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# Fatigue induced phase transformation in extruded Zn-Al based alloy

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## Abstract

In this paper the fatigue behavior of extruded eutectoid Zn–Al based alloy is studied, presenting S–N curves and employing scanning electron microscopy and X-ray diffraction techniques to identify microstructural change and phase transformation of the extruded alloy after fatigue testing.  $\bigcirc$  1998 Elsevier Science S.A.

Keywords: Zn-Al alloy; Phase transformation; Fatigue-induced

## 1. Introduction

Eutectoid Zn–Al alloy has been known as an example of superplastic material for many years. With the addition of about 3% copper, this traditional superplastic alloy has become a potential structural material, which is able to be extruded and rolled. The fatigue behavior of this alloy has been of common interest, as most industrial alloy material failures arise from fracture after being subjected to a large number of external stress cycles [1,2].

The fatigue behavior of an extruded eutectoid Zn–Al based alloy  $(Zn_{78}Al_{22}Cu_2 \text{ in wt.\%})$  is studied in this report by subjecting specimens to a number of identical stress cycles until failure occurs. The specimens of 6 mm diameter with a 35 mm gauge length were tested for several different maximum stress levels, i.e. peak stresses of 95, 75, 50, 35, and 30 MPa with a fluctuation frequency of 0.1 Hz at 100°C. Both the number of cycles and the time to rupture the specimen at various peak stresses are listed in Table 1. The peak stresses were then plotted against the logarithm of the number of cycles to failure, i.e. S–N curves, as shown in Fig. 1.

The number of cycles to failure increased as the peak stress decreased. The fatigue limit for the extruded eutectoid Zn-Al based alloy was about 27 MPa at 100°C, below which the eutectoid alloy works safely without fatigue damage.

Scanning electron microscopy (SEM) and X-ray diffraction (XRD) techniques were applied for the identification of microstructural changes and phase transformations in the extruded eutectoid Zn–Al based alloy specimens after fatigue testing. A back-scatter electron image was used for detecting the atomic number contrast of the phases involved in the examination. XDF with nickel-filtered Cu K $\alpha$  radiation was employed.

Table 1

Number of cycles and time to rupture the specimen at various peak stresses

Stress (Mpa)	Number of cycles to rup- ture	Time to rupture (h)
95	478	1.3
75	846	2.5
50	3799	10.5
35	15780	43.8
30	35858	99.6

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Fig. 1. S–N curve for the extruded eutectoid Zn–Al based alloy at  $100^{\circ}$ C.

#### 2. Results and discussion

Shown in Fig. 2(a) are the X-ray diffractograms of the specimen before fatigue testing and various parts of the 95 MPa fatigue tested specimen: the bulk part (no plastic deformation); the neck zone; and the rupture part. After fatigue testing, there were four phases:  $\alpha$ , T' phase and two zinc-rich hcp phases,  $\eta'_{\rm E}$ and  $\eta$ , that existed in the bulk part of the specimen and appeared similar to those of the specimen before fatigue testing, as shown in Fig. 2(a-i) and (a-ii).

After 95 MPa fatigue at 100°C, decomposition of the metastable  $\eta'_{\rm E}$  phase was observed in the neck zone and the rupture part of the specimen. Upon approaching the rupture frontier, the intensity of the (0002) diffraction of the  $\eta'_{\rm E}$  phase decreased at  $2\theta =$ 36.98° (d-spacing 0.2436 nm) and at the same time the intensity of the (0002) diffraction of the final stable  $\eta$  phase increased at  $2\theta = 36.45^{\circ}$  (d-spacing 0.2465) nm), as shown in Fig. 2. Obviously the decomposition of the metastable  $\eta'_{\rm E}$  phase was related to increase in the fatigue induced stress. In the rupture part, the  $\eta'_{\rm E}$ phase decomposed almost completely during the fatigue process and left the diffraction of the (0002) crystal planes of the final stable  $\eta$  phase at  $2\theta =$ 36.45°. It has been found that a discontinuous precipitation inside the metastable  $\eta'_{\rm E}$  phase occurs in the following solid reaction:  $\eta'_{\rm E} \rightarrow \eta + \alpha + T'$  [3].

It was observed that phase transformations induced by fatigue under both high and low peak stress (95, 75, 50, 35 and 30 MPa) were essentially similar in the neck zone and the rupture part of the specimen. For comparison, the X-ray diffractograms of the extruded specimen after 50 and 30 MPa fatigue testings are shown in Fig. 2(b) and (c).

With lower peak stress (e.g. 50, 35 and 30 MPa) fatigue testing, the decomposition observed in the bulk part of the specimen was due to the low stress fatigue testing being a slow fatigue process at 100°C, i.e. a thermal-ageing process. Shown in Fig. 3 are the X-ray diffractograms of the bulk parts of the specimen for fatigue testing at 95, 50 and 30 MPa. It is observed clearly that the decomposition of the  $\eta'_{\rm E}$  phase in the bulk parts of the specimens developed with decreasing peak stress, i.e. with increasing time to rupture.

Microstructural change in the extruded alloy occurred accompanying the decomposition of the metastable  $\eta'_{\rm E}$  phase during the fatigue processes. The microstructure of the extruded eutectoid Zn-Al based alloy consisted of the decomposed  $\beta'_{s}$  phase region as matrix, the light contrast decomposed Zn-rich  $\eta'_{\rm E}$ phase (appearing as fine particles), the dark-contrast decomposed  $\alpha'_{s}$  phase particles and colony rods and the T' phase. The SEM photographs of 95 MPa peakstresses fatigue-tested specimens are shown in Fig. 4. Segmentation and fracture of the decomposed  $\alpha'_{s}$ phase particles and rods were observed in the neck zone and became serious in the rupture part of the specimens due to the increasing stress, as shown in Fig. 4(a) and (b). Precipitates were observed inside the light-contrast  $\eta'_{\rm E}$  phase in both the bulk part and the rupture part of the specimens and became more obvious in the rupture part of the specimens, as indicated by the arrow in Fig. 4(a).

Fatigue is a slow tensile-creep process under fluctuating stresses. The decomposition of the  $\eta'_{\rm E}$  phase and the microstructural change, that occurred during fatigue testing at 100°C were essentially similar to that induced by tensile creep and mechanical milling. As discussed in the previous publications [4-9], the external stress induced by tensile creep, milling and fatigue enhanced the interfacial energy, which resulted in the decomposition of the metastable  $\eta'_{\rm E}$  phase. However the decomposition of the  $\eta'_{\rm E}$  phase appeared more obvious in the rupture part of the fatigue-tested specimens than in the short-term tensile-tested specimen. As can be seen the X-ray diffactograms of various parts of the same extruded alloy after fatigue testing and tensile testing at 100°C [8], it is observed that the (0002) X-ray diffraction peak of the metastable  $\eta'_{\rm E}$ phase has shifted almost completely to that of the



Fig. 2. X-ray diffractograms of extruded eutectoid Zn-Al based alloy before and after fatigue testing under various peak stresses at 100°C: (a) 95 MPa peak stress, (i) before fatigue testing, (ii) bulk part of the tested specimen, (iii) and (iv) neck zone, (v) rupture part of the tested specimen; (b) 50 MPa peak stress, (i) bulk part of the tested specimen, (ii) and (iii) neck zone, (iv) rupture part of the tested specimen; (c) 30 MPa peak stress, (i) bulk part of the tested specimen, (iv) rupture part of the tested specimen; (c) 30 MPa peak stress, (i) bulk part of the tested specimen, (iv) rupture part of the tested specimen; (c) 30 MPa peak stress, (i) bulk part of the tested specimen, (iii) and (iii) neck zone, (iv) rupture part of the tested specimen; (c) 30 MPa peak stress, (i) bulk part of the tested specimen, (ii) and (iii) neck zone, (iv) rupture part of the tested specimen.



final stable  $\eta$  phase after fatigue processes, whilst this transformation has not yet been completed in the tensile deformation, as shown in Figs. 2 and 5, respectively [8]. Obviously the long-term fatigue process, i.e. an ageing process under a large number of fluctuating stress, promoted considerably the decomposition of the metastable  $\eta'_{\rm E}$  phase.

It was observed that there were two kinds of microstructure in the extruded eutectoid Zn-Al based alloy, i.e. mainly fine grain structure or mainly lamellar structure, depending on the extrusion temperature



Fig. 3. X-ray diffractograms of the bulk parts of the specimens after fatigue testing under peak stresses of 95, 50, 30 MPa

[10]. Under further external stress (in cases of tensile and creep deformations [5,8,9]) the lamellar structure which was derived originally from the decomposed  $\beta'_{s}$ phase spheroidized into fine grain structure in the extruded specimen (temperature of extrusion above 268°C), improving the elongation of the material [5,8,9], whilst with the extruded specimen of mainly fine grain structure (temperature of extrusion below 268°C), the further external stress imposed on the decomposed  $\alpha'_{s}$  phase particles and colony rods resulted in fracture and segmentation of the particles and colony rods, as discussed in the present work and shown in Fig. 4. Therefore the fatigue process is essentially subjected to a combination of external stress induced phase transformation and microstructural change processes, and also depends in a sensitive way on the temperature at which the fatigue process is performed. The temperature dependence of the fatigue behavior of the alloy will be an interesting subject for further investigation.





Fig. 4. SEM graphs of the 95 MPa peak-stress fatigue-tested specimen: (a) rupture part of the specimen, (b) neck zone, (c) bulk part of the specimen.

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Fig. 5. X-ray diffractograms of various parts of the extruded eutectoid Zn-Al based alloy after tensile testing at 100°C: (i) bulk part of the specimen, (ii) neck zone, (iii) rupture part of the specimen.

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