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The effect of homogenization on microstructure and hot ductility behaviour of AZ91 magnesium alloy

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Abstract

Grade AZ91 is a casting magnesium alloy for ambient temperature applications. Because of good castability, formability and also for economical considerations, this alloy has vast applications in automotive industries. Microstructure of this alloy after casting includes α solid solution, β precipitates ($Mg_{17}Al_{12}$) in grain boundaries and $\alpha+\beta$ eutectic phase. Morphology, distribution and the amount of precipitates play the crucial role during deformation process and their behaviour can be controlled by selection the appropriate heat treatment procedure. In this study, homogenization heat treatment on AZ91 alloy at 415 °C for 9, 18 and 24 hours was performed and the influence of heat treatment on hot deformation behaviour was investigated. Hot tensile test in the temperature range of 380–440 °C and initial strain rate of 0.001 and 0.1 s^{-1} was carried out and the results revealed that the highest tensile strength and simultaneously the maximum ductility were obtained in the sample held at 415 °C for 24 hours and drawn at 400 °C with initial strain rate of 0.1 s^{-1}. Furthermore, microstructure and the extension of precipitates presence after homogenization heat treatment also were investigated by metallographical examination.

Key words: AZ91, homogenization heat treatment, β precipitates, hot deformation, microstructure

1. Introduction

AZ91 alloy contains approximately 9 and 1 wt.% of aluminium and zinc, respectively, as alloying elements. Under equilibrium solidification conditions, microstructure of this alloy contains α solid solution with β precipitates ($Mg_{17}Al_{12}$) in the matrix. However, under non-equilibrium solidification conditions, microstructure of the alloy has a dendritic form in which brittle β phase exists between dendrite arms. In addition to dendritic structure, other non-homogeneities can also be observed in casting specimen, which can cause brittleness and reducing formability [1–4]. Therefore, to improve the formability properties and preparing the alloy for subsequent processes such as rolling, extrusion, etc., homogenization treatment is inevitable. The most important parameters during homogenization are holding temperature, holding time and cooling rate of the specimen to the room temperature. For instance, increasing holding time during homogenization increases tensile strength and decreases the ductility [5].

In various research works, which have been carried out on magnesium alloys including AZ91, different homogenization processes were performed. Yakubtsov et al. studied the influence of heat treatment on tensile properties of AZ80 alloy at room temperature and showed that dissolving β precipitates causes an increase in ductility in room temperature [6]. Čížek et al. investigated the microstructure and the extension of recrystallization by deformation after casting and heat treatment condition T4 [7]. Their results demonstrated that DRX value for the T4 specimen was remarkably higher than the cast one. Kiełbus et al., on the other hand, showed that homogenization process does not change significantly the hardness value of cast specimen [2]. In spite of these outcomes, however, researches on studying the influence of ho-

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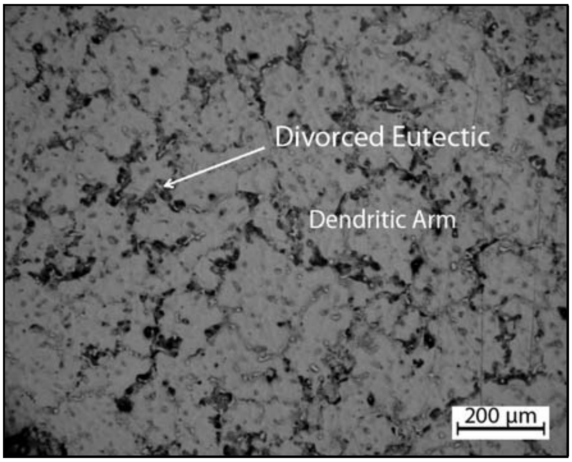


Fig. 1. As-cast ingot microstructure.

Fig. 2. Sample dimensions for tensile test.

mogenization parameters on hot deformation properties of AZ91 are scarcely available. In this study, influence of homogenization holding time on microstructure and precipitates of AZ91 is investigated. Moreover, to study the influence of homogenization process on deformation behaviour and the ductility of AZ91, hot tensile test was also performed.

2. Materials and methods

AZ91 ingot with the following chemical composition (in weight percent) was used: Al 7.69, Zn 0.5, Si 0.03, Cu 0.007, Fe 0.002, Mg bal. The ingot was obtained by gravitational casting process and Fig. 1 demonstrates the primary microstructure of ingot cast as a dendritic structure. The $\alpha + \beta$ eutectic phase exists between dendrites, which is surrounded by the matrix as magnesium supersaturated α phase. To

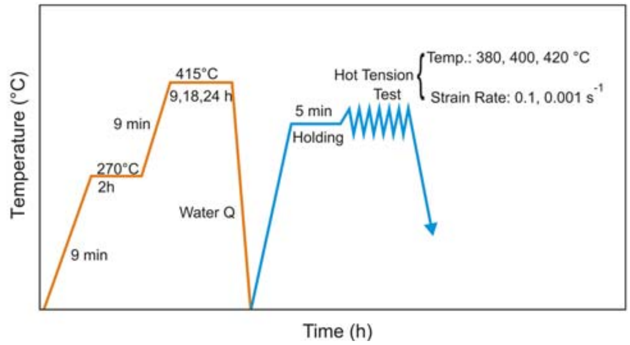


Fig. 3. Scheme of total homogenization process.

perform the experiments, hot tensile subsized specimen based on ASTM-B 557M was extracted and machined from primary ingot in longitudinal direction, as shown in Fig. 2. Performed homogenization process was shown schematically in Fig. 3. An electric furnace with SiC element with temperature accuracy of ±7 ℃ was used. As can be seen, a two-stage homogenization heat treatment was carried out. First cycle was based on holding the specimen at 270 ℃ for 2 hours. This stage was performed to avoid liquefying of low melting point eutectic phases and formation porosity [5]. The second stage was holding the specimen at 415 ℃ for 9, 18 and 24 hours. After the homogenization operation, the samples were water quenched.

To illustrate the influence of homogenization process on hot deformability, hot tensile test at 380, 400 and 420 ℃ with constant stage speed of 0.02 and $2\ \mathrm{mm\,s^{-1}}$ was performed. Zwick/Roll tensile test instrument equipped by an electric furnace with accuracy of ±5 ℃ was used. Considering the high conductivity of magnesium alloys, the small size of samples and the suggested previous works [1–4], 5 minutes are required for temperature equalization. Therefore, to equalize the sample temperature, prior to run the tensile test, the specimens were held for 5 minutes at the measured temperature, and then the hot tensile test was carried out. The specimen was then water quenched. Prior and after homogenization, the microstructure of specimens was examined by normal optical microscopy.

3. Results and discussion

3.1. Microstructure

The microstructure of primary ingot, shown in the previous section in Fig. 1, contains α phase, a supersaturated magnesium phase as matrix, and eutectic $\alpha + \beta$ phase formed among the dendrite arms. The space between the dendrite arms is shown in details in Fig. 4. In this figure, α phase, eutectic $\alpha + \beta$ phase,

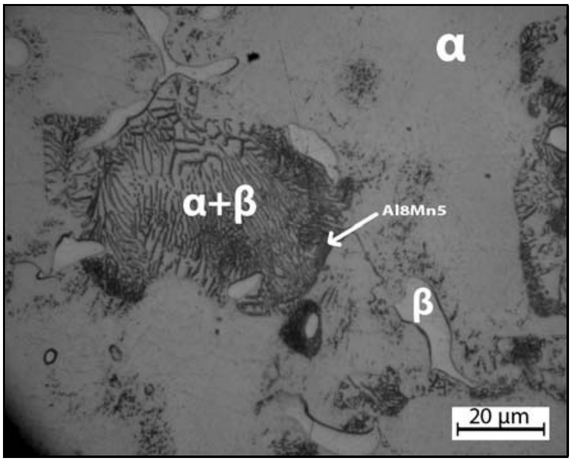


Fig. 4. Microstructure of space between dendritic arms in as-cast specimen.

$Mg_{17}Al_{12}$ as β precipitates and Al_8Mn_5 as Al-Mn intermetallic precipitates can be clearly observed. The β and Al_8Mn_5 precipitates are distributed arbitrarily in α matrix. While Al_8Mn_5 precipitates are generally small and round in shape, the β phase precipitates reveal different shape and size.

Microstructure of specimen after homogenization is shown in Fig. 5. Figure 5A represents the microstructure of the specimen after 9 hours homogenization. In comparison with the cast sample, in this sample, eutectic $\alpha + \beta$ phase and fine β phase precipitates were dissolved. However, the coarse β phase precipitates did not change significantly. Figure 5B represents the microstructure after 18 hours homogenization. As can be seen, dendritic structure entirely disappeared and equiaxed grains were formed. A vast amount of β phase precipitates were also dissolved. Figure 5C represents the microstructure after 24 hours homogenization. The average grain size is about 250 µm and β phase precipitates could not be observed at magnification 100×. Higher magnification image in Fig. 6 reveals that even homogenization for 24 hours can not completely dissolve the β phase precipitates. It has been previously reported that the β phase precipitates begin to dissolve in the matrix from the temperature 330 ℃ [8]. Its dissolving rate depends on the homogenization time and temperature. At the temperature 415 ℃, after 24 hours, there is still a small fraction of undissolved β phase precipitates. The remaining undissolved β phase precipitates are very small in size, placed in grain boundaries. In

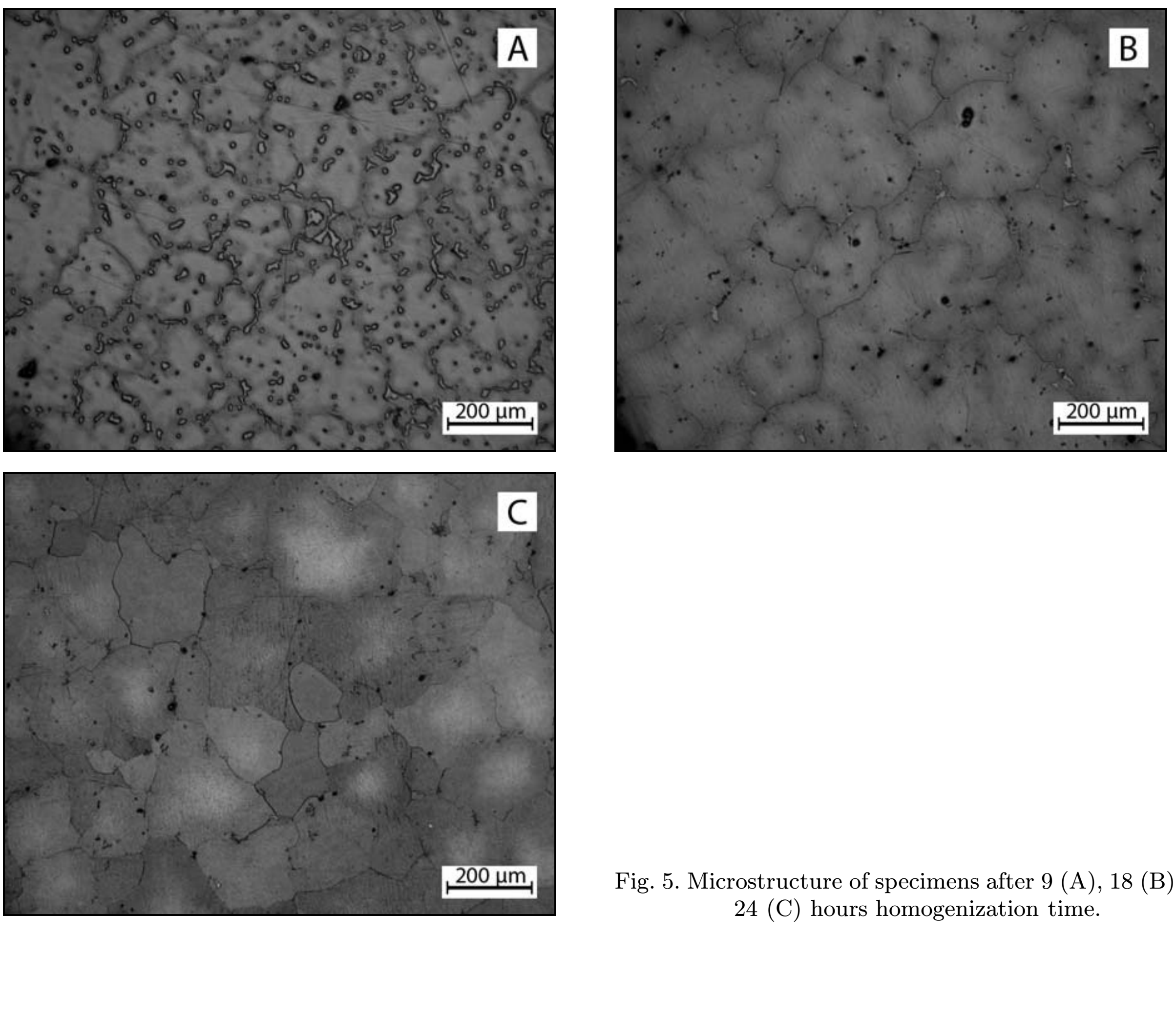


Fig. 5. Microstructure of specimens after 9 (A), 18 (B) and 24 (C) hours homogenization time.

summary, increasing the homogenization time at fixed temperature led to dissolving the eutectic $\alpha+\beta$ phase, coarse and worm-shape β phase precipitates in the α matrix phase. Noticeably, increasing the homogenization holding time does not affect significantly the dissolving or coarsening of Al_8Mn_5 as shown in Fig. 7.

3.2. Engineering stress-strain curve

To investigate the influence of homogenization process on hot deformability and obtaining the optimum homogenization holding time, hot tensile test was carried. Figure 8 represents engineering stress-strain curves for the specimens after 9, 18 and 24 hours homogenization at 415 °C. Clearly, by increasing the strain, the stress also increased which was an indication of an initial strain hardening until reaching a peak value. Consequently, a partial softening and then, at the final stage, the fracture occurs. In all conditions, by increasing the temperature, flow stress decreased. This is because dislocations movement is more feasible at higher temperatures. Moreover, by increasing the strain rate, flow stress level also increases. More interestingly, by increasing the homogenization holding time, flow stress at different tensile test temperatures and various strain rates increased. This can be related to increasing β phase precipitates dissolving

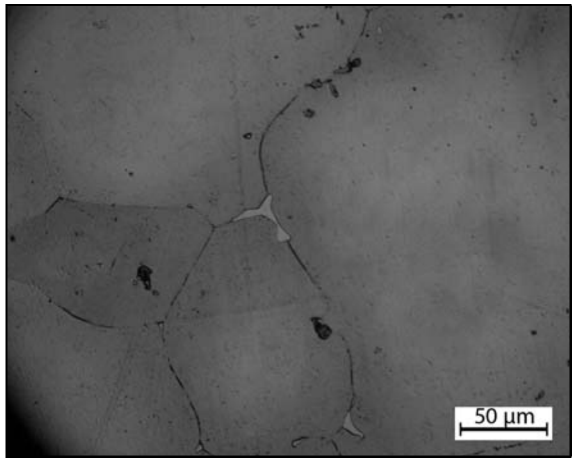


Fig. 6. Size and distribution of precipitates in specimens after 24 hours homogenization time.

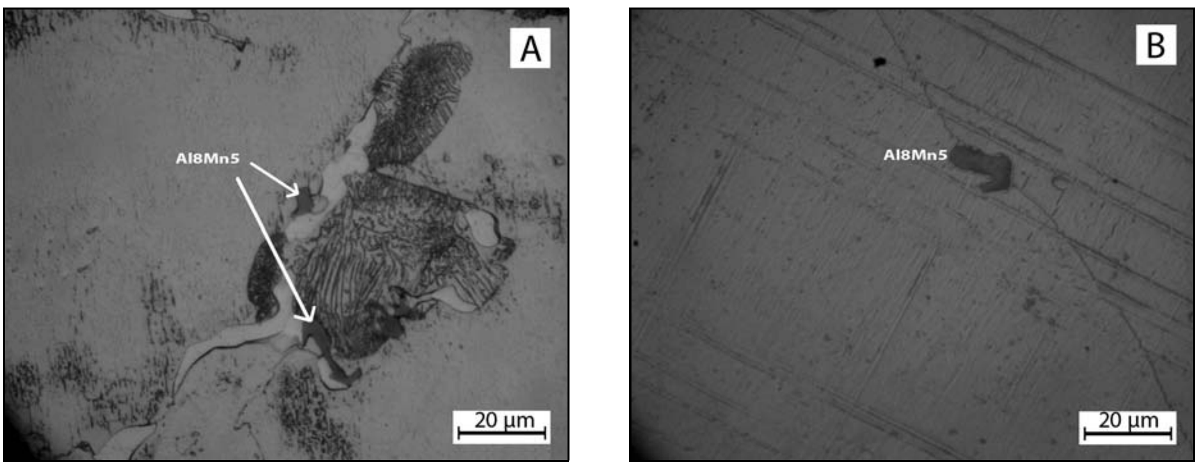


Fig. 7. Distribution of Al_8Mn_5 precipitates before homogenization (A) and after 24 hours homogenization time (B).

Table 1. Summary of hot tensile properties of AZ91

		380 °C				400 °C				420 °C			
HT*	Strain rate (s^{-1})	UTS (MPa)	YS (MPa)	ε_{UTS}	D** (%)	UTS (MPa)	YS (MPa)	ε_{UTS}	D (%)	UTS (MPa)	YS (MPa)	$h\varepsilon_{UTS}$	D (%)
9 h	0.001	62	52.5	0.016	1.45	60	53	0.014	1	49	44	0.014	1.15
	0.1	82.5	72	0.017	0.95	80	62	0.023	1.88	65	59	0.015	0.75
18 h	0.001	70	65.3	0.015	1.45	63	53	0.017	1.7	52	50	0.014	1
	0.1	85	67.2	0.023	2.05	79	60	0.031	3	79	60	0.021	2
24 h	0.001	78	67.2	0.019	1.8	69	61.5	0.018	1.6	61	54	0.016	1.45
	0.1	94	76	0.022	1.45	86	70	0.052	3.6	93	70	0.031	2.7

* Homogenization Time, ** Ductility

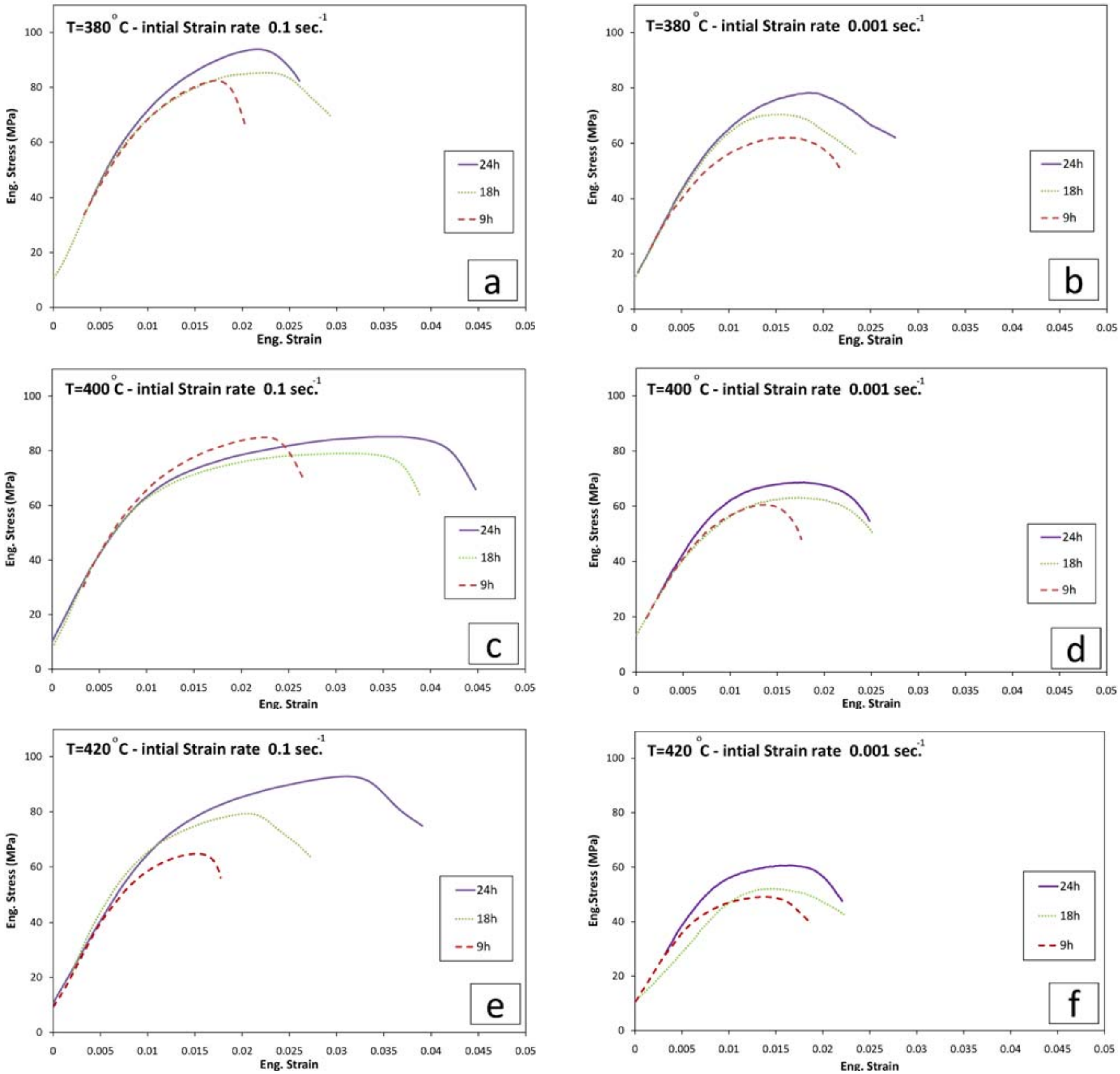
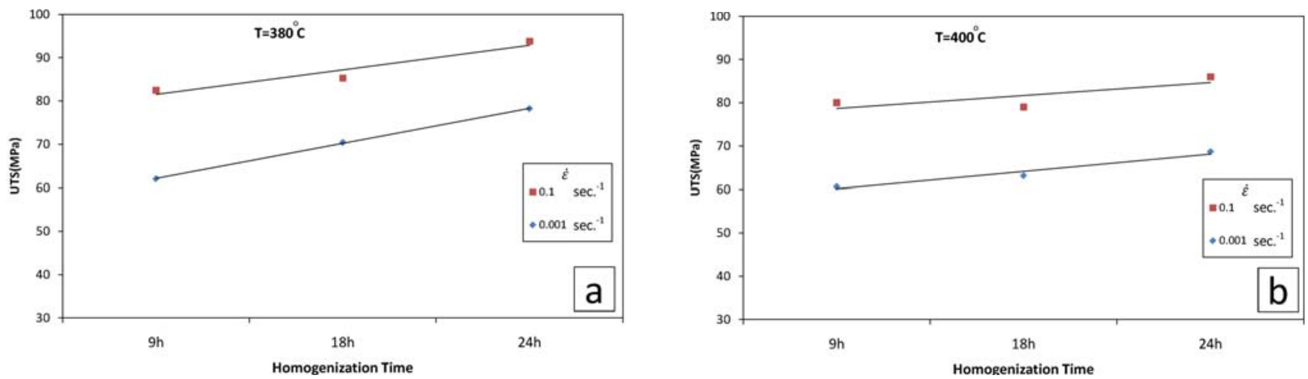


Fig. 8a–f. Engineering stress-strain curves of homogenized specimens in various test temperatures and strain rates.

by increasing homogenization holding time which led to an increase in the matrix strength. In magnesium alloys, deformation at high temperature is sustained on non-basal slip systems activation. It has been previously proved that activation of these slip systems is strongly related to the amount of dissolved atoms in the matrix phase [9]. Considering this fact illuminates that higher strain rate confines the time for the dissolved atoms to get away from the matrix, therefore the flow stress increases. The measured properties: tensile strength, corresponding strain, ductility, yield stress have been summarized in Table 1. Ultimate tensile strength (σ_{UTS}) is a parameter, which can be affected by the homogenization parameters. As shown in Fig. 9, by increasing the homogenization holding time, in all test temperatures and strain rates, σ_{UTS} value increases. The presence of small distributed precipitates and dissolved atoms in matrix restricts the dislocations movement and increases the strength. Increasing the temperature and simultaneously applying the stress on structure makes dissolved atoms more feasible to escape from the matrix. These two factors also assist β phase precipitates to grow up faster. Consequently, the presence of coarse β phase precipitates in microstructure significantly reduces the strength level.

Interestingly, in all samples, by increasing the test temperature and strain rate, the value of strain re-



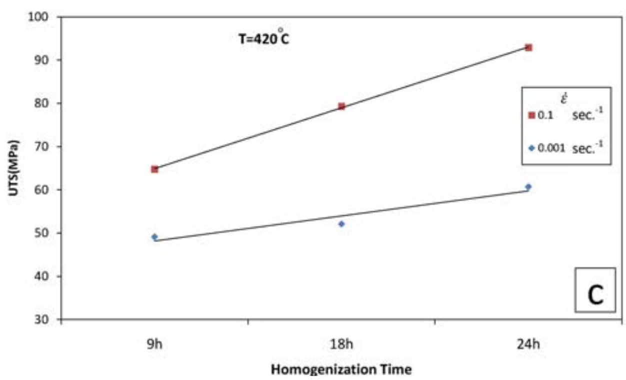
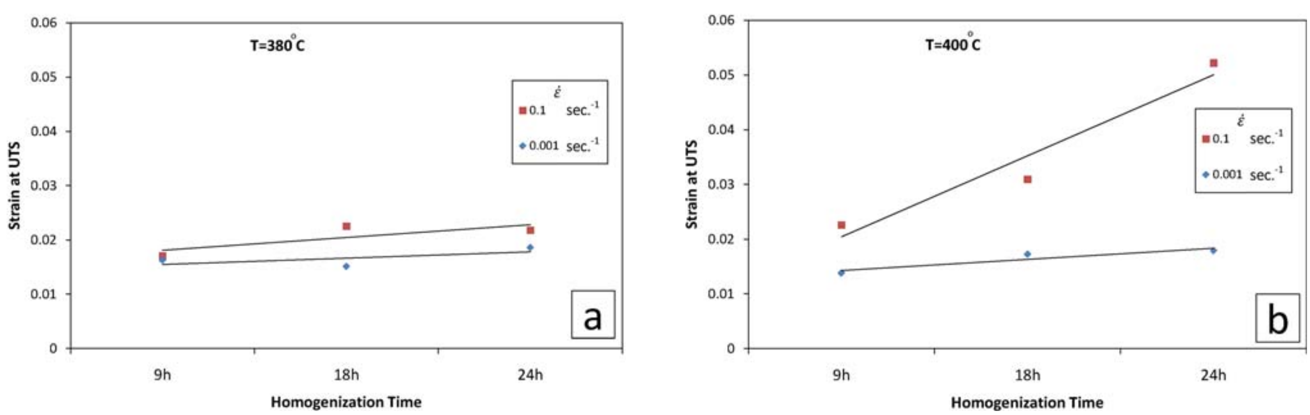


Fig. 9a,b,c. Effect of homogenization time on the UTS in various test temperatures and strain rates.



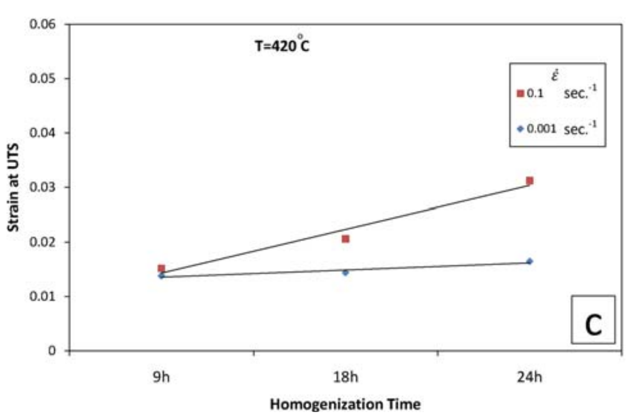


Fig. 10a,b,c. Effect of homogenization time on the strain of UTS in various test temperatures and strain rates.

corded at σ_{UTS} increases (Fig. 10). However, this increase in test temperature of 380 ℃ is not significant. Generally, increasing the homogenization holding time does not change significantly the strain value recorded at σ_{UTS}. Increasing the test temperature and strain rate also does not change significantly the strain

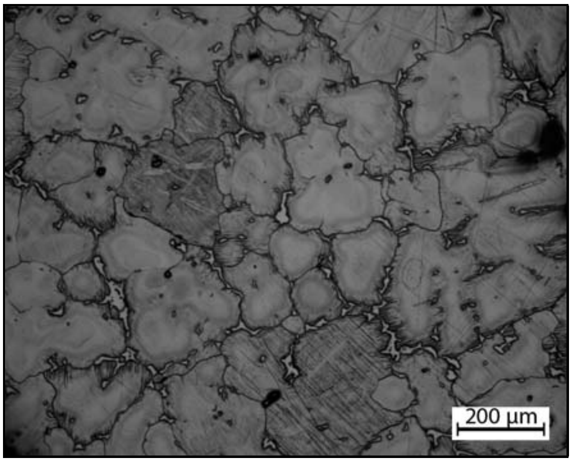


Fig. 11. Microstructure of deformation zone in specimen homogenized for 24 hours, tensile tested at temperature 380 ℃ with strain rate of 0.001 s^{-1}.

value recorded at σ_{UTS}. Tensile strength and ductility at strain rate 0.001 s^{-1} are less than the ones in 0.1 s^{-1}. In the sample which has been tested at higher strain rate, the time for the dissolved atoms to run away from the matrix is less and the volume of β phase precipitates formed by dynamic precipitation is also less. In several investigations, dynamic formation of β phase precipitates has been reported [3, 10, 11]. Longitudinal cross section of deformed area for the specimen after 24 hours homogenization holding time, tested at 380 ℃ with strain rate of 0.001 s^{-1} was shown in Fig. 11. The image indicates formation of new β phase precipitates in grain boundaries and triple cross points. Although Kumar et al. showed that the formation of β phase precipitates did not affect significantly the torsion behaviour of AZ91 [12], however, other research showed that the microcracks in AZ91 initiated from an interface between β phase precipitates and the matrix [3], and connecting of these microcracks led to the fracture occurrence. According to the present study, formation and increasing the volume of β phase precipitates has significant effect on the tensile behaviour of AZ91.

On the other hand, in all conditions, the value of recorded strain at σ_{UTS} point was remarkably small and the fracture quickly occurred after σ_{UTS} point. The character of fracture in this alloy and in these test temperatures is a quasi-cleavage type [13]. The nature of occurred fracture can be associated to following parameters: A – dynamic precipitates at grain boundaries and triple cross points causing stress concentration in these points, B – remaining precipitates from homogenization process and C – precipitates formed during preheating stage. Taking into account that the test temperature was laid in single phase region of AZ91, some studies showed that during preheating stage at the vicinity of grain boundaries, formation and growth of very small and lath shape of β phase precipitates could take place [11]. Considering image in Fig. 9 which indicates the formation of large amount of new β phase precipitates (comparing Fig. 5c and Fig. 11), it can be concluded that during hot tensile test of AZ91, presence of dynamic β phase precipitates definitely has foremost effect on fracture behaviour.

4. Conclusions

The effect of homogenization on microstructure and hot ductility behaviour of AZ91 magnesium alloy has been investigated. The conclusions can be summarized as follows:

1. By increasing the homogenization holding time at 415 ℃, the amount of dissolved β phase precipitated in Mg matrix increases.
2. By increasing the strain rate in hot tensile test, due to reducing dynamic precipitation time, both strength and ductility values increase.
3. Too low hot ductility was attributed to the dynamic precipitation process.
4. Utilizing 24 hours homogenization holding time, ultimate tensile strength increases due to dissolving β phase precipitates in the Mg matrix.

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