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Mechanical Milling Induced Preferred Orientation in a Zn-Al Based Alloy

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Abstract

Mechanical milling resulted in the preferred orientation of the Zn-rich metastable hcp ε, η'_{FC} and η'_{T} phases in the furnace cooled (FC) eutectoid Zn-Al based alloy filings, after hot rolling or elevated temperature tensile-creep. The milling induced preferred orientation for the Zn-rich ε phase, and the η'_{FC} and η'_{T} phases in the eutectoid alloy were at (0002) and $10\bar{1}0$ crystal planes respectively, which reverted the original, 0 h milling state orientation as the directional external stress was destroyed during further milling.

1.0 Introduction

Many physical properties vary in magnitude with direction in crystals. The crystal orientation is strongly related to microstructure and the stress imposed on the alloy material. Most deformation processes tend to induce anisotropy by virtue of the intrinsic crystallographic nature of deformation. Therefore, the stress induced preferred orientation is of importance for the technological application of alloy materials.

Mechanical milling imposes a resultant external stress of compression, impact and grinding on materials. No detailed information has been previously reported on the preferred orientation of Zn-Al based alloys under milling condition. The purpose of the present work is to investigate the milling induced preferred orientation in a furnace cooled eutectoid Zn-Al based alloy and compare these resulted with previously studied rolling and tensile creep induced preferred orientation in the alloy.

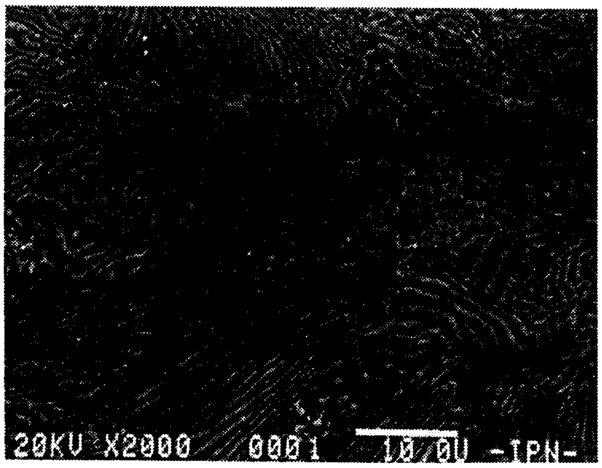
2.0 Experimental

Ingots of an eutectoid Zn-Al based alloy, Zn76Al22Cu2 (wt.%), were solution treated at 350°C for 4 days, then furnace cooled (FC) to room temperature. Alloy filings were produced from the FC eutectoid alloy with water cooling. The alloy filings were milled at room temperature in a stainless steel vial under argon atmosphere. The grinding medium was 12.7 mm diameter stainless steel balls and methanol solution was added as surfactant agent. The temperature of the vial remained near room temperature. A ball to alloy filings weight ratio of 36:1 was selected for the milling. Small quantities of the alloy filings were withdrawn from the vials at regular time intervals to follow the progress of milling via x-ray diffraction (XRD) and scanning electron microscopy (SEM) techniques. The normal metallographic procedures, such as optical, SEM and XRD examinations described previously, (1,2) were conducted. The powder filings were placed in a specimen holder with the same exposure area for x-ray diffraction in order to keep the diffraction intensity of phases quantitatively comparable.

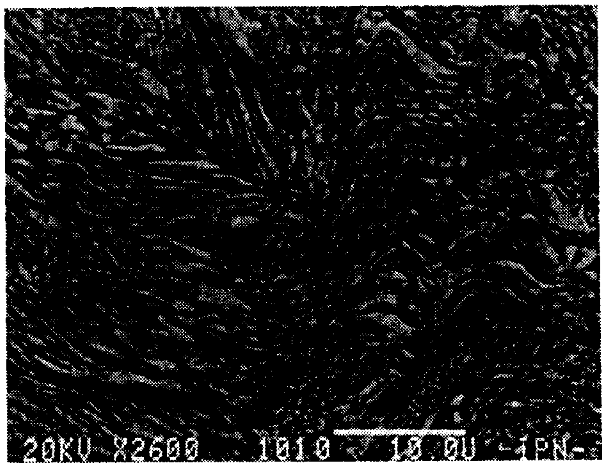
3.0 Results and Discussion

In the previous work, external stress, such as tensile and tensile-creep, induced microstructural change and phase transformation were investigated in detail (1-4). During mechanical milling, similar microstructural changes and phase transformations occurred. The microstructure of the furnace cooled eutectoid Zn-Al alloy filings changed from lamellar structure to fine grain structure of 0.2-0.4 μm in diameter during milling at room temperature. The microstructural change became obvious during prolonged milling, as shown in Figure 1 (5). At the same time the mechanical milling accelerated the decomposition of the metastable η'T and the four phase transformation, $\alpha + \varepsilon \rightarrow T' + \eta$ considerably (5-10).

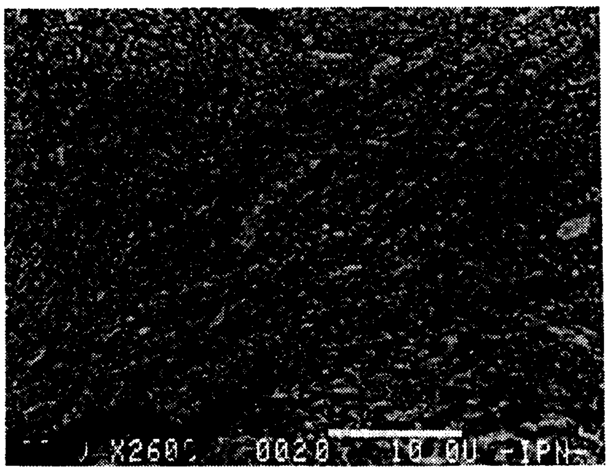
Accompanying the decomposition of the metastable η'_{FC} phase to η'_T phase and the microstructural change during 20 h milling, preferred orientations were observed. As shown in Figure 2, the x-ray diffraction peaks of (0002) crystal planes of the ε phase at 2θ 42.2° and $(10\bar{1}0)$ crystal planes of the metastable η'_{FC} and η'_T phases at 2θ 39.1° increased in intensity after 1 h milling, while both the diffraction peaks of the $(10\bar{1}0)$ crystal planes of the ε phase at 2θ 37.7° and the (0002) crystal planes of the metastable η'_{FC} and η'_T phases at 2θ 36.9° and 36.6° respectively decreased in diffraction intensity. After 5 h milling, the x-ray diffractions of the (0002) crystal planes of the ε phase and the $(10\bar{1}0)$ crystal planes of the metastable η'_{FC} and η'_T phases increased to a maximum. The discontinuous decom-position of the metastable η'_{FC} to η'_T phase was completed after 10 h milling. The diffraction peak of the (0002) crystal plane of the η'_{FC} phase vanished from the



(a)



(b)



(c)

Figure 1: Microstructural change induced by mechanical milling (a) after 1 h milling, lamellar structure, (b) after 10 h milling, partial lamellar structure, partial fine grain structure, and (c) after 20 h milling, fine grain structure.

x-ray diffractograms, and did not affect the (0002) diffraction peak of the η'_T phase at 2θ 36.5°, as shown in Figure 2.

After 20 h milling, the x-ray diffraction of the (0002) crystal planes of the ε phase and the (0002) crystal planes of the η'_T phase returned to their original, 0 h milling relative diffraction intensities. This relative interchanging of the intensities of the x-ray diffraction reflected the co-existance of preferred orientations of both the Zn-rich ε phase, and the η'_{FC} and η'_T phases during 20 h milling, as shown in Figure 2.

It was not difficult to understand the interchanging of the diffraction intensities after observing the plastic deformation resulted by mechanical milling. Shown in Figure 3 is the plastic deformation evolution of the alloy filings during 20 h milling. The alloy filings were flattened during early stage of milling, e.g. 1 h, 5 h and 10 h millings, due to a directional impact, grinding and compression. This directional external stress resulted in the relative interchanging of the x-ray diffraction from the Zn-rich ε phase, and the η'_{FC} and η'_T phases, in other words, the preferred orientation in the Zn-rich ε phase, and the η'_{FC} and η'_T phases. After 20 h milling, the filings had changed from the previous flattened forms to intergranular fine particles, i.e. the directional external stress was eliminated, thus the preferred orientation disappeared and the x-ray diffraction returned to their original, 0 h milling relative diffraction intensities. It was clear to see that the milling induced preferred orientations for the Zn-rich ε and the metastable η'_T phases were at the (0002) and $(10\bar{1}0)$ crystal planes respectively.

A similar preferred orientation was observed in the rolled eutectoid Zn-Al based alloy. The same eutectoid Zn-Al based alloy was solution treated at 350°C for 4 days, then quenched into cold water. The solution treated and quenched alloy material was rolled to different reductions after heating up to 250°C. Shown in Figure 4 are the x-ray diffractograms of the specimens after rolling to various reduction (11). It was observed that the x-ray diffraction of the $(10\bar{1}0)$ crystal planes of the ε phase decreased in intensity during rolling, which was accompanied by an increase in diffraction intensity of the (0002) crystal planes of the ε phase. The x-ray diffraction of the (0002) crystal planes of the metastable η'_T phase decreased in intensity after rolling to 90% reduction, and accordingly the x-ray diffraction of the $(10\bar{1}1)$ of the metastable η'_T phases increased considerably in intensity. Apparently the rolling, a mechanical process with one direction compression, resulted in the preferred orientation of the Zn-rich ε and the metastable η'_T phases. Both milling and hot rolling resulted in a similar preferred orientation of the ε phase in the eutectoid Zn-Al based alloy.

It was interesting to find that the preferred orientation of the metastable Zn-rich η'_T phase in the milled alloy specimen was not as clear as that in the rolled alloy specimen. This was not difficult to understand from the deformation evolution of the alloy specimen. As shown in Figure 4, accompanying a pronounced increase in diffraction intensity of the $(10\bar{1}1)$ crystal planes of the metastable η_T phase, the diffraction intensity of the

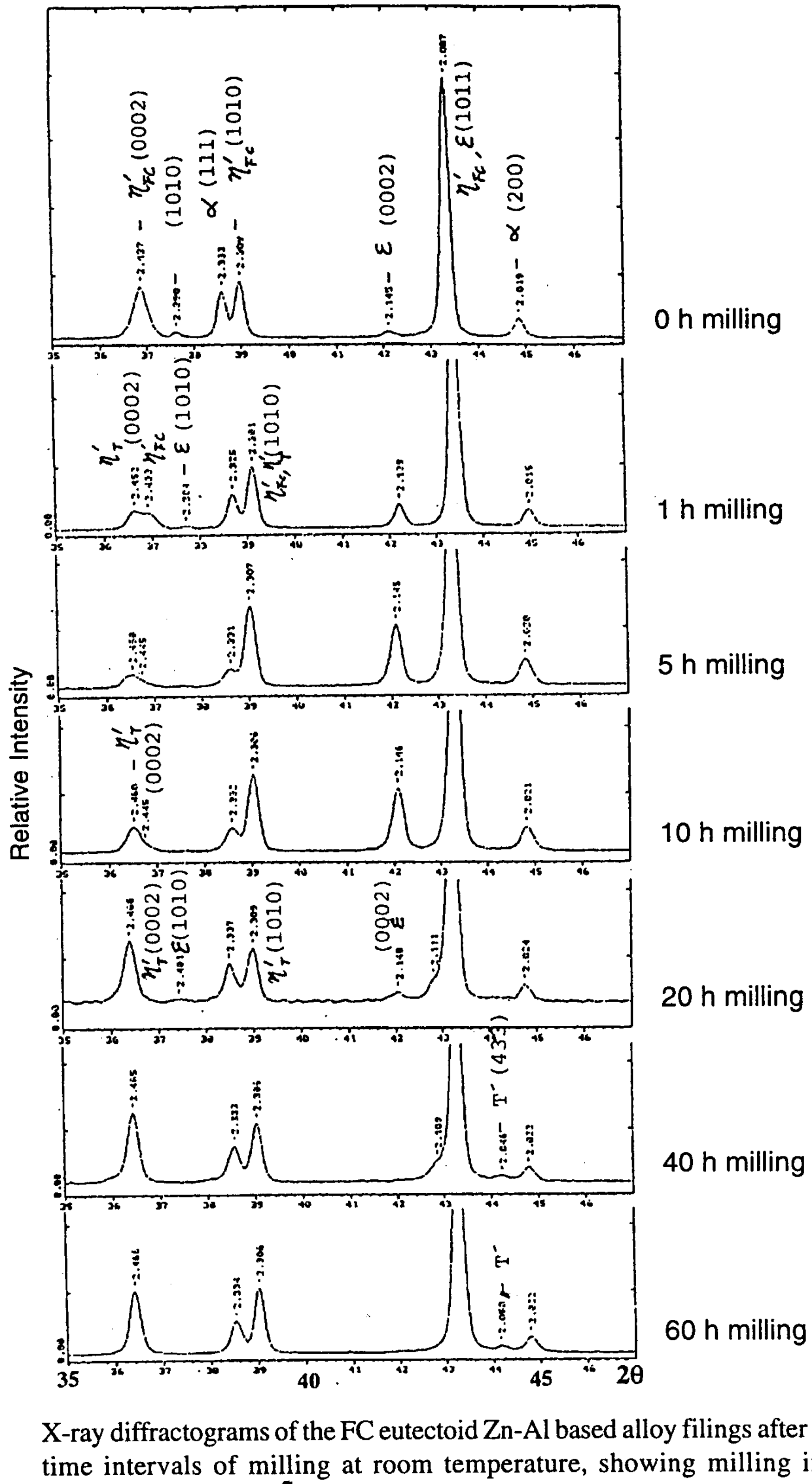
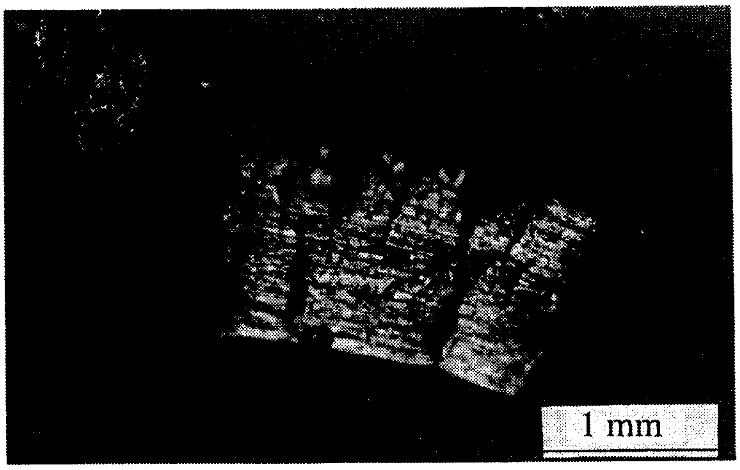
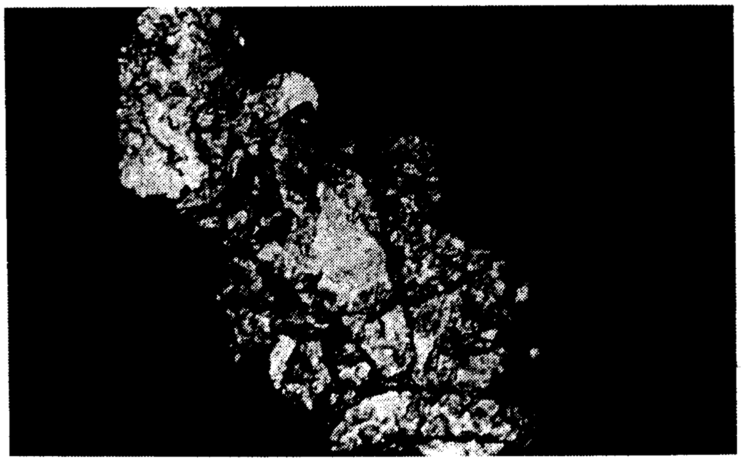


Figure 2: X-ray diffractograms of the FC eutectoid Zn-Al based alloy filings after various time intervals of milling at room temperature, showing milling induced preferred orientation.[3]



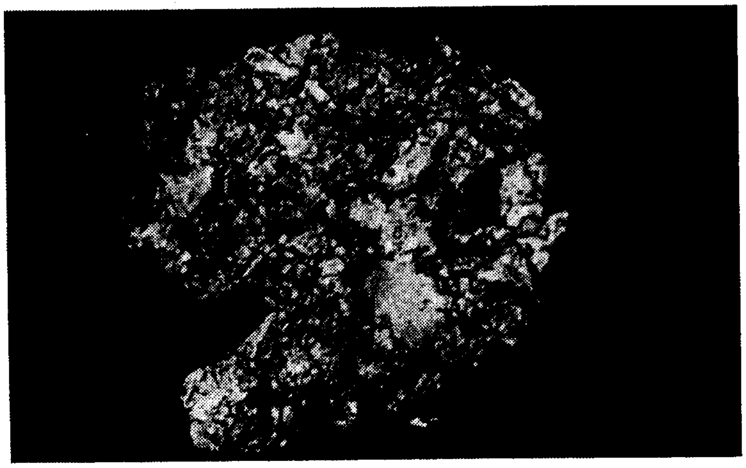
0 h milling

1 h milling



5 h milling

Figure 3: Deformation evolution of the FC eutectoid Zn-Al based alloy filings during milling. See Figure 3 continued in next page.


10 h milling


20 h milling

Figure 3: Continued.

(0002) crystal planes of the η'_T phase decreased apparently in intensity after rolling to 90% reduction, but not at lower reductions. This meant that the preferred orientation of the metastable η'_T phase occurred after a deep plastic deformation. However during prolonged milling, e.g. 20 h milling, the directional external stress was destroyed as the flattened particles were ground into fine particles, and the preferred orientation returned to the 0 h milling original state. In other words, there was not a sufficient directional external stress during prolonged mechanical milling to produce the pronounced preferred orientation at the $(10\bar{1}1)$ crystal planes of the metastable η'_T phase.

Shown in Figure 5 are x-ray diffractograms from various parts of the extruded - creep tested specimen of the eutectoid Zn-Al based alloy tested under uniaxial tension (12). Compared to the bulk of the creep tested specimen, the

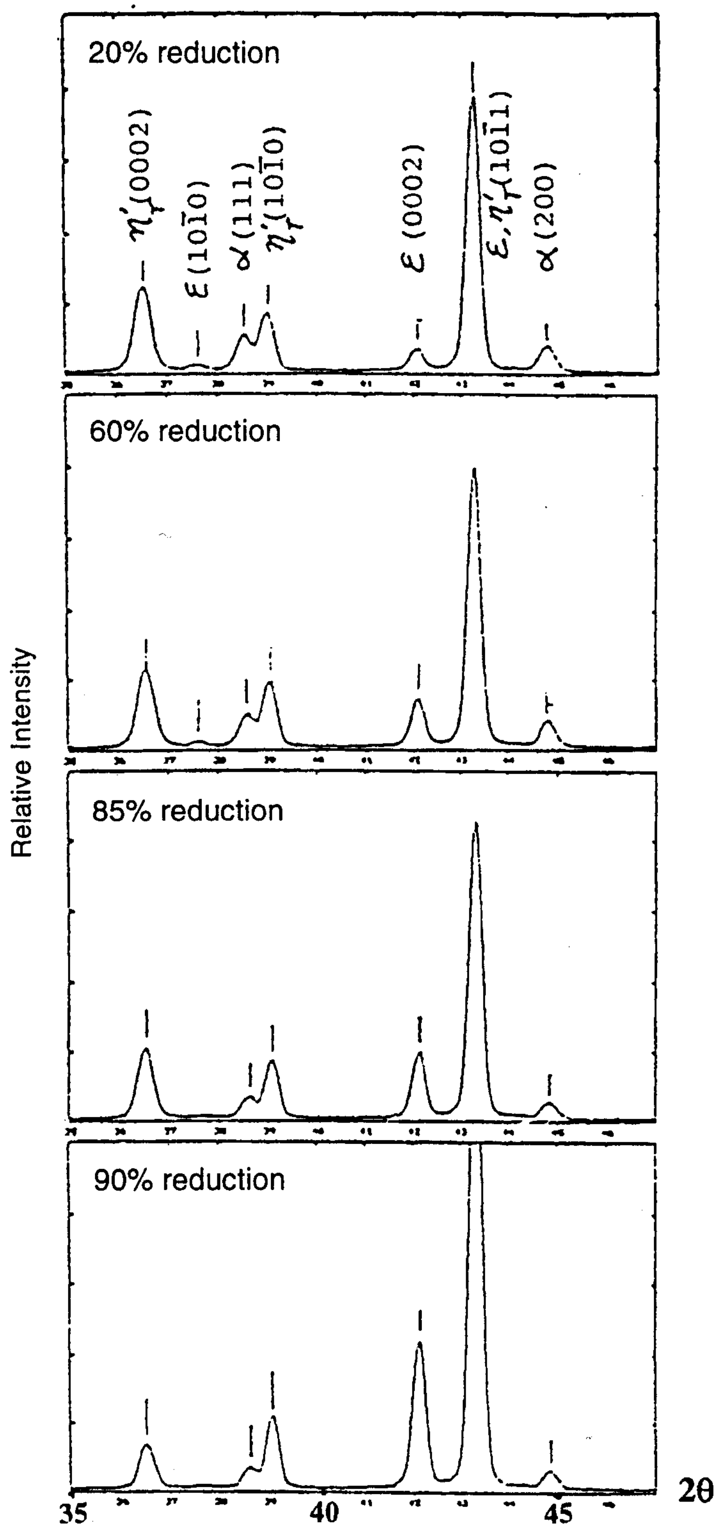


Figure 4: X-ray diffractograms of the solution treated-quenched eutectoid Zn-Al based alloy after various reduction rolling at 250°C, showing the rolling induced the preferred orientation.

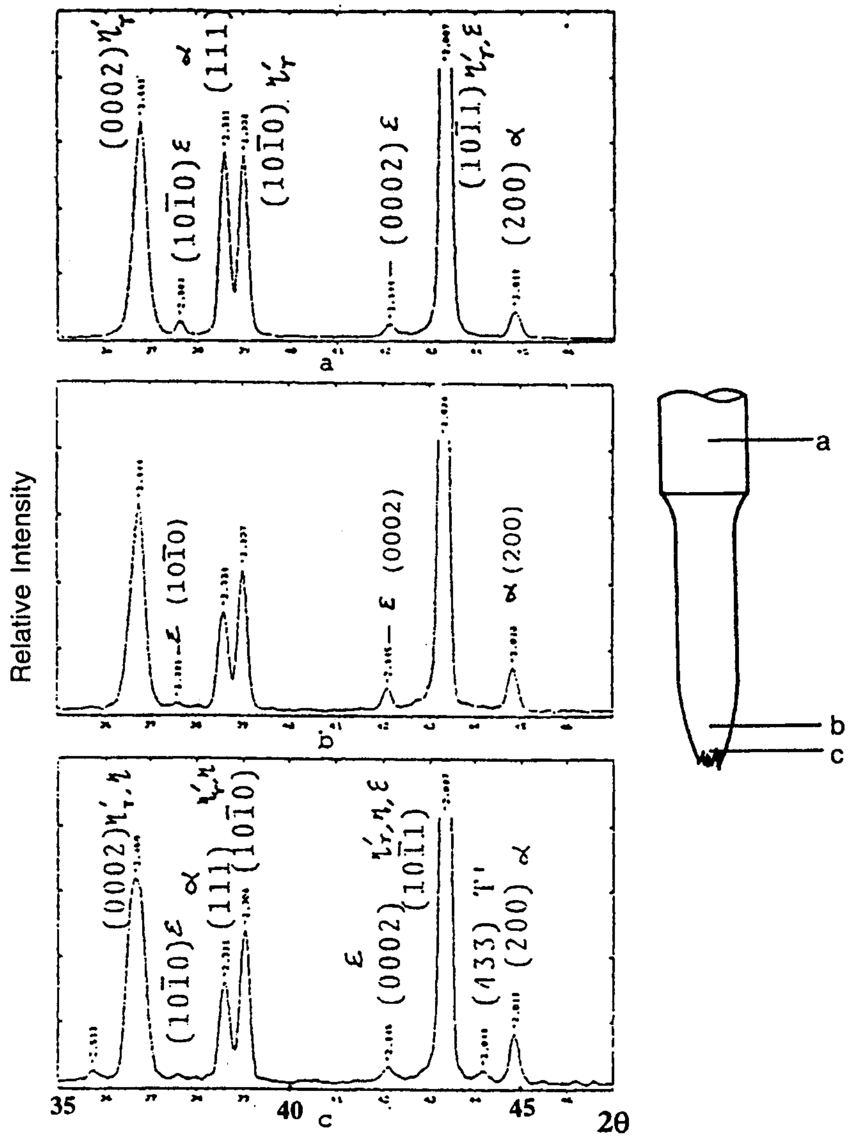


Figure 5: X-ray diffractograms of various parts of the specimen after creep test. (a) bulk part of the specimen, (b) neck zone of the specimen, (c) rupture part of the specimen.

x-ray diffraction intensity of the (0002) crystal planes of the ε phase increased in the neck zone of the creep tested specimen, which accompanied the decrease in diffraction intensity of the $(10\bar{1}0)$ crystal planes of the ε phase, as shown in Figures 5a and 5b. Obviously the creep induced preferred orientations for the Zn-rich ε phase was at the (0002) crystal planes. As distance from the rupture part of the specimen decreased, the more concentrated directional stress resulted in the four phase transformation, $\alpha + \varepsilon \rightarrow T' + \eta$, and both diffraction peaks of the (0002) and $(10\bar{1}0)$ crystal planes of the ε phase decreased in diffraction intensity and the diffraction peak of T' phase formed, as shown in Figure 5c.

4.0 Conclusions

1. Mechanical milling resulted in the preferred orientation of the Zn-rich metastable hcp ε, η'_{FC} and η'_{T} phases in the FC eutectoid Zn-Al based alloy, which was observed in the eutectoid Zn-Al based alloy after hot rolling or elevated temperaure tensile-creep. The preferred orientation reverted to the original, 0 h milling state orientation as the directional external stress was destroyed during further milling.
2. The milling induced preferred orientation of the Zn-rich ε and the metastable η'_{FC} and η'_{T} phases in the furnace cooled eutectoid Zn-Al based alloy were at the (0002) and $(10\bar{1}0)$ crystal planes respectively.

5.0 References

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