

Fatigue induced microstructural change in furnace cooled Zn-Al based alloy

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During the last few decades, a new family of monotectoid and eutectoid Zn-Al based alloys have been developed to serve as structural material in industrial applications [1, 2]. In most cases, contents of aluminum and copper have been increased in order to enhance mechanical properties of traditional Zn based alloys. Structural evolution of the Zn-Al based alloy have been studied accordingly for various thermal and thermo-mechanical processes [3–7]. In most structural alloys, fatigue occurs in their manufacturing and in subsequent services. A large amount of stress cycles imposed by external loading on the structural alloy always results in failure of the materials. Therefore, the fatigue induced microstructural changes become important for practical application of the alloys. In the present letter fatigue induced microstructural changes

and phase transformation of a furnace cooled (FC) eutectoid Zn-Al based alloy (the nominal chemical composition is Zn76Al22Cu2 in wt %) are reported.

Prior to fatigue testing, the alloy was solution treated at 350 °C for 4 days, then cooled to ambient temperature inside the furnace. The heat-treated alloy was machined into specimens of 6 mm in diameter with a 35 mm gauge length. Fatigue testing was carried out at 100 °C with a fluctuation frequency of 0.1 Hz in an Instron machine. Various peak stresses, i.e., maximum stress levels, 90, 65, 40 and 30 MPa, were applied. The logarithm number of cycles to rupture the specimens was plotted against the peak stresses, i.e., *S-N* curve, as shown in Fig. 1. It was clearly observed that the number of cycles to rupture the specimen increased when the peak stress decreased. The fatigue limit was

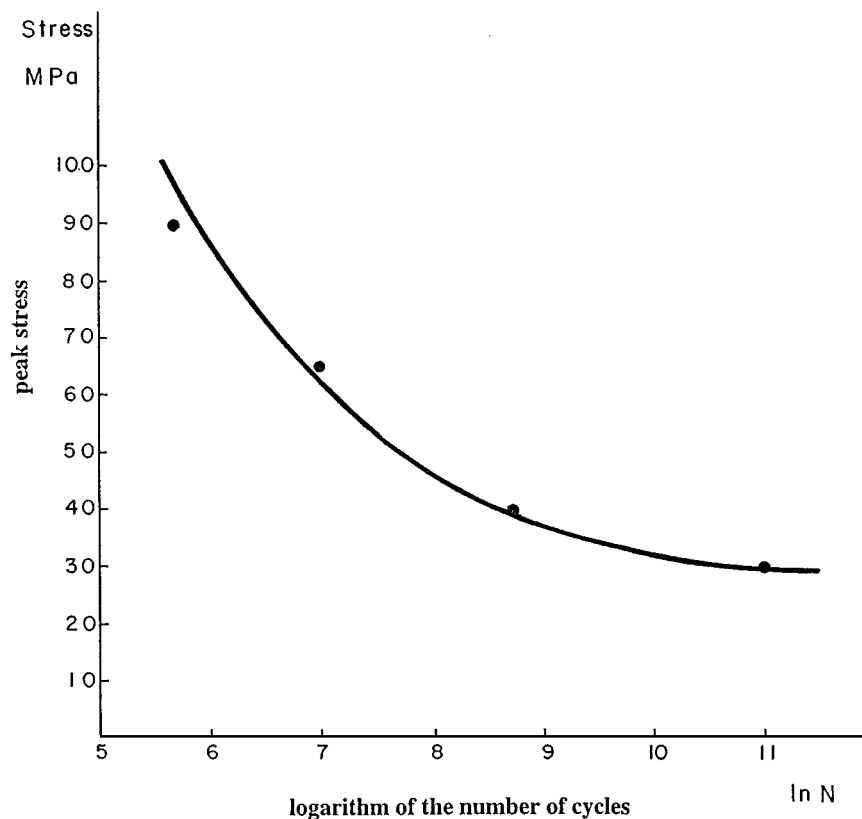


Figure 1 *S-N* curve for furnace cooled eutectoid Zn-Al based alloy at 100 °C.

about 30 MPa for the FC eutectoid Zn-Al based alloy at 100 °C. This implied that below this stress limit of 30 MPa, the FC eutectoid Zn-Al based alloy could serve as structural material at 100 °C without fatigue rupture.

The various parts of fatigue-tested specimens were examined using X-ray diffraction (XRD), scanning microscopy (SEM) and transmission electron microscopy (TEM) techniques. The XRD with nickel-filtered $\text{CuK}\alpha$ was performed on a flat polished specimen. The characteristic X-ray diffraction was collected within a range of diffraction angle 2θ from 35 to 47°, at a scanning speed of 1° min^{-1} in a Philips X-ray diffractometer. Back-scattered electron imaging was applied for detecting microstructural changes. Conventional TEM was used to produce high-resolution electron images of both bright field and dark field, and selected-area diffraction pattern (SADP).

The X-ray diffractogram of the as-FC eutectoid Zn-Al based alloy is shown in Fig. 2a for comparison [8]. Three phases (η'_{FC} , α and ε) were found in the FC alloy specimens. After fatigue testing, the η'_{FC} phase had partially decomposed to η'_T phase. This was clearly demonstrated in the XRD of the bulk part of the fatigue-tested specimen with different peak stresses: 65, 40 and

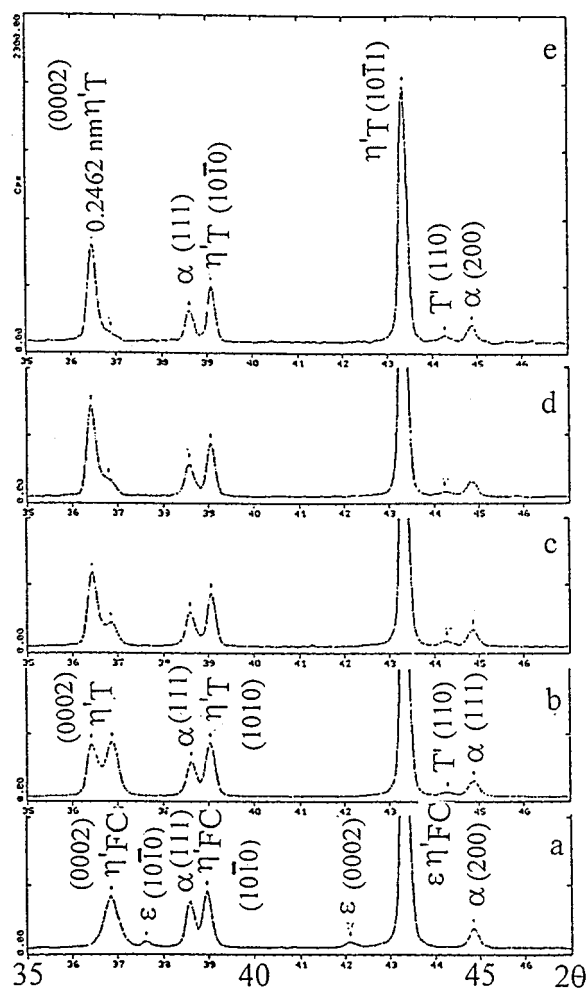


Figure 2 X-ray diffractograms of the as furnace cooled eutectoid Zn-Al based alloy specimen and the various parts of 65 MPa peak stress fatigue tested specimen: (a) as FC state; (b) bulk part; (c) and (d) neck zone; and (e) rupture part of the fatigue-tested specimen.

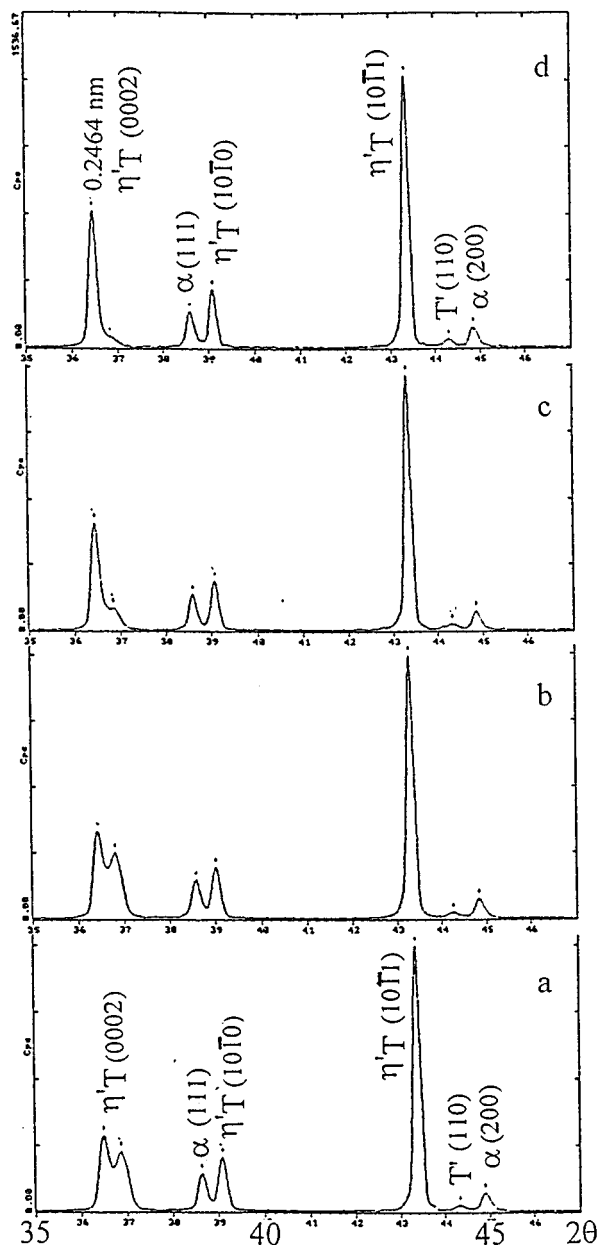


Figure 3 X-ray diffractograms of various parts of the fatigue-tested specimen with peak stress of 40 MPa: (a) bulk part; (b) neck zone; and (c) rupture part.

30 MPa, as shown in Figs 2b, 3a and 4a respectively. The decomposition of the η'_{FC} phase was characterized by a shifting of the (0002) η'_{FC} phase diffraction peak to a lower 2θ angle, accordingly d-spacing was increased from 0.2437 nm of the (0002) η'_{FC} phase to about 0.2465 nm of the (0002) η'_T . It was reported that the η'_{FC} phase decomposed in a discontinuous manner: $\eta'_{\text{FC}} \rightarrow \eta'_T + \alpha + \varepsilon$ during ageing at 100 and 170 °C [8]. It was also found that the decomposition of the η'_{FC} phase in the bulk part of the fatigue-tested specimens occurred even at the lower peak stress of 30 MPa, as shown in Fig. 5. It was because under the lower peak stress, e.g., 30 MPa the fatigue testing had lasted for a longer period of ageing at 100 °C than that under a higher peak stress.

Another phase transformation was observed during fatigue testing. Diffraction of (10T0) and (0002) crystal

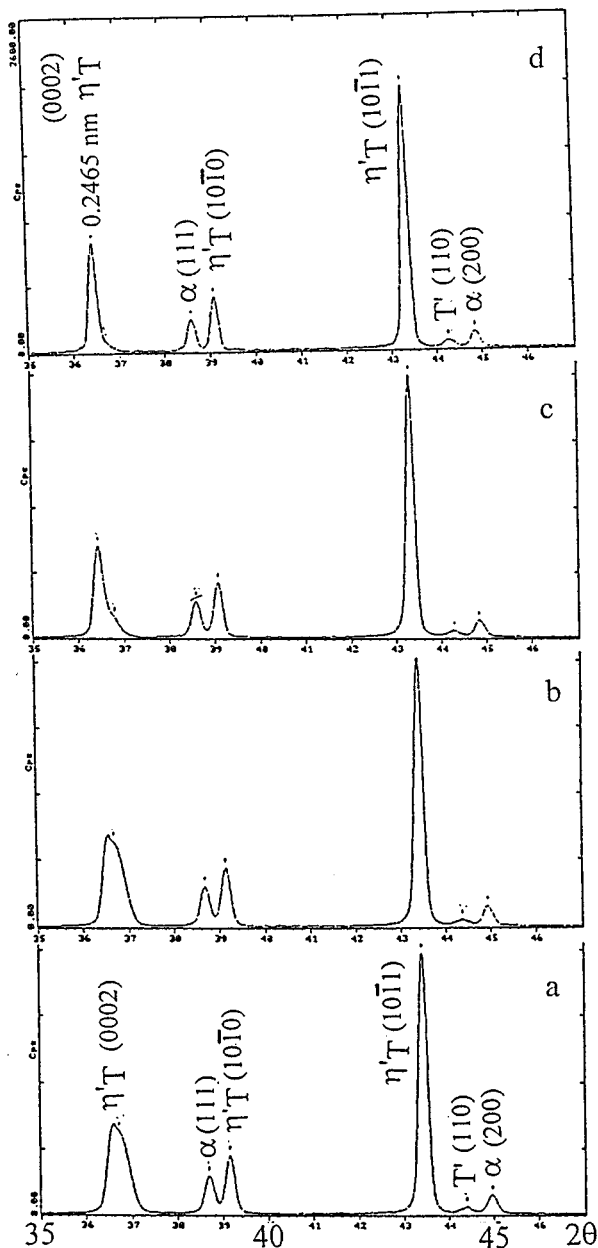


Figure 4 X-ray diffractograms of various parts of the fatigue-tested specimen with peak stress of 30 MPa: (a) bulk part; (b) neck zone; and (c) rupture part.

planes of the ε phase had greatly reduced in intensity, while a diffraction peak of (433) crystal planes of T' phase emerged, as shown in Figs 2b, 3a and 4a. It was well recognized that the four-phase transformation, $\alpha + \varepsilon \rightarrow T' + \eta$, can occur [3–10].

During fatigue testing the specimens were plastically deformed. The locally concentrated strain resulted in necking which accelerated the decomposition of the η'_{FC} phase. The diffraction of the (0002) η'_{FC} phase was reduced in intensity when the distance from the rupture frontier was decreased, as shown in Figs 2b–e, 3a–d and 4a–d. In the rupture part of the fatigue-tested specimen, the d-spacing of the (0002) η'_T phase was about 0.2465 nm, which was very close to that of the (0002) final stable η phase: 0.2466 nm. It was because the external tensile stress repeatedly imposed on the specimen and speeded up the decomposition of the η'_{FC} phase.

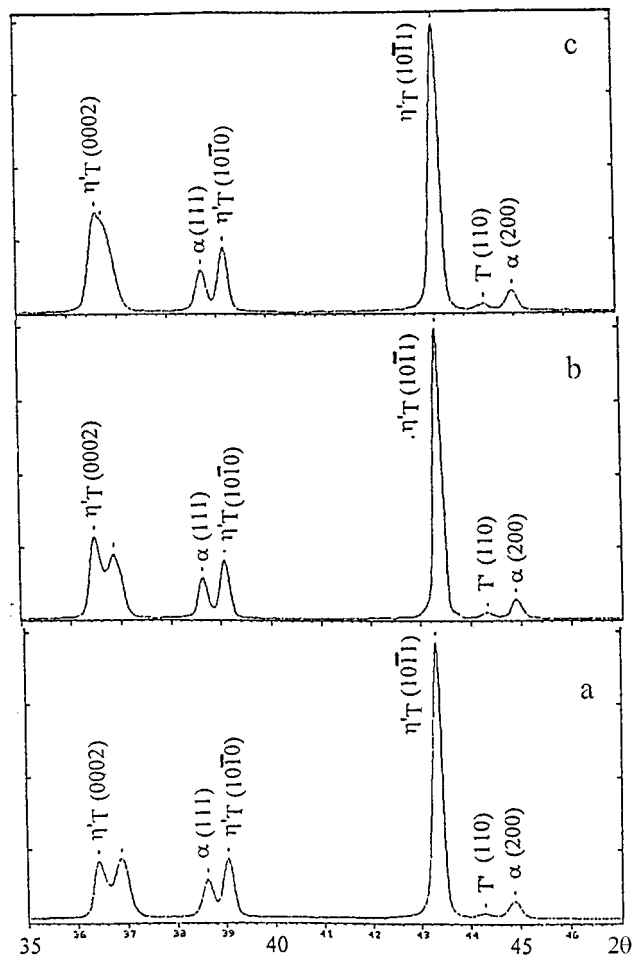


Figure 5 X-ray diffractograms of bulk parts of the fatigue-tested specimen with peak stresses of 64 (a), 40 (b) and 30 MPa (c).

Moreover, it was found that the intensity of the X-ray diffraction (433) T' phase was increased approaching the rupture part of the specimens. This implied that the four-phase transformation, $\alpha + \varepsilon \rightarrow T' + \eta$, was enhanced as the local strain was increased.

These two kinds of phase transformation have been observed previously in the specimens of cast, extruded and furnace cooled eutectoid Zn-Al based alloy in various thermal and thermo-mechanical processes, such as tensile and creep etc. [5, 6]. However the decomposition of the η'_{FC} phase and the four-phase transformation were accelerated considerably in the long term fatigue tests.

The original lamellar structure of the as FC eutectoid Zn-Al based alloy changed to a particulate structure after a large number of fluctuating tensile cycles. Shown in Fig. 6 are the back-scattered SEM photos of the 65 MPa peak stress fatigue-tested specimen. With increasing of the strain, the decomposed α'_s phase rods in the bulk part of the specimen were fractured in the neck zone and the rupture part of the specimen, as shown in Fig. 6b–c.

The TEM examination of the fatigue-tested specimens showed clearly decomposition of the Al-rich fcc α phase in both the bulk part and the rupture part of the specimens. According to the identification of the SADP, the precipitates inside the α phase were hcp η

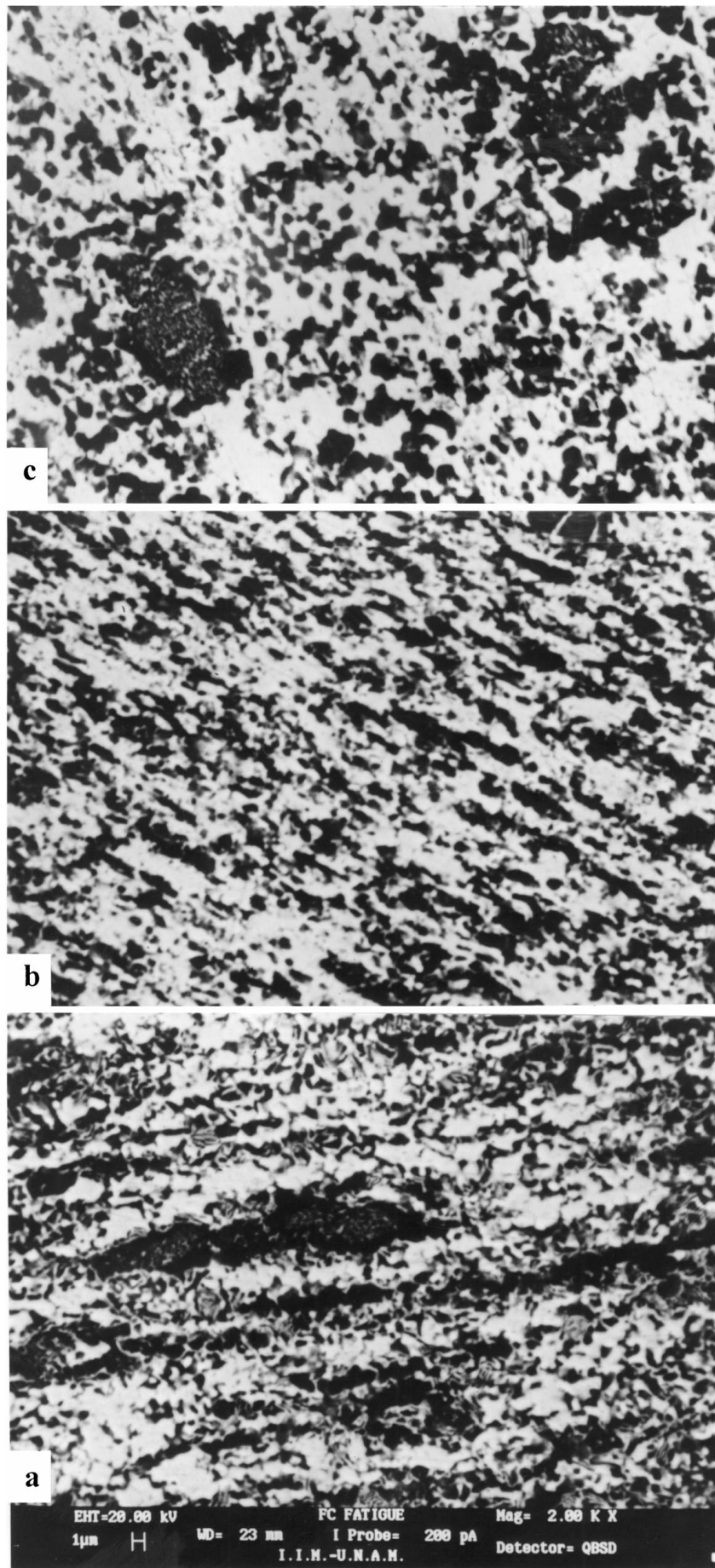


Figure 6 Back scattered SEM photos of various parts of 65 MPa peak stress fatigue-tested specimen: (a) bulk part; (b) neck zone; and (c) rupture part.

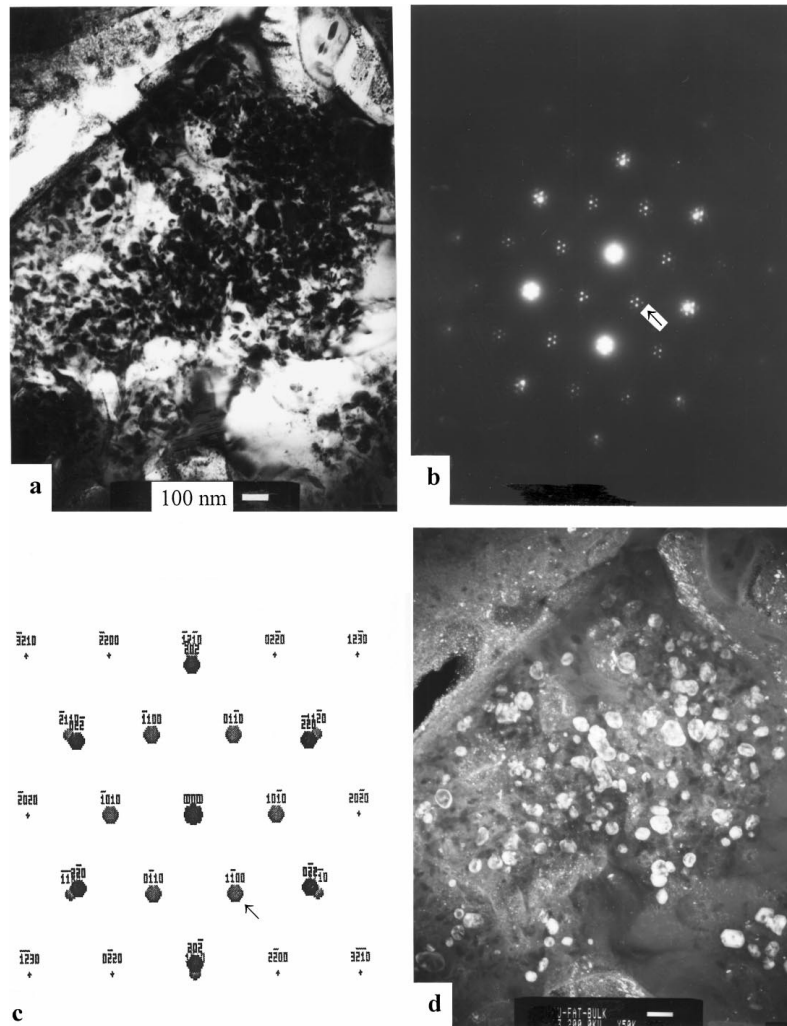


Figure 7 TEM bright field (a), the SADP from [0001] zone of the precipitate η phase inside the α phase (b) together with the indexing of the diffraction pattern (c), and the dark field using {1 1 00} (d) of the bulk part of the 65 MPa peak stress fatigue-tested specimen.

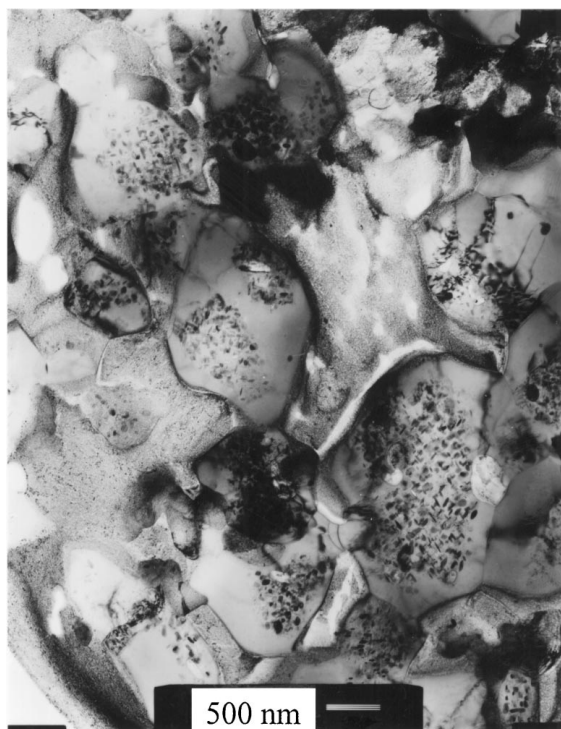
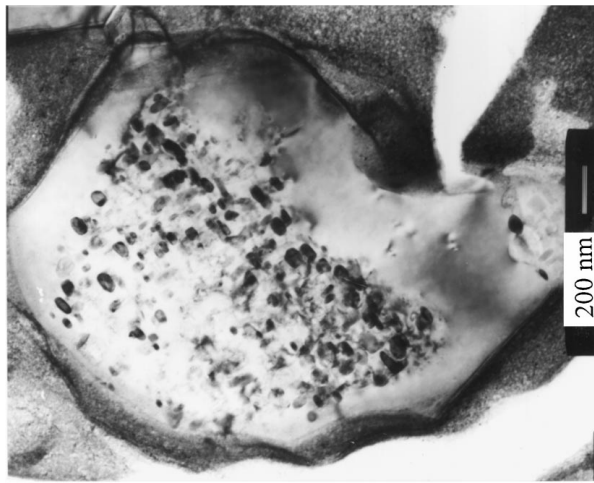
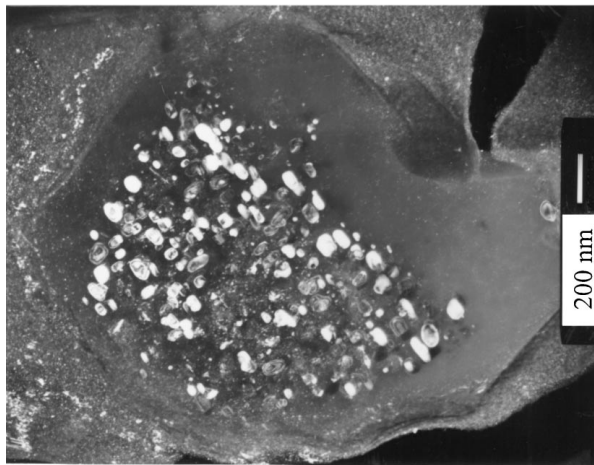


Figure 8 Transmission electron micrograph of the rupture part of the 65 MPa peak stress fatigue-tested specimen.

phase. The TEM bright field of the bulk part of the specimen is shown in Fig. 7a. Both the corresponding electron diffraction patterns were identified from [1 1 1] of the α matrix and [0001] of the η precipitates, as shown in Fig. 7b together with indexing of the diffraction pattern in Fig. 7c. In the TEM dark field, the light image of the η precipitates inside the α phase was diffracted from {1100}, as shown in Fig. 7d. The same hcp η phase precipitates inside the α phase were detected in the rupture part of the specimen using TEM, as shown in Fig. 8. Shown in Fig. 9a and b are the TEM bright field and the dark field of the rupture part of the fatigue-tested FC eutectoid Zn-Al based alloy respectively. Compared with the TEM examination of the as FC eutectoid Zn-Al based alloy [8], it was found that the precipitation of the Zn-rich η phase inside the Al-rich α phase was considerably developed in the fatigue-tested alloy specimens.



(a)



(b)

Figure 9 Transmission electron micrograph of the rupture part of the 65 MPa peak stress fatigue-tested specimen: (a) the bright field; (b) the dark field.

Acknowledgments

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