STRUCTURAL NON-HOMOGENEITIES ANALYSIS OF Zn-22 wt. % Al ALLOY BY NANO-INDENTATION

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Abstract. In the present paper the structure and properties of different phases/constituent of the eutectoid Zn-Al based alloys obtained by gravitational casting in brick moulds have been investigated. The microstructural analyses were performed by scanning electron microscopy which was followed by energy dispersive X-ray spectroscopy analysis. Nano-indentation testing has been used to determine the phase/constituents mechanical properties like elastic modulus and hardness for a 22 wt. % Al alloy composition. The structural analysis and the nano-indentation results have revealed the intensity of the segregation phenomena during alloy solidification at smaller cooling rates.

Keywords: Zn-Al, alloys, microstructure, nano-indentation, elastic modulus, hardness indentation

1. Introduction

Over the last years the category of industrial alloys based on the Zn-Al system has widely expanded with high aluminium contents (10 - 30 %), processed by various technologies. The resulting alloys have technological and good exploitation properties that render them eligible for application in the most varied fields.

The disadvantage of these materials is the instability of the structure in time, they undergo heat treatments meant to contribute to an increased structural stability during deployment. At the same time compositions with 18-40% aluminium have superplastic properties [1-5].

The analysis of phase transformation considered the Zn-Al thermal equilibrium diagram established by Presnyakov, Figure 1 [3].

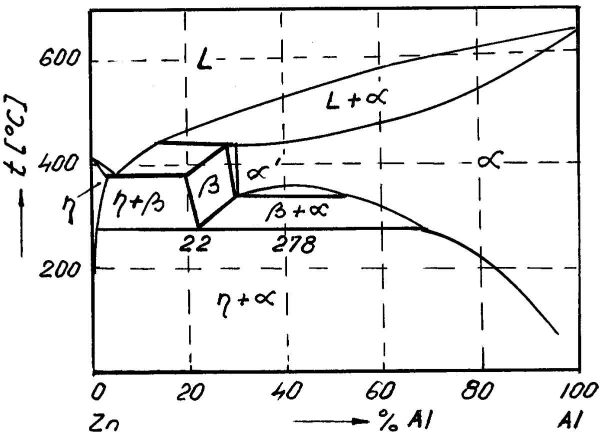

Figure 1. Zn-Al thermal equilibrium diagram, after Presnyakov

The paper analyses the phase transformations and mechanical properties of the eutectoid Zn-Al based alloy cast gravitationally in brick moulds and mention that the cooling rate was 0.64 ºC/s [6].

2. Experimental determinations

Binary eutectoid alloy with wt. % 22 Al has been used for the experimental investigations. The alloy was prepared by melting quantities of Zn and Al (purity > 99.99%) in an electric graphite crucible with silit bars (electrical resistors), at a melting temperature of 650 ºC. After the alloy was melted, the melt was immediately poured gravitationally into a brick moulds (C).

For the structural observations, the samples were prepared using grinding with sand papers with different grit sizes (80 - 1500) and polishing operations with alumina (Al_2O_3) suspension and etched for a few minutes with a HNO_3 + H_2O solution to distinguish the phases/constituent.

Then, the microstructural and morphological characterization of Zn-Al alloys were performed scanning electron microscope (SEM) equipped with an energy dispersive X-ray spectroscopy (EDX) to determine the compositional analysis of each phase/constituents.

Nano-indentation analysis was performed to determine the elastic modulus (E_{IT}) and indentation hardness (H_{IT}) of the phases/constituent of the alloy. The tests were performed using a Nanoindentor CSM Instruments NHT – 2 with a three – sided pyramidal diamond Berkovich indenter.

A series of ten indentation measurements were made on each phases/constituent (α-Al solid solution (a), eutectic (b) and eutectoid (c)) formed on the structure of Zn-22 wt.% Al alloy, with a maximum load of 10 mN, Figure 2. An approach speed of 2000 nm/min. was used. The measurements of mechanical properties were conducted according Oliver-Pharr method [7].

3. Results and discussion

Figure 3 (a) reports the SEM micrograph of the cast gravitationally Zn-22 wt. % Al alloy in brick moulds.

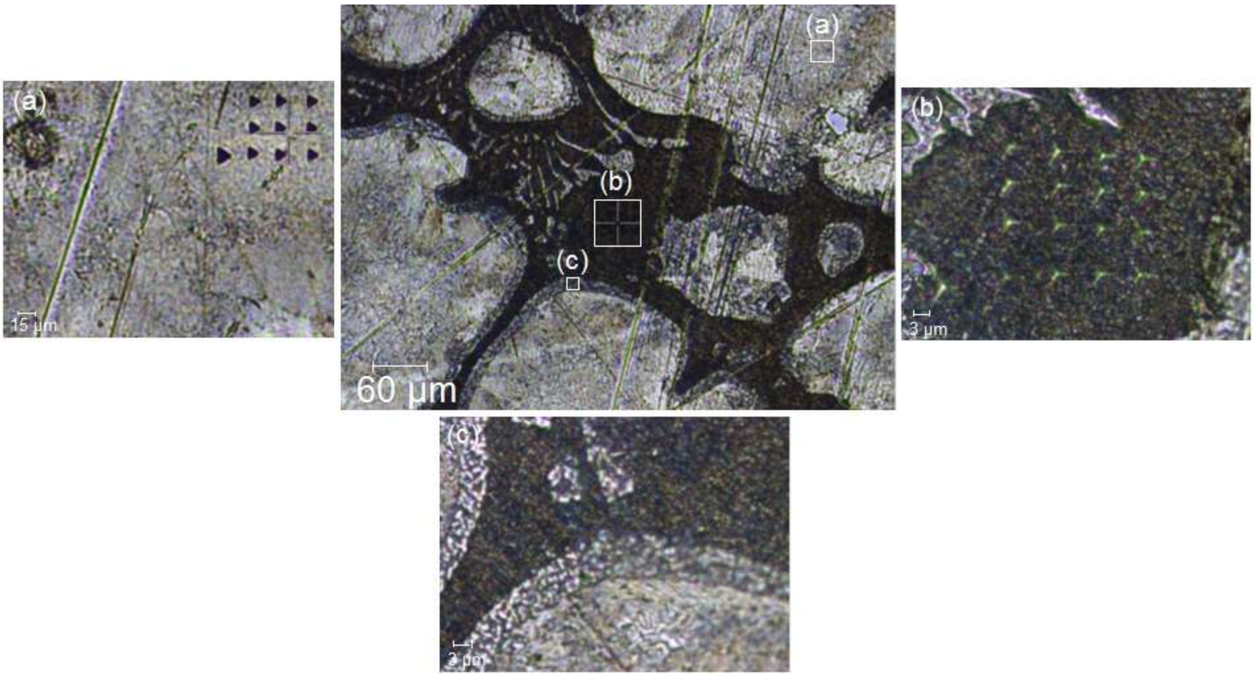

Figure 2. Nano-indentation areas of phase/constituents of the Zn-22 wt.% Al alloy
(a) s. s. α; (b) eutectic [η + β(η + α)]; (c) eutectoid (β → η + α)

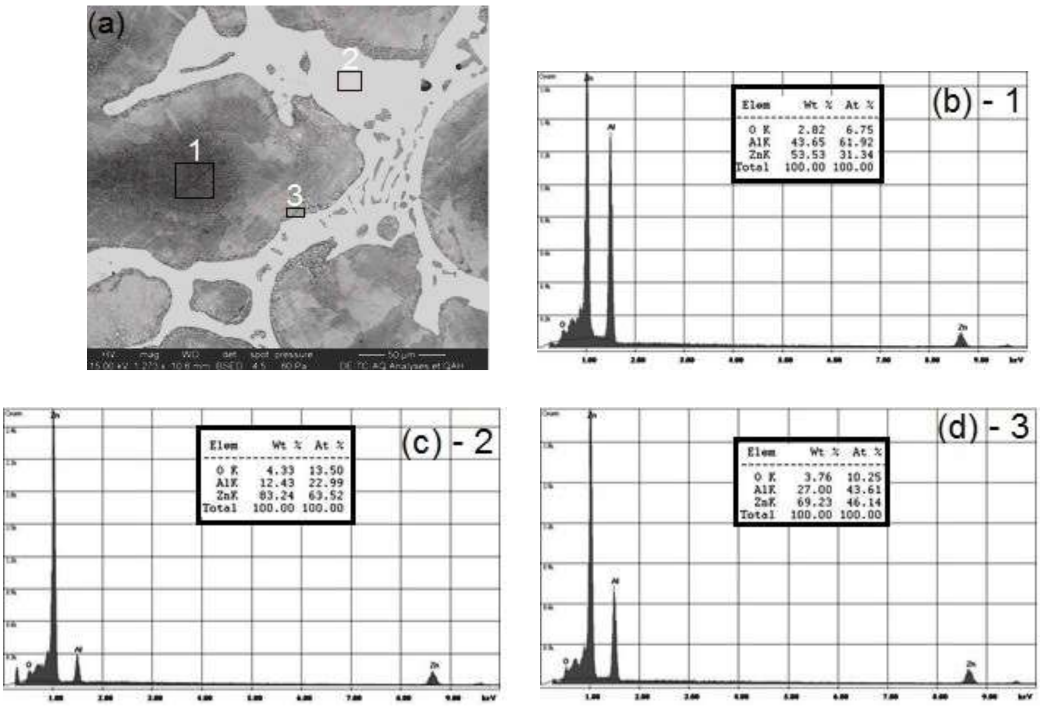

Figure 3. SEM microstructure (a) and EDX spectra (b), (c), (d) on phase/constituents
of the cast Zn-22 wt.% Al alloy in brick moulds
(1) s. s. α; (2) eutectic [η + β(η + α)]; (3) eutectoid (β → η + α)

SEM-EDX analysis was performed to confirm the chemical composition of each phase/constituents, Figure 3 (b, c, d) respectively. Figure 3 (a) shows the alloy exhibited dendritic structure consists of phase and constituents, i.e. α-Al solid solution, eutectic and eutectoid respectively. The main grain value of alloy is about 60 µm. Figure 4 reports the indentation normal force - penetration depth curves, which show us the structural changes that occur in each phase/constituents.

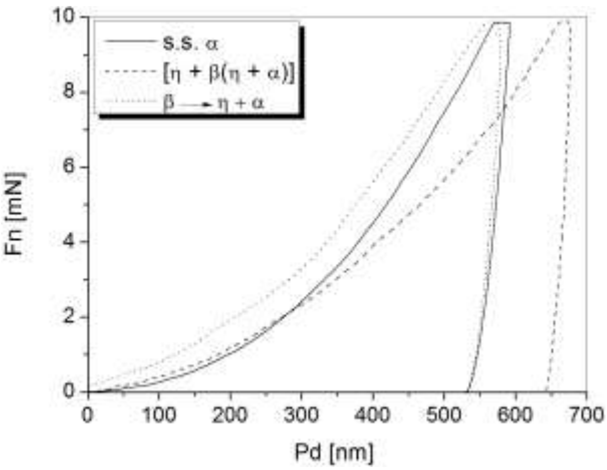

Figure 4. Indentation normal force in function of penetration depth for phase/constituents of Zn-22 wt.% Al alloy (applied load 10 mN)

From those curve it can be seen that aren't no major differences in indentation curves for all phase/constituents tested. Figure 4 reports that the softest phase from the eutectoid alloy is eutectic [η + β(η + α)] with a peak depth of approximatively 650 nm. In comparison with the other peaks, the hardest phase/constituent are the solid solution α and eutectoid (β → η + α), which was penetrated on a depth of approximatively 530 nm. It also shows, that the verticals of the unloading curves of all phase/constituents confirm a smaller elastic deformation. Figure 5 (a, b and c) show the variation of mechanical properties of Zn-22 wt. % Al alloy according the indentation measurements for individual phase/constituent.

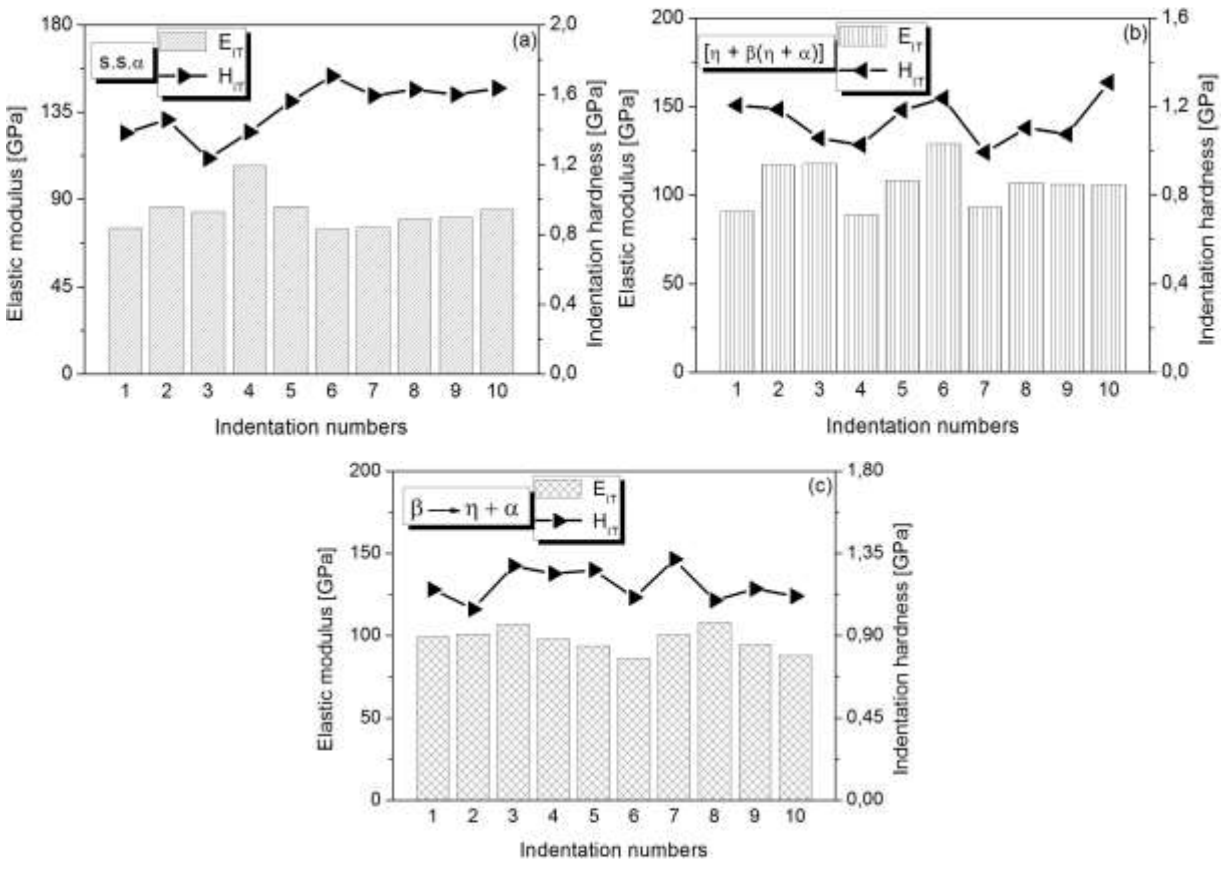

Figure 5. Variation of indentation hardness (H_{IT}) and elastic modulus (E_{IT}) for phase/constituents of Zn-22 wt. % Al alloy
(a) s. s. α; (b) eutectic [η + β(η + α)]; (c) eutectoid (β → η + α)

Figure 6 shows the mean value of the indentation hardness and elastic modulus.

In Figure 6 can be observed that the elastic modulus allow an increasing trend from α solid solution to the eutectic constituent and an decreasing trend from the eutectic to the eutectoid constituent.

Also, the indentation hardness allows a decreasing trend from the s. s. α phase to the eutectic and an increasing trend from the eutectic to the eutectoid constituent. The higher hardness of the solid solution can be explained by the intensity of the segregation phenomenon, leading to the solid solutions formation with high an aluminium content

(43.65%, Figure 3b).

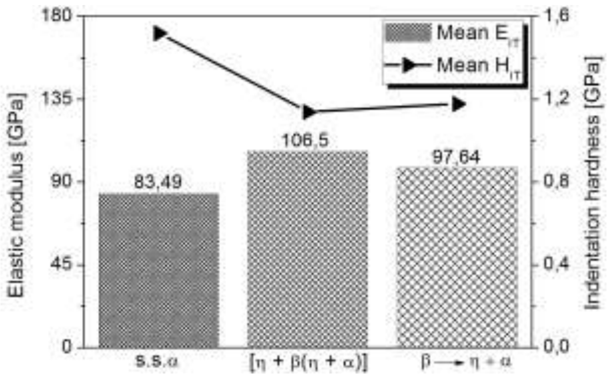

Figure 6. Mean indentation hardness and elastic modulus for phase/constituents of Zn-22 wt. % Al alloy
(a) s. s. α; (b) eutectic [η + β(η + α)];
(c) eutectoid (β → η + α)

The lower aluminium concentrations values from the eutectic and eutectoid constituents, with 12.43 and 27% Al respectively, are confirmed by the indentation hardness measurements.

4. Conclusion

The eutectoid Zn-Al alloy cast in brick moulds report significant structural deviations from the thermal equilibrium diagram.

According to the thermal equilibrium diagram, the eutectic constituent formation in the alloy structure occurs at lower concentrations of 17.2% Al.

The results of the SEM analysis for the phase and constituents reveal higher concentrations aluminium than the indicated values by the thermal equilibrium diagram.

Because of the segregation phenomena α solid solution shows higher indentations hardness values than eutectic and eutectoid constituents.

And also, because of the higher aluminium concentration, the hardness of the eutectoid is higher than that of eutectic constituent.

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