EFFECTS OF HEAT TREATMENT TIME ON THE MICROSTRUCTURE AND SOME MECHANICAL PROPERTIES OF C36000 BRASS*

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Abstract
This study aims to evaluate the influence of different annealing times on the microstructure and mechanical behaviour of C36000 brass. The specimens where heat-treated for 30 min to 480 min at 790 °C, followed by quenching in water. The microstructural and mechanical characterization was performed. The microstructural characterizations reveal that all specimens exhibit a microstructure consisting α + β, and microporosities, as well as the presence of a well-defined edge region. With increasing annealing time, there is a logarithmic increase in both the grain size of the α and β phases and in the length of the edge region, evidencing a dezincification process. The mechanical characterization revealed that the samples subjected to 30 and 90 min of annealing showed a reduction in mechanical strength, while longer treatment times at 790°C resulted in an increase in mechanical strength.
Keywords: Brass alloys; Heat treatment; Microstructure Characterization; Mechanical Properties

EFEITOS DO TEMPO DE TRATAMENTO TÉRMICO NA MICROESTRUTURA E ALGUMAS PROPRIEDADES MECÂNICAS DO LATÃO C36000

Resumo
Este estudo tem como objetivo avaliar a influência de diferentes tempos de recozimento na microestrutura e no comportamento mecânico do latão C36000. As amostras foram recozidas por 30 minutos a 480 minutos a 790 °C, seguido por resfriamento em água. Foi feita a caracterização microestrutural e mecânica. As caracterizações revelaram que as amostras apresentam uma microestrutura composta por α + β, além de microporosidades, e a presença de uma região de borda bem definida. Com o aumento do tempo de recozimento, há um aumento tanto no tamanho de grão das fases α e β quanto no comprimento da região de borda, evidenciando um processo de dezinficação. A caracterização mecânica revelou que as amostras submetidas a 30 e 90 min de recozimento apresentaram uma redução na resistência mecânica, enquanto tempos de tratamento mais longos a 790°C resultaram em um aumento na resistência mecânica.
Palavras-chave: Ligas de latão; Tratamento térmico; Caracterização da microestrutura; Propriedades mecânicas.

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1 INTRODUCTION

Brass refers to copper alloys incorporating zinc (Zn), prized in industry for their exceptional blend of physical and mechanical attributes, including high corrosion resistance, minimal friction coefficient, non-magnetic properties, superior thermal and electrical conductivity, excellent malleability, and ease of machining [1, 2]. Brass alloys are mainly composed of Cu and Zn, with copper content ranges from 50% to 95%, while zinc content varies from 5% to 50%. In addition to these elements, other elements such as Al, Fe, Pb, Sn, Ni, and/or Mn may be present in low concentrations in brass compositions [3].

In general, the differentiation between brass alloys is based on the composition of copper and zinc, so that the main commercial alloys can be divided into [4]:

- **α brass alloys:** Alloys containing up to 36% zinc. These are alloys containing only one phase (the (α phase), where one or more alloying elements are dissolved in the copper matrix in the form of homogeneous solid solution. Within this alloy, red brass (with 5 to 20% Zn) and alpha-yellow brass (with 20 to 36% Zn) are found.
- **β brass alloys:** Brass alloys with a high zinc content. These are copper alloys, generally harder and less ductile than the alpha phase. The beta phase makes the alloy more ductile when worked at high temperatures and less ductile when worked at low temperatures.
- **Dual phase brass alloys (α + β brasses):** These are copper-zinc alloys containing approximately 54 to 63% Cu and the rest mainly zinc, exhibiting a microstructure composed of two microconstituents.

Dual phase brass alloys are those with a microstructure composed of two phases (α phase and β phase), usually containing zinc levels between 40% and 60% by mass [5]. However, some alloys may have a biphasic microstructure even with slightly lower zinc levels, around 35% Zn, such as C35300 and C36000 alloys [5]. The α phase, rich in copper, has a face-cantered cubic (FCC) crystal structure with low stacking fault energy and good plasticity, providing better room-temperature deformability than at high temperatures (better cold formability). Additionally, the α phase is responsible for the corrosion resistance of these alloys [6, 7, 8]. On the other hand, the presence of the β phase provides excellent hot plasticity to dual phase alloys and allows the forming of components with complex geometries through hot deformation [8].

Among dual phase brass alloys is UNS-C36000 brass (CuZn36Pb3), sometimes referred to as free-cutting brass, which contains about 3% Pb. Being a dual phase brass alloy, in addition to exhibiting the typical properties of this type of brass, the addition of lead contributes to the improvement of machinability due to its lubricating action and stress concentrator effect, promoting the formation of short chips (which increases machining speed) and minimizing tool wear [9, 10]. The characteristic microstructure of these alloys consists of three microconstituents: the α matrix, the β' phase, and lead (Pb) nodules.

In general, lead does not significantly affect the strength of the alloy; however, brass alloys containing lead may exhibit some degree of difficulty in forming, with alloys with lower lead content showing better formability [4]. The morphology and distribution of insoluble lead phase affect not only the machinability of C36000 brass

but also its formability. An increase in the size of lead particles (nodules), as well as non-uniform distribution, leads to reduced machinability and the appearance of surface cracks during forming [2].

This study evaluated the influence of different annealing times on the microstructure and mechanical behaviour of C36000 brass. The results were compared with the characteristics of the same material in the as-received condition (as extruded condition).

2 MATERIALS AND METHODS

2.1 Specimens preparation

Extruded wires of C36000 brass alloys in M30 temper condition (as extruded), with an initial diameter of 9.4 mm, were cut and machined to obtain samples measuring 80 mm in length and 6 mm in diameter. For the preparation of specimens intended for metallographic and mechanical characterization, after the heat treatment the samples were cut using a precision cutter (labcut 1010), resulting in specimens with diameter of 6 mm and a height of 8 mm.

The heat treatment to assess the impact of time on the properties of the C36000 alloy was carried out in a muffle-type electric resistance furnace under air atmosphere, located in the Materials Laboratory 505-1 at UFABC. The specimens where heat treated for 30 minutes, 90 minutes, 150 minutes, 270 minutes and 480 minutes at 790 °C (extrusion temperature), followed by immediate quenching in water.

2.2 Microstructure Characterization

For microstructural characterization, the specimens were embedded in Bakelite resin and subsequently mechanically polished using abrasive SiC paper (#250, #400, #600, and #1000 grit) and colloidal silica of 0.5 µm. After preparation, the heat-treated samples were etched with 30% HNO_3 and photographed using an Observer.Z1m optical microscope, manufactured by Zeiss, located in the Metallurgical Processes Laboratory at the Institute of Technological Research of the State of São Paulo (LPM/IPT). Both the as-received sample and the heat-treated samples were also characterized with the aid of a Scanning Electron Microscope (SEM), a compact model JSM-6010 LA manufactured by JEOL, equipped with Energy Dispersive Spectroscopy (EDS), located at the Multiuser Center of UFABC (CEM/UFABC), Santo André campus.

X-ray diffraction was utilized for the characterization of phases and structures present in the microstructures of the specimens. The data from the diffractograms were collected using a Bruker D8-Focus diffractometer, with a copper anode tube of wavelength λ = 1.5418 Å (Cu Kα), operating at 40 kV and 40 mA of tube voltage and current, respectively. Scans were conducted in the range between 20° and 80° (2θ). The determination of the volumetric fraction of phases in the heat-treated samples was performed using the method of grid superimposed on three micrographs and grain size (GS) determination was carried out by the Linear Intercept method.

2.3 Mechanical Characterization

The Vickers hardness test was performed on the specimens after polishing. Measurements were carried out using a Buehler microhardness tester located at the LPM/IPT. Hardness measurements were taken on the cross-section of the cylindrical samples with a diameter of 6.0 mm. Following ASTM E384-17 [11], a 300 g indentation load was applied for a total period of 15 seconds, and 6 measurements were taken per sample, discarding the first measurement.

Compression tests were conducted following ASTM E9-19 [12] in the Materials Laboratory **505-1** at UFABC using the INSTRON 3369 universal testing machine. A 50 kN load cell was used, with a displacement rate of 1.0 mm/min. For each experimental condition, tests were performed in duplicate. Stress and strain data were then obtained and subsequently used to plot stress-strain curves for mechanical properties analysis.

3 RESULTS AND DISCUSSION

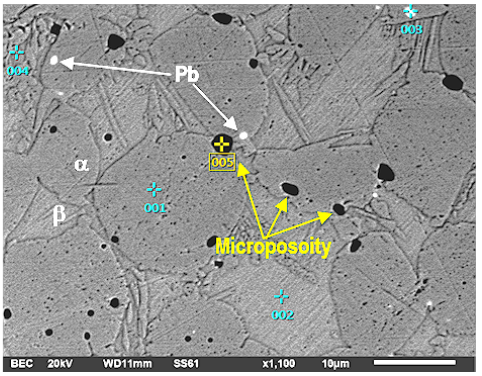
3.1 Microstructural Characterization

Figure 1 presents SEM micrographs of specimens annealed for 30 minutes, 150 minutes, and 480 minutes, all treated at 790°C. The temperature of 790°C was chosen because it is the extrusion temperature for the C36000 brass (in the as-supplied condition). Semi-quantitative EDS-SEM analyses showed that both specimens consist of α phase (with ~34.4% Zn) and β phase (with ~39.5% Zn).

In the analysis of the specimen annealed for 30 minutes, it is observed that along the grains of the α phase, typical annealing twins, known as stacking faults [13], are present. These stacking faults are found in brasses with zinc contents close to 33%. On the other hand, it is observed that the β phase dissolves alloying elements such as Sn, Fe, and Al; according to the literature [14], Sn, Fe, and Al are beta (β) stabilizing elements, meaning they promote the formation of the β phase. However, the presence of these elements appears to induce the formation of dendritic morphologies within the β phase, as well as induce the precipitation of a secondary α phase (in lamellar form) within the β phase [13]. The presence of dendrites and secondary α phase lamellae within the β phase (matrix) is not well understood; however, it may be associated with a condition of β phase instability, as in specimens treated for longer annealing times, where there is more time for chemical homogenization as well as grain growth, no dendrites or secondary α phase lamellae were observed except for a small region in the specimen annealed at 790°C for 150 min (**Figure 1b**).

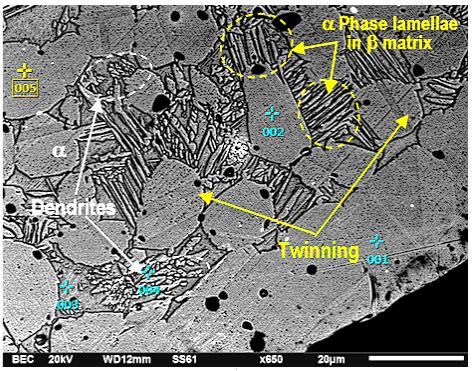
Analysed point	% wt / EDS-SEM				Phase
	Cu	Zn	Pb	Sn/Fe	
+001	65,7	34,3			α
+002	60,3	39,6		0,1	β
+003	54,5	31,5	12,0	0,3	Pb
+004	58,5	41,2			β
+005	57,8	32,9		Others (O, Al, Si, P, S, Ca)	porosity

specimens: 790 ºC / 30 min – Center
(a)



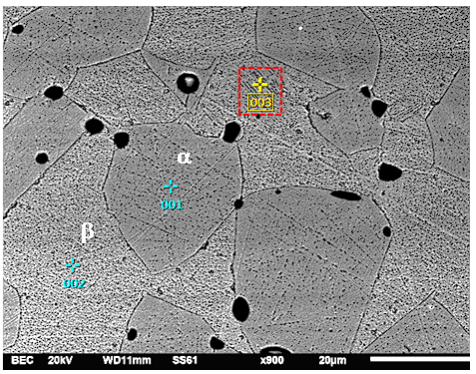
Analysed point	% wt / EDS-SEM				Phase
	Cu	Zn	Pb	Sn/Fe/Al	
+001	67,2	31,8			α
+002	65,6	34,5			α
+003	60,3	39,5		0,2	β
+004	62,3	37,5		0,2	β
+005	65,7	34,3			α

specimens: 790 ºC / 30 min – edge
(a1)



Analysed point	% wt / EDS-SEM				Phase
	Cu	Zn	Pb	O/Si/Sn	
+001	65,3	34,7			α
+002	60,6	39,2		0,2	β
+003	60,2	39,5		0,2	α Phase lamellae in β

specimens: 790 ºC / 150 min – Center
(b)



Analysed point	% wt / EDS-SEM				Phase
	Cu	Zn	Pb	O/Si/Sn	
+001	65,4	34,6			α
+002	64,3	35,4		Others	porosity
+003	60,5	39,3	0,2		β

specimens: 790 ºC / 480 min – Center
(c)

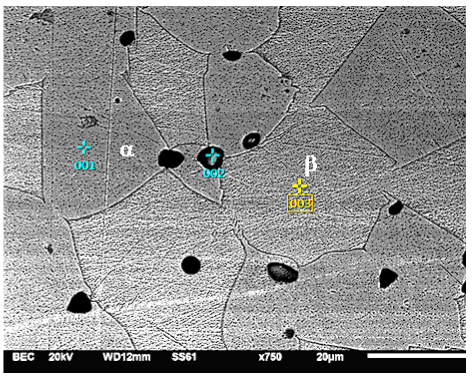


Figure 1. SEM (backscattered electron mode) images of C36000 brass: (a) Central region and (a1) Edge region - annealed for 30 minutes, (b) Central region of specimen annealed for 150 minutes, and (c) Central region of specimen annealed for 480 minutes, all at 790°C.

Figure 2 presents optical micrographs of C36000 brass specimens after heat treatment at 790°C for different times. It provides some characteristics observed in the SEM images (backscattered electron mode). All specimens exhibit a biphasic microstructure consisting of a β phase in low relief, a α phase in high relief, and black

spots corresponding to microporosities, as well as the presence of a well-defined edge region in the cylindrical samples. The specimen annealed for 30 min also shows the precipitation of secondary α phase in lamellar form within the β phase matrix. This precipitation was not observed in samples with longer annealing times.

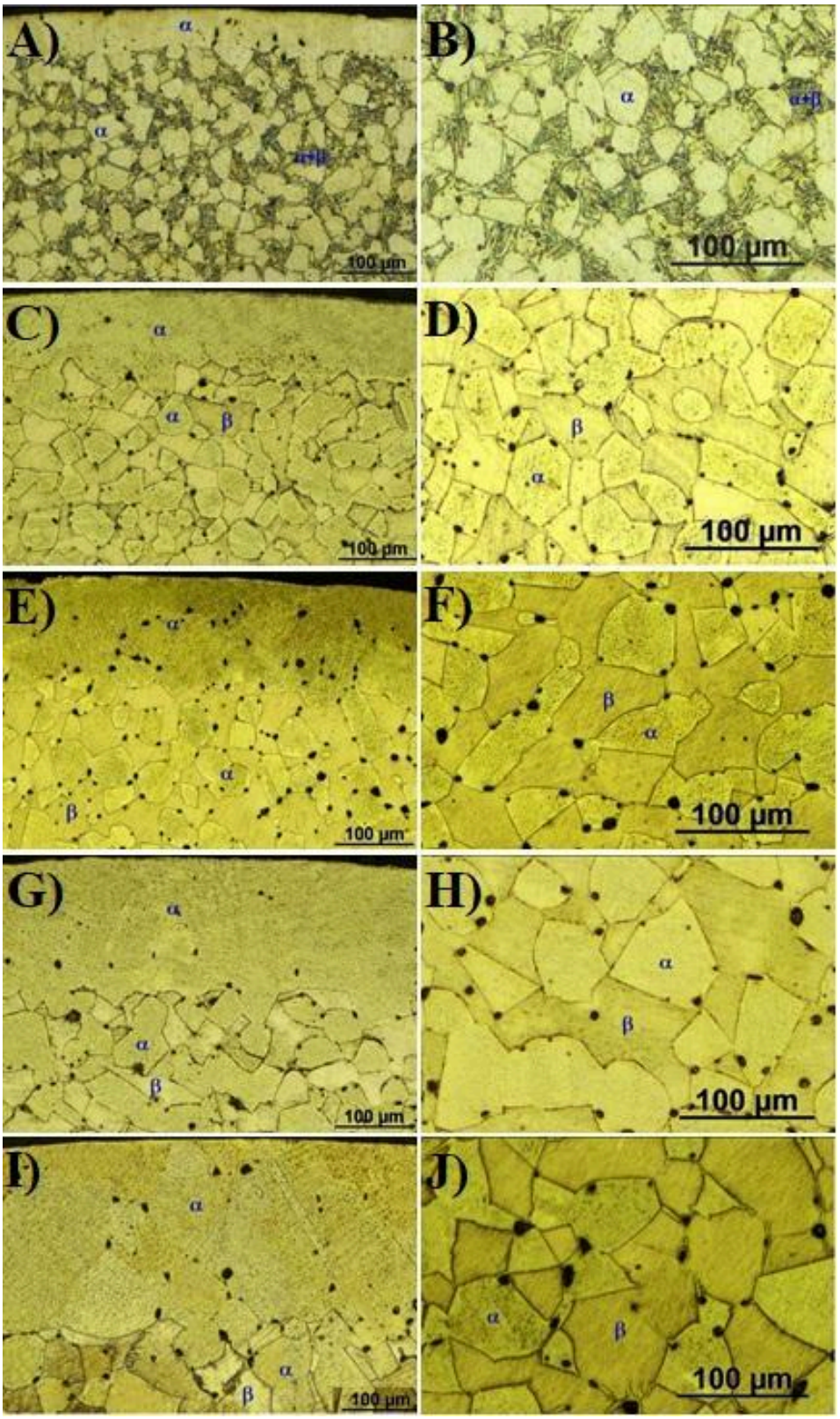


Figure 2. Optical micrographs of C36000 brass after heat treatment at 790°C for A) 30 min - Edge region, B) 30 min - Center Region, C) 90 min - Edge region, D) 90 min - Center Region, E) 150 min -

Edge region, F) 150 min - Center Region, G) 270 min - Edge region, H) 270 min - Center Region, I) 480 min - Edge region and J) 480 min - Center Region,

For each of the specimens, the average grain size and the average length of the edge region (dezincification region) were measured, as shown in **Figure 3a**. It is evident that with increasing annealing time, there is a logarithmic increase in both the grain size of the α and β phases and the length of the edge region. The grain size of the α phase in the as-received sample (without heat treatment - ST), with a grain size of around 9.6 µm, logarithmically increases to sizes around 59 µm for an annealing time of 480 minutes. The same trend of grain size growth was observed for the β phase, although slightly smaller than for the α phase. It was not possible to measure the size of the β phase in the as-received sample (ST) due to the presence of deformed grains resulting from the extrusion operation of the C36000 brass.

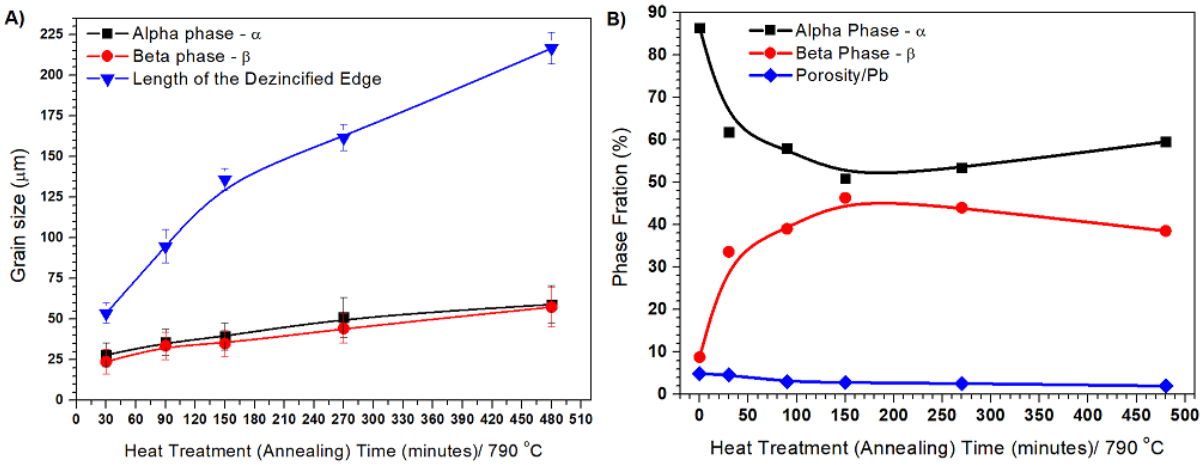


Figure 3. Evolution of (a) Grain size, and (b) Volume fraction of the α, β phases, and microporosity of specimens annealed for 30, 90, 150, 270, and 480 minutes at the temperature of 790°C, and the specimen in the as-received condition extruded at 790°C.

It is also observed that during the different annealing times at a temperature of 790°C, the formation of a coarse layer with varied composition occurs; however, as it approaches the outer edge, the chemical composition of this region becomes richer in copper and poorer in zinc, but still corresponds to the α phase. This can be observed in the EDS-SEM analyses of the sample annealed for 30 minutes in **Figure 1a1**, where point 1 (α phase) exhibits a higher copper content (67.2% Cu and 31.8% Zn) than point 2 (α phase: 65.6% Cu and 34.5% Zn). Although a highly copper-rich regions were not observed in samples with longer annealing times, this behaviour suggests that the formation of these coarse α phase layers (zinc-poor) is associated with the dezincification mechanism. According to Moriarty, M. and colleagues [8], the dezincification process (i.e., loss of Zn present in the alloy) in brasses is favoured by the presence of the β' phase and by the increase in grain size.

In **Figure 3b**, the evolution of the volume fraction of α and β phases as a function of annealing time is observed. The specimen in the as-received condition exhibited a volume fraction of ~8.6% of β phase and, when subjected to annealing, it is observed that with increasing annealing time, it increases rapidly until a maximum value in the condition of 150 min/790°C. This increase in volume fraction is accompanied by a reduction in the volume fraction of the α phase, suggesting the existence of $\alpha \rightarrow \beta$ phase transformation and grain growth. Also, it is observed that for times greater than 150 minutes, there appears to be a stabilization in the volume fraction of both α

and β phases, since at the time of 150 minutes the volume fraction for both phases is close to 48%, and at longer times, there is a non-significant reduction in the β phase.

In **Figure 4**, the X-ray diffraction patterns are presented. In this figure, both the as-received sample in the extruded condition at 790°C and samples annealed for 30, 150, and 480 minutes at a temperature of 790°C exhibit only peaks of higher intensity of the α phase, followed by peaks of the β phase and insignificant peaks of the Pb element, suggesting that both the C36000 brass as received and the annealed samples present a biphasic α-β microstructure.

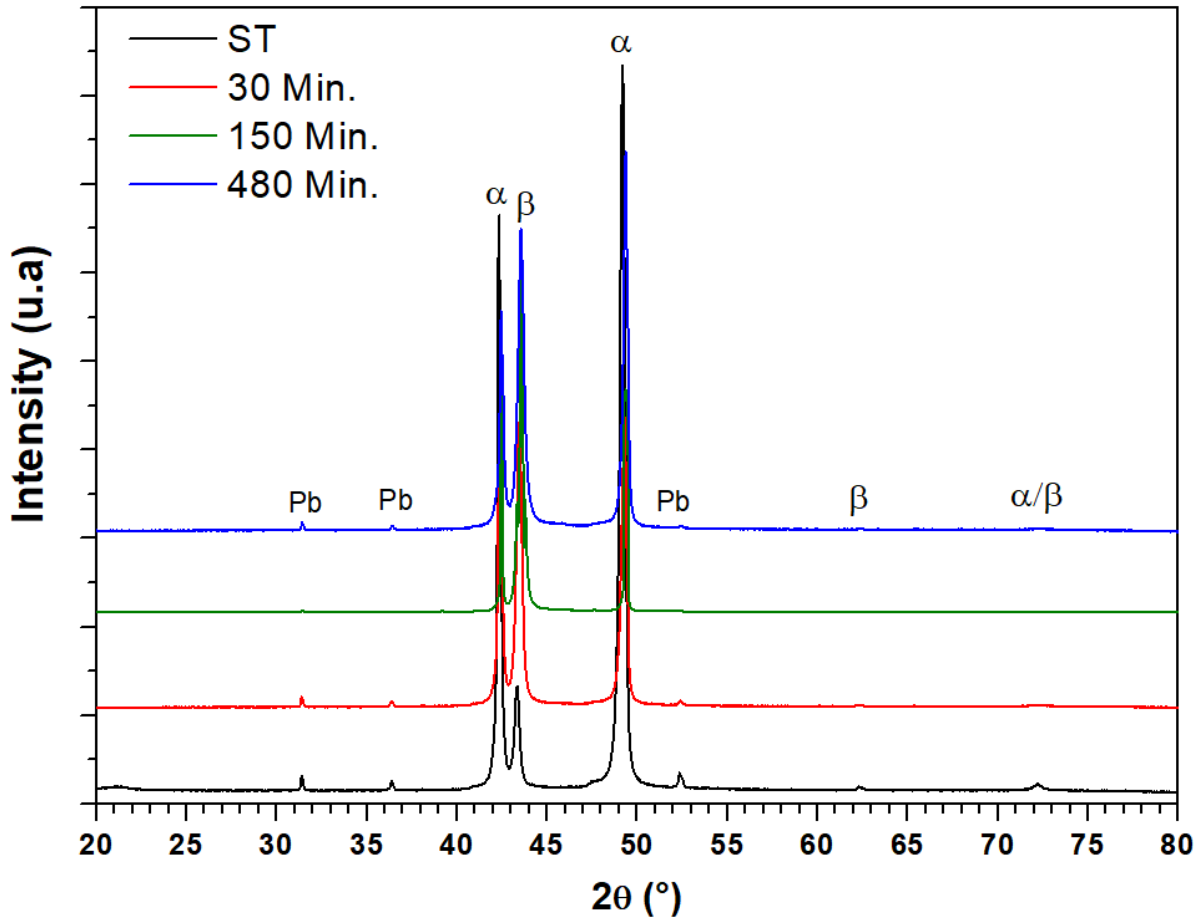


Figure 4. XRD diffractograms of the as-received specimen and specimens annealed for 30, 150, and 480 minutes at 790°C of the sample extruded at 790°C - C36000 Brass.

3.2 Mechanical Characterization

Figure 5 presents the Stress vs. Compressive Strain curves of the as-received specimens (ST) and specimens annealed for 30, 90, 150, 270, and 480 minutes at 790°C. It is observed that, in general, the specimens exhibit a remarkably high maximum compressive strength when compared to the maximum tensile strength values (338 to 469 MPa) reported in the literature [4] for the same C36000 alloy. Additionally, a high strain hardening rate in compression (n = Δlogσ/Δlogε) and high compressive deformation (between 50 to 60% - **Figure 6a**) are noted, indicating that the C36000 alloy exhibits a high plastic deformation capacity before material failure [15]. It is observed that the as-received specimens (ST) in the extruded condition at high temperature (i.e., deformed) exhibit higher compressive strength than the annealed specimens. This fact may be related to their refined microstructure and

residual stresses due to extrusion deformation, which increase mechanical strength and hardness, as observed in **Figure 6.**

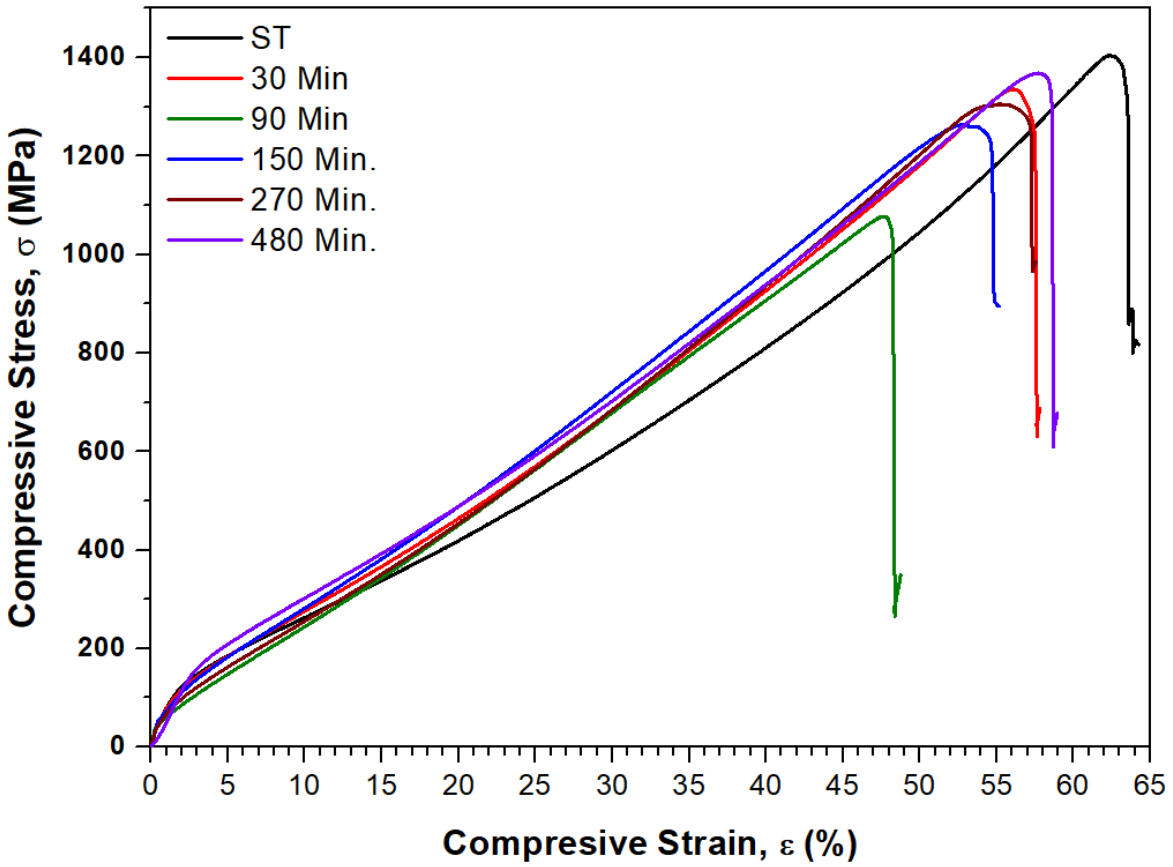


Figure 5. Compressive Stress vs. Compressive Strain Curves for the as-received specimen (ST) and specimens annealed for 30, 90, 550, 150, 270, and 480 minutes at 790°C.

In **Figures 6a** and **6b**, it is observed that there was a reduction in mechanical strength and hardness in the specimens annealed for 30 and 90 minutes at a temperature of 790°C. This effect may be associated with stress relief and atomic arrangement favoring a higher volume fraction of the β phase, as well as grain growth in the β phase, as observed in **Figure 3**. Between 90 and 480 minutes of annealing, a slight increase in mechanical strength is observed, which may be associated with the formation of the maximum volume fraction of the β phase (~45%) and solid solution hardening effects (greater dissolution of zinc atoms in copper in the CCC structure). However, there is a slight decrease in hardness, contrary to the mechanical strength. This decrease in hardness would be associated with the larger grain growth of the α and β phases. However, for annealing times exceeding 270 min/790°C, further studies are needed to confirm the behavior of increased mechanical strength and decreased hardness.

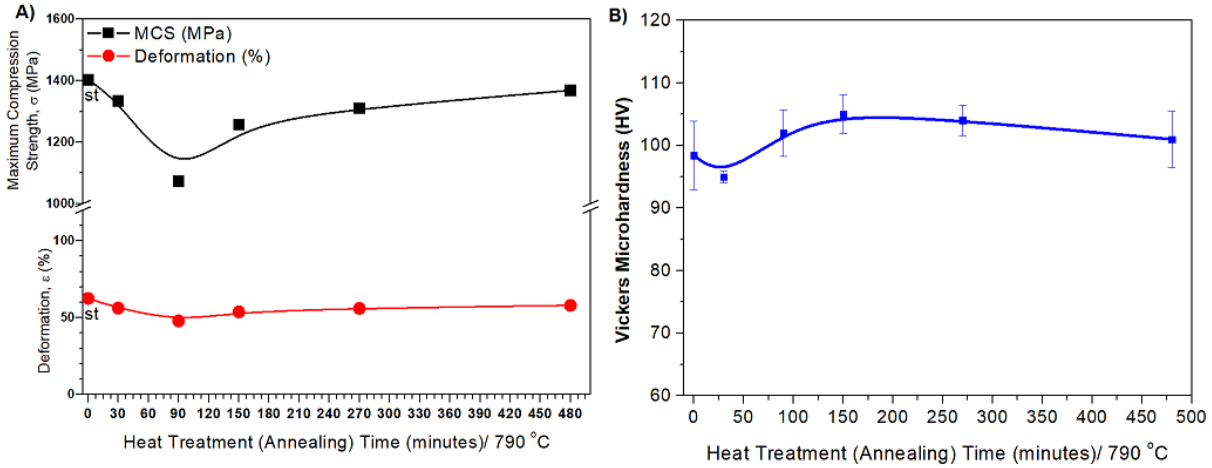


Figure 6. a) Maximum compressive strength and strain as a function of annealing time, and b) Vickers microhardness as a function of annealing time, for specimens annealed for 30, 90, 550, 150, 270, and 480 minutes at 790°C.

4 CONCLUSION

After conducting the heat treatment to investigate the effect of heat treatment (annealing) time at 790°C on the microstructure and mechanical properties, the following conclusions can be drawn:

1. All specimens, regardless of heat treatment time, exhibited a biphasic microstructure composed of α phase and β phase, as observed through various microstructural characterizations.
2. An increase in grain size and edge length with increasing heat treatment time was noted, indicating dezincification (loss of zinc in certain regions) and the formation of a coarse layer on the outer edge of the samples richer in copper and poorer in zinc (α phase).
3. Specimens subjected to 30 and 90 minutes of treatment showed a reduction in mechanical strength, suggesting stress relief during this period.
4. Longer treatment times at 790°C resulted in the maximum fraction of the β phase, leading to a slight increase in mechanical strength. The slight decrease in hardness can be associated with the increase in grain size. Further studies are needed to better understand the observed trend of increased mechanical strength and decreased hardness.

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