Creep and Corrosion Behavior of SiC Nanoparticles Dispersed Squeeze-cast Mg-5.0Al-2.0Ca-0.3Mn Alloy

Dissertation submitted in partial fulfilment

of the requirement of the degree of

Doctor of Philosophy

in

Metallurgical Engineering

by

Purnendu Nasker

(Roll Number: 18141005)

Under the supervision of

Dr. Ashok Kumar Mondal

Department of Metallurgical Engineering,
Indian Institute of Technology (BHU) Varanasi,
Varanasi - 221005, India.

February 2025

Chapter 9

Conclusions and Scope for Future Research

9.1 Conclusions

In the present thesis, microstructural characterization, tensile and compression, creep and corrosion behavior of the squeeze-cast Mg-5.0Al-2.0Ca-0.3Mn (AXM520) alloy and nanocomposites with 0.5, 1.0, 2.0 and 3.0 (wt.%) of SiC_{np} is investigated. The nanocomposites (NCs) were abbreviated as NC0.5SiC, NC1.0SiC, NC2.0SiC and NC3.0SiC. Further, the microstructural evolution and creep characteristics of the squeeze-cast age-hardened Mg-5.0Al-2.0Ca-0.3Mn (AXM520HT) alloy, as well as AXM520HT+1.0SiC (NC1.0SiCHT) and AXM520HT+2.0SiC (NC2.0SiCHT) (wt.%) nanocomposites have been investigated. The major conclusions drawn from the present thesis are as follows.

Conclusions on microstructures, tensile, compression, creep, and corrosion behavior of the squeeze-cast AXM520 alloy and NCs

i. The microstructures of AXM520 alloy and NCs consist of a primary solid solution (α-Mg), a eutectic of α-Mg and $(Mg,Al)_2Ca$ (C36) phases, and an Al_8Mn_5 phase. Additionally, the SiC phase was also present in the NCs.

ii. The grain size of the NCs reduced as the concentration of the SiC_{np} increased in the AXM520 alloy, and the same decreased by 36.8% in the NC3.0SiC.

iii. The C36 phase formed a continuous network in the AXM520 alloy. However, the same in the NCs was fragmented and became discontinuous with the increase in the fraction of the SiC_{np}. Thus, the C36 phase was the most fragmented in the NC3.0SiC.

iv. The α-Mg and C36 phase present in the AXM520 alloy exhibited an orientation relationship of $(0001)_{\alpha\text{-Mg}} \,||\, (0001)_{C36}$, $[10\bar{1}0]_{\alpha\text{-Mg}} \,||\, [11\bar{2}0]_{C36}$. The lattice parameter values for the α-Mg and C36 phases were found to be a = 3.19 Å, c = 5.23 Å, and a = 5.92 Å, c = 9.87 Å, respectively

v. All the NCs exhibited superior tensile properties than the AXM520 alloy. The NC2.0SiC with 37.2 and 69.8% enhancement in YS and UTS exhibited the most superior tensile properties. The %El of the AXM520 alloy and NC2.0SiC were 1.03±0.03 and 3.1±0.02%, and it decreased to 1.29±0.02% in the NC3.0SiC. The UTS and %El of the NCs declined with more than 2.0 (wt.%) SiC_{np} to the AXM520 alloy because of the nanoparticle agglomeration.

vi. The superior compressive properties were exhibited by the AXM520 alloy with the SiC_{np} additions. The presence of thicker shear bands in the fractograph of the NC3.0SiC indicated its higher energy absorption capability during failure than the AXM520 alloy.

vii. The strengthening from CTE mismatch contributed the most to the overall strengthening of all the NCs, and the same was 146.0% greater in the NC3.0SiC than the NC0.5SiC. The contribution from Orowan strengthening also increased by 121.2%. However, the Hall-Petch strengthening increased only by 18.1%with the increase in nanoparticle content from 0.5 to 3.0 (wt.%) in the NCs.

viii. The NCs revealed improved creep performance compared to the AXM520 alloy under the experimental parameters utilized. The creep resistance of the NCs increases with the increase in the SiC_{np} content. The NC2.0SiC exhibited an increase in creep resistance by 73.2% compared to the alloy. However, the creep resistance deteriorated with a further increase in the fraction of the SiC_{np} in the NC3.0SiC, due to the agglomeration.

ix. The stress exponents varied from 5.0 to 6.7, and activation energies varied from 89.8 to 101.8 kJ/mol, implying the deformation at elevated temperature in the AXM520 alloy and NCs was dominated by the climb of dislocation assisted by the pipe diffusion.

x. The pile-ups of dislocations took place around the C36 phase and near the SiC_{np}. The additional strengthening owing to the presence of the SiC_{np} in the NCs was responsible for their improved creep performance compared to the AXM520 alloy.

xi. The corrosion resistance of the NCs measured in the hydrogen evolution test was superior to the AXM520 alloy, and the improvement was 91.1% in the NC3.0SiC.

xii. The OCP of all NCs shifted to more noble values with the increased quantity of SiC_{np} in AXM520. The polarization resistance determined from the EIS increased with the increase in the SiC_{np} content in the NCs.

xiii. The potentiodynamic polarization scans further confirmed the superior corrosion resistance of the NCs to the AXM520 alloy. Among the fabricated NCs, NC3.0SiC exhibited the highest corrosion resistance, and it was 91.3% lower in comparison to the AXM520 alloy.

xiv. The corrosion products predominantly consist of $Mg(OH)_2$. The addition of SiC_{np} reduced the formation of $Mg(OH)_2$ and increased the content of the $Al(OH)_3$ in the NCs leading to the higher stability of the corroded film formed on them.

xv. The α-Mg phase was severely damaged owing to the galvanic corrosion between the α-Mg and C36 phases. However, the same was reduced due to a decrease in the volta potential between α-Mg and C36 phases in the NCs, resulting in their superior corrosion resistance.

Conclusions on microstructures and creep behavior of the squeeze-cast age-hardened AXM520 alloy and NCs

i. The age-hardened AXM520HT, NC1.0SiCHT, and NC2.0SiCHT contain the primary α-Mg grains, the discontinuous (Mg, Al)$_2$Ca (C36) phase at the grain boundaries, and randomly present spherical-shaped Al_8Mn_5 phase. All the NCs additionally contain the SiC phase.

ii. The NCs revealed superior creep resistance compared to AXM520HT, and it was the best in the NC2.0SiCHT. The improvement was 76% in NC2.0SiCHT compared to AXM520HT.

iii. The creep deformation was dominated by dislocation climb facilitated by pipe diffusion at the chosen test temperature and stress ranges. The newly constructed maps could be used as a reference to predict the creep deformation mechanism for a particular stress, temperature, and grain size range for the Mg-Al-Ca-based alloys and NCs.

iv. The C36 phase was utterly broken in the AXM520HT, whereas the same remains intact in the NC2.0SiCHT. The percentage of twins in the creep-tested specimen of NC2.0SiCHT was lower than in the AXM520HT alloy. The density of the <c> type dislocations was much higher than that of the <a> type dislocations in the NCs.

v. The significant improvement in creep resistance of all the NCs over the AXM520HT alloy was attributed to the age-hardening as well as dispersion strengthening from the nanoparticles.

To conclude, the tensile, compression, creep, and corrosion behavior of the NC0.5SiC, NC1.0SiC, NC2.0SiC, and NC3.0SiC are superior to that of the AXM520 alloy. Thus, the use of NCs is beneficial over the AXM520 alloy. Among the fabricated NCs, NC2.0SiC is the best, considering the mechanical properties and corrosion behavior.

9.2 Scope for future research

The following research works on the squeeze-cast Mg-5.0Al-2.0Ca-0.3Mn (AXM520) alloy, and nanocomposites might be carried out in the future.

1. The tensile and compressive properties were evaluated at ambient temperature. It would be interesting to evaluate both of these properties at elevated temperatures.
2. The tensile, compressive, and corrosion properties were evaluated in as-cast condition. It would be interesting to evaluate these properties after aging.
3. The wear behavior of the AXM520 alloy and NCs in both as-cast and aged conditions may be evaluated.
4. The fatigue and creep-fatigue interaction of the AXM520 alloy and NCs in both as-cast and aged conditions may be evaluated.
5. The dislocation twin interaction revealed in the present study can be expanded further in future work, which may help to improve the understanding of the strengthening mechanisms in detail.
6. The creep mechanism map developed in the present study for AX alloys and composites needs to be addressed with more experimental data, which may help improve the map and make precise load and temperature range prediction possible.
7. Ultrasonic vibration may be employed for dispersing a higher volume fraction of SiC_{np}, and the tensile, creep, and corrosion behaviour may be evaluated with a higher content of SiC_{np}.