

Characterization of SiC-Reinforced AZ91 Magnesium Alloy Composites Produced Using In situ Microwave Casting



Radha Raman Mishra, Parvej Alam, Jitendra Yadav, Gaurav Kumar, and Apurbba Kumar Sharma

1 Introduction

In recent years, time compression and energy-saving attributes of microwave energy over conventional heating processes while processing metallic materials have been explored in various researches [1, 2]. It has been reported that microwaves induce two-directional heating during microwave hybrid heating (MHH) of metallic materials that results in more uniform heating [3]. Various processes such as sintering [4–10], joining [11], cladding [12], and casting [13, 14] have been developed to process metallic materials using MHH technique. Microwaves couple with metallic powder particles rapidly at room temperature due to availability of higher surface area and comparable particles size and microwave penetration depth [1].

On the other hand, bulk metallic materials reflect almost all microwaves from their surfaces, owing to very small skin depth and negligible heating of bulk material occurs at room temperature [15]. However, the increase in temperature of bulk metals allows microwaves to penetrate deeper in the target metal and enables the

R. R. Mishra (✉) · A. K. Sharma

Design Innovation Center, Indian Institute of Technology Roorkee, Roorkee, Uttarakhand 247667, India

e-mail: rmishra@me.iitr.ac.in

A. K. Sharma

e-mail: aks@me.iitr.ac.in

P. Alam · J. Yadav · G. Kumar · A. K. Sharma

Department of Mechanical and Industrial Engineering, Indian Institute of Technology Roorkee, Roorkee, Uttarakhand 247667, India

e-mail: parvejalam33@gmail.com

J. Yadav

e-mail: jkyadav1@hotmail.com

G. Kumar

e-mail: grv.kmr2015@gmail.com

material to attain microwave absorbing characteristics beyond its critical temperature [11–13]. Microwave casting of metallic materials is one of the novel applications of microwave energy. Prior to casting, melting of bulk metallic materials using microwave energy was demonstrated [16–20]. For better utilization of microwave energy during the melting of metals, understanding of microwave-metallic material interaction phenomena is required. Experimental and theoretical studies were reported to explore physics of microwave melting of metallic materials [17, 19, 21]. Microwave melting of metals in the inert atmosphere was reported, and a comparison was also carried out with conventional furnaces melting [17]. Microwave melting was found faster and significant energy-saving process than conventional melting processes. The thermal profile of aluminum alloy 7039 while exposed to microwaves in the ambient environment was studied [19, 21]. It was reported that the electromagnetic properties of the oxide layer, which forms on the surface of alloy influences the microwave heating significantly at elevated temperatures. Various metals/alloys such as aluminum, copper, steel, titanium, lead, and tin were melted using microwave energy; however, melting of magnesium was hardly reported. The lower ignition temperature of the magnesium might result in limited research in the casting of magnesium. In spite of the fact, magnesium and its alloys offer ease of recycling, superior specific mechanical properties, high damping capability, and significant electromagnetic shielding capability. These properties place them in highly demanded industries, including aerospace and automotive. In the present work, microwave energy at 2.45 GHz was used to cast AZ91 magnesium alloy-based silicon carbide metal matrix composite (AZ91 Mg alloy/SiC MMC) inside a multi-mode microwave applicator. The produced composites cast were characterized. Microstructural properties of the composites vis-à-vis micro-indentation hardness were discussed.

2 Materials and Method

Magnesium alloy AZ91D was used as charge material for the experimental trials. The optical image of the alloy indicating the microstructure is shown in Fig. 1a. The elemental composition of as-received alloy is presented in Table 1.

The silicon carbide (SiC) powder particle of 800 grit size (average diameter of 6.5 μm) was used as a reinforced constituent in the process. The SEM image of SiC powder particles is shown in Fig. 1b. A microwave applicator (Mode: multi-mode, Model: MH-1514–101-V6, Make: Enerzi Microwave Systems Pvt. Ltd.) and a casting setup including a base, split mold, pouring basin, susceptor, and sprue were used for experimental trials. A schematic diagram of the in situ microwave casting setup is shown in Fig. 2. The ceramic crucible acts as a pouring basin and absorbs microwave energy rapidly during microwave irradiation. The charge was hybrid heated using the pouring basin. The alumina sprue was used to facilitate self-pouring of the melt into a mold cavity. The split mold made of graphite was used for solidification of the melt. A ceramic plate of low dielectric loss was used for the base

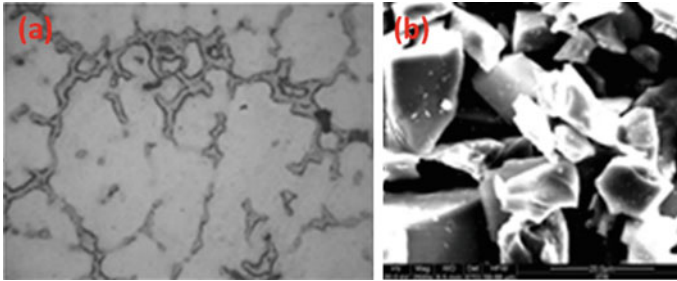


Fig. 1 **a** Optical image of as-received AZ91 alloy (500 ×) and **b** SEM image of the SiC powder particles

Table 1 Elemental composition of the as-received alloy

Elements	Al	Fe	Zn	Cu	Mn	Mg
% Wt	6.81	0.01	0.35	0.03	0.01	Bal

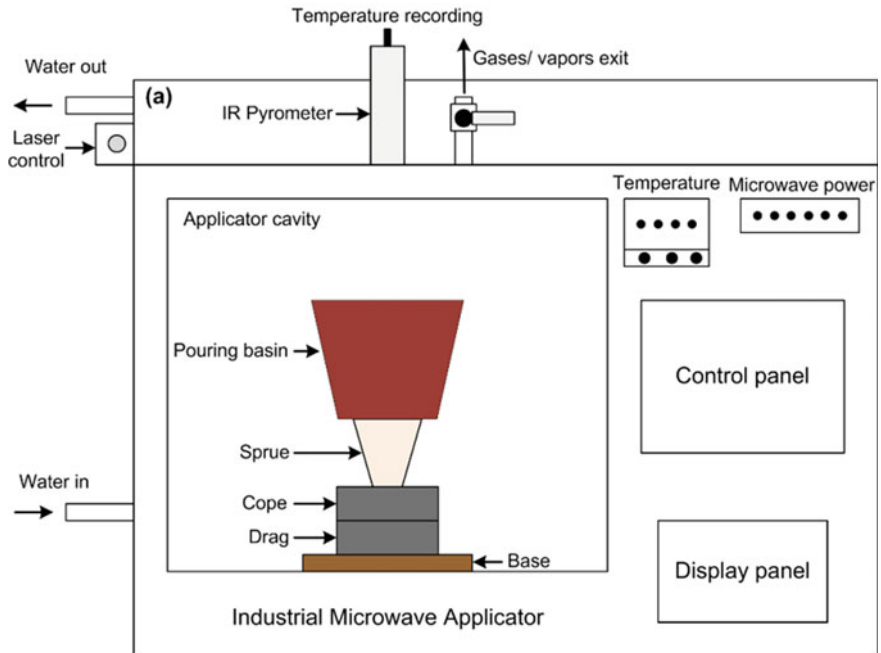


Fig. 2 Schematic diagram of the microwave casting setup

to avoid any possibility of thermal damage in the cavity. The setup was insulated using a microwave transparent material to reduce heat losses from mold assembly to the cavity environment. The mold assembly was placed inside the cavity. The position of the charge (50 g) and mold assembly was fixed using a laser control. The SiC powder works as a susceptor and also accelerates the microwave heating process. Monitoring of temperature at the top surface of charge was carried out using a built-in infrared (IR) pyrometer (range: 350–1800 °C, least count: 1 °C). The charge and SiC mixture was exposed to microwaves at 2.45 GHz and 1400 W in the ambient cavity environment. The temperature of the top surface of the charge was monitored up to its melting. A sharp decrease in charge temperature indicated self-pouring of the melt inside the cavity. The experiments were repeated at least thrice to ensure that the data were reproducible.

The produced casts were sectioned and polished using standard metallographic techniques. The polished samples were cleaned with acetone and dried. The microstructural analysis was carried out using an optical microscope (Make: Dewinter, Model: LT-23B) and a software tool (Dewinter Material plus, version 4.2). Moreover, a field emission scanning electron microscope (FE-SEM, Make: Carl Zeiss, Model: Ultra Plus) equipped with energy-dispersive X-ray spectroscopy (EDS, Make: Oxford X-max) was also used to see the various phases present in the cast and their chemical compositions. The phase analysis was carried out at room temperature using X-ray diffraction (XRD) in a Bruker AXS diffractometer with Cu-K α X-ray with a scan rate at 1° min⁻¹ within the scan range of 5–100°. A Vickers microhardness tester (Make: Chennai Metco, Model: Economet VH 1MD) was used to measure the hardness of the developed casts using the load of 100 g with dwell time 30 s.

3 Results and Discussion

3.1 Microstructural Analysis

Figure 3a shows the optical image of a selected area of the cast cross section. It is evident from Fig. 1a that the grain size of the cast is lesser as compared to as-received materials. The presence of SiC particles in the cast cross section is also visible. The high-resolution image (Fig. 3b) of the cast cross section indicates the presence of various phases at grain boundaries and distribution of SiC particles inside the grains and along the grain boundaries. The dark areas in the SEM micrograph correspond to α -Mg phase, whereas the lighter areas indicate the precipitated intermetallic phases. The shiny particles are traces of the η -phase which predominantly contains aluminum.

Distribution of various elements which are present in the developed cast along a selected line (Fig. 4a) on the cast cross section is shown in Fig. 4b. The EDS analysis along the line clearly shows the presence of elements Mg, Al, Si, Ni, Zn and C in the

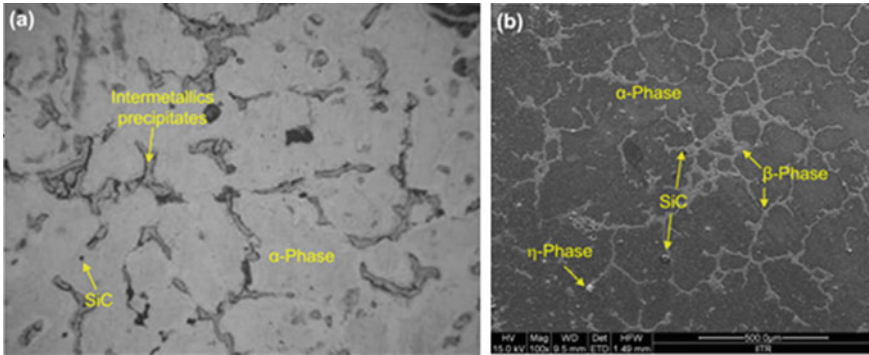


Fig. 3 a Optical image b Fe-SEM micrograph of the cast AZ91 alloy

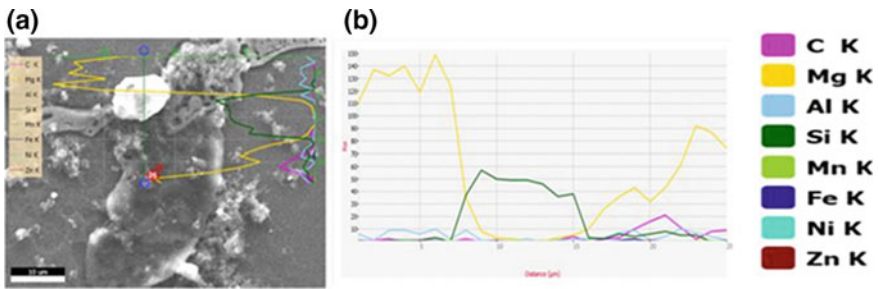


Fig. 4 a SEM image of a selected area of the cast and b distribution of various elements present along a specified length in a

cast. It indicates the possible presence of SiC particles, α -Mg phase, Mg_2C , Mg_2C_3 , $Mg_{17}(Al, Zn)_{12}$, and the phase β - $Mg_{17}Al_{12}$.

3.2 X-Ray Diffraction Analysis

The X-ray diffraction pattern of the various produced composite cast samples is shown in Fig. 5. It indicates that α -Mg phase is mainly present in the composite cast. Also, the presence of SiC, phase Mg_2Si , and phase β - $Mg_{17}Al_{12}$ was observed. However, no trace of metallic carbides was observed.

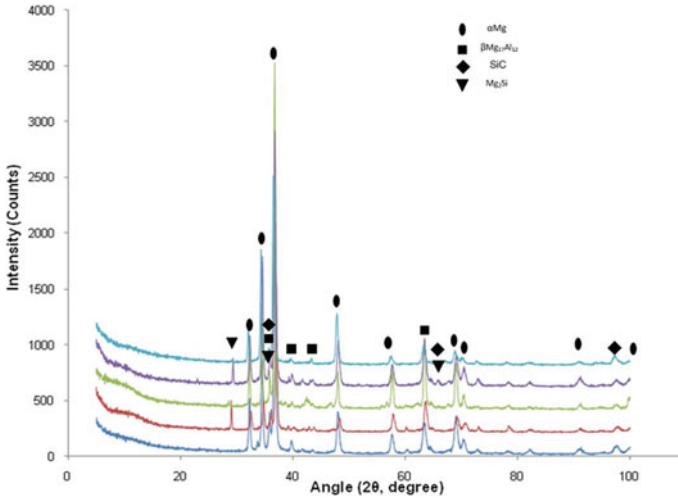


Fig. 5 Image indicating the X-ray diffraction pattern of different composite casts of alloy AZ91/SiC

3.3 Microhardness Analysis

Microhardness analysis was carried out on the developed composite casts and as-received alloy. The average value of microhardness for the base alloy was found to be 115 ± 10 HV, whereas microhardness of the composite cast was observed 206 ± 28 HV. The increase in hardness of the composite cast is due to the presence of harder SiC and Mg_2Si intermetallic phase. Moreover, the decrease in grain size as evident from Figs. 1a and 3a also affects the hardness. According to the Hall–Petch relationship, the decrease in grain size enhances the hardness of a material. Therefore, in the present study, the increase in hardness of the cast composite as compared to the base alloy is attributed to (a) presence of SiC particles and intermetallic phases and (b) finer grains of the composite cast.

4 Conclusions

In the present work, a new approach called in situ microwave casting is used for casting the magnesium alloy (AZ91)-based SiC-reinforced metal matrix composites. The following may be concluded from the study -

- (a) Microwave hybrid heating approach can be utilized for the casting of metal matrix composites.
- (b) The microstructural analysis-indicated finer grains are formed in the composite cast as compared to the base alloy.

- (c) The presence of SiC particles in the composite cast was observed; however, more iteration is required to achieve uniform distribution of SiC particles.
- (d) Presence of various phases— α -Mg, β -Mg₁₇Al₁₂, Mg₂C, Mg₂C₃, Mg₁₇(Al, Zn)₁₂, and Mg₂Si and SiC were confirmed by the EDS and XRD results.
- (e) Presence of SiC, Mg₂Si phase, and finer grains in the cast composite results in higher microhardness.

References

1. Oghbaei M, Mirzaee O (2010) Microwave versus conventional sintering: a review of fundamentals, advantages and applications. *J Alloy Compd* 494(1–2):175–189. <https://doi.org/10.1016/j.jallcom.2010.01.068>
2. El Khaled D, Novas N, Gazquez JA, Manzano-Agugliaro F (2018) Microwave dielectric heating: applications on metals processing. *Renew Sustain Energy Rev* 82:2880–2892. <https://doi.org/10.1016/j.rser.2017.10.043>
3. Gupta M, Wong WLE (2005) Enhancing overall mechanical performance of metallic materials using two-directional microwave assisted rapid sintering. *Scripta Mater* 52(6):479–483. <https://doi.org/10.1016/j.scriptamat.2004.11.006>
4. Roy R, Agrawal D, Cheng J, Gedevisanishvili S (1999) Full sintering of powdered-metal bodies in a microwave field. *Nature* 399(6737):668–670
5. Mahmoud MM, Link G, Thumm M (2015) The role of the native oxide shell on the microwave sintering of copper metal powder compacts. *J Alloy Compd* 627:231–237. <https://doi.org/10.1016/j.jallcom.2014.11.180>
6. Muthuchamy A, Kumar R, Annamalai AR, Agrawal DK, Upadhyaya A (2016) An investigation on effect of heating mode and temperature on sintering of Fe-P alloys. *Mater Charact* 114:122–135. <https://doi.org/10.1016/j.matchar.2016.02.015>
7. Mondal A, Upadhyaya A, Agrawal D (2010) Microwave and conventional sintering of 90W–7Ni–3Cu alloys with premixed and prealloyed binder phase. *Mater Sci Eng: A* 527(26):6870–6878. <https://doi.org/10.1016/j.msea.2010.07.074>
8. Sueyoshi H, Hashiguchi T, Nakatsuru N, Kakiuchi S (2011) Effect of surface oxide film and atmosphere on microwave heating of compacted copper powder. *Mater Chem Phys* 125(3):723–728. <https://doi.org/10.1016/j.matchemphys.2010.09.066>
9. Rajkumar K, Aravindan S (2009) Microwave sintering of copper–graphite composites. *J Mater Process Technol* 209(15–16):5601–5605. <https://doi.org/10.1016/j.jmatprotec.2009.05.017>
10. Xu L, Srinivasakannan C, Peng J, Guo S, Xia H (2017) Study on characteristics of microwave melting of copper powder. *J Alloy Compd* 701:236–243. <https://doi.org/10.1016/j.jallcom.2017.01.097>
11. Bansal A, Sharma AK, Kumar P, Das S (2014) Characterization of bulk stainless steel joints developed through microwave hybrid heating. *Mater Charact* 91:34–41. <https://doi.org/10.1016/j.matchar.2014.02.005>
12. Zafar S, Sharma AK (2014) Development and characterizations of WC–12Co microwave clad. *Mater Charact* 96:241–248. <https://doi.org/10.1016/j.matchar.2014.08.015>
13. Mishra RR, Sharma AK (2017a) Structure-property correlation in Al–Zn–Mg alloy cast developed through in-situ microwave casting. *Mater Sci Eng, a* 688:532–544. <https://doi.org/10.1016/j.msea.2017.02.021>
14. Singh S, Gupta D, Jain V (2016) Novel microwave composite casting process: theory, feasibility and characterization. *Mater Des* 111:51–59. <https://doi.org/10.1016/j.matdes.2016.08.071>
15. Mishra RR, Sharma AK (2016a) Microwave–material interaction phenomena: heating mechanisms, challenges and opportunities in material processing. *Compos a Appl Sci Manuf* 81:78–97. <https://doi.org/10.1016/j.compositesa.2015.10.035>

16. Agrawal D (2006) Microwave sintering, brazing and melting of metallic materials. In: Sohn international symposium on advanced processing of metals and materials. New improved and existing technologies, Non-Ferrous materials extraction and processing, vol 4. pp 183–192
17. Chandrasekaran S, Basak T, Ramanathan S (2011) Experimental and theoretical investigation on microwave melting of metals. *J Mater Process Technol* 211(3):482–487. <https://doi.org/10.1016/j.jmatprotec.2010.11.001>
18. Moore AF, Schechter DE, Morrow MS (2006) Method and apparatus for melting metals. United States Patent No. US 7,011,136 B2
19. Mishra RR, Sharma AK (2016b) On mechanism of in-situ microwave casting of aluminium alloy 7039 and cast microstructure. *Mater Des* 112:97–106. <https://doi.org/10.1016/j.matdes.2016.09.041>
20. Lingappa MS, Srinath MS, Amarendra HJ (2017) Microstructural and mechanical investigation of aluminium alloy (Al 1050) melted by microwave hybrid heating. *Mater Res Expr* 4(7):076504
21. Mishra RR, Sharma AK (2017b) On melting characteristics of bulk Al-7039 alloy during in-situ microwave casting. *Appl Therm Eng* 111:660–675. <https://doi.org/10.1016/j.applthermaleng.2016.09.122>