are seen which may be due to the presence of hard particles at the interface, on the other hand, relatively low and smooth evolutions are observed while annealing from high temperatures with the last three heat treatments. Wear scars were also analysed using FE-SEM, showing some interesting results in good agreement with the frictional behaviour. For the first three heat treatments, the unstable evolution of the friction coefficient is due to the presence of a rough area containing protrusions generated from the severe abrasive and adhesive wear mechanism. The stable evolution of friction coefficient of the last three heat treatments (Figure 4) is obviously due to the smooth wear scars, confirming the dominant adhesive wear mechanism. Generally, wear loss decreases with high temperatures (Table 4) generating low precipitates content (Table 5), Tao et al. [13] also confirmed that the increase in phases increases the wear rate of specimens.

EDS analysis were performed considering the typical surfaces (rough and smooth), the results are depicted in Figures 5a-5f.

At higher applied temperatures (furnace cooled heat treatments), the

Phase	Chemical composition						
	Cu	Al	Fe	Ni	Mn		
α	89.32904	6.364469	1.035875	2.563847	0.706772		
Retained ß	84.27795	7.323211	3.514275	4.154185	0.730382		
k _{II}	21.62 ± 7.69	18.98 ± 2.13	35.75 ± 6.03	21.53 ± 3.68	2.09 ± 0.46		
k _{III}	37.80 ± 11.33	19.49 ± 6.94	19.83 ± 5.28	20.92 ± 4.22	1.94 ± 0.49		
k _{IV}	27.62 ± 3.91	19.63 ± 4.01	30.13 ± 4.45	20.75 ± 3.64	1.85 ± 0.24		

 Table 3. Chemical composition of different phases encountered with the applied heat treatments.



Figure 4. Friction coefficient evolutions under the heat treatments considered in this study: a) Tempered at 600°C-1 h; b) Tempered at 700°C-2 h; c) Quenched from 850°C-1 h, Tempered at 720°C-5 h; d) Furnace cooled from 930°C-1/2 h; e) Furnace cooled from 980°C-2 h; f) Furnace cooled from 980°C-2 h to 600°C-air cooled.

Table 4. Results of mechanical and tribological properties with the applied heat treatments.

	Friction coefficient	μ Wear Volume (mm ³)	Tensile strength (Mpa) Hardness V _н	Elongation%
As-Built Sample	0.2	0.191	479	393	1.49
Heated-600°C-1 h	0.252	0.100	794	267	3.38
Heated-700°C-2 h	0.244	0.106	654	205	14.81
Q-850°C-1 h-T-720°C-5h	0.216	0.097	571	205	18.21
Furnace cooled from 930°C-1/2 h	0.191	0.108	524	193	17.91
Furnace cooled-980°C-2 h	0.179	0.03	531	162	18
980°C-2 h furnace cooled to 600°C + air cooled	0.184	0.016	525	167	23

Table 5.	Phase's	quantification	for each	applied	heat	reatment

Phase	Heat treatn	Heat treatment							
	600°C-1 h	700°C-2 h	Qd850°C-Tp-720°C	Furnace cooled- 930°C-1/2 h	Furnace cooled- 980°C-2 h	980°C-2 h- Furnace cooled to 600°C -air-cooled			
α	75.14%	88.1%	83.5%	89%	89%	96.04%			
k _{II}			9.63%	11%	10%	2.97%			
k _{III}	23.12%	16.4%			1%	0.99%			
k _{IV}	1.73%		7.02%						



Figure 5. Wear scars observation under the heat treatments considered in this study: a) Tempered at 600°C-1 h; b) Tempered at 700°C-2 h; c) Quenched from 850°C-1 h-Tempered at 720°C-5 h; d) Furnace cooled from 930°C-1/2 h; e) Furnace cooled from 980°C-2 h; f) Furnace cooled from 980°C-2 h to 600°C and air cooled.

alumina formed before testing is very adherent to the substrate making the evolution of friction coefficient very stable as we can see in Figure 4d-4e, in that case (smooth surfaces case), an increase of both iron and oxygen concentrations is observed suggesting formation of a transfer film from the counter body. On the other hand, with lower heat treatments temperatures, alumina film breaks up easily as we can see from EDS mapping, owing to its hardness, three body abrasive wear mechanism takes place leading to fluctuating friction evolution (Figure 6).





The results of tensile tests and hardness measurements after each heat treatment are shown in Table 4, a maximum of strength is obtained by tempering at 600°C for 1 hour, according to XRD and phase quantification results (Table 5), this heat treatment generates more precipitates than others, progressive decrease in strength is observed with decreasing the total precipitates content regardless their nature. This may be interpreted

by the decreased effect of strengthening due to decreased content of precipitates, moreover, if we refer to Figure 3, we can see clearly that with globular precipitates, the microstructure is characterized by voids suggesting detachment of precipitates, however, with lamellar precipitates, no voids are found, this observation may lead to think that the lamellar precipitates cannot be detached assuming their high resistance to deformation, thus imparting the material high strength.

Similarly, the hardness is shown to decrease with increasing heat treatment temperatures, if we refer to Table 5, the decrease of hardness is accompanied by decreased precipitated particles, we may even confirm that the particles nature is unlikely to play a role, since both lamellar and spherical particles if precipitated by the same amount, same hardness value is obtained, viz: HV=205 (tempering at 700°C yield 16% of lamellar precipitates vs. Quenching from 850°C+tempering at 720°C yielding also 16% of precipitates globular in nature), however, the maximum hardness is obtained with the as-built sample, this means that the β generated upon the very fast cooling rates is harder than a microstructure of combined with the precipitated phases . With heat treating, transformation of β to α to kphases is induced, thus reducing the amount of retained β or martensitic β phase, as a result, lower hardness is obtained. The elongation, on the other hand, is influenced by the nature of precipitates as we can see with both heat treatments with same amounts of precipitates: tempering at 700°C and Quenching from 850°C+tempering at 720°C, the globular precipitates impart higher elongation to the material compared to the lamellar ones. The

maximum elongation is obtained with the highest amount of α -phase.

Comparing the heat treatment at 930°C for 30 min followed by furnace cooling to the study of Kamran et al. [9] who applied the same heat treatment, L-PBF provide higher hardness with a microstructure containing and phases against only obtained with the hot forged sample.

ZRD analysis

After each heat treatment, X-ray diffraction (SmartLab, Rigaku, Japan) tests were conducted without any pre-processing to avoid altering the microstructure orientations and stress state, the scans were performed in the 2°C range from 5°C to 120°C, using Cu radiation. PDXL software was used to identify (based on fitting the peaks with that of ICCD, PDF-2 release 2014 RDB) and quantify the precipitated phases (based on Reference Intensity Ratio method), the results are shown in Figure 7 and Table 5.



Figure 7. XRD patterns of heat treated samples. Note: (_____) Tempered-600-1 h; (_____) Tempered 700-2 h; (_____) Quenched-tempered; (_____) Furnace-cooled-930-30 min; (_____) Furnace-cooled-980-2 h; (_____) Furnace-cooled-980-600 air cooled.

The main peak of all samples is that of copper rich phase with various magnitudes depending on heat treatment applied, k phases are represented by Fe₃Al and NiAl. The quantification in Table 5 supports X-ray diffraction analysis, only the Cu-rich phase, Fe₃Al and NiAl peaks are present and detected. The increase of heat treatment temperatures shifts the Fe₃Al peak to higher angles, from 30°C with annealing from 930°C to 95°C and 100°C with annealing from 980°C. The pattern of the annealed sample from 980°C till 600°C followed by air cooling (980°C-2 h -Furnace cooled to 600°C -air-cooled) reflects the peaks of the Cu-rich phase, according to the quantification results, this alloy contains 96% of α phase.

Discussion

In his review, Brezina [3] confirmed that quenching followed by tempering of nickel aluminium bronze produces the highest mechanical properties, he also stated that redistribution of stacking faults along with annealing of dislocations occur at first stages of tempering allowing obtention of maximum strength values, slightly above this critical time the strength decreases accompanied with important improvement in ductility. In our study the maximum tensile strength and hardness are obtained with heat treating by tempering for 1 hour at 600°C, upon which a very fine microstructure of $\alpha + k_{111}$ was generated. On the other hand, a rich microstructure with and was obtained with quenching and tempering imparting lower mechanical properties to the material. These results show that L-PBF followed by heat treatments yield different microstructures than expected while processing with conventional manufacturing techniques. This discord may only originate from the use of L-PBF that guarantees formation of a microstructure with the highest amount of dislocations (marten site in the as-built sample) comparing to other manufacturing techniques, thus serving as nucleation sites of fine precipitates upon heat treatments.

Regardless the heat treatment applied (tempering, quenching, annealing...), as much as the temperature increases, the friction coefficient decreases along with the wear rate and the hardness, whereas the strength goes to its maximum with a tempering at 600°C for one hour (up to \approx 800 MPa) before following the same trend as other properties with increasing the heat treatment temperature. On the other hand, the elongation keeps improving with increasing the temperature with a maximum of 23% with the furnace cooled from 980°C till 600°C followed by air cooling heat treatment, this improvement is proportional to the increased amount of phase generated by heat treatments.

Relationship between precipitates and material's properties

Many studies tackled the subject of Stacking Fault Energy (SFE) in copper alloys and its sensitivity to alloying [14], solute concentration [15] and temperature [16] on one hand, and its effect on friction [15], wear and hardness [17] on the other hand. In general, alloying with a higher valence solute along with the increase in solute concentration [14] decrease the SFE. Remy et al. [16] (Figure 8a) explained the decrease in SFE with increasing the temperature by the thermal activation of dislocations motion, he also stated that in case the increase of temperature leads to segregation of a solute, this will impede the dislocations motion, leading to local increase of shear forces, thus increasing the friction coefficient. Buckley [15] (Figure 8b) studied the effect of increasing the solute concentration at room temperature in order to induce segregation, a decrease in SFE simultaneously with increase in shear forces and friction coefficient were observed. Wert et al [17] (Figure 8c) similarly to Buckley, studied the combined effect of increasing solute concentration and the resulting decrease in SFE, he found that wear rate and bulk hardness along with the hardness in the wear scars increase accordingly.



Figure 8. Correlations of material properties vs. SFE: a) SFE vs. temperature; b) SFE vs. Hardness and solute concentration; c) SFE vs. solute concentration, critical shear forces and friction coefficient.

Knowing that the precipitates play similar role on dislocations motion as solutes and with analogy to previous results, we will attempt to establish a correlation between the precipitate concentration and nickel aluminium bronze properties such as tensile strength, friction, wear and hardness.

The material properties were plotted as a function of precipitates content. Similar trends are obtained as for the correlation with solute concentration; the mechanical properties are shown to improve at the expense of the tribological ones. For the friction coefficient, we deduce that increasing precipitates content increases the friction coefficient, while it decreases with more phases in the microstructure. In the case of two different heat treatments (tempering at 700°C for 2 hours and quenching tempering) with the same precipitates content, similar friction coefficient is obtained. The hard k phase alters the embeddability of the soft phase generating an abrasive wear process along with high friction coefficients. On the other hand, enhancement of tensile strength and hardness is granted by the precipitation strengthening effect (Figure 9).





This can be a deciding tool on whether the targeted properties are the mechanical or tribological ones. Keeping more guaranties higher tribological properties while increasing -phases improves mechanical properties. Li et al. [18] found that the optimum volume ratio of the soft to hard phases is 67:33 besides a grain size between 33-46 µm; adhesive wear is found with higher ratio while abrasive wear dominates with lower ratio. In the same way, Yuanyuan et al. [19] correlated the tribological behaviour and mechanical properties to the content of phase and the corresponding grain size, he found that the optimal combination of properties is obtained when the volume percent of phase is 67% and its grain size is about 35 µm. they referred the sharp increase in wear rate and friction coefficient with a volume percent of phase from 70% to 76% compared to the case with less than 63% to the sudden decrease in the yield strength of the alloy at these ranges [20]. He also stated that, if the adhesive wear is dominant, increasing the hardness and lowering the plasticity will have an advantageous effect on friction coefficient and wear rate, on the other hand, if the abrasive wear is dominant, increasing the hardness and lowering the plasticity will have a negative effect on tribological properties [21].

Conclusion

As we investigated our alloy upon the applied heat treatments, we first of all elucidated the precipitation hardening effect of different phases compared to the as-built sample as follows:

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Fine (165% tensile strength)

\alpha + k_{111}

\alpha + k_{11} + k_{1\nu}

\alpha + k_{11}
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 $\alpha + k_{111} + k_{11}$

Strengthening is related to the amount and size of precipitates. The hardness is shown to decrease with increased heat treatment temperature; this is due to recrystallization and coarsening of grains with the applied heat treatments and stresses relaxation compared to the as-built sample. Yuting et al. investigated the difference in micro hardness of friction stir processed NAB (Cu₉.5Al₄.2Ni₄Fe₁.2Mn) using TEM and showed that the obtained microstructure consisted of high quantity of dislocations and high amount of β phase, and that annealing leads to discontinuous static recrystallization and coarsening, decreased amount of β phase and decreased dislocations

quantity thus decreasing the work-hardening effect, which explains the decrease in the micro hardness of the stirred zone. Shen et al. showed that heat treatments leading to significant density of k phases in NAB fabricated by WAAM yield samples with higher hardness compared to the as-fabricated one, on the other hand, if the density of phases is low, lower hardness is to be expected. The reverse is observed in our case, the asbuilt samples not containing any k phases showed the highest hardness, upon heat treatments, different types of phases nucleate resulting in lower hardness, in our case β phase transforms completely to α +k, β does not seem to contribute to the hardness improvement.

If we refer to the microstructures obtained with different heat treatments, we can see that the single phase offers the best tribological properties at the expense of the mechanical ones, associated with (930°C for 30 min furnace cooled) improves moderately the hardness and strength while the friction coefficient and wear rate increase. With the addition of phase (Quenched-tempered HT), further improvement of mechanical properties at the expense of tribological ones is observed. A microstructure consisting of $\alpha + k_{111}$ (tempered at 700°C for 2 hours) causes raising of friction coefficient along with the hardness and yield strength, while the worst tribological properties and the highest strength are obtained with the finest microstructure (tempered at 600°C for 1 hour).

Ranking of friction coefficient regarding HT applied:

 $\alpha < \alpha + k_{11} < \alpha + k_{11} + k_{1\nu} < \alpha + k_{111} < \alpha + k_{111} + k_{1\nu}$

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