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Strengthening of Zn–22Al–2Cu alloy wire by artificial ageing

J. Hinojosa-Torres [a,*], Y.H. Zhu [a], A. Herrera-Vázquez [a], V.M. Castaño [b]

[a] *Facultad de Estudios Superiores Cuautitlán, Posgrado, U.N.A.M., Ave. 1ro. de Mayo, Atlanta, Cuautitlán Izcalli, Mexico 54700, Mexico*
[b] *Instituto de Física, U.N.A.M., Apartado Postal 1-1010, Querétaro, Querétaro 76000, Mexico*

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Abstract

Changes in hardness, measured at room temperature, were detected after continuous artificial ageing at 250°C, for different periods, in a Zn–22Al–2Cu (wt.%) alloy wire. The presence of the ternary stable τ' phase precipitate (Al_4Cu_3Zn) and the decomposition of the Zn-rich hcp metastable η'_T phase as a consequence of the ageing, are shown in this work. Then, X-ray diffraction showed that after 1 h of ageing the hcp η phase is present in the microstructure of the wire, this phase remaining stable along the heat treatment. The wire was prepared by rapid solidification by using the semicontinuous casting technique, followed by extrusion of the semicontinuous casting rods and, finally, wiredrawing of the extruded material.

Keywords: Semicontinuous casting; Extrusion; Wiredrawing; Artificial ageing; Strengthening; Metastable; Phase transformation

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1. Introduction

Al–Zn alloys have attracted the attention of many researches in the last few decades, as a result many variations of these binary alloys have been reported and their properties characterised. One interesting variation of this family of alloys, namely the Zn–22Al–2Cu (wt.%) alloy prepared by semicontinuous casting, has been recently reported [1]. This novel material is basically the eutectoid alloy of the Al–Zn system but modified with copper and many of its potential applications still remain to be explored in detail. In this context, the analysis of the hardening mechanisms, under different conditions, of this new alloy, represents an interesting enterprise, especially because of the possibility of using artificial ageing processes at low temperatures for improving the toughness of these materials. Accordingly, thin (3 mm in diameter) wires were produced with the Zn–22Al–2Cu (wt.%) alloy and a relatively low temperature heating procedure (250°C) for different periods was applied to the specimens, aiming to understand, with the aid of X-ray diffraction and hardness measurements, the hardening mechanisms that this material undergoes under those low temperature conditions.

2. Experimental

2.1. Specimen preparation and artificial ageing procedure

Circular rods of a Zn–22Al–2Cu (wt.%) alloy were obtained by the semicontinuous casting technique [2]. The use of this technique allows a more

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[*] Corresponding author.

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homogeneous distribution of the alloy elements, with the additional advantage that the resulting grain size is smaller than that attained by the conventional casting technique. Then, the rods were cut in 1-m-long sections and heated at 250°C by half-hour prior to extrusion. After heating, each rod section was placed in the extrusion chamber and, by employing an 18:1 extrusion rate, a gross wire of 9.5 mm in diameter was produced. This gross wire was cold-wiredrew to 3 mm in diameter wire. Finally, pieces of the 3 mm wire were placed in an electric furnace, preheated at 523 ± 5 K. Samples of the wire pieces were withdrawn from the furnace chamber each half-hour up 6 h. In every case, the hot wire pieces were left to cool down in air.

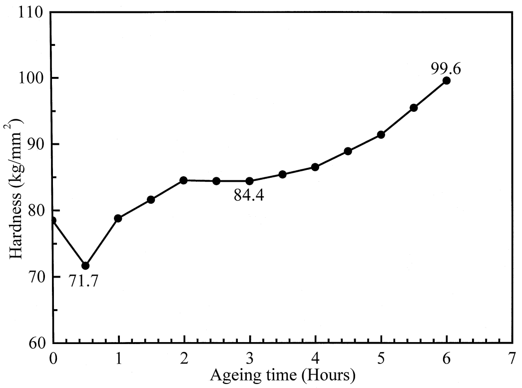

Fig. 1. Hardness curve of the Zn–22Al–2Cu alloy wire aged at 250°C.

3. Characterisation techniques

Vickers hardness testing was carried out on the heat-treated wire longitudinal section with a WOLPERT hardness tester by using a square-based diamond pyramid as indenter. X-ray powder diffraction was performed in a D-5000 SIEMENS machine with Cu Kα radiation, at a scanning rate of 1 deg/min.

4. Results and discussion

The mean values of the Vickers hardness numbers obtained from the hardness measures along the longitudinal section of the aged wires are graphically shown in Fig. 1, for different periods of the heat treatment. The plot of Fig. 1 shows that, after the first half hour of the heat treatment, the wire has already suffered a softening; likely caused by the release of the internal stresses introduced by the previous mechanical forming [3–6]. Subsequently, after an ageing period from 0.5 to 6 h, the wire presents a hardening in two stages: the first occurring from 0.5 to 3 h and the second stage from 3 to 6 h of treatment. Since the hardening is a consequence of the heat treatment only, one mechanism is expected to be taking place; namely, phase transformation. This hypothesis is sustained by the X-ray analysis, which showed the presence of the metastable η'_T phase and the appearance of the stable η phase from 1 h of ageing.

In the first stage mentioned above, the hardening can be explained by the presence of the η and η'_T phases in the Zn-rich grains which reduces the possibility of dislocation motion. Based on the X-ray analysis for the (0002) *d*-spacing and considering that the introduction of Cu in the zinc structure can result in a shrinking of the *c* parameter [4], the reduction of the hardening rate at the end of the first stage can be explained by a high coherency attained among the supersaturated η phase and the η'_T phase as observed on the diffraction pattern of Fig. 4, since the number of physical obstacles for the dislocation motion is reduced, with just a small number of τ' precipitates, remaining. As for the second stage, the hardening can be explained by assuming the chief hardening mechanism to be piling-up of groups of dislocations as a consequence of the presence of τ', η and η'_T phases in the Zn-rich grains, considering that along this stage a poor coupling prevails between the η phase and the η'_T phase (Table 1).

The diffraction patterns obtained from the heat-treated wire samples by different periods showed the presence of the α, η and η'_T phases of the Al–Zn system (according to Presnyakov et al. [7] and modified by Goldak and Parr) and the τ' phase of the Al–Cu–Zn system [8] (Figs. 2–4). The closest packed (111) plane of the α phase produced a diffraction reflection at 38.6°, whereas the closest packed (0002) plane of η and η'_T phases produced diffraction peaks at 36.4° and 36.8°, respectively. Additionally, the

Table 1
Values of the c lattice parameter at the end of each ageing time of η and η'_T phases obtained from the X-ray diffraction analysis
The last column shows the Δc disagreement of this parameter among these phases.

Ageing time (h)	η Phase c parameter (nm)	η'_T Phase c parameter (nm)	Δc (nm)
1.0	0.4930	0.4892	3.8×10^{-3}
1.5	0.4924	0.4872	5.2×10^{-3}
2.0	0.4918	0.4884	3.4×10^{-3}
2.5	0.4922	0.4870	5.2×10^{-3}
3.0	0.4920	0.4860	6.0×10^{-3}
3.5	0.4914	0.4866	4.8×10^{-3}
4.0	0.4924	0.4878	4.6×10^{-3}
4.5	0.4926	0.4882	4.4×10^{-3}
5.0	0.4924	0.4872	5.2×10^{-3}
5.5	0.4916	0.4864	5.2×10^{-3}
6.0	0.4918	0.4858	6.0×10^{-3}

$(10\bar{1}1)$ planes, corresponding to both η and η'_T phases, produced a very intense reflection at 43.3°. The presence of the η phase in the alloy was detected in the samples aged for 1, 1.5, 2, 2.5, 3, 3.5, 4, 4.5, 5, 5.5 and 6 h only. Finally, a weak Bragg reflection located at 44.2° was identified as corresponding to the (110) planes of the rhombohedral τ' phase. This last diffraction peak was present in all the diffraction patterns of all the heat-treated wire samples.

The α phase was identified as an Al-rich phase with an fcc crystallographic structure, with an estimated cell parameter $a = 0.4032$ nm. The η phase was identified as a Zn-rich phase with a hexagonal lattice with estimated lattice parameters $a = 0.266$ nm and $c = 0.492$ nm; whereas for the η'_T, also a Zn-rich phase with hexagonal lattice and $a = 0.2667$ nm and $c = 0.487$ nm were found (the a and c lattice parameters for the η and η'_T phases changes with the solute atoms content [4]).

By analysing the diffraction intensity as a function of the ageing time, it can be observed that variations on the intensity, over all the heat treatment, indeed exist. It is known that the diffraction intensity is directly proportional to the structure factor; then, as the solute content vary in each phase this factor is modified since the scattering factors of Zn, Al and Cu atoms are different. In view of the last mentioned and considering only the diffraction intensity produced by the closest packed layers of each phase (the scattering power of an atom decreases as

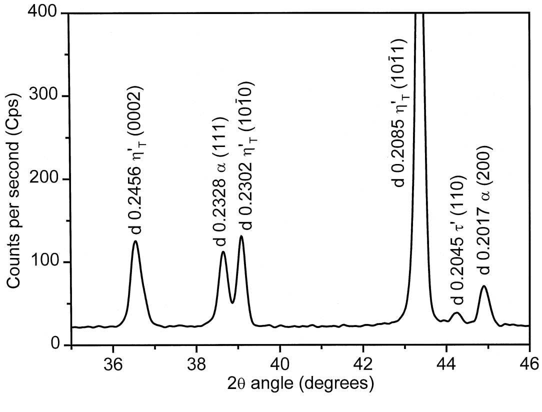

Fig. 2. X-ray diffraction pattern of the Zn–22Al–2Cu alloy wire aged at 250°C for 0.5 h.

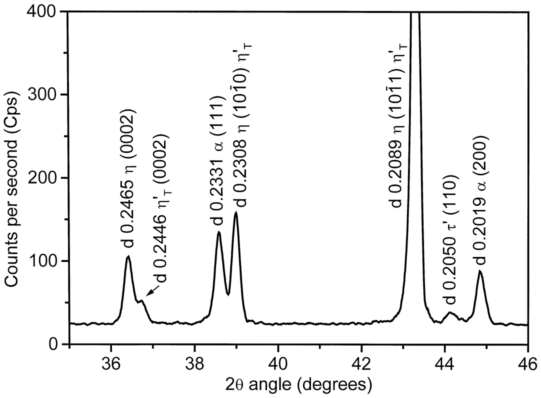

Fig. 3. X-ray diffraction pattern of the Zn–22Al–2Cu alloy wire aged at 250°C for 1 h.

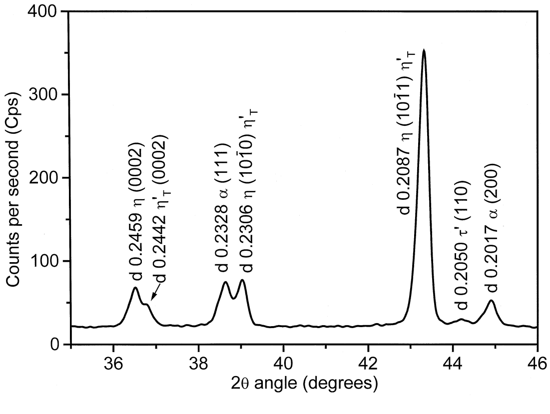

Fig. 4. X-ray diffraction pattern of the Zn–22Al–2Cu alloy wire aged at 250°C for 2 h.

Table 2
Values of the relative diffraction intensity change of the closest packed layers of each phase in the Zn–22Al–2Cu (wt %) alloy wires aged from 0.5 to 1 h deduced from the X-ray diffraction intensity analysis

Time period (h)	Relative increasing (+) or decreasing (−) of diffraction intensity (%)			
	η (0002)	η'_T (0002)	α (111)	τ' (110)
0.5–1	+100	−73	+17	+12

θ increases), it was observed particularly in the interval from 0.5 to 1 h of ageing that the diffraction intensity changes as summarised in Table 2 suggesting the possibility of reaction (Figs. 2, 3).

It must be clarified, however, that the above values were estimated with the residual phases observed at room temperature. Nevertheless, if the air cooling described in Section 2 can be considered as a sort of gentle quenching, in the 0.5–1 h interval the transformation $\eta'_T \rightarrow \alpha + \eta + \tau'$ would effectively occur as reported [9–11].

By comparing the estimated crystallographic data of the η and η'_T phases obtained by the X-ray experiments, it is possible to conclude that the lattice parameter a of the η'_T phase is 0.26316% larger than the corresponding one of the η phase, and that the crystallographic axis c of the η'_T phase is 1.01626% smaller than that of the η phase. By considering the (0001) slip planes in the hexagonal closest packing structure, the smaller size of the c axis in the η'_T phase make semi-coherent this phase with the η phase, and, in consequence, the dislocation movement through the interface are difficult. Moreover, it is well known that the introduction of solute atoms into solid solution in the solvent-atoms lattice invariably produces an alloy which is stronger than the pure metal [5]; then, the frictional resistance to dislocation motion in the η'_T phase would be higher than that one in the η phase as a consequence of the solute atoms content in this phases.

On the other hand, some previous studies on the elemental analysis of both the dendrites and the interdendritic zones in the same alloy as-cast, detected the presence of copper in the interdendritic zones (Zn-rich zones) [1]. This leads to think that the τ' phase precipitate forms preferentially in these sites in the non-extruded material. Now, if the cellular reaction $\eta'_T \rightarrow \alpha + \eta + \tau'$, is really occurring in the wire aged at 250°C, the τ' would form in the Zn-rich zones mainly (Fig. 5), promoting the hardening of these zones. Moreover, the extrusion and wiredrawing process produces an increase in internal energy that, in addition to the thermal energy, allows the alloy to reach the required activation energy for that the cellular reaction to occur.

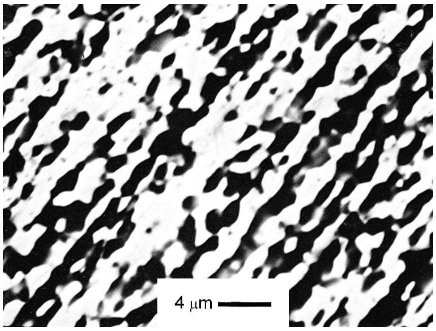

Fig. 5. Scanning electron micrograph of the Zn–22Al–2Cu alloy wire showing an elongated grains structure as a consequence of the mechanical forming with very fine τ' precipitates (medium grey) inside the Zn-rich grains (white) from the sample aged at 250°C for 6 h.

5. Conclusions

The X-ray diffraction analysis of Zn–22Al–2Cu (wt.%) alloy wire prepared by the semicontinuous casting technique followed by extrusion and wiredrawing showed the presence of the α, η'_T and τ' phases in the samples aged at 250°C for different periods. Also, the presence of the metastable η'_T phase and the stable η phase on the phase transformation hardening was determined experimentally.

The low copper content in the alloy restrains the size and number of the τ' precipitates and the potential effect of this type of precipitates on the mechanical properties of the wire is not significant.

Acknowledgements

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