

Low Cycle Fatigue Behavior of a T6-treated Mg-Nd alloy Tested at an Elevated Temperature



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Abstract

The cyclic stress amplitude of the T6-treated Mg-3.74wt%Nd-0.59wt%Zr alloy tested at RT (room temperature) shows first increases and subsequently decreases. In contrast, the alloy tested at 150°C shows cyclic constant followed cyclic softening during fatigue. Both $\langle a \rangle$ and $\langle c \rangle$ or $\langle c + a \rangle$ dislocations-slip and twinning are the main deformation modes of the Mg-Nd alloy under high temperature fatigue. The shearing of the dislocations on the β_2 and β_1 precipitates or twins boundaries reduced the hindrance of the precipitates relative dislocations and caused the change of the precipitates, which leads to the micro softening. Different from RT fatigue, a large number of incoherent twin boundaries and cross-slip bands were formed in the microstructure of the alloy during high temperature fatigue.

Keywords: Fatigue deformation behavior; Incoherent twin boundaries; High temperature; Dislocations-slip; Cast Mg alloy

Introduction

Fatigue properties of cast Mg alloys is correlated with the defects and microstructural characteristics, which are strongly dependent on casting methods [1,2], heat treatment conditions [3,4], alloy compositions [5] as well as test temperatures [6]. It has been reported that the Mg-Nd alloys have demonstrated excellent comprehensive mechanical properties which are good for engine block applications [7,8]. As an example, our previous works pointed out that the T6-treated (540°C × 8h + 200°C × 14h) Mg-3Nd-0.2Zn-1Zr (NZ30K) alloy tested at RT (room temperature) exhibits a trend of first work hardening and then cyclic softening during low cycle fatigue (LCF) [9,10]. It is widely known that the stress response behavior of the metallic materials shows a strong response to movement characteristics of the dislocations and the evolution of the microstructure [11].

Test temperature influences significantly the low cycle fatigue behavior of the metallic materials. Benaarbia et al. [12] pointed out that the MarBN steels tested at elevated temperatures show continuous cyclic softening and stress relaxation during the

low cycle fatigue process. Zhang et al. [13] suggested that the cyclic softening is determined mainly on the laths and dynamic recrystallization at RT and grain rotation at elevated temperature, respectively. For a laser powder bead fused AlSi10Mg alloy [14], the cyclic responses also exhibit cyclic softening and decreased stress relaxation and energy dissipated for each cycle. Rae et al. [15] suggested that for the 12%Cr turbine steel, the mutual annihilation of the dislocations and rearrangement of the residual dislocation in the low energy structures leads to the softening behaviour. The dislocations-slip, grain boundary sliding and coarsening of β_2 precipitates cause the cyclic softening of the Mg-10Gd-2.0Zn-0.46Zr alloy during fatigue at 300°C [16]. This work aims to study the deformation behavior and microstructure evolution of the T6-treated Mg-3.74wt%Nd-0.59wt%Zr alloy during low cycle fatigue at 150°C, which shall provide technical support to the application of Mg-Nd-based alloys at elevated temperatures. The tensile properties and fatigue behavior for the T6-treated alloy tested at RT were also included as a comparative reference.

Experimental Procedure

The shape and size of the castings and casting process for the Mg-3.74wt%Nd-0.59wt%Zr alloy prepared by gravity permanent mold casting process is referred to Ref. [9]. The tensile samples (diameter of 6mm and gauge length of 30mm) and fatigue samples (diameter of 6.25mm and gauge length of 12.5mm) were machined from the T6-treated ($550^{\circ}\text{C} \times 7\text{h} + 200^{\circ}\text{C} \times 18\text{h}$) castings. The tensile and fatigue specimens were heated to 150°C and kept for 30 min prior to testing. The tensile tests were performed on an Instron tensile machine using a strain rate of $6.67 \times 10^{-4} \text{ s}^{-1}$. Fatigue tests were performed on an Instron fatigue machine with a split resistance furnace (at $150 \pm 3^{\circ}\text{C}$) under the strain amplitudes of 0.3, 0.4 and 0.6% until the specimens runout after 104 cycles. At the strain amplitude of 0.4%, the fatigue tests of the alloy tested at 150°C were terminated after 100, 300, 4000 and 9300 cycles to investigate the precipitates, dislocations and twins using JEM-2100 TEM and FEI TECNAL G2 20 high resolution projection electron microscope. The samples for TEM analysis were electropolished by double spraying at the temperature of -25°C , working voltage

of 45V and electrical current of 0.2A.

Results and Discussion

Microstructure

The typical microstructure of the T6-treated Mg-3.74wt%Nd-0.59wt%Zr alloy (average grain size of $67\mu\text{m}$) is shown in figure 1a. The result of EBSD analysis in figure 1b indicates that the orientation of grains varies randomly in the cast Mg-Nd alloy. Figure 1c shows the typical morphology of the precipitates in the T6-treated alloy ($B = [0001]_{\alpha}$). Figure 1d shows the high-resolution bright field image of the precipitates in the T6-treated alloy. In general, the microstructure characteristics of the alloy are similar to those of the T6-treated NZ30K alloy, which consist of finer β'' precipitates (Mg_3Nd , hcp, D019, $a = 0.64\text{nm}$, $c = 0.52\text{nm}$, hexagonal prism) located on the $\{1-100\}_{\alpha}$ and $\{11-20\}_{\alpha}$ planes [11]. The β'' precipitate located on the prismatic planes has a completely coherent relationship with the matrix, which can more effectively hinder the dislocation-slip.

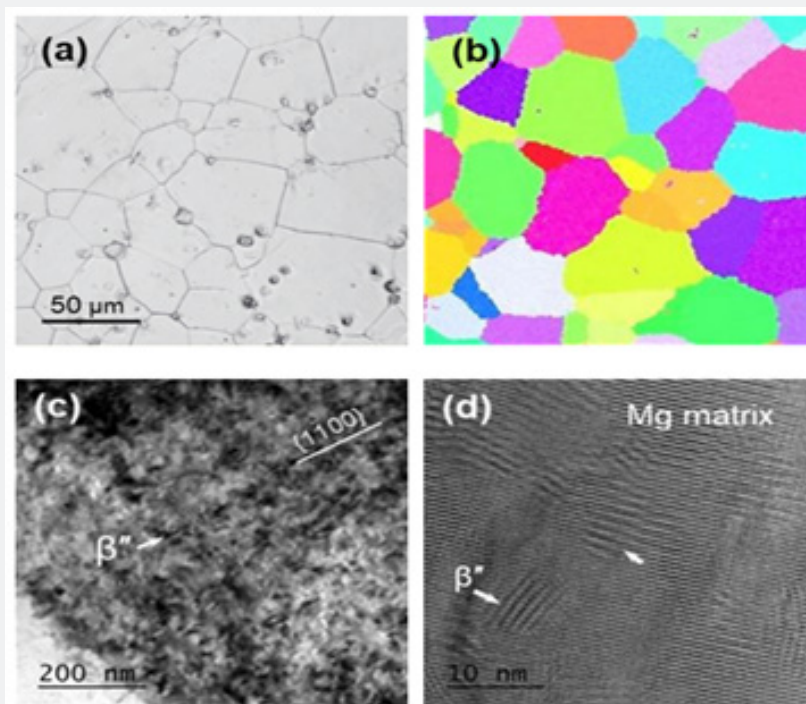


Figure 1: (a) Optical micrograph (OM) image and (b) EBSD image of the T6-treated Mg-3.74Nd-0.59Zr alloy. (c) Bright field image and (d) high-resolution image showing the precipitates in the T6-treated specimens before fatigue test ($B = [0001]_{\alpha}$).

Tensile properties

The tensile properties of the T6-treated alloy tested at RT and 150°C are summarized in table 1. Increasing testing temperature leads to a significant decrease of the yield strength (YS, 0.2% proof stress) and ultimate tensile strength (UTS) but the ductility

increases. As the test temperature is increased from RT to 150°C , the YS and UTS decrease from about 154 to 131MPa and from about 283 to 267MPa, respectively. In contrast, the elongation of the T6-treated alloy increases from about 6.3 to 12.6%. Compared with RT, the slip bands are activated easily under elevated

temperature environment, resulting in lower yield strength and ultimate tensile strength of the specimens at 150°C. In other words, more deformation mechanisms are activated and stress

concentration is released with the increase of test temperature, leading to the improvement of the plasticity.

Table 1: Tensile properties of the Mg-3.74wt%Nd-0.59wt%Zr alloy tested at RT and 150°C.

Test Temperature	Young Modulus Parameter E (N/mm ²)	YS (MPa)	UTS (MPa)	Elongation A (%)
RT	44083	154 ± 1.9	283 ± 3.8	6.3 ± 2.1
150°C	43515	131 ± 2.4	267 ± 3.2	12.6 ± 0.9

Stress amplitude and plastic strain amplitude

For the T6-treated alloy tested at 150°C and RT, the evolution curves of the cyclic stress amplitude and plastic strain amplitude with the increase of the number of cycles are shown in figure 2. With the increase of the test temperature, the cyclic stress amplitudes of the T6-treated alloy decrease at all strain amplitudes. The alloy tested at RT exhibits cyclic hardening and then cyclic softening until the fatigue failure (Figure 2a). In contrast, the counterpart tested at 150°C shows cyclic stabilization followed by cyclic softening

for the entire fatigue lifetimes. At the strain amplitude of 0.4%, the inflection point of the stress amplitude reduction of the T6-treated alloy tested at 150°C and RT occurs at about 150 and 450 cycles, respectively. In addition, the maximum stress amplitude of the alloy tested at 150°C is reduced by 20% in comparison with that of the counterpart tested at RT. Generally, for most materials, increasing test temperature decreases the fatigue resistance. The different in cyclic stress amplitude change between the samples tested at RT and 150°C is mainly attributed to different matrix strength of the specimens at both temperatures.

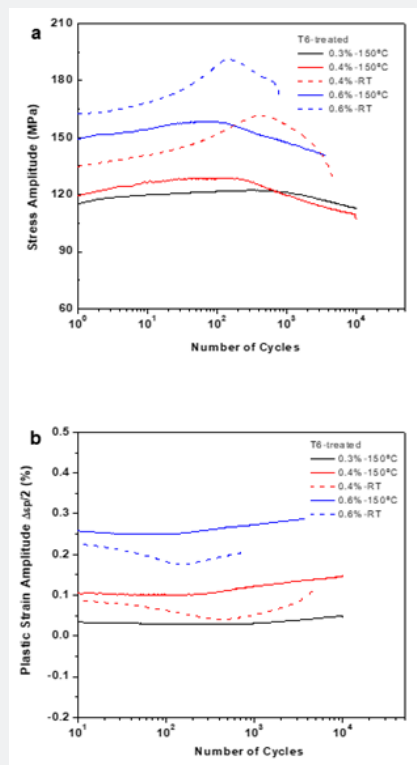


Figure 2: The evolution curves of (a) cyclic stress amplitude and (b) plastic strain amplitude for the T6-treated Mg-3.74Nd-0.59Zr alloy tested at 150°C and RT with the increase of number of cycles.

It has been reported that the cyclic stress response behavior of the T6-treated alloy tested at RT is determined mainly by the competition between micro-hardening and micro-softening. The shearing of the dislocations through the precipitates or twins and the transformation of precipitates lead to the micro softening. In contrast, work hardening is attributed to the dislocation

entanglement and the interaction of dislocations with precipitates and twin boundaries. For the T6-treated alloy tested at 150°C, the cyclic stress amplitude first keeps constant followed by a gradual decrease during fatigue. This is probably due to different hardening behavior and plastic deformation mechanism in the T6-treated alloy tested at RT and 150°C.

As shown in figure 2b, the plastic strain amplitudes of the alloy tested at 150°C are higher than those tested at RT and the same strain amplitudes. For the T6-treated samples tested at the strain amplitude of 0.2%, the plastic strain amplitude is near zero at both test temperatures. At 150°C and high strain amplitudes ($\geq 0.3\%$), the plastic strain amplitude of the alloy firstly keeps saturated and then increases slightly before final failure. In contrast, the plastic strain amplitude of the T6-treated alloy tested at RT decreases in the initial cyclic loading stage, and then increases until failure occurs. Moreover, with the increase of total strain amplitude, the plastic strain amplitude of the specimens decreases under both test temperatures.

Fatigue failure behaviour and fatigue life

As shown in figure 3a and figure 3b, the cracks are primarily

initiated from the sheared grains (RT) or oxide films (150°C) near the surface of the alloy samples, respectively. Compared with the samples tested at RT, more oxide films can be observed in the crack initiation region of the counterparts tested at 150°C. When test temperature is increased from RT to 150°C, as shown in figure 3c, both young oxide films and slip bands are also observed at the crack propagation region. This is due to the interaction between elevated temperature environment and dislocations-slip. Our previous research [9] has reported in detail the interaction mechanism between the dislocations-slip and environment for the T6-treated NZ30K alloy. Compared with the alloy tested at RT, more dimples (marked by white arrow in figure 3d) can be observed on the fracture surface of the counterpart tested at 150°C, corresponding to the increase of the ductility under high temperature environment ($A\% \geq 12\%$).

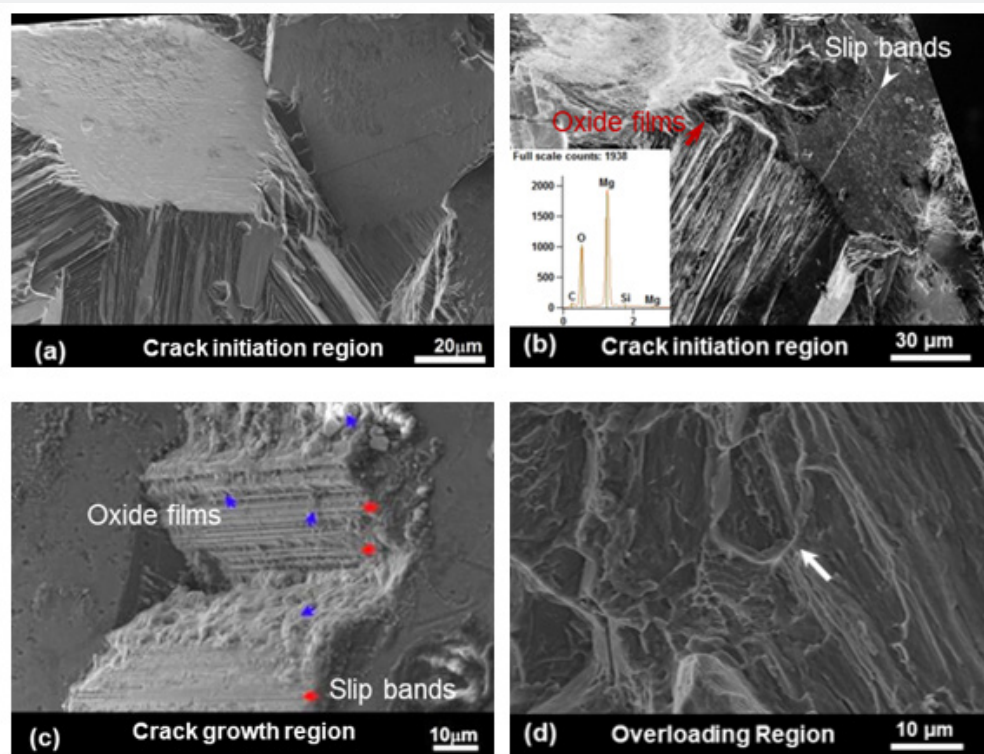


Figure 3: SEM images showing (a) grains and (b) oxide films initiating the fatigue cracks for the T6-treated alloy tested at RT (after 4524 cycles) and 150°C (after 9300 cycles) and the strain amplitude of 0.4%, respectively. (c) SEM image showing the relationship between slip bands and oxide films at the fatigue crack propagation region. (d) SEM image from the overloading region of the T6-treated samples.

Evolution of precipitates

The morphology of the precipitates of the T6-treated alloy tested at 150°C after 100, 300 and 4000 cycles are shown in figure 4. After 100 cycles, the microstructure the alloy contains many finer β_2 precipitates (Figure 4a) and some band-shaped β_1 precipitates (Mg_3Nd , fcc, $a = 0.74\text{nm}$, on the $\{1-100\}$ plate) [11] (Figure 4b). Compared with RT fatigue, more β_1 precipitates can be formed

in the samples tested at 150°C after 100 cycles. After 300 cycles, as shown in figure 4c, the part of the β_2 precipitates transformed into the β_1 precipitates. There are massive β_1 precipitates (length of 150-1000nm, width of 100-300nm) after 4000 cycles (Figure 4d). The stress response behavior of the alloy is related to the transformation of the precipitates during the low cycle fatigue process. Our previous research [17] suggested that for the T6-treated NZ30K alloy tested at RT, the interaction between shearing

of dislocations through precipitates and dynamic evolution of precipitates lead to the cyclic softening [18-20]. During plastic deformation, the precipitates hinder the movement of the dislocations [21], which can accelerate the diffusion of solute atoms and provide more nucleation sites for dynamic precipitates

[22-24]. In addition, high temperature accelerates the diffusion of solute atoms providing more driving force and nucleation sites for the dynamic precipitates. The atoms rearrange and eventually from a metastable strengthening phase.

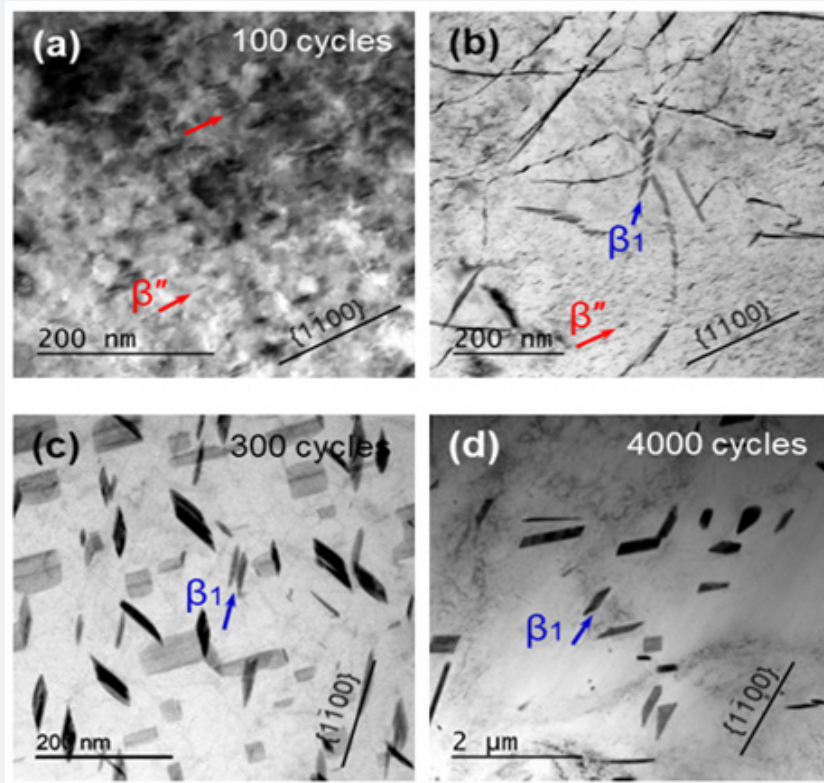


Figure 4: The β'' precipitates (a) and β_1 precipitates (b) in the T6-treated Mg-3.74Nd-0.59Zr alloy tested at 150°C after 100 cycles (at the strain amplitude of 0.4%), respectively. The β_1 precipitates in the T6-treated alloy tested at 150°C (c) after 300 cycles and (d) after 4000 cycles.

Plastic deformation mechanism

Figure 5a shows that typical dislocation structures in the alloy tested at 150°C after 100 cycles (at the strain amplitude of 0.4%). Some edge dislocations located on the $\{0001\}_\alpha$ basal plane can be observed in the matrix. Figures 5b-5d show the typical morphologies of the dislocations and the twins at the same location but different basal vectors in the alloy specimens at 150°C after 300 cycles. Under the condition of $g = 1-102$, as shown in figure 5b, a large number of the $\langle a \rangle$ dislocations [25] at the basal plane (marked by yellow arrows) and twins were formed in matrix of the T6-treated alloy. After 300 cycles the appearance of thin and straight PSBs (persistent slip bands) located on $\{1-100\}_\alpha$ prism plane (marked by blue arrow) is a sign that the dislocations cut through the precipitates repeatedly [26-28]. In addition, it is seen that there are few dislocations in the $\{10-12\}_\alpha$ twin. In the case of $g = 0002$, the specimens contain a large number of the $\langle a + c \rangle$ or $\langle c \rangle$

dislocations located on the $\{1-100\}_\alpha$ plane (Figure 5c). Moreover, some dislocations were also activated in $\{10-12\}_\alpha$ twin. Under the condition of $g = 1-102$, however, some $\langle a \rangle$ and $\langle c + a \rangle$ or $\langle c \rangle$ dislocations are observed on the basal and non-basal planes and within the twins (Figure 5d). The density of $\langle c + a \rangle$ dislocations will increase with the increase of test temperature, which may be attributed to the decrease of CRSS (critical resolved shear stress) required to active the $\langle c + a \rangle$ cone plane dislocations [29,30].

The high-magnification image in figure 6a shows the typical morphology at location A in figure 5c. Some short parallel lines (marked by red and yellow arrows) similar to moire fringes can be seen in the matrix of the alloy. In terms of the typical morphology in figure 6b, the lines in the alloy mainly consists of the ITBs (incoherent twin boundary) [31-34]. The $\{10-12\}$ twinning causes the plane to rotate 86-88° around a specific $\langle 1-210 \rangle$ axis [35,36]. Therefore, the included angle of the basal plane on both sides of

the $\{10\text{-}12\}$ twin boundary is 86° (Figure 6b) after twinning. The interface of $\{10\text{-}12\}$ twin tip is not completely flat but stepped at some sites. The twin interfaces marked by white dots are basically parallel to the $(10\text{-}12)$ cone plane. The stepped interfaces (marked by yellow lines in figure 6b) parallel to the $(1\text{-}100)$ prismatic plane forms a 45° angle with the $(10\text{-}12)$ cone plane. When the interface reaction between dislocations (marked by red arrows) and twins occurs, the sawtooth twin plane at the edge can form and hinder the slip of the twin dislocation. Once twins are formed, they can act as the obstacles, which further promote the dislocation stacking and twin nucleation. The dislocations pass through the incoherent twin boundary and react with the twin boundary to form a large number of movable twin boundary dislocations, which in turn enhances the migration of twin boundary [37,38]. The strengthening mechanism dominated by dislocation stacking and dislocation crossing the twin boundary transforms into the

softening mechanism dominated by nucleation and movement of the incomplete dislocations. Figure 6c shows the typical morphology of the moire fringes marked by red arrow in figure 6a. It can be seen that these moire fringes are similar to the twin tip [39-41]. After 9300 cycles, there are many $\{10\text{-}12\}$ twins on the surface of the fatigued samples tested at 150°C (Figure 6d). It is thus believed that for the T6-treated alloy tested at 150°C , the size of the twins increases gradually during fatigue testing. This result is obviously different from the evolution characteristics of the twins in the T6-treated NZ30K alloy tested at RT [17]. The effect of the twinning on deformation decreases gradually with the increase of the number of cycles. The plastic deformation of the NZ30K-T6 alloy tested at RT mainly depends on the dislocations-slip. In contrast, for the T6-treated Mg-Nd alloy tested at 150°C , both dislocations-slip and twinning participate in the plastic deformation.

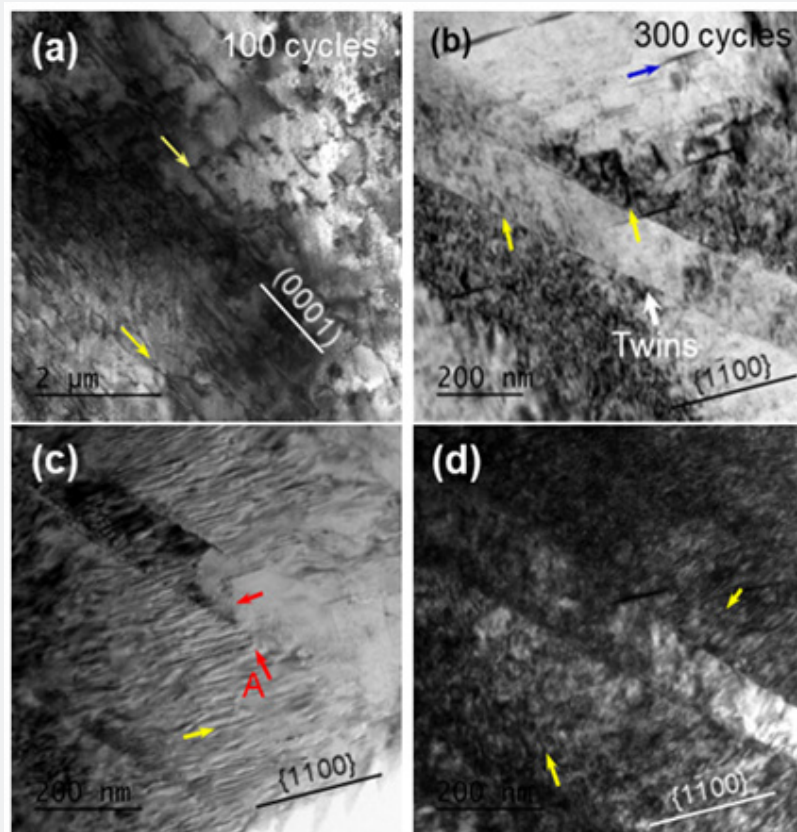


Figure 5: (a) Typical morphology of the dislocations in the T6-treated Mg-3.74Nd-0.59Zr alloy tested at 150°C after 100 cycles (at the strain amplitude of 0.4%). After 300 cycles, the morphology of the dislocations and twins located at same position and different base vectors ($B = [11\text{-}20]a$): (b) $g = 1\text{-}100$, (c) $g = 0002$, and (d) $g = 1\text{-}102$.

At the strain amplitude of 0.6%, the typical microstructure of the dislocations in the alloy tested at 150°C after 100 cycles is shown in figure 7a. Large numbers of the dislocations (marked by yellow arrows) were observed on the $\{0001\}_a$ base plane for the alloy after 100 cycles. Moreover, the twins also participate in

the plastic deformation of the alloy (Figure 7b). The band-shaped β_1 precipitates have been also cut by the dislocations in the alloy after 100 cycles (Figure 7c). After 2000 cycles, there are some massive β_1 precipitates with large size formed in the alloy tested at 150°C (Figure 7d).

Crack initiation and propagation mechanism

During fatigue, the formation of cracks tends to experience four development stages including dislocation-slip in the matrix, micro persistent slip lines, initiation and propagation of the PSBs, cracking of the PSBs. After 100 cycles, some dislocations located on both basal plane and {1-100} prism plane were formed in the matrix of the T6-treated alloy tested at 150°C (Figure 8a). As a

result, the dislocation-slip continuously shears the precipitates, leading to the transformation of the precipitates and the formation of the PSBs on the surface of the fatigued samples (Figure 8b). On the fracture surface of the T6-treated samples, in addition to the basal plane fringes parallel to each other, some wavy slip fringes caused by non-basal planes can be also observed (Figure 8d). The wave slip bands are considered to be the result of dislocation movement on multiple slip planes [25].

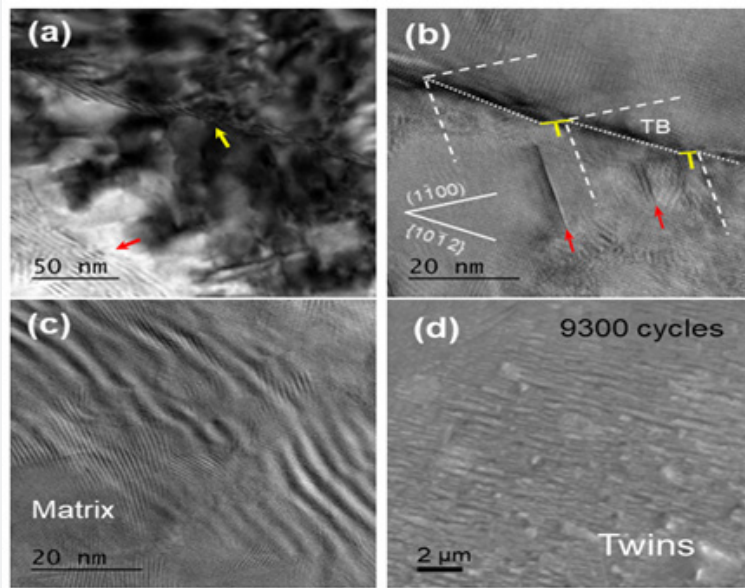


Figure 6: (a) High-magnification image of location A in figure 6c showing the moiré fringes and the dislocations. (b) High-resolution bright field image showing the typical configuration of the incoherent twin boundary (ITB). (c) High-resolution bright field image showing the typical morphology of the moiré fringes. (d) After 9300 cycles (at strain amplitude of 0.4% and 150°C), typical twin morphology of the surface of the T6-treated alloy specimens.

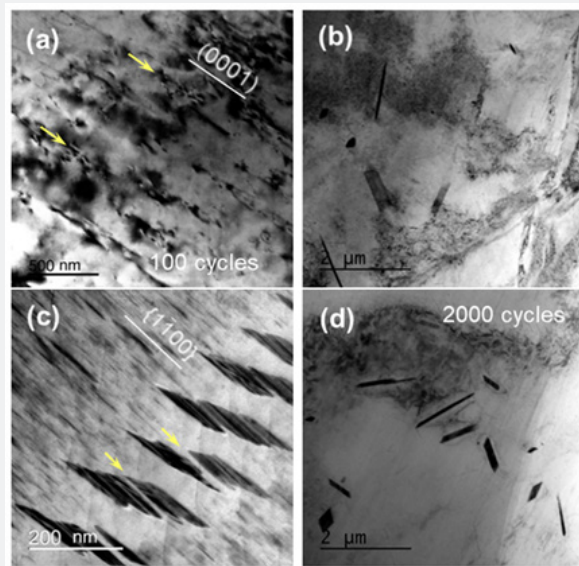


Figure 7: (a) Typical dislocation morphology and (b) the twins in the T6-treated alloy tested at 150°C and the strain amplitude of 0.6% after 100 cycles ($B = [11-20] \alpha$). (c) The β_1 precipitates were cut by the dislocations in the alloy after 100 cycles. (d) The β_1 precipitates in the matrix of the alloy after 2000 cycles.

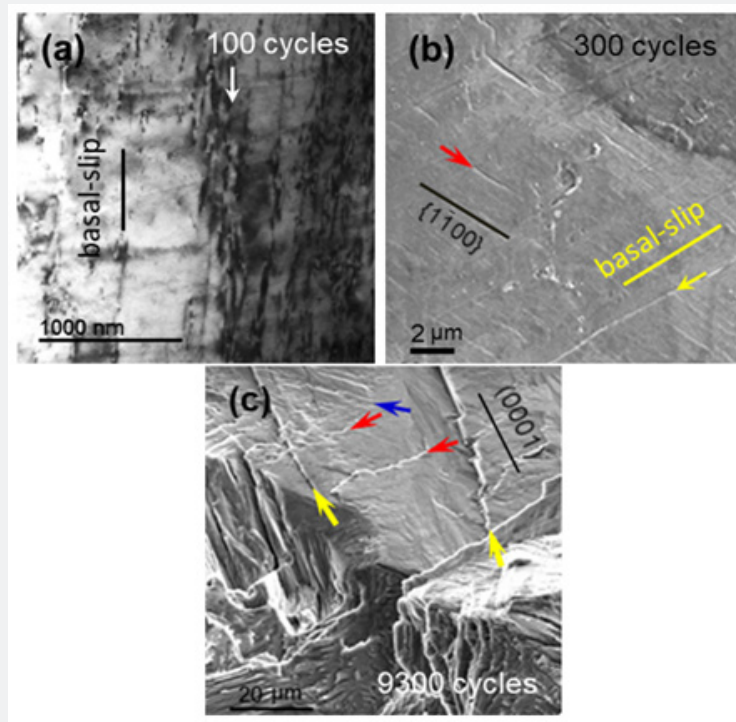


Figure 8: (a) The dislocations-slip in the T6-treated Mg-3.74Nd-0.59Zr alloy tested at 150°C after 100 cycles (strain amplitude of 0.4%). (b) The PSBs on the surface of the T6-treated samples after 300 cycles. (c) Slip bands in crack initiation region of the T6-treated samples after 9300 cycles.

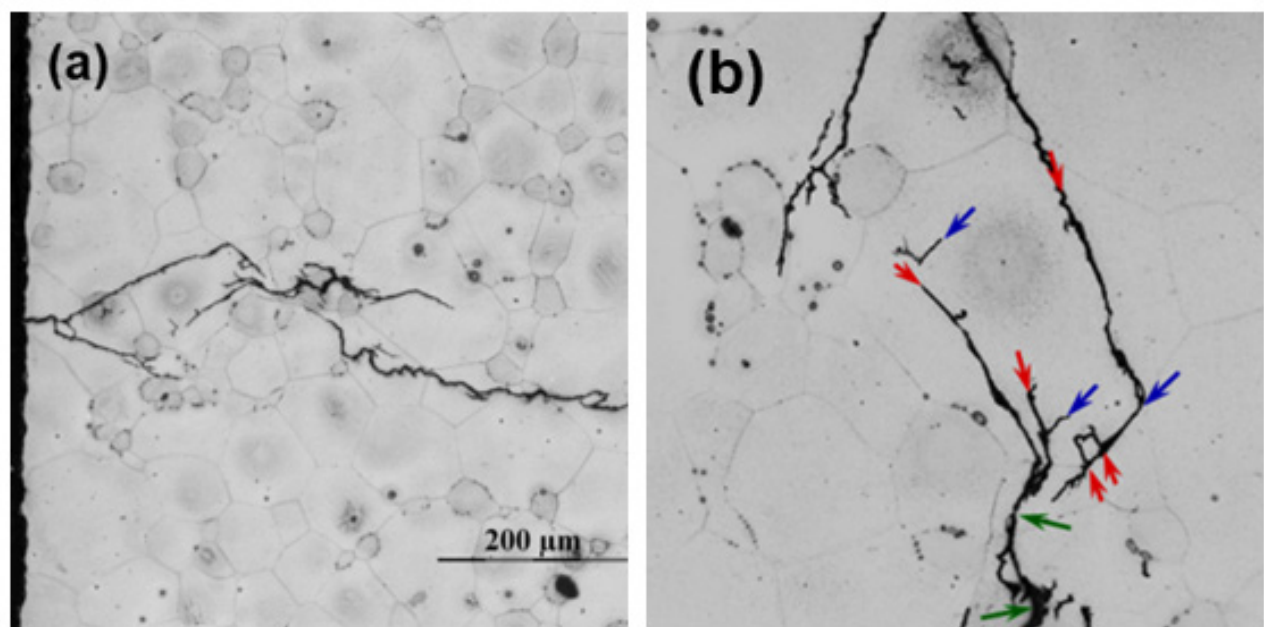


Figure 9: (a) OM image showing the characteristic of fatigue crack initiation and propagation of the T6-treated specimens after 2000 cycles (at the strain amplitude of 0.4% and 150°C). (b) The typical morphology of fatigue crack propagation for the T6-treated alloy.

Several studies have been also conducted to understand the influence of the dislocations and twins on crack initiation of the Mg alloys [42-46]. Pan et al. [42] suggested that for the T6-treated Mg-8Gd-3Y-Zr alloy during fatigue testing, the basal dislocation-slip was observed on the surface of the fatigue specimens and the basal slip deformation only limited on several PSBs. It is believed that the basal dislocation-slip is the dominant fatigue damage mechanism. Yue et al. [44] suggested that the fatigue cracks of the as-cast NZ30K alloy initiate from the PSMs (persistent slip markings) and twinning bands. For an extruded Mg-Gd-Y magnesium alloy [45], it has been reported that the fatigue cracks are mainly initiated at the grain boundaries when the strain amplitude is higher, while those are found to initiate at the PSBs and grain boundaries at the lower strain amplitudes. Briffod et al. [46] pointed out that the secondary cracks in an extruded Mg-Al-Ca-Mn alloy are mainly originated from the basal slip bands, twin bands, grain boundaries, and non-metallic inclusions. However, these research results are mainly focused on room temperature fatigue of the Mg alloys. In contrast, there are few studies on the fatigue deformation mechanism and fatigue crack mechanism of magnesium alloys at high temperature.

Figure 9a shows the initiation site and growth path of the micro-cracks in the T6-treated Mg-3.74Nd-0.59Zr alloy samples. For the T6-treated alloy, the small microcrack is first initiated from the cracked PSBs and then quickly propagates within a grain by shearing along slip bands under cyclic loading. When the microcrack tip approaches the grain boundary, its growth rate decreases significantly, or it even stops growing. It is mainly due to the different orientations of slip systems in adjacent grains (Figure 1b). Only when the small microcracks pass through the grain boundaries, they can connect with the small micro-cracks in the adjacent grains or expand into the adjacent grains to form macro scale fatigue. From the characteristics of the small microcracks propagation (Figure 9b), it is seen that the cross slip was also involved in plastic deformation of the alloy samples in the high temperature environment. Due to special crystallographic orientation relationship between grains on both sides of ITB, the dislocation-slip can pass through the twin boundary meanwhile the twin boundary can migrate under the action of the dislocations, which can slow down the fatigue damage and improve the crack propagation resistance.

Conclusion

1. The T6-treated Mg-3.74wt%Nd-0.59wt%Zr alloy shows a significant response to test temperature. Compared to those of the alloy tested at RT, the tensile strengths and cyclic stress amplitudes of the counterpart tested at 150°C reduce significantly. Different to RT fatigue, the alloy tested at 150°C exhibits an initial cyclic stabilization followed by a gradually softening until fatigue failure.

2. For the T6-treated alloy, in addition to dislocations, the twins are more involved in plastic deformation at 150°C until fatigue failure, which is different from RT fatigue. Environment-

induced fatigue damage behavior tends to cause the crack initiation in the samples at the elevated temperature.

3. At high temperature, the alloy shows longer fatigue lifetimes at same strain amplitudes in comparison with at RT, which is mainly attributed to more cross slip and twins participating in cyclic deformation.

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