

Creep behaviour and micro-structural evolution in Mg-5Zn-3Al-0.2Mn alloy

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Abstract

In a standard creep testing machine, the creep behavior and microstructural evolution of a gravity-cast Mg-5Zn-3Al-0.2Mn (wt%) alloy were studied under conditions of stress and temperature ranging from 70 MPa to 110 MPa for 100 hours. The findings demonstrate that when temperature and stress increase, so does the creep strain of the Mg-5Zn-3Al-0.2Mn alloy. The Mg-5Zn-3Al-0.2Mn alloy has exceptional creep resistance under 523 K, with steady-state creep rates at 523K/70 MPa and 523K/90 MPa being just $2.3611108s^{-1}$ and $3.9194108 s^{-1}$, respectively. The creep strain after 100 h is only 0.735% and 1.751%. When the stretching temperature is constant, the alloy's grain size grows as tensile strength rises. When temperature rises while maintaining the same tensile strength, the grain size of the alloy obviously grows. Under the creep circumstances of 70 MPa-110 MPa, the creep stress exponents (n) are 0.84 at 473 K, 2.58 at 498 K, and 5.13 at 523 K. Additionally, as stress levels rise, the creep activation energy Q_c rises as well. For 70 MPa, 90 MPa, and 110 MPa, respectively, the creep activation energies Q_c are 96.85 kJ mol⁻¹, 113.48 kJ mol⁻¹, and 181.24 kJ mol⁻¹. According to the results of stress exponents and creep activation energies, when a Mg-5Zn-3Al-0.2Mn alloy ages, the creep mechanism shifts from grain boundary slip to dislocation slip as creep temperature and stress rise.

1. Introduction

Low density, high stiffness and specific strength, strong damping, electromagnetic shielding, and high recovery are all benefits of magnesium alloys [1-6]. The widespread use of magnesium alloys at high temperature is severely constrained by the classic magnesium alloys' weak creep resistance at high temperatures and their maximum allowable working temperature of 473 K [2-7]. As a result, one of the key areas of magnesium alloy study is enhancing the alloy's creep resistance at high temperatures. One of the simplest and most straightforward methods for enhancing the characteristics of magnesium alloys is alloying [2-9]. AZ91D and AM60 Mg-Al alloys cannot be used in environments with temperatures higher than 120 °C [10-11]. According to studies, the Al atoms' solubility causes the unfavorable dynamic precipitation of the phase (Mg₁₇Al₁₂), which is the cause of the Mg-Al alloys' impaired anti-creep performance [12-15].

As a result, the alloy Mg-5%Zn-3%Al-0.2%Mn (mass fraction) was created. It was intended for the Mg-5%Zn-3%Al-0.2%Mn alloy to have high zinc contents, and the Al content has been reduced to about 3%, which is far below the solubility of Al in the magnesium matrix. In order to determine the relationship between the creep resistance capability and the thermo-

stability, location, and crystalline morphology of second phases in magnesium alloys with a high zinc content, the creep behaviors and microstructures of as-cast and pre-solution treated Mg-5%Zn-3%Al-0.2%Mn alloys were compared. This work also examined the morphology and chemical composition changes in intermetallic compounds that occur during creep deformation.

2. Experimental Procedure

The experimental alloy was made from high pure Mg (99.9 wt%) and Zn (99.95 wt%), Mg15wt% Al, and Mg30wt% Mn master alloy ingots. The alloy's target compositions were Mg-5Zn-3Al-0.2Mn in wt%. First, a steel crucible was used to melt pure magnesium ingots while being shielded by a gas mixture of CO₂ and 2 vol% SF₆. Second, at 730 ± 5 °C, pure Zn and Mg15 wt% Al master alloy were added, and the mixture was thoroughly stirred for 10 to 15 minutes. The melt temperature was raised to 760 ± 5 °C and maintained for 10 minutes, after which Mg30 wt% Mn master alloy was added and thoroughly stirred for 10 minutes. The melt temperature was lowered to 720 ± 5 °C, then held still for 30–40 minutes. The melts were then poured into a copper mold that had been heated to 250 °C. Using an inductivity coupled plasma atomic emission spectroscopy (ICP-AES), the practical chemical composition of the experimental alloy was found to be Mg5.16Zn3.62Al0.23Mn (wt%).

Tensile creep specimens with gage sections ranging from 6 to 75 mm were created using standard lathe machining methods. The RC-1130 creep testing machine is used to conduct the creep tests. Testing is done at 473 K, 498 K, and 523 K. With a creep

time of 100 hours, the experimental stresses are 70 MPa, 90 MPa, and 110 MPa. For each condition, three samples were measured, and the average was computed. Each creep time-strain curve was used to calculate the total creep strain and steady state creep rate.

The microstructures were examined using optical microscopy (OM Olympus GX71), field emission scanning electron microscopy (FESEM Hitachi S4800), transmission electron microscopy (TEM FEI-TECNAI G2), and an X-ray energy-dispersive spectrometer (EDS EDAX GENESIS) analysis system. Following a traditional mechanical polishing procedure, samples for OM and SEM analyses were etched in a solution of 35 ml ethanol, 2 ml acetic acid, 2.2 g picric acid, and 3 ml deionized water. Punching 3mm diameter discs with 30–50 μm in thickness produced the TEM samples. Additionally, these foils were thinned using a low-energy ion beam and a liquid nitrogen cooling system (milling parameters: Ar, 4.6 kV, 90 min).

3. Results and Discussions

3.1. Microstructure

SEM images of the as-cast microstructure of the Mg-5Zn-3Al-0.2Mn alloy are shown in Fig. 1. In the interdendritic regions, a network of phase (Mg₃₂(Al,Zn)₄₉, BCC, a = 1.416 nm) has formed between dendrites with dendrite arm spacing of about 30–40 μm. The secondary intermetallic particles made up about 16.6% of the total volume, according to the measurements.

The SEM images and EDS analysis of the Mg-5Zn-3Al-0.2Mn alloy after 100 hours of creeping at 473 K, 498 K, 523 K/70 MPa, 90 MPa, and 110 MPa are shown in Figure 2 and Table 1. The observed plane is parallel to the tensile strength direction. The diagram shows that the black -Mg matrix and white particle second phase make up the majority of the alloy's microstructure. The grain size clearly increases with an

increase in tensile strength when the creep temperature is constant, and similarly, the grain size clearly increases with an increase in temperature. The above structure has changed, and the creep properties have also changed. As the creep specimens at 473K and 498K are not broken, no macro cracks are found in the microstructures (figures 2a - b).

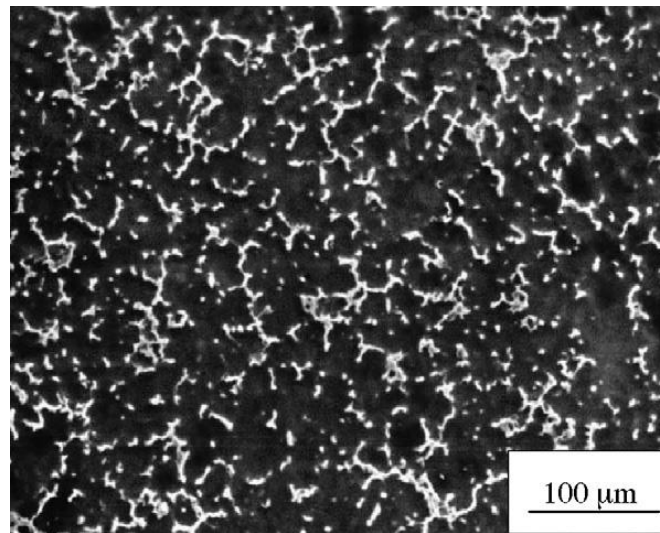


Fig 1: SEM micrograph of as-cast Mg-5Zn-3Al-0.2Mn alloy showing interdendritic τ phase.

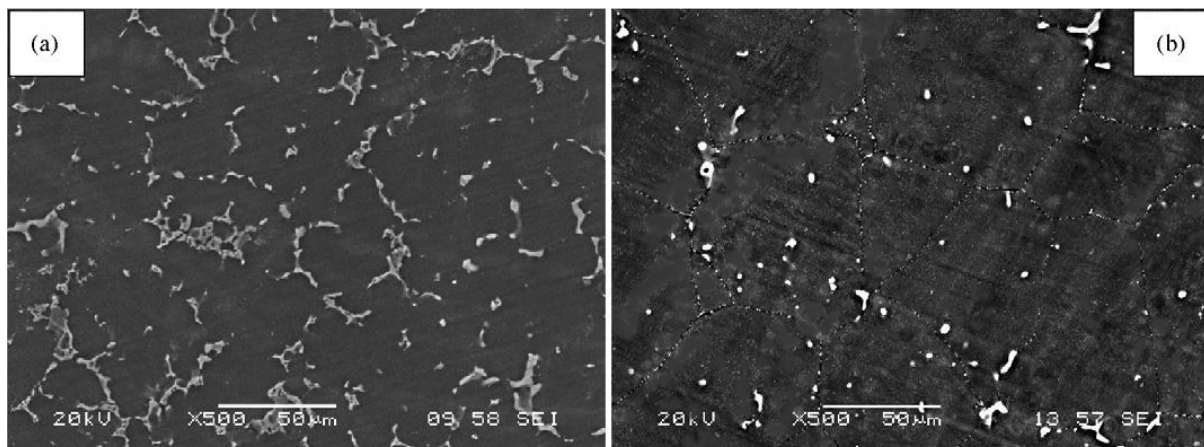


Fig 2: SEM photographs of Mg-5Zn-3Al-0.2Mn alloy crept for 100 h at different temperature under different stress a) 70 MPa and b) 110 MPa.

3.2. Creep Behaviour

Figure 3 depicts the Mg-5Zn-3Al-0.2Mn alloy's creep curves at 473 K, 498 K, 523 K/70 MPa, 90 MPa, and 110 MPa. The primary creep stage and steady-state creep stage are both included in the creep curve. The linear steady-state creep stage, with a low steady-state creep rate, follows the primary creep stage, which lasts for about 10 to 15 hours. Conclusion: The deceleration creep stage lengthens with increasing creep stress at 473 K, the alloy's steady creep rate increases gradually, and the creep strain at 100 h increases correspondingly. However, at 100 hours, the steady-state creep rate and creep strain are negligible. The creep strain at 100 hours is only 0.493%, despite the fact that the steady-state creep rate is only 3.3333109s^{-1} at 473K/110 MPa. It has a lower steady-state creep rate than the

Table 2 summarizes the creep characteristics of the alloy Mg-5Zn-3Al-0.2Mn at 473 K, 498 K, 523 K/70 MPa, 90 MPa, and 110 MPa. Generally speaking, the alloy's 100 h creep strain and steady creep rate increase gradually as the creep temperature rises while maintaining the same tensile strength. This is primarily because magnesium alloy creep at high temperatures is a heat-activated process. The slip mechanism of the magnesium alloy changes during the creep process, creep, and steady creep when the temperature rises to a certain level.

commercial WE43 alloy at 523K/60 MPa (4.46108), even at 523K/90 MPa (3.9194108) [16]. As a result, the alloy Mg-5Zn-3Al-0.2Mn exhibits excellent creep resistance at 473 K. The primary creep stage and steady-state creep stage at 498 K are additional stages of the creep curve. At 70 MPa, 90 MPa, and 110 MPa, the creep rate of the alloy Mg-5Zn-3Al-0.2Mn is $4.6388\ 109\ \text{s}^{-1}$, $8.6666\ 109\ \text{s}^{-1}$, and $1.5250\ 108\ \text{s}^{-1}$, respectively. In comparison to 473 K, the creep rate of the alloy Mg-5Zn-3Al-0.2Mn is significantly higher. Creep curves at 110 MPa 473-523 K are depicted in Figure 3(c). When the temperature is between 473 K and 498 K, it can be seen that the alloy's creep is still in the primary creep stage and steady-state creep, but the amount of deformation increases noticeably. The alloy has a 42-hour creep life when the temperature is 523 K.

Table 1: EDS results of the second phases in wt%.

Elements (wt%)	x(Mg)/%	x(Y)/%	x(Nd)/%	x(Sm)/%	x(Zr)/%
figure 4(a)	44.14	31.13	03.74	20.25	00.74
figure 4(b)	48.53	29.62	01.31	19.94	00.60
figure 4(c)	45.51	29.63	01.10	22.98	00.63
figure 4(d)	40.12	25.14	05.86	28.45	00.43
figure 4(e)	45.45	33.86	02.74	17.58	00.37
figure 4(f)	44.72	24.56	03.67	26.66	00.39
figure 4(g)	45.54	26.70	03.66	23.48	00.62
figure 4(h)	47.47	24.56	06.29	20.99	00.69
figure 4(i)	46.67	27.68	03.69	21.43	00.53

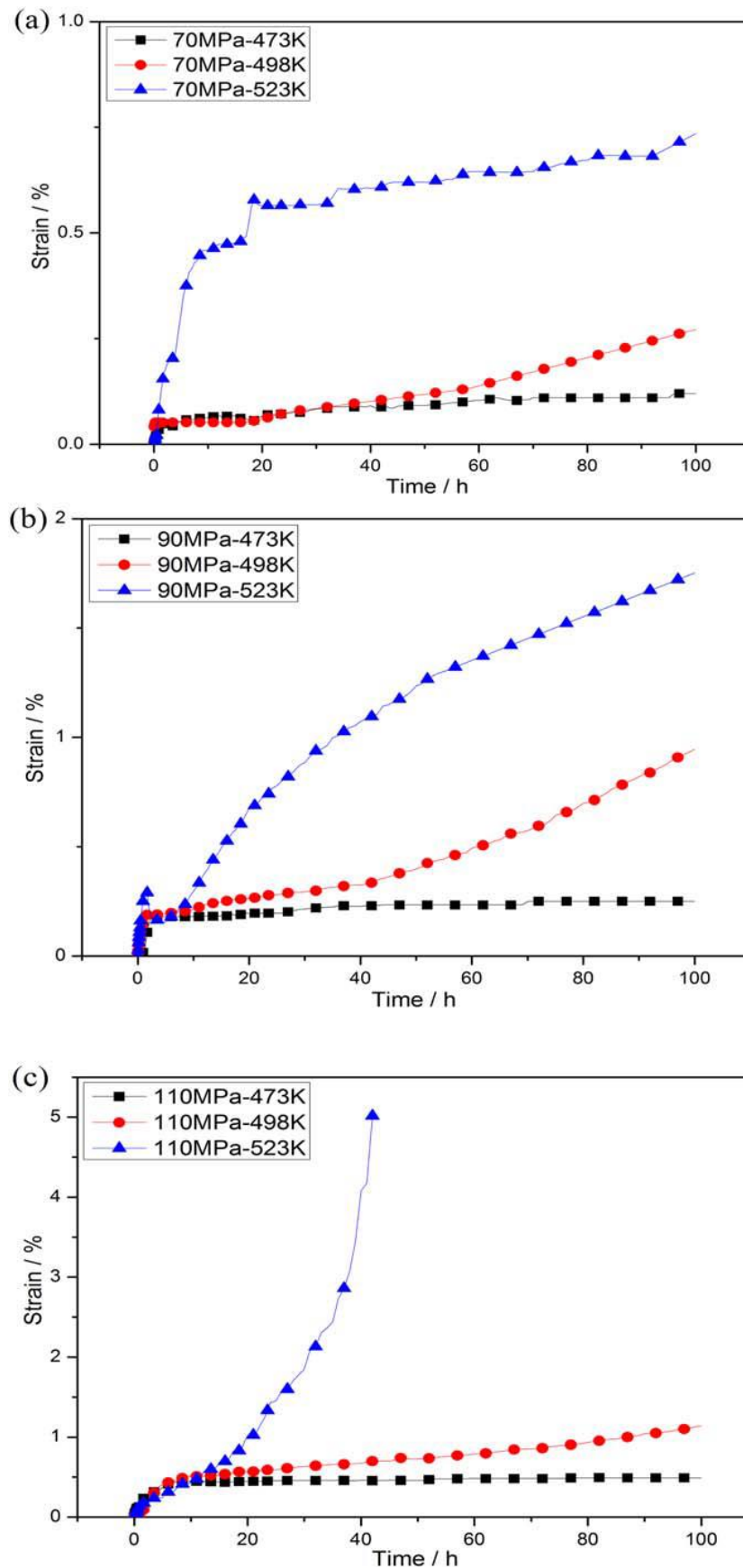


Fig 3: Creep curves of Mg-5Zn-3Al-0.2Mn alloy. (a) 70 MPa 473–523K) (b) 90 MPa 473–523K) (c) 110 MPa 473–523K).

Significant growth in the rate was observed [16–21]. The 100-hour creep strain and constant creep rate of the alloy gradually increase at the same temperature as the creep stress increases. This is because as the tensile strength rises during the creep process, a local stress yielding phenomenon happens when a certain value is exceeded. Larger steady-state creep rates and creep deformation values are the result, as more dislocations that were initially pinned in the grain boundaries and hard points of the crystal are restarted [21–23].

The steady-state creep strain rate of the alloy under various stresses and temperatures is compared, as shown in figures 4(a) and (b), in order to more clearly illustrate how temperature and stress affect the creep properties of the alloy. The graph shows that, at a particular creep temperature, the creep strain rate rises as the creep stress rises. According to the analysis of the front creep curve, when the creep stress is constant, the creep strain rate rises as the temperature rises.

Figure 5 depicts a graph that was created based on the $\ln\dot{\epsilon}$ - $\ln\sigma$ value of the Mg-5Zn-3Al-0.2Mn alloy during tensile creep at

The stress exponent n reflects the extent to which the steady creep rate of the alloy is affected by the loading stress level [16-30]. The higher the value of n , the faster the steady creep rate

various temperatures. Figure 4 depicts the relationship between stress and steady-state rate in logarithmic coordinates. The slope of the Mg-5Zn-3Al-0.2Mn alloy at various temperatures can be calculated from the fact that it equals n . Table 3 presents the outcomes. The table shows that, under the conditions of 473-523 K/70-110 MPa, the creep stress exponent n of the alloy rises with temperature.

Figure 6 depicts the relationship between the logarithm of steady creep rate ($\ln\dot{\epsilon}$) and reciprocal temperature ($1/T$) of the alloy Mg-5Zn-3Al-0.2Mn under various stresses. The figure illustrates the linear relationship between \ln and $1/T$ in the range of 473-523 K ($1/T=0.0019-0.0021$). We can calculate the alloy's creep activation energies Q_c at 70 MPa, 90 MPa, and 110 MPa using the slope value in figure 6, which is equal to $R=8.314 \text{ J mol}^{-1}\text{K}$, as shown in table 3. It can be seen from table 5 that the creep activation energy Q_c increases with the increase of stress in the range of 70–110 MPa. The creep activation energy Q_c is $96.85 \text{ kJ mol}^{-1}$ and $113.48 \text{ kJ mol}^{-1}$ for 70 MPa and 90 MPa, respectively, in the range of 473 K-523 K.

Table 2: Creep properties of Mg-5Zn-3Al-0.2Mn alloy.

Temperature (K)	Load (MPa)	Creep life (h)	Strain rates (%)	Steady-state creep strain rate (s^{-1})
473	70	>100(389h)	0.14	2.2887×10^{-9}
	90	>100(346h)	0.34	2.5437×10^{-9}
	110	>100(311h)	0.49	3.3333×10^{-9}
498	70	>100(313h)	0.27	4.6388×10^{-9}
	90	>100(308h)	0.95	8.6666×10^{-9}
	110	>100(290h)	1.14	1.5250×10^{-8}
523	70	>100(279h)	0.74	2.3611×10^{-8}
	90	>100(212h)	1.75	3.9194×10^{-8}
	110	42	5.02	2.6255×10^{-7}

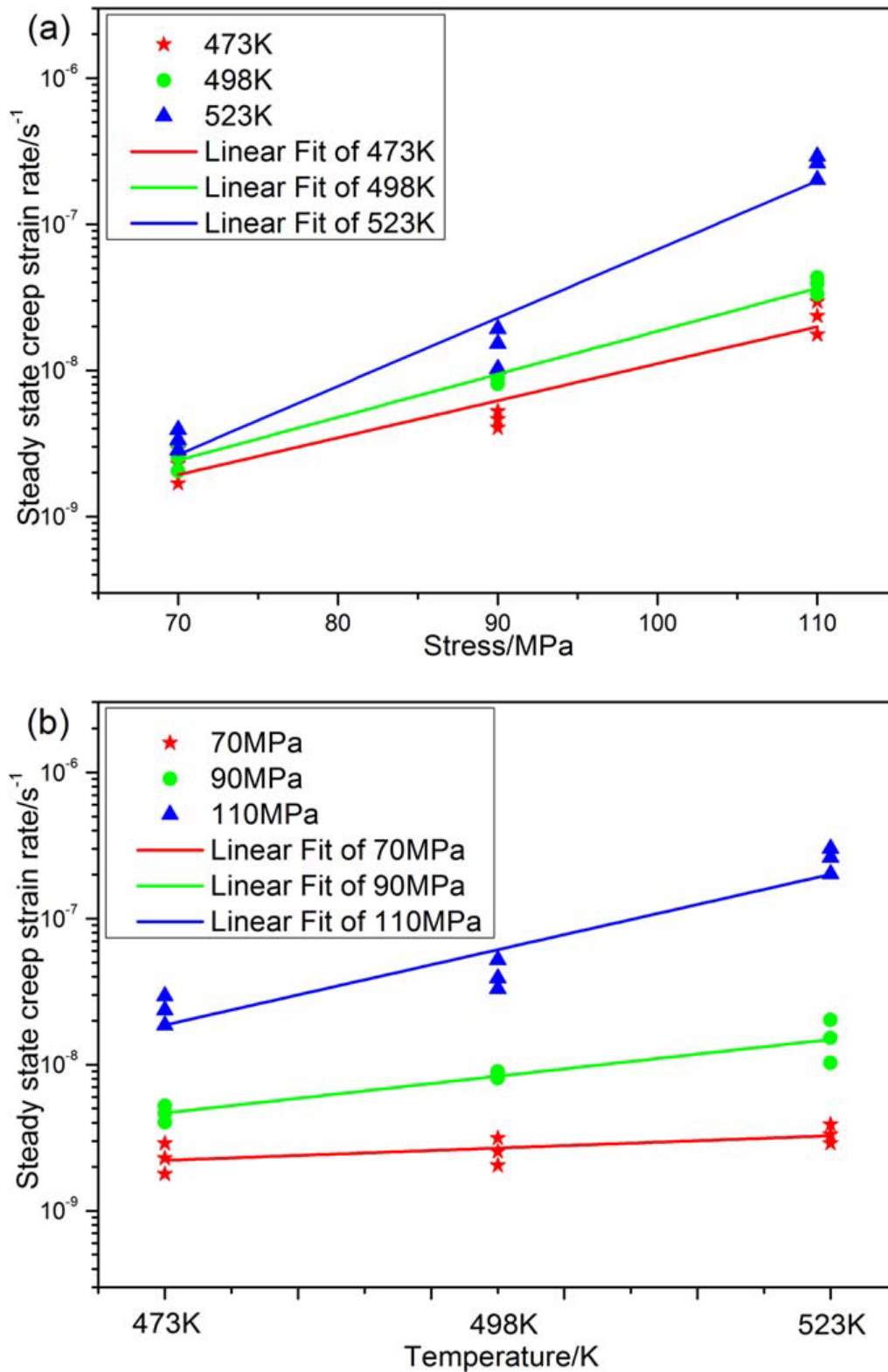


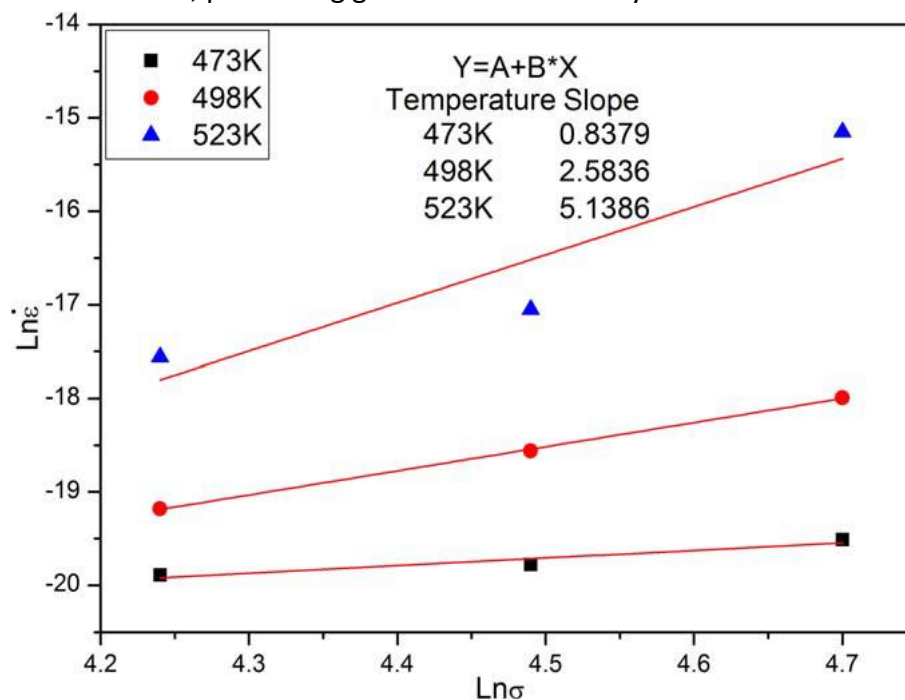
Fig 4: Comparison of steady-state creep strain rate of Mg-5Zn-3Al-0.2Mn alloy under (a) different stress (b) different temperature.

The amount to which the loading stress level affects the alloy's steady creep rate is indicated by the stress exponent n [16–30]. The steady creep rate increases with stress more quickly and is more sensitive to stress the higher the value of n . The alloy's creep mechanism may be indirectly reflected by the stress exponent n [22–30]. The findings demonstrate that the diffusion mechanism is the primary regulating mechanism when n is 1. When $n=2$, grain boundary sliding acts as the alloy's primary control mechanism. When $n=3$, the dislocation slip mechanism controls it, and when $n=4$, the dislocation climb mechanism controls it. The second phase particle strengthening mechanism is used when $n > 6$ [16–31]. The Mg-5Zn-3Al-0.2Mn alloy's creep stress exponent is 0.84 at 473 K/70-110 MPa, and the creep is regulated by the diffusion slip

At room temperature, magnesium alloys consist of tightly packed hexagonal crystals with few sliding systems and significant strain levels at grain boundaries [20–32]. As a result, under high temperature stress, grain boundary slip of magnesium alloys is easily possible. Therefore, preventing grain

mechanism [16-32]. The creep is governed by dislocation slip and grain boundary slip when the temperature is 498 K and the n value is 2.58 [20-32]. Dislocation slip and dislocation climb are the two mechanisms that control the creep at 523 K, where the n value is 5.13 [20–32]. The Mg-5Zn-3Al-0.2Mn alloy has an activation energy that is very similar to that of magnesium (100 kJ mol⁻¹) [21–32]. Dislocation slip and grain boundary slip are the main factors controlling the alloy [21–33]. The creep activation energy is 181.24 kJ mol⁻¹ at a stress of 110 MPa. The alloy is primarily controlled by grain boundary slip under high stress at this point because the activation energy is significantly greater than the cross-slip activation energy and the grain boundary diffusion activation energy of magnesium [22–33].

boundary slip and dislocation movement is crucial when designing magnesium alloys that are resistant to creep at high temperatures. Rare earth magnesium alloys' second phase is difficult to soften and coarsen at high temperatures, which effectively

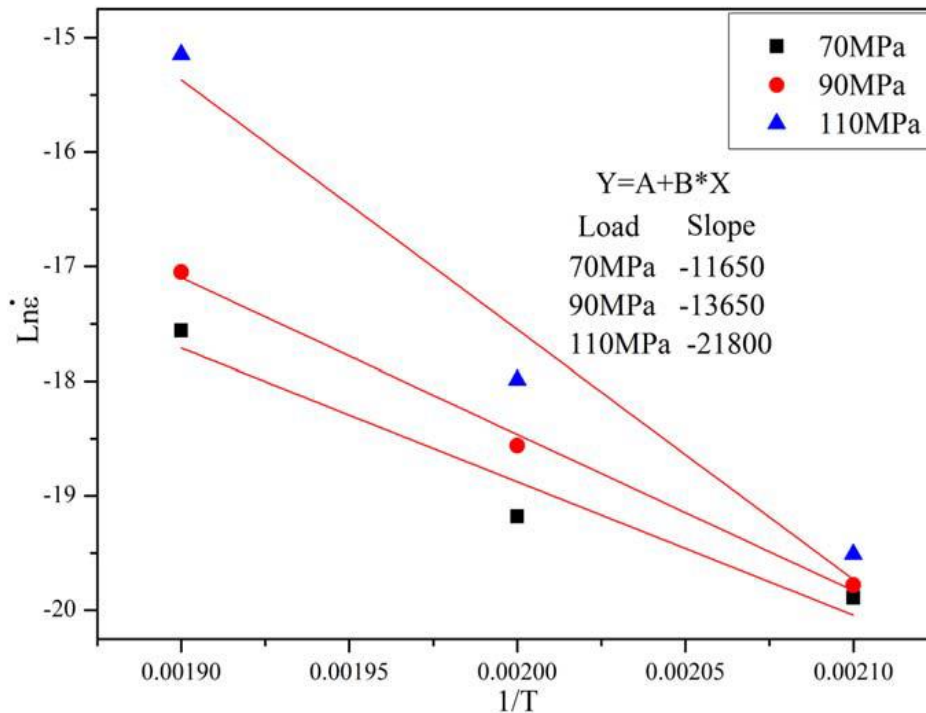


prevents dislocation movement over time and significantly increases the alloys' heat resistance.

Fig 5: The relationship between steady creep rate and stress of Mg-5Zn-3Al-0.2Mn alloy.

According to the research of Murayama [34] and colleagues, there are various ways that alloying elements can affect the creep behavior of magnesium alloys. These methods include direct strengthening of the grain boundaries, solid solution strengthening, and precipitation hardening. Creep strength can also be strengthened by solute and matrix distortion, as well as by raising the eutectic

five rare earth magnesium alloys with various microstructure characteristics were examined by Koundinya et al. [35]. According to studies, the dislocation structure of the alloy at the minimum creep rate is observed to be similar in all five alloys, with the majority of them being found on the base. Higher load carrying capacity and greater layer thickness of the thick layered eutectic result in greater



temperature. The creep characteristics of creep resistance.

Fig 6: The relationship between the logarithm of the steady creep rate and the reciprocal of temperature of Mg-5Zn-3Al-0.2Mn alloy under different stress.

Table 3: Creep stress exponents (n) of Mg-5Zn-3Al-0.2Mn alloy at different temperature.

Temperature (K)	473	498	523
Load (MPa)	70-110		
n	0.84	2.58	5.13

Table 4: Creep activation energies (Q_c) of Mg-5Zn-3Al-0.2Mn alloy under different stress

Load (MPa)	70	90	110
Temperature (K)	473-523		
Q_c (kJ/mol)	96.85	113.48	181.24

The comprehensive analysis in this paper demonstrates that there are grain boundary slip and dislocation slip phenomena in Mg-5Zn-3Al-0.2Mn alloys when combined with numerical and microscopic analysis of stress index and creep activation energy. According to the dislocation theory, the dislocation slip mechanism regulates the creep of the studied alloys as test temperature rises. It was investigated how different stresses at 473-523 K affected the creep process of cast rare earth magnesium alloys. Different temperatures and loading stresses resulted in a different creep mechanism for magnesium alloys. However, one thing is certain: the strengthening phase formed in the grain is much larger than that of the grain boundary and improves the creep properties [24-35]. This phase prevents creep deformation of the alloy grain boundary and dislocation slip.

4. Conclusions

The creep behavior of the Mg-5Zn-3Al-0.2Mn alloy was investigated, and the creep mechanism was examined, at 473-523 K/70-110 MPa for 100 h. The following conclusions can be drawn:

1. Under the creep conditions of (473K, 498K, 523K)/(70MPa, 90MPa, 110MPa), the creep strain of the Mg-5Zn-3Al-0.2Mn alloy increases as the temperature and stress increase. At 523K/70 MPa and 523K/90 MPa, the

steady-state creep rates are only 2.3611 s^{-1} and 3.9194 s^{-1} , respectively, and the creep strain after 100 h is only 0.735% and 1.751%. Under 523 K, the alloy Mg-5Zn-3Al-0.2Mn exhibits excellent creep resistance.

2. The grain size of the alloy increases with the increase in tensile strength at the same creep tensile temperature. When temperature rises while maintaining the same tensile strength, the grain size of the alloy obviously grows.
3. The Mg-5Zn-3Al-0.2Mn alloy has a creep stress exponent of 0.84-5.13 at 473-523 K/70-110 MPa, and the creep activation energy Q_c is $96.85 \text{ kJ mol}^{-1}$ for 70 MPa and $113.48 \text{ kJ mol}^{-1}$ for 90 MPa in the range of 473 K-523 K, respectively. According to the results of stress exponents and creep activation energies, as a Mg-5Zn-3Al-0.2Mn alloy ages, the creep mechanism shifts from grain boundary slip to dislocation slip as creep temperature and stress rise.

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