Tensile fracture of as-cast and hot rolled Mg-Zn-Y alloy with long-period stacking phase

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Abstract: An experimental $Mg_{97}Zn_1Y_2$ (molar fraction, %) alloy was produced by rolling the as-cast alloy. The microstructure of the alloy is composed of the α-Mg (also marked as 2H-Mg with reference to long-period stacking structure according to hexagonal close packed structure) and long-period stacking (LPS) phase. Tensile tests of $Mg_{97}Zn_1Y_2$ alloy in comparison with pure Mg were conducted. The fracture morphologies of the tensile specimens were characterized and the microstructures near fracture surface were observed. The results show that the rolled $Mg_{97}Zn_1Y_2$ alloy shows a mixed fracture mode including dimples indicating a ductile fracture pattern and a small fraction of cleavage planes indicating a brittle fracture pattern, which is different from the single brittle fracture of the as-cast alloy. In addition, the plastic deformation is mainly from dislocations induced strain with small strengthening effect during plastic deformation in the as-cast $Mg_{97}Zn_1Y_2$ alloy, and the strain hardening rate is similar to that of the as-cast pure magnesium. The deformation mechanism of $Mg_{97}Zn_1Y_2$ alloy is different from that of the pure magnesium according to a metallographical observation that whether twins are found or not. The strengthening effect hardly exists in the rolled $Mg_{97}Zn_1Y_2$ alloy under the same dislocations induced strain, which is different from that of the as-cast alloy with moderate strengthening effect.
Key words: magnesium-yttrium-zinc alloy; long-period stacking phase, rolling; mechanical properties; fracture

1 Introduction

Due to strong demand for mass reduction of transportation vehicles for better fuel efficiency, magnesium alloys have recently received much research interest in applications to various structural components of automobile and aircraft industries[1]. Inter-metallic phases can be found in almost every magnesium alloy and play a very important role in optimizing micro-structure and mechanical properties[2]. Mg-Zn-RE-Zr system is regarded as a high strength magnesium alloy system because of evident strengthening effect of rare earth (RE) addition on magnesium alloys. PADEZHNOVA et al[3] found X-$Mg_{12}ZnY$ (molar fraction, %) as ternary equilibrium phase. LUO et al[4−5] identified X-$Mg_{12}ZnY$ phase as an 18R long-period

stacking (LPS) structure by electron diffraction technique. KAWAMURA et al[6−8] fabricated a high strength Mg-based $Mg_{97}Zn_1Y_2$ alloy with a novel LPS structure by rapid solidification (RS) techniques such as powder metallurgy and melt-spinning, and variation and formation process of LPS structures were reported later[9−11]. Since the strength of conventionally cast $Mg_{97}Zn_1Y_2$ alloy is lower than that of commercial AZ91 at room temperature, investigations to improve the properties of Mg alloys with LPS structure as second phase were focused on refining the LPS structure or applying deformation treatment[12−13]. While the tensile strength and fracture characteristics of $Mg_{97}Zn_1Y_2$ alloy, as one of the promising engineering magnesium alloys, have not been reported clearly. In the current work, tensile tests of the as-cast and the rolled $Mg_{97}Zn_1Y_2$ alloys were conducted and the results were

compared with those of the as-cast and rolled pure magnesium.

2 Experimental

The nominal composition of the alloy was $Mg_{97}Zn_1Y_2$ (molar fraction, %) in this study. The experimental conditions such as the casting and the hot-rolling process parameters can be obtained by referring to our previous work[14]. The ingot was prepared by melting Mg-19.9%Y (mass fraction) master alloy and pure magnesium in a graphite crucible at 775 °C with protecting atmosphere of SF_6+CO_2 gas mixture, and adding pure zinc particles after melting completely and holding at 775 °C for 30 min, and then casting the alloy into a steel mould of 190 mm× 95 mm×20 mm. Plate type samples for rolling were cut from the ingot. The samples for deformation were heat-treated at 400 °C for 30 min before rolling. The thickness of the plates after nine-pass hot-rolling was reduced by 74.8% at 12%−17% deformation strain per pass. All rolled specimens were etched with acetic-picric acid solution (5.5 mL CH_3COOH, 2.1 g picric acid, 5 mL water, 2 mL nitric acid and 90 mL alcohol). The tensile specimens were prepared from the as-cast and the rolled materials and the tensile axis of the rolled materials was kept to be along the rolling direction. The gauge size was 4 mm in width, 1 mm in thickness and 10 mm in length. Tensile tests were performed on an Instron tensile test machine at a strain rate of $6.7×10^{−4}$ $s^{−1}$. The fracture morphologies of tensile specimens were characterized using a scanning electron microscope (JSM5600, JEOL). The microstructures near fracture surface were observed by a laser optical microscope.

2.1 Microstructure of as-cast and hot-rolled $Mg_{97}Zn_1Y_2$ alloy

The microstructures of the as-cast and hot rolled $Mg_{97}Zn_1Y_2$ alloys were studied[14]. The microstructure of the as-cast $Mg_{97}Zn_1Y_2$ alloy consists of α-Mg dendrites and the X-$Mg_{12}ZnY$ phase. Besides the eutectic LPS phases on grain boundaries, part of which are devoiced eutectic phases, there are some fine acicular phases within the α-Mg phase and near grain boundaries which are precipitated from α-Mg along basal planes during cooling.

The deformation texture of the $Mg_{97}Zn_1Y_2$ alloy after hot rolling can be observed to be along rolling direction as presented in Ref.[15]. No deformation twins are found and deformation bands can nearly be visible after hot rolling. The existing weak contrast in the deformation microstructure confirms that the partial recrystallization behavior and the extended recovery occur during cooling after hot rolling at an elevated

temperature of 400 °C in the α-Mg matrix. The recrystallized grains are difficult to be identified because of small orientation difference between them.

2.2 Tensile test curves

The graphs of true stress—true strain obtained from tensile test are shown in Fig.1. The yield strengths of as-cast and rolled pure magnesium specimens are 18 and 108 MPa and those of $Mg_{97}Zn_1Y_2$ alloys are 85 and 282 MPa, respectively. The strengthening effect of as-cast $Mg_{97}Zn_1Y_2$ alloy with the LPS phase is significantly obvious in comparison with the as-cast pure magnesium. The working hardening resulting from the rolling deformation is different for pure magnesium and $Mg_{97}Zn_1Y_2$ alloy. The elongation prior to ultimate rupture of $Mg_{97}Zn_1Y_2$ alloy is lower than that of pure magnesium. The elongation of rolled $Mg_{97}Zn_1Y_2$ alloy presented in Fig.1 is slightly larger than that of the as-cast alloy.
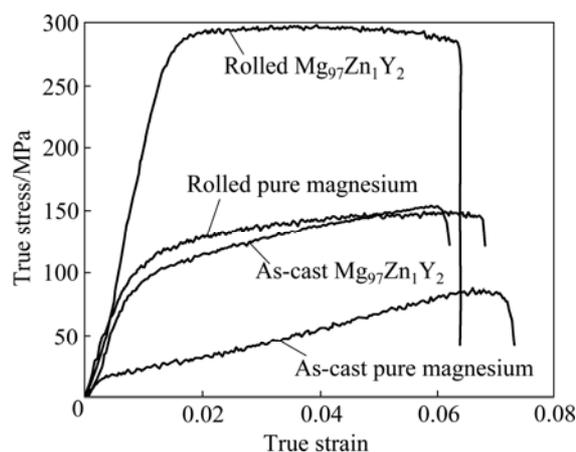

Fig.1 True stress—true strain curves of as-cast and rolled pure magnesium and $Mg_{97}Zn_1Y_2$ alloy in uniaxial tensile test at room temperature

2.3 Fracture morphologies

The fracture morphologies of the tensile specimens from as-cast and rolled pure magnesium and $Mg_{97}Zn_1Y_2$ alloy are shown in Fig.2. The fracture of the as-cast pure Mg is composed of two grain boundaries and cleavage planes. Therefore, a mixed brittle pattern of trans-granular and inter-granular cleavage fractures is its fracture mode (as shown in Fig.2(a)). The fracture of the rolled pure Mg is made up of tear ridges and vein pattern as shown in Fig.2 (b) and the fracture mode is almost trans-granular brittle pattern. Fig.2(c) shows the fracture characteristics of the as-cast $Mg_{97}Zn_1Y_2$ alloy, which can be described as a brittle quasi-cleavage mode including mainly cleavage planes from 2H-Mg phase and inter-granular interfaces between the 2H-Mg and 18R X-$Mg_{12}ZnY$ phases. The fracture of rolled $Mg_{97}Zn_1Y_2$ alloy is mainly characterized by dimples, a shear angle

existing from the midline of the sample to two surfaces of RD-TD section and localized tear ridges as shown in Fig.2(d), which indicates a ductile fracture mode attached by cleavage fracture.

2.4 Microstructures near fracture

The microstructures of all tensile samples are shown in Fig.3. There are many big primary twins in the as-cast pure magnesium, which are originated from the grain boundaries and extend to the interior of grains, and some secondary twins inside the primary twins as shown in Fig.3(a) and the inset image. Twins occur more severely on grain boundary than within the interior, thus a crack initiates on the grain boundary and even develops along it. The fracture is judged to originate from the grain boundary indicated by fine recrystallization grains which may be obtained by recrystallization during processing into the inset samples at 150 ˚C. Fig.3(b) shows that the
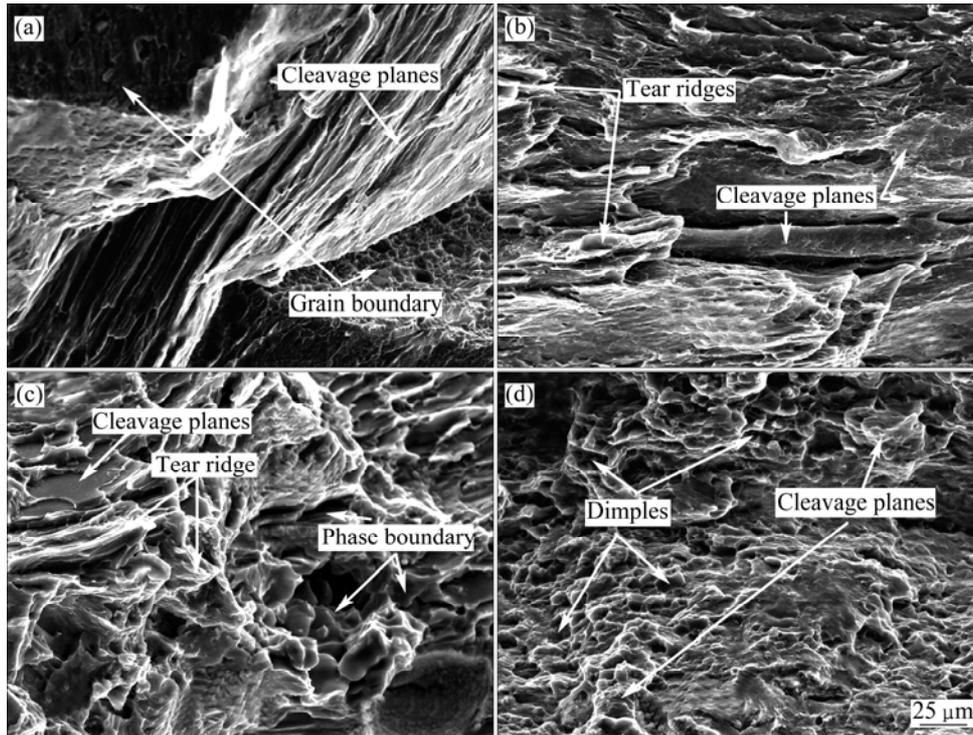

Fig.2 Fracture images of as-cast and rolled pure magnesium and Mg$_{97}$Zn$_1$Y$_2$ alloy using SEM: (a) As-cast pure magnesium; (b) Rolled pure magnesium; (c) As-cast Mg$_{97}$Zn$_1$Y$_2$ alloy; (d) Rolled Mg$_{97}$Zn$_1$Y$_2$ alloy
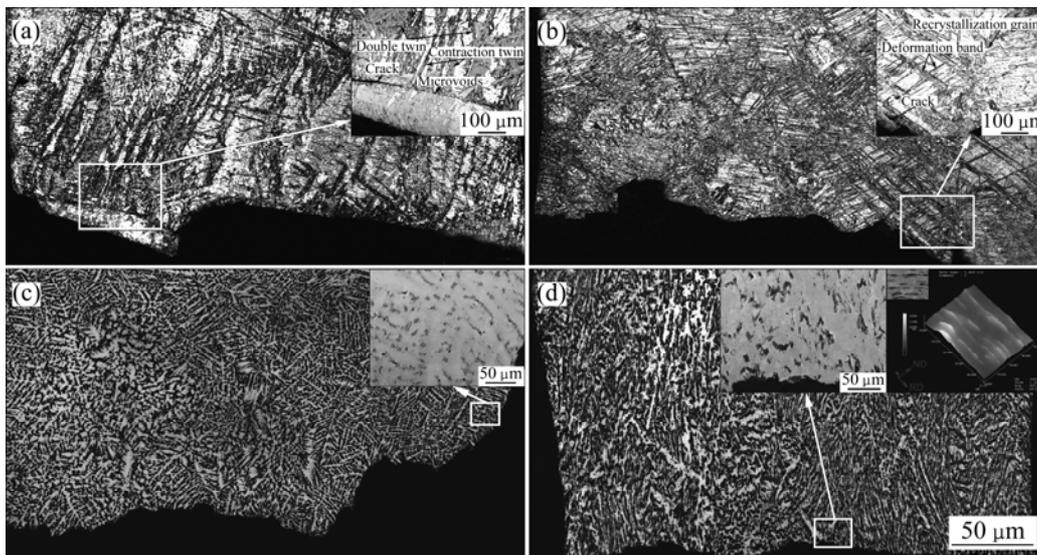

Fig.3 LOM micrographs including inset images near fracture of as-cast and rolled pure magnesium and Mg$_{97}$Zn$_1$Y$_2$ alloy by uniaxial tensile test (at TD-RD section of deformation samples): (a) As-cast pure magnesium; (b) Rolled pure magnesium; (c) As-cast Mg$_{97}$Zn$_1$Y$_2$ alloy; (d) Rolled Mg$_{97}$Zn$_1$Y$_2$ alloy with one inset image from a 3D LOM micrograph at ND-RD section

fracture of the rolled pure magnesium travels whether among the very fine recrystallized grains or along deformation bands. Twins are hardly found in the hot worked samples, and it can be understood as follows. The tensile strain comes from movement of dislocations within shear bands of deformation texture during nine passes of thermo- machining at 400 ˚C in which other slip systems than basal plane slide are activated and the mechanical twins are suppressed by refined grains and dense shear deformation bands. In Fig.3(c), the final fracture is ended by a shear angle near fringes of the sample. In case of the rolled $Mg_{97}Zn_1Y_2$ alloy, as shown in Fig.3(d), the neck prior to rupture occurs during tensile test based on the deformation texture. While a straight fracture in the transverse direction is imposed nearly with zigzag.

3 Discussion

When tensile test is conducted on a dual-phase or multi-phase alloy, crack initiation generally occurs at the phase interfaces. This is different from the crack initiation resulting from deformation twins in the as-cast pure magnesium. In case of the as-cast $Mg_{97}Zn_1Y_2$, the crack initiates at the interfaces between α-Mg and X-$Mg_{12}ZnY$, which is judged by different deformation mechanisms of two phases though they have an orientation relationship of $a_{LPS}//a_{2H}$, $b_{LPS}//b_{2H}$ and $c_{LPS}//c_{2H}$[5]. In addition, the dislocation types are different for α-Mg grains with or without LPS phases[16]. The fracture mode of as-cast $Mg_{97}Zn_1Y_2$ alloy is a quasi-cleavage pattern with cleavage plane across 2H-Mg grains and inter-granular pattern between two HCP structures as shown in Fig.2(c). Furthermore, twins are not visible for as-cast $Mg_{97}Zn_1Y_2$ alloy as shown in Fig.3(a). MATSUDA et al[17] pointed out that the LPS phase contributed strengthening effect to the as-cast $Mg_{97}Zn_1Y_2$ alloy and the twins were deflected or arrested in the region with high density of bundled LPS phases. In comparison with the tensile profile of the pure magnesium, the hardening effect induced by mechanical twins is not marked. A yield strength of 85 MPa is due to the mechanism of the second phase strengthening in as-cast $Mg_{97}Zn_1Y_2$ alloy, and the strain hardening of the as-cast $Mg_{97}Zn_1Y_2$ alloy in Fig.1 during tensile test is mainly from dislocation multiplication.

The fracture of the rolled pure Mg is mainly made up of vein pattern because of the hard orientation resulting from rolling. While, when the tensile test was performed upon rolled $Mg_{97}Zn_1Y_2$ alloy, a mixed fracture morphology including dimples, tear ridges and cleavage planes can be observed in Fig.2(d). The size of localized cleavage plane regions is much bigger than that of dimples, which is below 10 μm usually, and almost equal to that of the grains of as-cast alloy, which is about 30 μm. The mean grain size of rolled alloy at normal direction is about 6 μm according to the inset 3D LOM images of Fig.3(d), which is close to the size of above mentioned dimples. The deformation texture of the rolled $Mg_{97}Zn_1Y_2$ alloy can be observed clearly in Fig.3(d). The existence of cleavage planes and dimples shows the inhomogeneity of rolling strain in this experiment in connection with developed recovery and partial recrystallization in 2H-Mg. The dimples can be understood as that recrystallization grains or deformation substructures below 10 μm rotate its orientation to accommodate further strain in order to obtain a uniform strain in a whole grain. Of course, the α-Mg dendrites as matrix in the as-cast $Mg_{97}Zn_1Y_2$ alloy is believed to be a Mg-Y binary alloy, in which elevating deformation temperature and increasing yttrium content can reduce the effect of twins as a deformation mode and increase the activity of non-basal slip mode[18]. The addition of high level of yttrium combined with a high deformation temperature up to 400 ˚C leads to a near random distribution of dynamically recrystallized grains in contrast to strongly textured parent grains. The LPS phase is fragmented during rolling and its dissolution resulting from the reaction with deformation defects such as dislocations leads to precipitation at grain boundaries and in the cell wall of substructure. The dimples would develop from small second phase particles. Thus, the mechanism of ductile fracture is explained. The neck prior to rupture exists in tensile test rests from the ductile mode of fracture. The big α-Mg grains are difficult to randomly shift their orientation to accommodate the same strain as that of very small grains, therefore a localized cleavage plane indicating brittle fracture also occurs during tensile test in rolled $Mg_{97}Zn_1Y_2$ alloy. From Fig.1, a balance between strain hardening and strain softening is maintained before fracture and the curve of rolled $Mg_{97}Zn_1Y_2$ alloy is much smoother than that of the as-cast $Mg_{97}Zn_1Y_2$ alloy, which indicates that the dislocations generated by rolling are activated and started up, and that annihilation and occurrence of dislocations at grain boundaries keep balance until rupture. After deformation, the tensile strength at room temperature is upgraded from 85 MPa to 282 MPa and the mechanisms of strengthening are derived from deformation texture and grain refinement strengthening.

4 Conclusions

1) In as-cast $Mg_{97}Zn_1Y_2$ alloy, the fracture is originated from phase interface and developed by cleavage planes across the α-Mg grains. A brittle fracture mode is obtained, which is similar to the mixed brittle fracture mode of the inter-granular and the trans-granular

patterns in as-cast pure magnesium.

2) In rolled $Mg_{97}Zn_1Y_2$ alloy, dimples, localized cleavage planes and tear ridges indicate a ductile mixed fracture mode, which is different from the single brittle fracture of the as-cast alloy and the rolled pure magnesium.

3) The deformation mechanism during tensile test is changed from mechanical twins induced strain with obvious strengthening effect in the as-cast pure magnesium to the strain with slight strengthening in the rolled metal which is jointly induced by the dislocation movement and partial mechanical twins, to dislocations functionally induced strain in the as-cast $Mg_{97}Zn_1Y_2$ alloy, and to dislocations induced strain without remarkable strengthening effect in the rolled alloy.

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