

## Article

# Enhanced Age Strengthening of Mg-Nd-Zn-Zr Alloy via Pre-Stretching

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**Abstract:** Pre-stretching was carried out to modify the microstructure of Mg-Nd-Zn-Zr alloy to enhance its age strengthening. The results indicated that more heterogeneous nucleation sites can be provided by the high density of dislocations caused by the plastic pre-stretching deformation, as well as speeding up the growth rate of precipitates. Comparison of microstructure in non-pre-stretched specimens after artificial aging showed that pre-stretched specimens exhibited a higher number density of precipitates. The fine and coarse plate-shaped precipitates were found in the matrix. Due to an increase in the number density of precipitates, the dislocation slipping during the deformation process is effectively hindered, and the matrix is strengthened. The yield strength stabilizes at 4% pre-stretching condition, and then the evolution is stable within the error bars. The 8% pre-stretched specimens can achieve an ultimate tensile strength of 297 MPa. However, further pre-stretching strains after 8% cannot supply any increase in strength. Tensile fracture surfaces of specimens subjected to pre-stretching strain mainly exhibit a trans-granular cleavage fracture. This work indicated that a small amount of pre-stretching strain can further increase strength of alloy and also effectively enhance the formation of precipitates, which can expand the application fields of this alloy.

**Keywords:** magnesium alloys; precipitation; pre-stretching; mechanical properties

## 1. Introduction

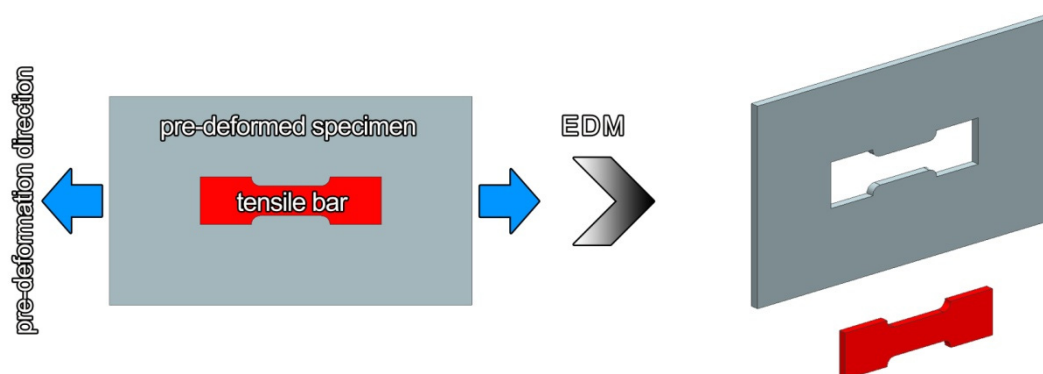
The addition of rare-earth elements (RE) is an effective way to improve the mechanical properties of magnesium alloys [1–4]. The Mg-Nd-Zn-Zr alloy is one of the most successful Mg-RE alloy systems. This alloy shows a highly desirable combination of relative good room-temperature tensile properties and high-temperature creep resistance, both of which are associated with structure, distribution and number density of precipitates [5]. The precipitation sequence in this alloy during isothermal aging is commonly accepted as: supersaturated solid solution (SSSS)  $\rightarrow$  ordered Guinier-Preston zones (G.P. zones)  $\rightarrow \beta'' \rightarrow \beta' \rightarrow \beta_1 \rightarrow \beta$  [6–8]. Despite many works regarding the precipitates evolution have been achieved in the past few years. However, further attempts to improve the strength of the alloy via controlling the nucleation and growth of precipitates during aging process are rarely reported. Micro alloying additions of some elements may considerably influence the precipitation during ageing in many magnesium alloy systems. Geng and Buha [9,10] have reported that the addition of Co and Cr to a Mg-Zn alloy can increase number density of precipitates and enhances age-hardening response. Elsayed [11] investigated the influence of Na on age-hardening response of the Mg-9.8Sn-3.0Al-0.5Zn alloy. The Na clusters refine the microstructure by acting as heterogeneous nucleation sites for Mg<sub>2</sub>Sn precipitates. However, the recognized micro alloying elements are still less and not applicable for all the magnesium alloy systems, especially for RE-containing or Zr-containing magnesium alloys. For this reason, it is necessary to find other effective methods to enhance the age strengthening for Mg-Nd-Zn-Zr alloy systems.

Another very efficient way to enhance the age strengthening for most magnesium alloy systems may be pre-deformation. The cold pre-deformation after solution treatment, and prior to aging treatment, has been attracted researchers lately due to a tremendous potential for enhancing the nucleation and growth of precipitates. Gazizov [12] has reported pre-deformation is effective in increasing the strength of Al-Cu-Mg-Ag alloy by modifying the normal precipitation sequence. Ozaki [13] studied the effects of pre-compressive strain on the fatigue life of the AZ31 magnesium. These results suggest that the pre-deformation introduces numerous dislocations and twins. The precipitates interact with dislocations during the aging treatment and therefore influence the performance of alloy, which is an area that has been much less explored. Moreover, the pre-deformation by stretching is rarely employed due to poor ductility of many magnesium alloys. In this research, the cold plastic pre-stretching at room temperature (RT) was performed on the Mg-Nd-Zn-Zr alloy system, and the microstructure, mechanical properties and fracture behavior were investigated in detail. It is aimed to further improve the strength of the alloy and, thus, to broaden its application fields.

## 2. Experimental Section

The nominal compositions of the investigated alloy was Mg-2.7Nd-0.4Zn-0.5Zr (wt. %). Solution treatment of as-cast ingots was performed at 530 °C for 14 h and followed by quenching in water. The specimens with dimensions of 3 mm × 60 mm × 120 mm were cut from the as-quenched ingots by electrical discharge machining (EDM) and cleaned in ethanol. Then the specimens were pre-stretched by 2%, 4%, 6%, 8%, and 10% by electronic material testing machine under a constant strain rate of  $1 \times 10^{-3} \text{ s}^{-1}$  at room temperature (RT), and subsequently aged at 200 °C for 8 h. The specimens without pre-stretching were also prepared for comparisons. After aging treatment, a series of tensile bars were prepared by EDM and polished to remove the oxide layer from the surface and cleaned with ethanol.

Tensile test was performed on material test machine under a constant strain rate of  $1 \times 10^{-3} \text{ s}^{-1}$  at RT. Tensile bars with a 10 mm gauge width and 20 mm gauge length parallel to the pre-stretched direction, as illustrated in Figure 1. Metallographic specimens were etched with a solution of 2.5 g picric acid, 2.5 mL acetic acid, 50 mL ethanol and 50 mL water, and observed by an optical microscope (GX71, OLYMPUS, Tokyo, Japan). The transmission electron microscopy (TEM) was used to study the microstructure in the pre-deformed and aged alloys. TEM specimens were prepared by ion-milling (691, Gatan, Pleasanton, CA, USA) and examined in a transmission electron microscope (JEM-2100, JEOL, Tokyo, Japan) operating at 200 kV. The fracture surfaces of specimens after a tensile test were observed by a scanning electron microscope (Quanta 200, FEI, Eindhoven, The Netherlands).

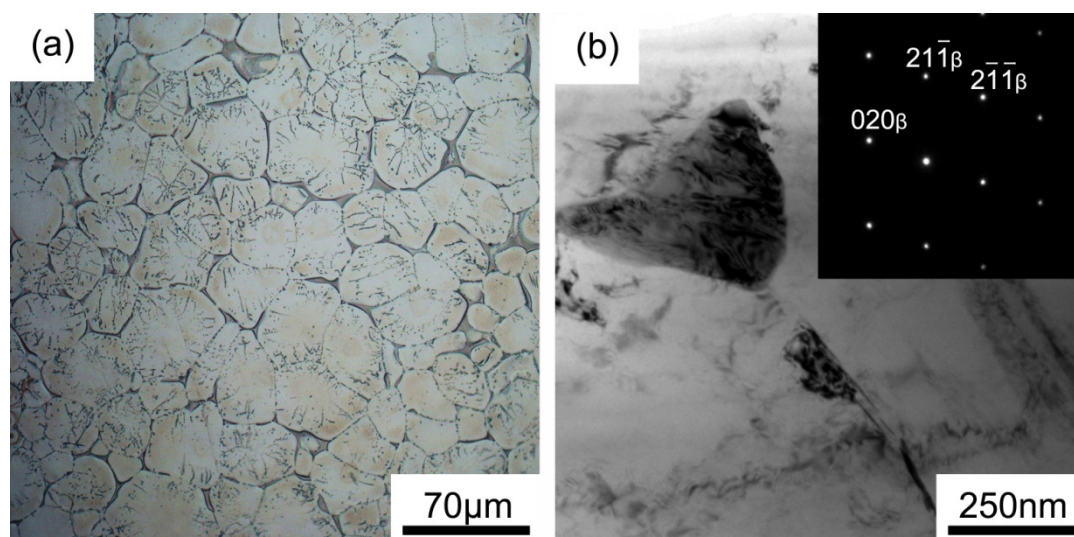


**Figure 1.** A schematic illustrating the preparation of tensile bar.

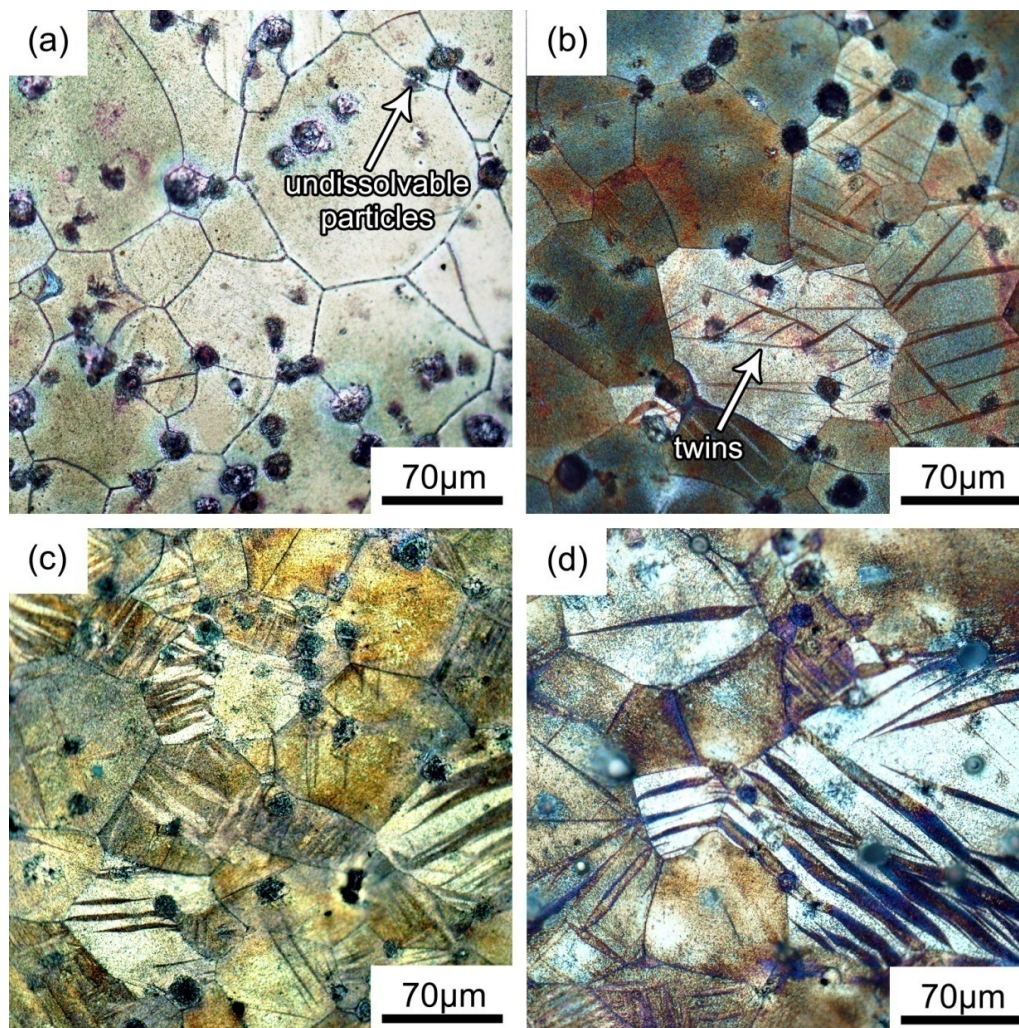
### 3. Results

#### 3.1. Microstructures

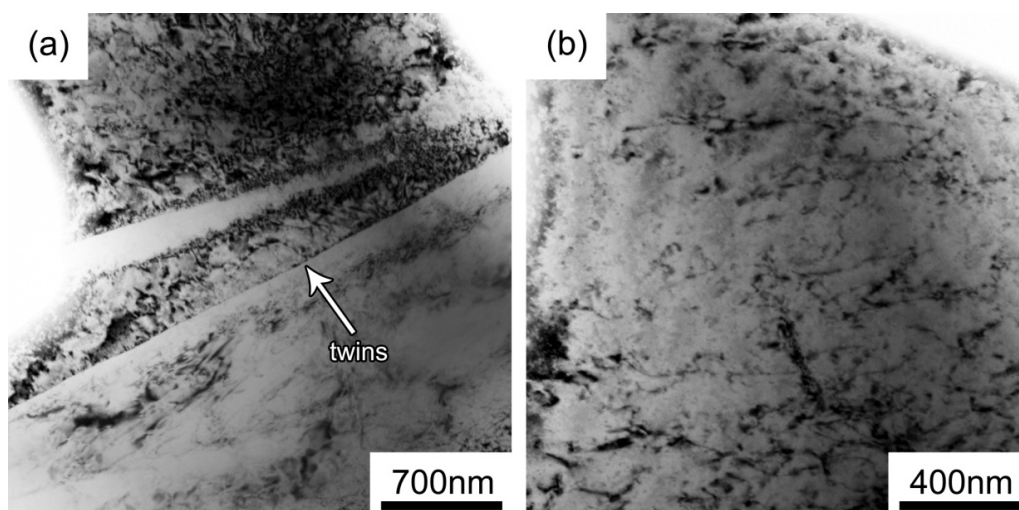
Figure 2 shows the microstructure for Mg-2.7Nd-0.4Zn-0.5Zr alloy in as-cast condition. The microstructure of as-cast specimen consists of  $\alpha$ -Mg matrix and eutectic compounds. The fine equiaxial  $\alpha$ -Mg grains are separated by eutectic compounds. The eutectic compounds mainly gather in grain boundaries, and some small eutectic compound particles also present within grains as is evident in Figure 2a. As previously reported [14], the eutectic compounds for the Mg-Nd system may be  $\beta$  phase, which is tetragonal lattice structure with  $a = 1.03$  nm and  $c = 0.59$  nm. The composition of the  $\beta$  phase is reported as  $\text{Mg}_{12}\text{Nd}$ , which is the main existing phase in the as-cast microstructure. Figure 2b shows a typical bright-field TEM image and corresponding selected area electron diffraction (SAED) patterns of the eutectic compounds on grain boundaries. The beam is parallel to  $[102]_{\beta}$  zone axis in this specimen. The eutectic compounds are identified to be the equilibrium  $\beta$  phase, which is irregular in shape and has no orientation relationships with the  $\alpha$ -Mg matrix [14]. Figure 3 shows the optical images of the quenched specimens with 0%, 2%, 8%, and 10% pre-stretching. After solution treatment at 530 °C for 14 h and quenching in water, the eutectic compounds completely dissolve into the matrix. It should be noted that many black regions distribute unevenly in a few grains as shown in Figure 3a. These regions contain numerous undissolvable particles, which precipitate during the solution treatment. Another interesting point is the presence of twins after pre-stretching. The number density of twins is varied from grain to grain due to size and orientation difference. The formation of twins is easier in coarse grains during deformation. It is revealed that the stress concentration along the grain boundaries triggers the nucleation of twins and, thus, the nucleation density of twins is sensitive to the grain size. Additionally, the size of twins is limited by grain size, which implies that grain size affects twin growth [15]. Figure 3b,d reveal that the number density and size of twins increase significantly as the strains rise from 2% to 10%. However, the further pre-stretching after 8% cannot supply any visible increase in twins. Figure 4 shows TEM bright-field images of the quenched specimen with 8% pre-stretching. Numerous dislocations distribute evenly in the twins and matrix. Hence, high density dislocations form during pre-stretching plastic deformation.



**Figure 2.** The microstructure of the as-cast specimen: (a) optical images and (b) TEM bright-field image and corresponding SAED patterns for  $\beta$  phase ( $B//[102]_{\beta}$ ).

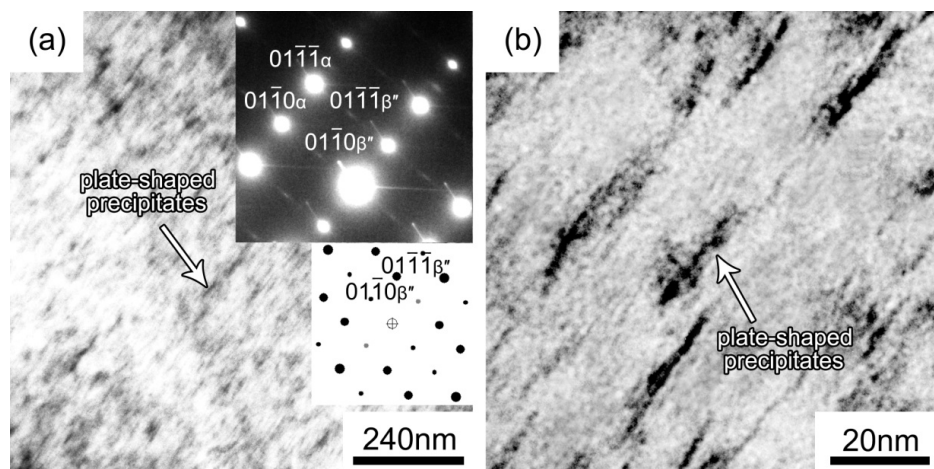


**Figure 3.** Optical images of quenched specimens with (a) 0%, (b) 2%, (c) 8%, and (d) 10% pre-stretching.



**Figure 4.** TEM bright-field images of the quenched specimen with 8% pre-stretching: (a) twins; (b) dislocations.

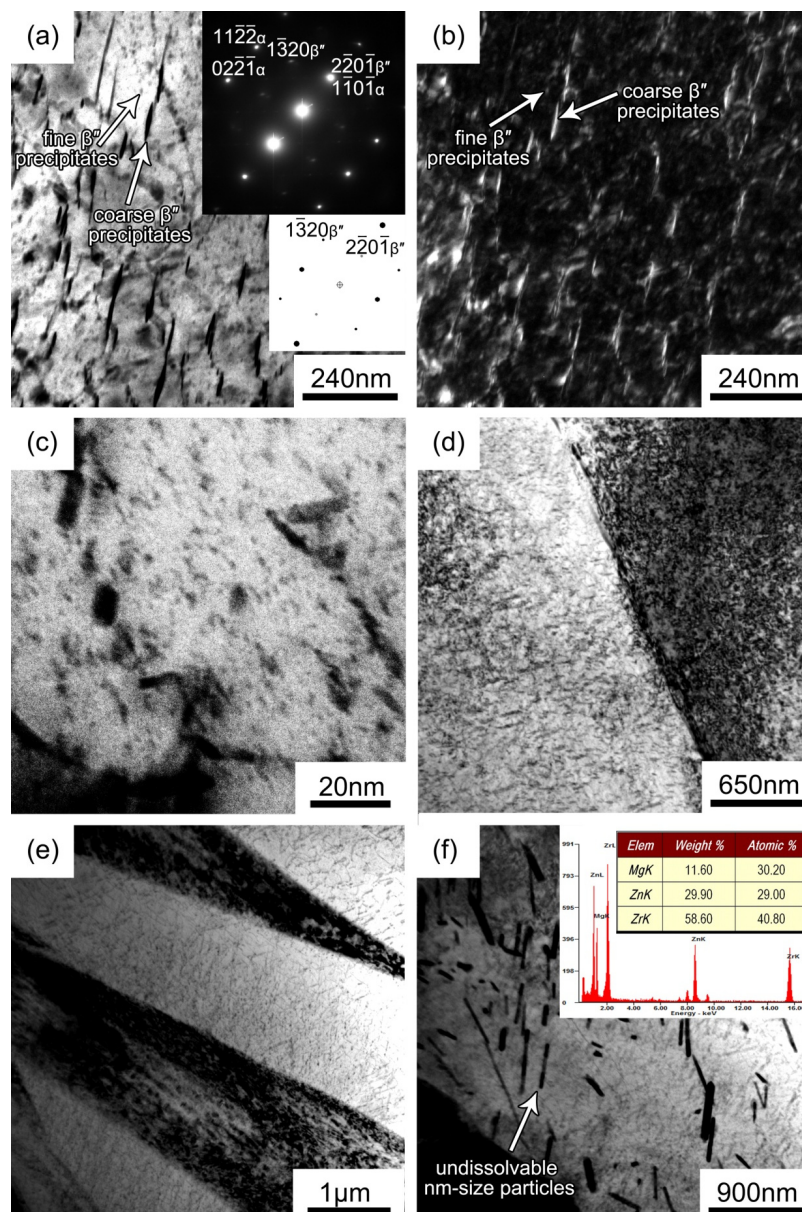
Figure 5 shows the TEM images of specimens without pre-stretching aged at 200 °C for 8 h. During the aging treatment, the Nd atoms in solid-solution within the matrix decrease due to precipitation. According to bright-field image with beam parallel to  $[11\bar{2}0]_{\alpha}$  direction (Figure 5a), the presence of plate-shaped precipitates distribute evenly in the matrix. The orientation relationship between plate-shaped precipitates and  $\alpha$ -Mg is such that  $[11\bar{2}0]_{\beta''} // [11\bar{2}0]_{\alpha}$  and  $(01\bar{1}0)_{\beta''} // (01\bar{1}0)_{\alpha}$ . The  $\beta''$  precipitates in Mg-Nd alloy usually form these kinds of SAED patterns. Moreover, the simulated SAED patterns (right lower quadrant in Figure 5a for this structure are in good agreement with the observed experimental results. Hence, the plate-shaped precipitates are  $\beta''$  precipitates. Figure 5b shows the high magnification TEM image of the plate-shaped precipitates. The average size and inter-spacing of plate-shaped precipitates are ~30 nm and ~20 nm, respectively, in the non-pre-stretched condition.



**Figure 5.** TEM images of specimens without pre-stretching aged at 200 °C for 8 h: (a) plate-shaped precipitates in the matrix and corresponding SAED patterns ( $B // [11\bar{2}0]_{\alpha}$ ); and (b) high magnification TEM image of the plate-shaped precipitates.

Figure 6 shows TEM images of specimen with 8% pre-stretching aged at 200 °C for 8 h. Two distinct types of precipitates morphologies are revealed under this condition as seen in Figure 6a. The first type of precipitates is the fine dispersion of plate-shaped  $\beta''$  precipitates. Compared with the specimen without pre-stretching strain, the size and inter-spacing of plate-shaped precipitates have been significantly reduced when the specimen subjected to pre-stretching strain of 8%. The other precipitates are a large number of coarse precipitates. The average size of this precipitates is ~80 nm in length. In order to determine the type of coarse precipitates, the SAED patterns of coarse precipitates are analyzed. The  $\beta''$  precipitates in Mg-Nd alloy usually form these kinds of SAED patterns. The orientation relationship between  $\beta''$  precipitates and  $\alpha$ -Mg is such that  $[5\bar{1}412]_{\beta''} // [5\bar{1}46]_{\alpha}$  and  $(2\bar{2}0\bar{1})_{\beta''} // (1\bar{1}0\bar{1})_{\alpha}$ . The simulated SAED patterns (right lower quadrant in Figure 6a) for  $\beta''$  precipitates are in good agreement with the observed experimental results. The dark-field morphology of  $\beta''$  precipitates are shown in Figure 6b. As it is evident, the fine and coarse precipitates display the same contrast in white. Although they have the different morphologies, they have the same structure. Hence, the coarse precipitates may be also the  $\beta''$  precipitates. The high magnification TEM image of fine precipitates is shown in Figure 6c, which reveals that the average size and inter-spacing of fine precipitates are ~3 nm and ~5 nm, respectively. Figure 6d shows the distribution of precipitates around grain boundary. After 8% pre-stretching and subsequent aging treatment, there is no visible precipitate-free zones around grain boundary. Figure 6e displays the distribution of precipitates around twins. It exhibits the presence of twins in the pre-stretched specimens even after the aging. Additionally, the distribution and number density of precipitates around the grain boundary and twins is same as the matrix. Figure 6f shows the TEM bright-field morphology of undissolvable rod-shaped

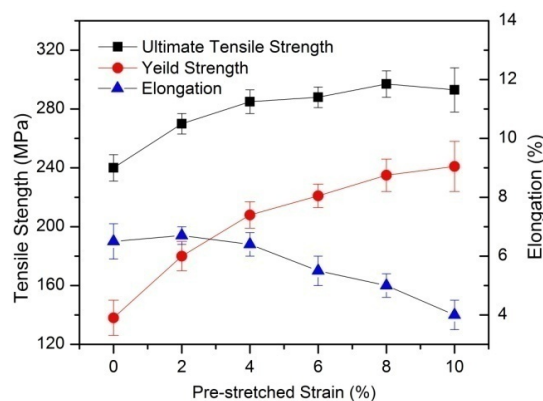
particles, which are also present in as-quenched specimens (Figure 3). The projected lengths of the undissolvable rod-shaped particles vary from 100 nm to 900 nm. The energy dispersive X-ray spectrum (EDXS) recorded from an undissolvable rod-shaped particle is shown in the Figure 6f upper right inset. The qualitative results show a prominent Mg K peak, together with significant intensities of Zn K and Zr K peaks. As can be noticed, the ratio of Zn (29.00 at. %) and Zr (40.80 at. %) is nearly two to three. Therefore, the undissolvable rod-shaped particles may be the  $\text{Zn}_2\text{Zr}_3$  phase similar to what was reported in the literature for this phase [16]. It should be mentioned that the distribution of plate-shaped  $\beta''$  precipitates is uniform around the  $\text{Zn}_2\text{Zr}_3$  particles. The  $\text{Zn}_2\text{Zr}_3$  particles precipitate during the solution treatment. Thus, the distribution of Nd atoms has been slightly affected by  $\text{Zn}_2\text{Zr}_3$  particles, and the  $\beta''$  precipitates can precipitate evenly around the  $\text{Zn}_2\text{Zr}_3$  particles.



**Figure 6.** TEM images of specimen subjected to 8% pre-stretching and subsequently aged at 200 °C for 8 h: (a) and (b) are bright-field and dark-field morphology of plate-shaped precipitates in the matrix ( $B//[5\bar{1}46]_{\alpha}$ ), respectively; (c) is high magnification TEM image of the fine precipitates; (d) and (e) are bright-field morphology of grain boundaries and twins, respectively; and (f) is bright-field morphology of undissolvable particles and corresponding EDXS recorded from an undissolvable particle.

### 3.2. Mechanical Properties

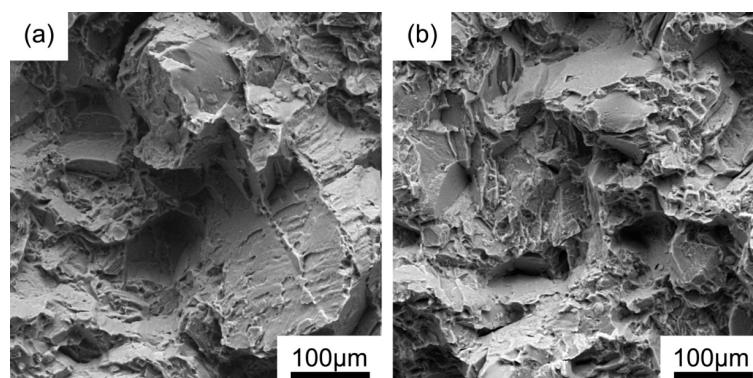
Figure 7 shows the evolution of mechanical properties with pre-stretching strains for all specimens aged at 200 °C for 8 h. It clearly shows that the ultimate tensile strength (UTS), yield strength (YS) and elongation change with the changes in the pre-stretching levels. For the case without pre-stretching, the UTS is low at 240 MPa. As pre-stretching rise from 2% to 8%, although the UTS increases from 270 MPa to 297 MPa, the elongation decreased significantly. The YS stabilizes at 4% pre-stretching condition, and then the evolution is stable within the error bars. It is noteworthy that the 8% pre-stretched specimen exhibits the highest UTS, whereas the increment rate of UTS decreases when the pre-stretching after 2%. As the pre-stretching strains increase from 8% to 10%, the average strength tends to slightly reduce, indicating that further pre-stretching strains after 8% cannot supply any increase in strength. Hence, the strength significantly increases after pre-stretching and aging, and ductility largely decreases.



**Figure 7.** Mechanical properties of specimens subjected to pre-stretch and subsequently aged at 200 °C for 8 h.

### 3.3. Fractography

Figure 8 shows the SEM images of typical tensile fracture surfaces for specimens with different pre-stretching strains and subsequently aged at 200 °C for 8 h. The examination of the microstructure reveals that alloys have mixed trans-granular and inter-granular fracture. However, the fracture mode is the mainly trans-granular fracture. The fracture surfaces consist of cleavage planes and steps. The size of cleavage planes in the specimen with pre-stretching strain of 8% is smaller than specimen with pre-stretching strain of 0%. Based on the above results, it is inferred that all the tensile fracture surfaces mainly exhibit a trans-granular cleavage fracture, and the cleavage plane size of the pre-stretched alloy is smaller than that of the non-pre-stretched alloy.

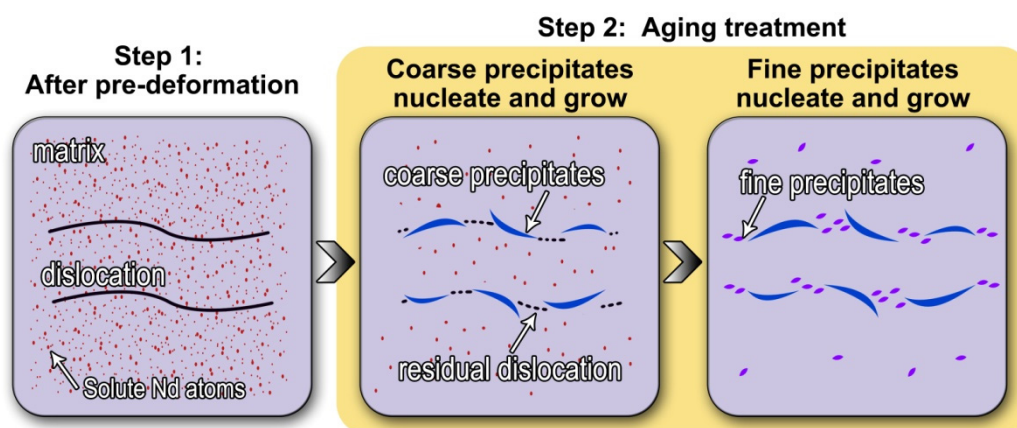


**Figure 8.** SEM images showing typical fracture surfaces of specimens with (a) 0% and (b) 8%, respectively, after tensile test at RT.

## 4. Discussion

### 4.1. Effect of Pre-Stretching on the Precipitation Evolution

The Mg alloys deformed at RT will promote the activation of twins and basal slip for the former, and the prismatic and pyramidal slip are activated for the later. Moreover, basal plane slip takes a high proportion during cold deformation in random orientated alloys [15,17]. That is why the density of dislocations and twins becomes pronounced when the specimen subjected to plastic pre-stretching deformation. During aging, the morphology, distribution, and number density of precipitates will be modified by dislocations. Compared with the specimen without pre-stretching strain, there are numerous coarse plate-shaped precipitates and fine plate-shaped precipitates exist in the matrix. Moreover, the number density of precipitates is higher than specimens without pre-stretching. This is attributed to heterogeneous nucleation on equilibrium state dislocations [18]. The schematic diagrams of precipitates evolution during aging treatment are shown in Figure 9. For a start, a large number of coarse plate-shaped precipitates heterogeneously nucleate on the dislocations and grow during the aging process. Most of the dislocations and a certain amount of solute Nd are consumed in this process. The continuous dislocations are interrupted and turn into numerous small segments. Subsequently, fine plate-shaped precipitates nucleate and grow on the matrix and the interrupted dislocation segments. It consumes the residual dislocation segments and solute Nd. Due to the growth of precipitates is limited by the residual dislocation size and solute Nd, the size of plate-shaped precipitates formed in the later aging process is smaller. Thus, the potential nucleation sites of precipitates are increased by dislocations, which increased the number density of precipitates. In addition, the length of coarse plate-shaped precipitates is ~80 nm, and the length of the plate-shaped precipitates forming in the non-pre-stretched specimen is ~30 nm. It is noticed that the size of the coarse plate-shaped precipitates is significantly larger than plate-shaped precipitates formed in non-pre-stretched specimen. The reason may be that the dislocations most likely act as fast diffusion pipes for the solute Nd. The high density dislocations considerably speed up the growth rate of coarse plate-shaped precipitates. Additionally, non-equilibrium vacancies formed in pre-stretching also increase the kinetics of solute diffusion. Therefore, the pre-stretching strain can greatly enhance the aging kinetics. As noticed in the above discussion, the microstructure includes numerous coarse plate-shaped precipitates, fine plate-shaped precipitates and twins, when the specimens subjected to pre-stretching strain and subsequently aging treatment.

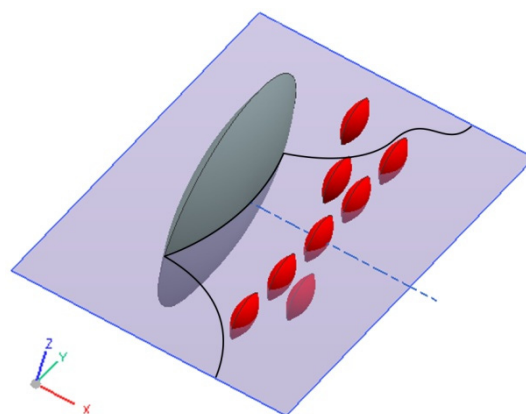


**Figure 9.** Schematic diagrams of precipitates evolution during aging treatment.

### 4.2. Effect of Pre-Stretching on the Mechanical Properties

The high strength of the pre-stretched and aged alloy is attributed to the superposition of different strengthening mechanisms. In the case of pre-stretched and aged specimens, the increment in yield

strength is determined by work strengthening and precipitation strengthening. The work strengthening is severely weakened by the occurrence of recovering. The density of dislocations gradually reduces during the aging at 200 °C. However, the numerous twins are retained in the matrix after aging treatment. The twin boundaries can effectively act as barriers for dislocation slips and become a source of work hardening. The precipitation strengthening in pre-stretched alloys occurs through the formation of precipitates, which form mainly as coarse and fine plate-shaped particles. It is worth noting that the numerous dislocations increase the number density of precipitates. Additionally, the strength is sensitive to the number density of precipitates, since the more precipitates will shorten the inter spacing of them according to the Orowan law [19]. Then the dislocations are more difficult to pass through them [20]. In addition to the Orowan strength, there is a contribution from the backstress caused by the strain incompatibility and modulus mismatch between precipitates embedded in a sheared matrix [21]. The dislocation motion is impeded simultaneously, rather than sequentially, by both coarse and fine precipitates, which leads to threshold stress higher than for a dislocation interacting with either type of precipitates. In order to illustrate it, a highly simplified geometry is assumed as shown in Figure 10. This situation occurs when the dislocations are pinned at the departure side of the coarse precipitates, while concurrently subjected to the elastic back-stress from 8 neighboring fine precipitates. The overall threshold stress is the sum of the true detachment stress from the precipitates and the back-stress [22]. As it is noticed in the above discussion, the combination of work strengthening and precipitation strengthening provides excellent strength to the alloy after pre-stretching and subsequent aging treatment.



**Figure 10.** Three-dimensional schematic, showing precipitates contributing to the back-stress on the dislocation.

## 5. Conclusions

(1) The more heterogeneous nucleation sites can be provided by the high density of dislocations caused by the plastic pre-stretching deformation, as well as speeding up the growth rate of precipitates. The comparisons of microstructure in non-deformed specimens after artificial aging shows that pre-stretched specimens contain not only fine and coarse precipitates but also a higher number density of precipitates.

(2) The dislocation slipping during the deformation process is effectively hindered, and the matrix is strengthened because of an increase in the number density of precipitates and twins. The YS stabilizes at 4% pre-stretching condition, and then the evolution of YS is stable within the error bars.

(3) Tensile fracture surfaces of pre-stretched and subsequently aged specimens mainly exhibit a trans-granular cleavage fracture. The cleavage plane size of the pre-stretched specimens is smaller than that of non-pre-stretched specimen.

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**Author Contributions:** E.G. and L.W. conceived and designed the experiments; S.Z. and T.W. performed the experiments. T.W. analyzed the data. S.Z. wrote the paper.

**Conflicts of Interest:** The authors declare no conflict of interest.

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