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Influence of strain rate and heat treatments on tensile and creep properties of Zn-0.15Cu-0.07Ti alloys

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Abstract
The use of Cu and Ti in Zn alloys improves mechanical properties as solid solution and dispersoid particles (grain refiners) may harden the material and reduce creep deformation. This is one of the main design problems for parts made with Zn alloys, even at room temperature. In this work the mechanical behavior of a Zn-Cu-Ti low alloy is presented using tensile tests at different strain rates, as well as creep tests at different loads to obtain the value of the strain rate coefficient *m* in samples parallel and perpendicular to the rolling direction of the Zn strip. The microstructure of the alloy in its raw state, as well as heat treated at 250°C, is also analyzed, as the banded structure produced by rolling influences the strengthening mechanisms that can be achieved through the treatment parameters.

Keywords: Zn alloys; creep; strain rate coefficient; solid solution hardening; texture.

Influencia de la velocidad de deformación y tratamientos térmicos en las propiedades de tensión y fluencia del Zn-0.15Cu-0.07Ti

Resumen
El uso de Cu y Ti como aleantes del Zn mejora las propiedades mecánicas a través de solución sólida y partículas dispersas (promoviendo el afino de grano), resultando en materiales duros y resistentes al creep, lo que es, incluso a temperatura ambiente, uno de los principales problemas al diseñar piezas con aleaciones de Zn. El trabajo presenta el comportamiento mecánico de una aleación Zn-Cu-Ti a través de pruebas de tensión a diferentes velocidades de deformación, así como ensayos de fluencia a diferentes cargas para obtener el valor del coeficiente *m* de velocidad de deformación tanto en muestras paralelas a la dirección de laminación del material como en muestras perpendiculares. También se analizó la microestructura de la aleación en su estado de laminación, así como muestras tratadas a 250°C, ya que la estructura bandeada producto del rolado influye en los mecanismos de endurecimiento que se pueden alcanzar a través del tratamiento térmico.

Palabras clave: aleaciones de Zn; fluencia; coeficiente de velocidad de deformación; endurecimiento por solución sólida; textura.

1. Introduction

Zn alloys microalloyed with Cu and Ti are mainly used by the construction industry in roofing, gutters and drains [1]. The material is usually supplied as strips that, after solidification, have undergone hot rolling (between 250 and 100 °C). In some cases, room temperature rolling is also performed. Concepts of hot and cold rolling must be carefully considered because at 300 K (27 °C) Zn alloys have a temperature equivalent to 0.43 T_M (the melting temperature is 649 K), which makes them behave like metals deformed at high temperatures (dynamic restoration, creep, static recrystallization after straining, etc.) [2].

A specific requirement for this material is its creep strength [3] when loaded at room temperature. For example, with stresses higher than 90 MPa, stationary strain rate must not reach or exceed $1 \times 10^{-7}\ s^{-1}$ in both parallel and perpendicular

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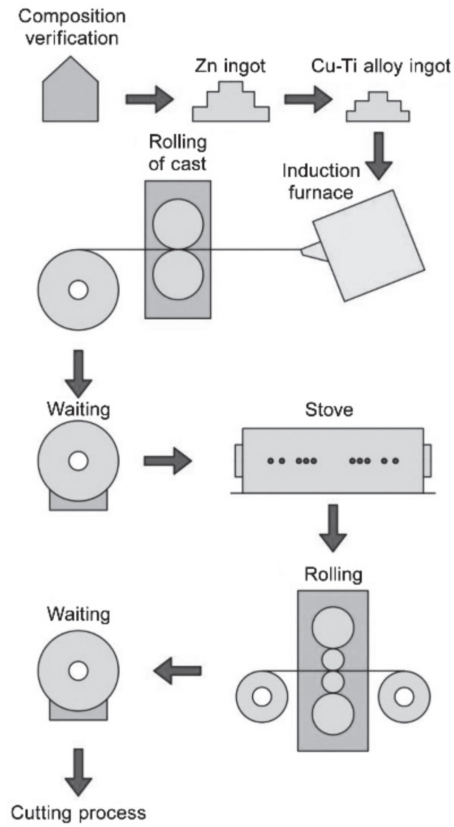

Figure 1. Schematic representation of the rolling process for a 0.7 mm strip at the ASLA facilities
Source: The authors

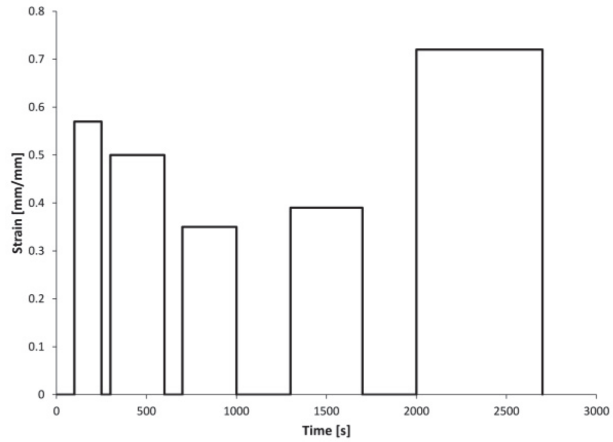

Figure 2. Strain amount during the rolling passes of 0.7 mm strips
Source: The authors

directions compared to the rolling axis (in strips with thickness of 0.7 mm). Adding the microalloying elements Cu and Ti improves hot deformation strength by solid solution of Cu in Zn or by grain refining of the dispersoid $Zn_{16}Ti$, which is formed during solidification [4-6]. The tensile and creep properties of this alloy are the product of hardening by grain refinement due to $Zn_{16}Ti$ [7-9], solid solution hardening by Cu in Zn [10-12] and also the increase in resistance in the direction perpendicular to the rolling axis caused by texture hardening [13-17].

The material analyzed in this work was manufactured by "Asturiana de Laminados (ASLA)" in facilities located at Asturias, Spain, using continuous casting equipment of the "Twin roll casting" type, which produces 8 mm thick slabs in the form of coils (Fig. 1).

After a 10-hour homogenization treatment at 250°C, hot rolling is undertaken in a reversible "Steckel" train at temperatures between 250 and 100°C in 5 phases. A final thickness of 0.7 mm is obtained (Fig. 2).

As the industrial application of parts manufactured with Zn alloys is conditioned by room temperature creep, this work analyzes the behavior of a Zn – Cu – Ti alloy that has been deformed at room temperature in tensile tests (different strain rates) or applied with a constant load (creep tests) in both the raw state and heat treated conditions. The anisotropy of the rolled strip is also considered.

2. Experimental procedure

The strip produced by ASLA that is 0.7 mm thick is a Zn-0.15% Cu- 0.07% Ti used in industrial applications. Samples for mechanical testing were obtained from the Zn strip in both parallel and perpendicular directions relative to the rolling axis and were then machined as tension test samples with a calibrated gage length of l_0 = 100 mm (ASTM E8-04). The samples were tested using an Instron 5583 standard universal machine, while varying crosshead speeds (Table 1) until total fracture of the specimens. Also, creep tests were performed in samples with the same geometries by applying a constant load until steady strain rate behavior was observed for a considerable period of time (Table 2).

Furthermore, tension tests changing the crosshead speed (from 0.1 mm/min to 1 mm/min) every 2 or 3 mm were undertaken in order to calculate the strain rate equation coefficient. To compare the behavior of the raw state rolled material with that of the samples modified by heat treatments, tension test specimens were heat treated at 250°C for periods of 1 and 24 hours.

Microstructural characterization of the samples was undertaken using a metallographic cutter to observe longitudinal sections of the thin slab casting as well as the parallel direction of the thin strip. Traditional grinding and polishing techniques were used to obtain a mirror finish, as well as etching using Palmerton reactive. Metallographic characterization was performed through optical microscopy with Nikon Epiphot equipment and the quantitative analysis of the grain sizes of the α-Zn grains was undertaken using a Buehler Omnimet image analyzer connected to the optic microscope. This allowed for automatic grain identification and ASTM grain size measurement to be used, although in the case of the heat treated samples manual counting techniques were employed.

3. Results

The microstructure of the thin slab casting is shown in Fig. 3. Columnar grains of α-Zn (disperse constituent) are surrounded by an eutectic matrix (α+$Zn_{16}Ti$), just as is expected from the phase diagram (Fig. 4) [18]:

$$L\,(0.07\%) \rightarrow 74\%\ \alpha_{proeut} + 26\%\ (\alpha + \mathrm{Zn_{16}Ti}) \quad (1)$$

The phase diagram indicates a 74% presence of proeutectic phase and 26% of eutectic, which results in approximately 1.1% of $Zn_{16}Ti$. It is, therefore, an abundant dispersoid capable of grain refinement and a barrier to the restoration and recrystallization processes during hot deformation.

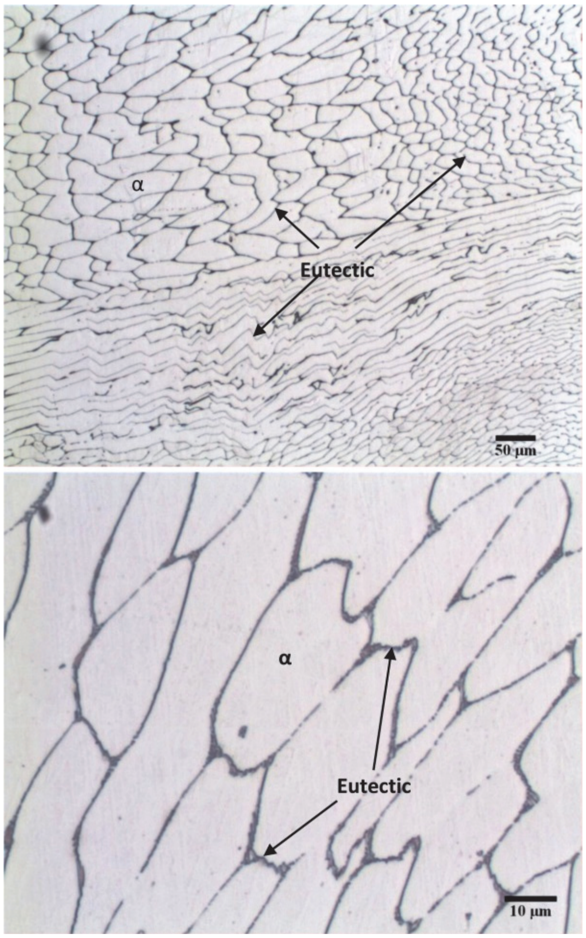

Figure 3. Solidification microstructure: α grains and eutectic phase (α + $Zn_{16}Ti$)
Source: The authors

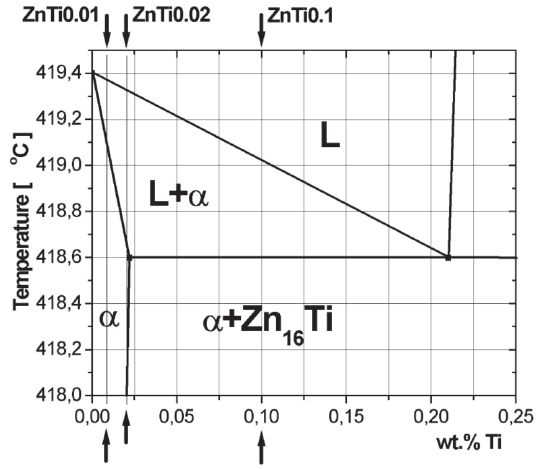

Figure 4. Zn-Ti phase diagram.
Source: [18]

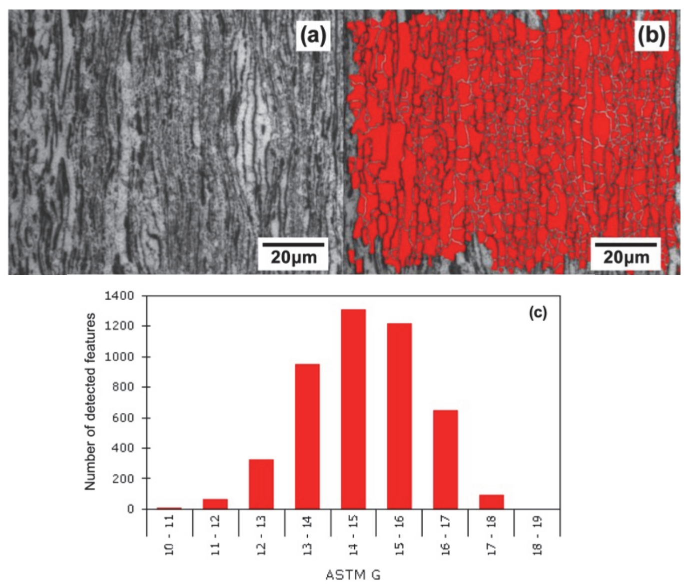

Figure 5. Raw state microstructure (a), detected grain pattern (b) and ASTM G grain size distribution histogram (c)
Source: The authors

Moreover, the microstructure of the raw hot rolled state strips (0.7 mm in thickness) observed in a section parallel to the rolling direction (Fig. 5a) is that of a restored material, partially recrystallized, and with bands in the rolling direction. Just as Kurz [19] observes, lighter zones are α-Zn grains and darker ones are α+$Zn_{16}Ti$ eutectics which were not separated nor spheroidized after the homogenization and hot deformation treatments. The optical microscopy does not show $Zn_{16}Ti$, neither at the boundaries nor at the interior of the α-Zn grains. Also, the size of the α-Zn grains where the eutectic is deformed is smaller than at the bands where the eutectic is not present. The quantitative measurements (Fig.s 5b-c) indicate a very small grain size with a mean length of α-Zn, close to 2 µm (14.5 ASTM).

In order to evaluate the response of this material to strain rate, tensile tests were made at different crosshead speeds in samples both parallel and perpendicular to the rolling direction, as indicated by Table 1. As expected, the higher the strain rate the higher maximum stress the material may withstand, as shown in the engineering stress-strain curves for perpendicular samples (Fig. 6). Though the sample tested at 10 mm/min may have fractured due to defects produced during machining, the rest of the samples have a tendency to deform by larger amounts (values as high as 37%) when low strain rates are used.

On the other hand, samples parallel to the rolling direction present lower maximum stresses (Table 1), although deformations between 50 and 55% are reached in all samples. This confirms the effect of the banded structure and crystallographic texture in the anisotropic mechanical behavior.

Table 1.
Tension stresses at various crosshead speeds for samples perpendicular and parallel to the rolling direction. RS – Raw State, 1h – 250°C + 1 hour heat treated, 24h – 250°C + 24 hours heat treated.

Crosshead speed		Tension stresses (MPa) 300 K			
		Perpendicular	Parallel		
mm/min	$\dot{\varepsilon} \times 10^{-5}$ (s^{-1})	RS	RS	1h	24h
0.1	1.67	143.3	97.2		
1	16.67	179.0	116.9		
2	33.33	188.5	119.0	129.3	123.3
5	83.33	201.8	133.2	135.5	132.0
10	166.67	207.4	140.3		
20	333.33	213.9			
50	833.33	223.0			

Source: The authors

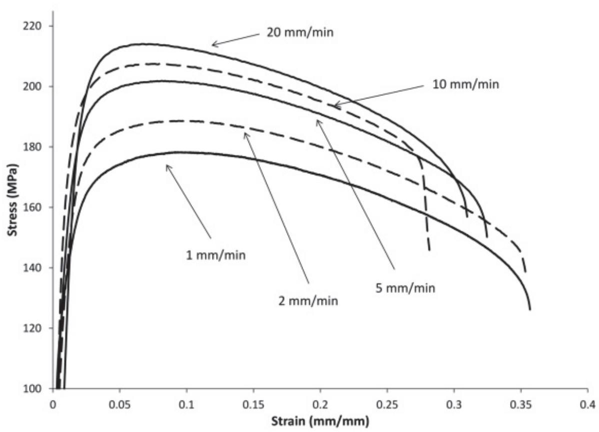

Figure 6. Effect of strain rate on engineering stress-strain curves for samples perpendicular to the rolling direction
Source: The authors

Table 2.
Steady state strain rates at constant stress values, for samples perpendicular and parallel to the rolling direction. RS – Raw State, 1h – 250°C + 1 hour heat treated, 24h – 250°C + 24 hours heat treated

Creep Stress (MPa)	$\dot{\varepsilon} \times 10^{-9}$ (s^{-1}) 300 K			
	Perpendicular	Parallel		
	RS	RS	1h	24h
70	3.2	210.0	30.0	10.0
80	9.8			
90	45.0	7100.0	250.0	1000.0
100	290.0			
110	1090.0			

Source: The authors

As the resistance of the material under constant load is very important in Zn alloys, creep tests were performed in order to determine the strain rate (steady state creep) [16,20,21] for the material (Table 2). Although samples with stresses above 90MPa were tested until they completely deformed and fractured, showing the typical creep curve [22] of strain vs. time (Fig. 7), lower stress levels showed very low strain values. These tests were only carried out until the steady state strain rate was evident.

If the values of stress (maximum stress for tensile tests and constant stress for creep) vs. strain rate are plotted for all the samples tested, it becomes evident that parallel and perpendicular samples have a similar profile: as strain rate is higher, stress reaches a plateau of approximately 190 MPa for the perpendicular samples and 125 MPa for the parallel ones (Fig. 8).

Furthermore, in order to analyze the possibility of improving creep behavior of parallel samples through the modification of microstructural features [23], heat treatments at 250°C were undertaken for periods of 1 and 24 hours. Fig. 9 shows evident microstructural changes due to the activation of diffusional processes [17]: the strip has recrystallized, grain size has grown (13 µm or 9 ASTM for 1 h and 15 µm or 8.5 ASTM for 24 h) and is more homogeneous, and the eutectic phase has fragmented which results in bands diminishing. Nevertheless, $Zn_{16}Ti$ are still not observed at either boundaries or at the interior of α-Zn grains.

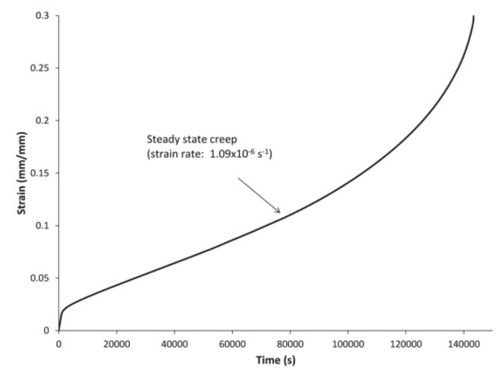

Figure 7. Creep curve for a sample perpendicular to the rolling direction with a constant stress of 110 MPa
Source: The authors

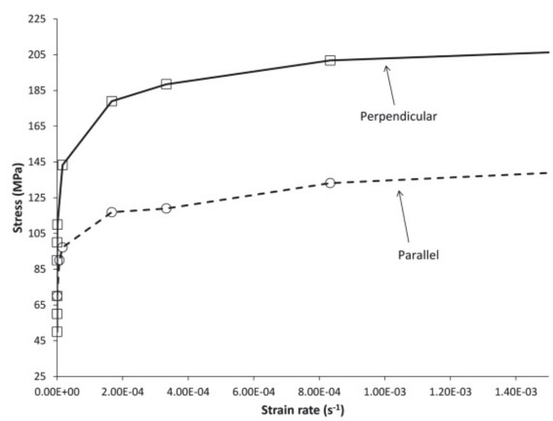

Figure 8. Maximum tension stress vs. strain rate for samples perpendicular and parallel to the rolling direction
Source: The authors

If tensile tests are compared in the case of samples parallel to the rolling direction (Fig. 10), the one hour treatment seems to increase mechanical properties (also indicated in Table 1) caused by the disappearance of the continuous eutectic bands. The 24 hour treatment can only slightly improve mechanical resistance (or even decrease it) depending on the strain rate (Fig. 9 and Table

1): grain growth and $Zn_{16}Ti$ coarsening during recrystallization softens the material [3] and the disappearance of continuous bands results in a continuous α-Zn deforming phase.

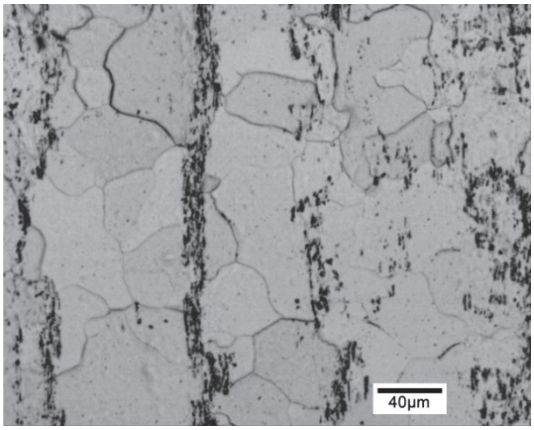

Figure 9. Microstructure of the material heat treated at 250°C
Source: The authors

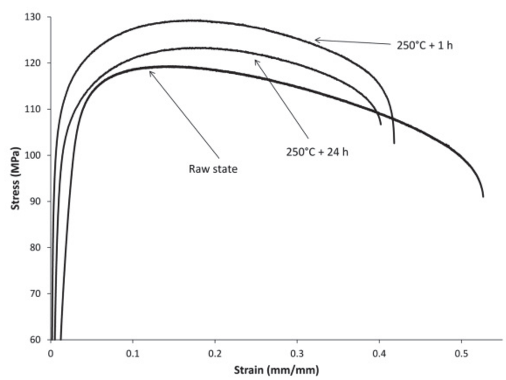

Figure 10. Effect of heat treatments on engineering stress-strain curves for samples parallel to the rolling direction that were tested with a constant crosshead speed of 2 mm/min
Source: The authors

4. Discussion

If the data for maximum stress (in the case of tensile tests) or constant stress (in the case of creep tests) is plotted vs. strain rate (strain rate of the tensile tests or steady state creep rate of the creep tests), using logarithm of both variables, a linear behavior is observed in the parallel and perpendicular directions (Fig. 11), especially above 90 MPa.

In order to confirm that the value of the m coefficient ($\sigma = K\dot{\varepsilon}^m$) is actually the one indicated by the data in Fig. 11 (slopes), a tensile test varying strain rate every 2 or 3 mm from 0.1 to 1 mm/min (Fig. 12) was undertaken. Consider the following equation:

$$m = \frac{\log(\sigma_2/\sigma_1)}{\log(\dot{\varepsilon}_2/\dot{\varepsilon}_1)} \quad (2)$$

where the $\dot{\varepsilon}_2/\dot{\varepsilon}_1$ ratio in this test is 10, σ_2 is the peak value of each step at the 1 mm/min curve sections and σ_1 is the last value of the 0.1 mm/min curve section before the crosshead speed changes. The m coefficient was calculated: for the samples in the parallel direction $m \approx 0.077$, while for the perpendicular direction $m \approx 0.080$. Both values are very similar to the ones observed in Fig. 11, and much lower (0.13~0.33) than the ones reported by Reed-Hill [24].

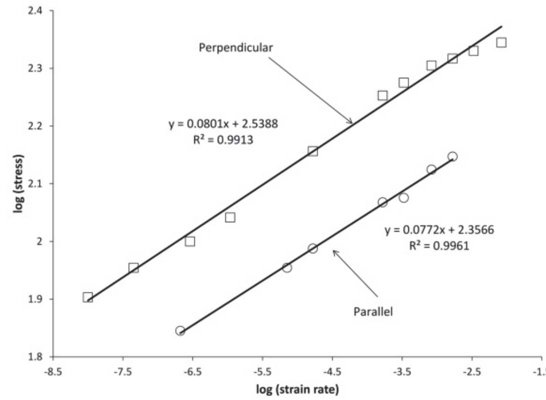

Figure 11. Tension stress vs. strain rate (log of both values) for samples perpendicular and parallel to the rolling direction
Source: The authors

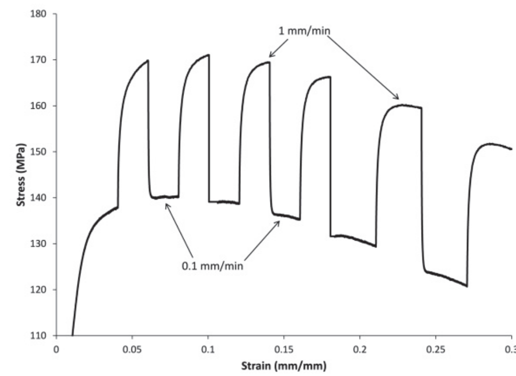

Figure 12. Stress-strain for a sample perpendicular to the rolling direction changing from 0.1 mm/min to 1 mm/min and vice versa every 2 mm (first 4 peaks) and then every 3 mm (last 2 peaks)
Source: The authors

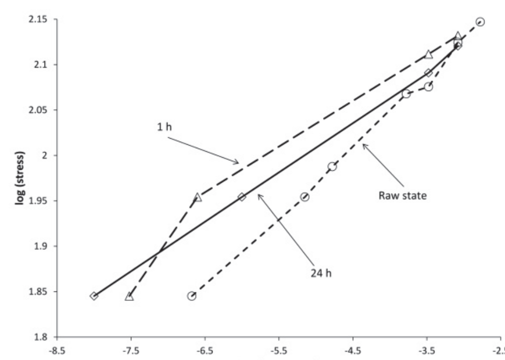

Figure 13. Tension stress vs. strain rate (log of both values) for samples parallel to the rolling direction in their rolled raw state and treated at 250°C for 1 and 24 hours
Source: The authors

In Fig. 13 Stress vs. Strain rate of samples heat treated for 1 and 24 hours at 250°C and the data of the material in its raw state is presented. Although at low strain rates the heat treated samples show higher strength and higher creep resistance than the raw state material, at high strain rates the values converge. This may be an indication that plastic mechanisms activated at low strain rates are different to the ones at high strain rates: at 300 K, creep in these type of alloys is characterized by intergranular sliding, thus the increase in grain size by the 250°C treatments will slow the effect of this mechanism [9].

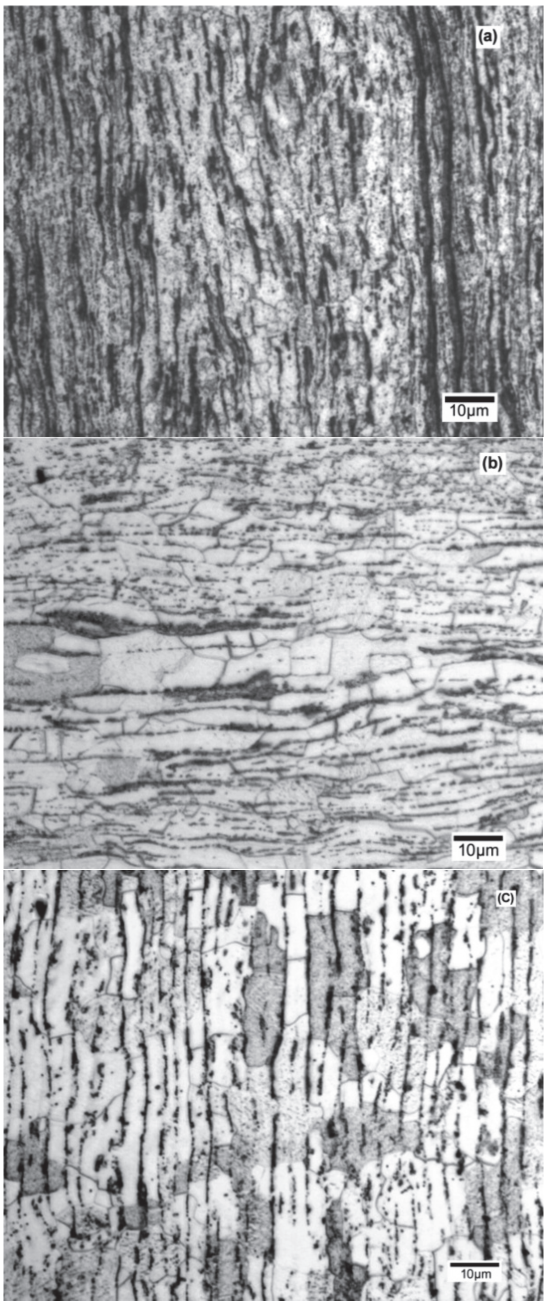

Figure 14. Microstructure of the material a) in its raw state and b) heat treated for 1hour at 250°C and c) heat treated for 24 hours at 250°C
Source: The authors

A comparison of microstructures in a raw state and after both heat treatments (250°C) for 1 and 24 hours is presented in Fig. 14. The increase in resistance for the one hour sample may be explained by the appearance of small, more homogeneously distributed dispersoids (precipitation hardening); however the 24 hour sample no longer presents the fine precipitates. Instead, there are coarser ones (overaging), which explains the decrease in mechanical properties. Furthermore, evidence of twinning is observed in the heat treated samples, which also explains the higher mechanical properties compared to the raw state ones [25-28].

5. Conclusions

Although the mechanical resistance of this alloy in the direction perpendicular to the rolling axis is higher than the parallel one, the strain rate coefficient m in both directions is very similar (0.07~0.08). Moreover, heat treatments at 250°C may increase mechanical properties (tensile and creep resistance), especially at low strain rates. Deformation mechanisms at high strain rates are very similar for raw state and heat treated samples.

The strengthening of heat treated samples deformed at low strain rates may be explained by the combination of the disappearance of continuous bands, twinning of the α-Zn grains and the appearance of homogeneously distributed precipitates.

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