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Empirical optimization of corrosion rate for magnesium-chromium composites

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In this study, optimization of the corrosion rate (CR) of Mg-composites has been evaluated by varying the concentration, reinforcement percentage, and immersion time. As prime material, pure Mg is preferred for this research and chromium (Cr) consider as a reinforcing material with different percentages. CR (miles/yr) has been optimized by varying parameters such as reinforcement percentage as 3%, 5% and 7% including NaCl immersion medium (%) as 2.4%, 3.5% and 4.7% with immersion time (h) such as 48h, 72h and 96h. By using, DOF, minimal CR has been measured with the assistance of Minitab Software having ANOVA and Taguchi approaches. Optimized results reveal that the percentage of corrosion solution is influenced upto 66.10%, reinforcement percentage contributed to 27.56% and immersion time influenced upto 2.81%. An optimized combination of CR is 7wt. % Cr with 2.4% NaCl for 96h. However, microscopy results illustrate shallow crack boundaries in Mg/Cr composites due to its chemical reaction in alkaline solution.

Keywords: Chromium, Corrosion Rate, Magnesium, Microscopy, Minitab, Optimization

Recently, fabricated aeroplane units, vehicle parts and marine applications have been dominated by magnesium-based alloys and composites[1-3]. In home appliances and medical instruments also, Mg composites have been enormously used and enhance the production rate in such sectors[4,5]. In comparison to Al-based composites, Mg composites possess high ductility with the light-weight in nature and efficiently formed small size dimensional parts[6]. Further, the incorporation of reinforcing particles enhances the strength of Mg composites and validates the specific processing application. The addition of reinforcement upgrades the usage life of the Mg-composites with optimum strength to weight ratio[7]. The fabrication of Mg-based composites is an ambitious (difficult) process because of its blazing (burning) action during fabrication but the specific fabrication process also enhances its properties[8]. A high concentration of magnesium or its alloy contents under an optimum vacuum environment is necessary to synthesize an effective and innovative composite structure. The proper dispersion of reinforcement in the prime material is an art that restores its favourable bonding nature and improves its efficacy[9]. Various fabrication techniques are involved to manufacture Mg-based composites but the suitable and simple method has been achieved by the stir casting process[10,11].

The vacuum-based stir casting process is conducted by blending both materials in ingot/powder form in a muffle furnace. A muffle furnace is a simple vacuum-based furnace in which argon gas is supplied. The stirrer attachment is connected to the muffle furnace which is rotated repeatedly with a specific period. This causes uniform and homogenous blending of both materials into the composite melt. Stirring speed and fabrication temperature are the key factors that influence uniform mixing[12,13]. Squeezed pressure is also involved to fabricate defect-free specimens by applying high rate squeezed pressure[14]. MS die is commonly used during Mg/Al composites from the composite melt into solid billets in cylindrical shaped[15].

This study intends to determine and empirically optimize CR parameters for the immersion test. The key objective of this paper is to reduce the CR for Mg/Cr composites.

Experimental Section

Materials

Pure Mg and its composites have weak resistant to strike of the alkaline medium as well as marine-water conditions[16]. Mg-composites having a yield strength range of 20-230 MPa and has a significant hardness value upto 65HB[17,18]. Mg-based composites mostly processed in car body parts, aeroplane-wings, electronic parts etc[19]. Chromium reinforced particles have been selected due to their silvery hard ductile material with good corrosion resistant properties and used as coating, plating and bathroom fittings etc[20, 21].

Immersion test

This investigation focussed to demonstrate optimum immersion CR of all Mg-based specimens. The Mg-based specimens are immersed in NaCl solution at varying level of concentration and immersion time. To find actual corrosion mass loss result, Mg-specimens are measured before and after the immersion test. The dimensions of Mg-specimens used for this experiment is 10mm X 10mm X 5mm along with its polished surfaces. 220-800 grit emery sheets are operated to finish the testing surfaces then clean with distilled water and dried[22]. Mg-samples are dipped in NaCl alkaline medium at the normal temperature of 30^{o}C and the CR equation as follows[23]

$$\text{CR} = \frac{87500 \times (m_{before} - m_{after})}{\rho \times A \times t} . \frac{miles}{year} \quad \ldots (1)$$

where,

CR: Corrosion Rate (miles/yr)

m_{before} :Weight loss before immersion (g)

m_{before} :Weight loss after immersion (g)

ρ : Density of composites (g/cm^3)

A: Surface area of composite Specimens (cm^2)

t: Immersion Time (hrs)

Table 1 — Processing parameters and its levels for CR of Mg/Cr composites

S. No.	Processing parameters	L1	L2	L 3
1	Reinforcement percentage	3	5	7
2	Percentage of corrosion NaCl medium	2.4	3.5	4.7
3	Immersion time in hrs	48	72	96

L means the Level

As per the literature review, optimizing factors have been evaluated through an ANOVA experiment with all processing factors and levels in tabulated form as shown in Table 1 (Ref. 24).

Results and Discussion

In this study, Table 2 is tabulated with optimized factors as immersion time, reinforcement percentage, CR, corrosion medium and signal to noise ratio (SNR). All factors are identified with the help of the literature study.

Further, Tables 3 and 4 represents optimized response data values of SNR and means of Mg/Cr composites corrosion factors. These data values intimate about analysis of the most influential factors based on the 'smaller is better' concept[25]. The least CR value illustrates the best result of optimization having highly influence as compared to other factors[26]. During optimization, the corrosion medium percentage is placed as first rank order whereas reinforcement percentage secured second rank order and immersion time gains third rank order.

Figure 1. demonstrates optimized main effect plots of Mg-composites CR factors. The increment in the percentage of corrosion medium percentage from 2.4% to 4.7% causes a reduction in CR. 3 wt. % of Cr reinforcement decreases the CR whereas, with the increase in Cr reinforcement percentage, CR behaves as positively.

Table 3 — Mean response

Level	Corrosion medium (NaCl %)	Reinforcement percentage (Wt. %)	Time of Immersion (h)
1	0.002214	0.002778	0.002406
2	0.002290	0.002309	0.002600
3	0.004171	0.002318	0.002588
Delta	0.000857	0.000459	0.000204
Rank	1	2	3

Table 2 — Output response of optimized corrosion parameters

No. of runs	Corrosion medium (NaCl %)	Immersion time (hrs)	Reinforcement percentage (Wt. %)	CR (miles/yr)	SNR
1	2.4	48	3	0.002406	58.2971
2	3.5	72	3	0.002504	58.9642
3	4.7	96	3	0.004603	53.0960
4	2.4	72	5	0.002204	59.5885
5	3.5	96	5	0.002105	60.6078
6	4.7	48	5	0.002704	57.9018
7	2.4	96	7	0.000948	62.0141
8	3.5	48	7	0.002108	60.2465
9	4.7	72	7	0.002908	56.6706

Figure 2 evaluates the residual plot graphs of optimized CR factors of Mg/Cr composites. These plots explain about distributed points lies nearer to the probability line having the best output response of CR factors. However, the results of order versus uniformly split histogram bars and junction lines graphs suggest an excellent optimized CR approach for Mg/Cr-based composites.

The derived ANOVA results of each CR factors are displays in Table 5. From the given factors, the percentage of corrosion medium is majorly influenced and its increment upto 66.10% whereas reinforcement percentage is contributed to 27.56% and the last i.e. immersion time influenced only 2.81%. The larger Fisher data i.e. F-value of corrosion medium is 12.75 as compared to other optimized factors. This signifies that the corrosion medium has dominantly influenced by this investigation.

Table 4 — SNR response

Level	Immersion Time (hrs)	Reinforcement percentage (Wt. %)	Corrosion medium (NaCl %)
1	58.28	60.42	56.58
2	58.20	59.78	59.82
3	58.48	55.26	59.95
Delta	0.87	4.26	2.42
Rank	3	2	1

Smaller is better

CR (miles/yr) of Mg/Cr composites has been evaluated through regression equation 2 also. Figure 3 (a-c) represents the counterplot of corrosion parameters of Mg/Cr composites with the varying colour combination as variable outputs. Figure. 3(a) represents that with the increase in corrosion medium percentage, CR increases. And with a low medium percentage having a moderate reinforcement level shows minimum CR. Figure 3b displays optimum CR under low corrosion solution medium and high immersion time. However, Figure 3c signifies low CR under moderate immersion time and minimum reinforcement percentage.

Table 5 — ANOVA Results of CR Parameters

Source	Degree of Freedom	Contribution%	Seq. SS	Adj. SS	Adj. MS	F	P
Corrosion medium (NaCl %)	1	66.10	0.000001	0.000001	0.0000	12.75	0.032
Reinforcement %	1	27.56	0.000001	0.000001	0.000001	3.83	0.152
Immersion time(hrs)	1	2.81	0.000001	0.000001	0.00000	0.32	0.556
Error	5	3.53	0.000001	0.000001	0.00000	-	-
Total	8	100.00	0.000002	-	-	-	-

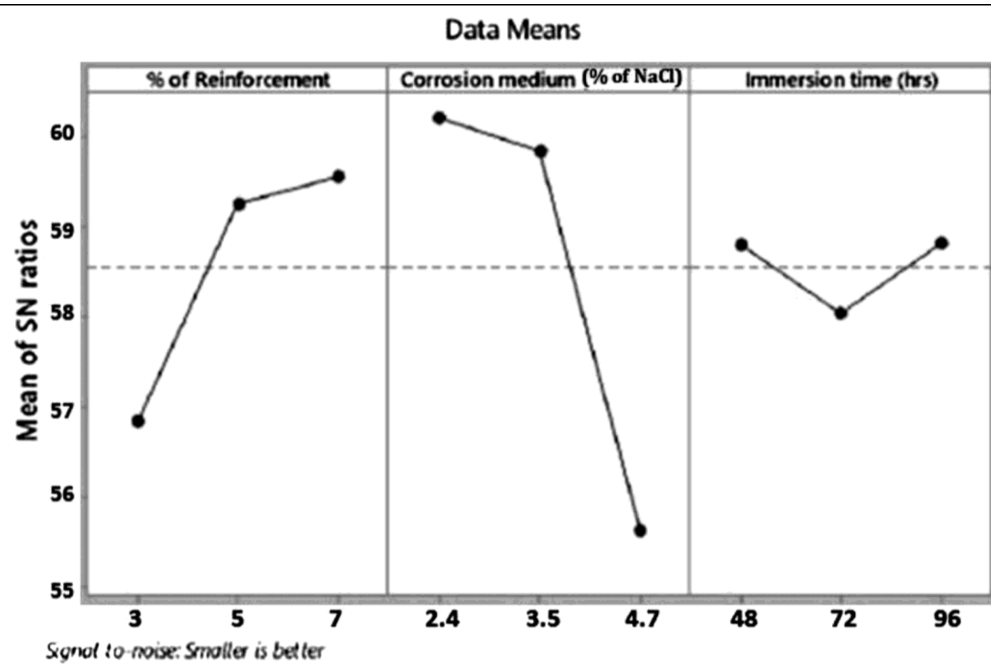

Fig. 1 — Main effects plots for SN Ratios

Regression Equation:

CR (MPY) = 0.00026 – 0.00042% (Reinforcement) + 0.000350 (Immersion Medium, NaCl %) + 0.00002 … (2)

Figs. 4 (a) & (b) represents the microscopy of corroded pure Mg and Mg/5 wt. % Cr composite in 96h of immersion time under 3.5% NaCl alkaline corrosion medium. The pure Mg-mono composite shows deep shallow cracks in form of crack boundaries whereas Mg/Cr composite displays the shallow cracks with little shines near the crack boundaries.

The resulting counterplots have been evaluated for each designing parameters that indicate the mean distributions and interactions of the response variables as presented in Fig. 1, 2 and 3.

It reflects from the counterplots that as immersion time increases upto a certain limit, the CR become stable, although the concentration of NaCl solution (immersion medium) is increased as shown in Fig. 3 (a). It is also revealed from the counterplots that maximum CR observed in immersion medium vs. wt. % chromium interaction.

The optimum corrosion rate is observed in Fig. 3(b) having a slight solution of corrosion medium under immersion time. From these counterplots, it is revealed that immersion time in NaCl solution is the key parameter that affects to maximum duration over the mean CR of Mg-composite material in the research lab. Similar, the optimum range of CR is observed in the area between 3wt. % -7 wt. % Cr and 48-72 hr immersion time of NaCl medium, Fig. 3(c). The diversion part of this counter plot explains that an increase in immersion time of alkaline has lead to more cracks of the Mg/Cr interface. From these counterplots, it is observed that the region of magnesium and chromium particles interface preserves for the maximum duration of immersion time, as selected 96h. This illustrates the significance of sorting Cr particles as reinforcement, being noble for Mg-based composites.

Lastly, Fig. 4(b) signifies the shining cracks are due to the incorporation of Cr reinforcement in the Mg-matrix. As Cr is having soft shinning nature property[27]. As Cr reinforcement increases, the shallow cracks reveal more shiny deep cracks with the formation of oxides. These deep cracks increase due to the chemical reaction in alkaline NaCl nature as an increase in the duration of time[28].

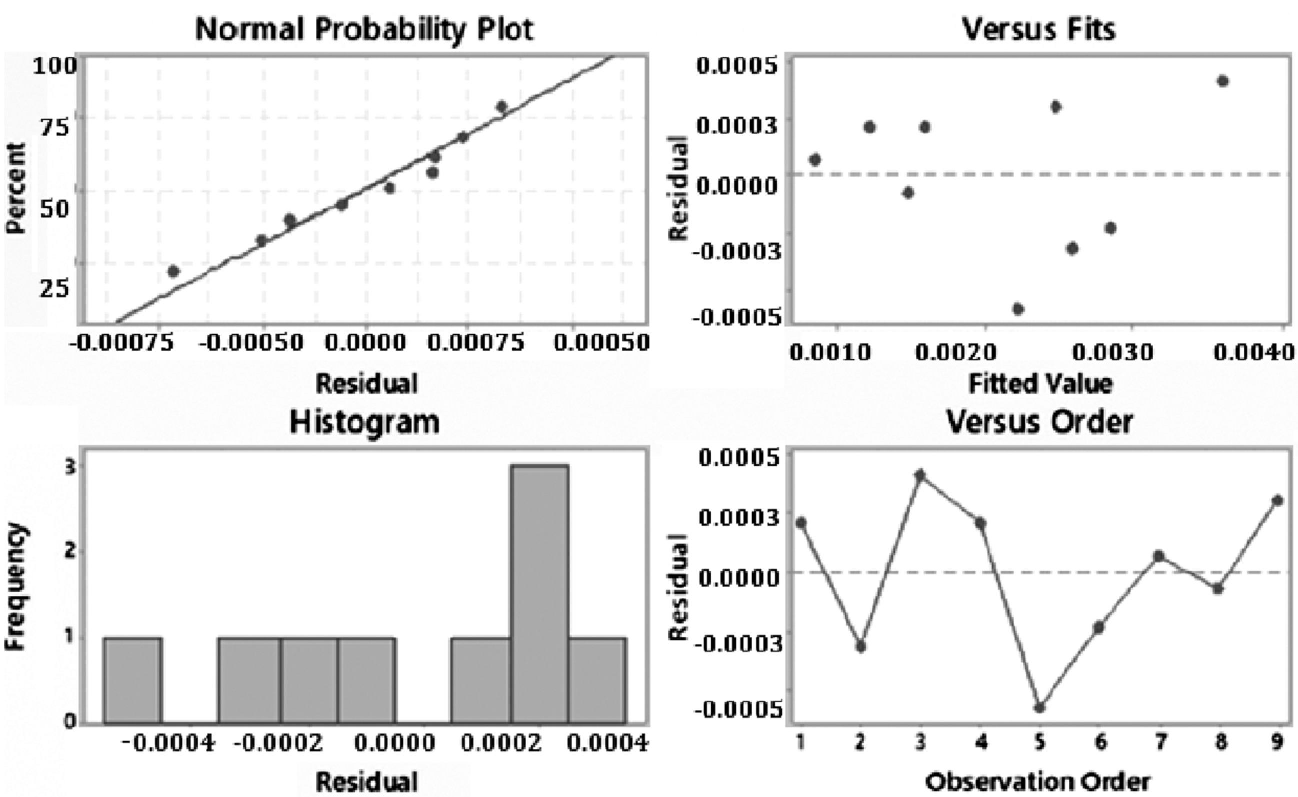

Fig. 2 — Residual plots for Corrosion Rate (MPv)

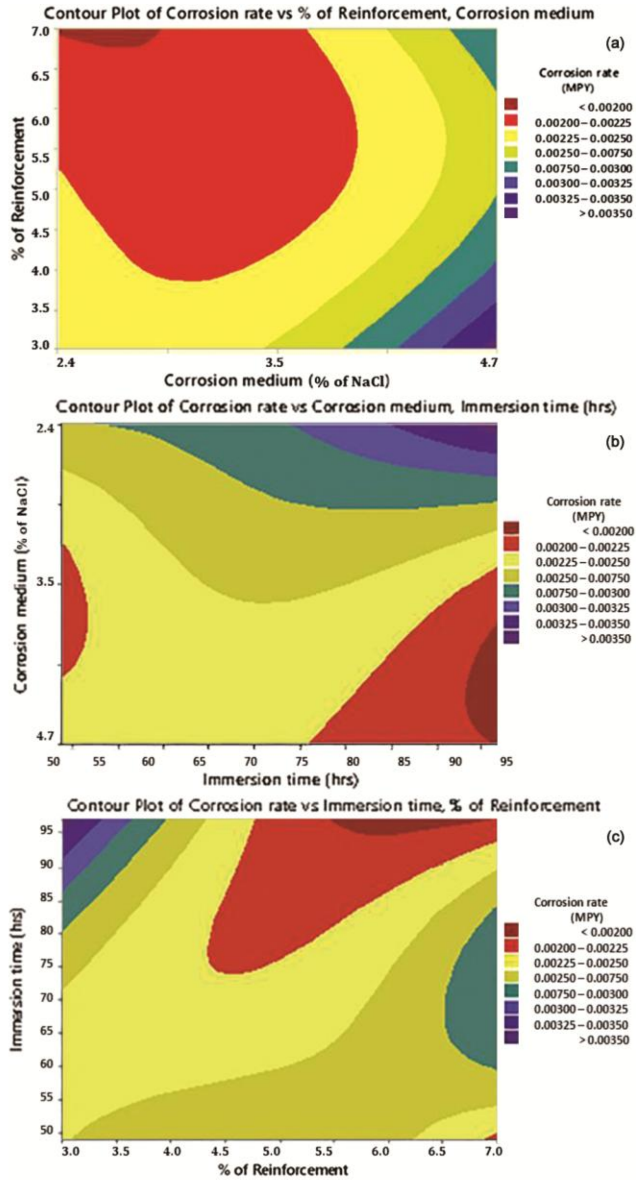

Fig. 3(a) — Plotting of CR of chromium % and Immersion medium; (b)-Plotting of CR of Immersion medium and time; (c)-Plotting of CR of Immersion time and Cr %

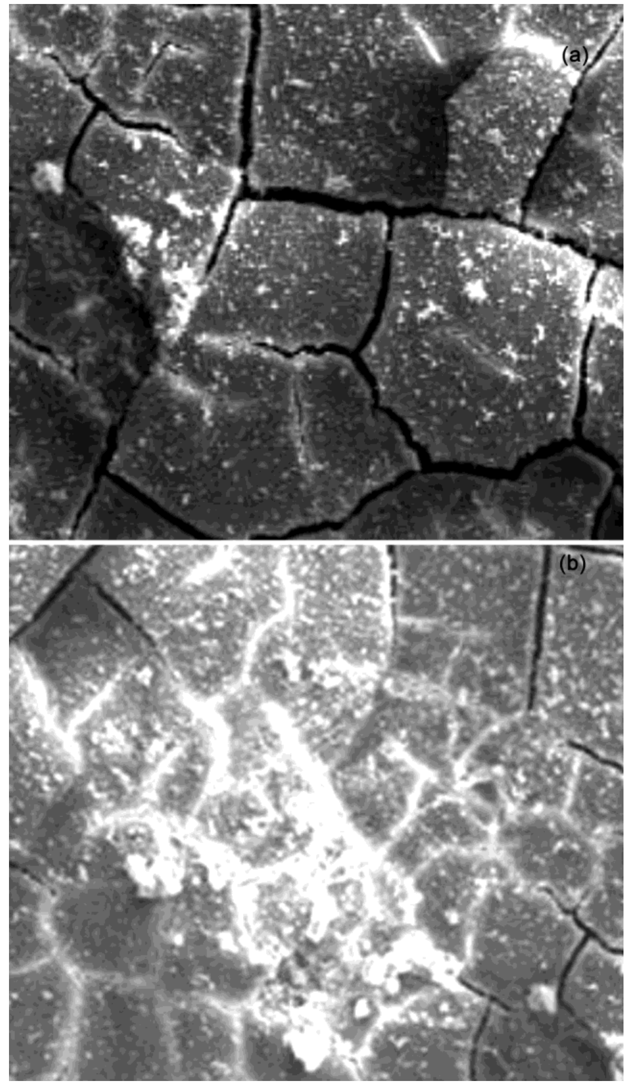

Fig. 4 — Corroded microscopy (50µm magnification) of (a) Pure Mg and (b) 5wt. % Cr-based composite under 3.5% NaCl solution for 96h

Conclusions

The CR of Mg/Cr-based composites through bottom pouring squeezed stir casting technique have been studied and optimized successfully.

The following optimized outcomes have been drawn as follows:

1. The percentage of corrosion medium factor is highly influenced as output response when compared to other factors.
2. From the above factors, the percentage of corrosion solution is influenced upto 66.10%, reinforcement percentage contributed to 27.56% and the last i.e. immersion time influenced only 2.81%.
3. However, counterplots explain the effective occurrence of the corrosion process of Mg/Cr composite surfaces.
4. The optimized combination of CR parameter is 7wt. % Cr reinforcement with 2.4% of alkaline solution (NaCl) under the immersion time of 96h.
5. Deep shiny cracks have been observed in Mg/Cr composites due to the chemical reaction between the composite and alkaline medium.
6. From these counterplots, it is observed that the region of the Mg/Cr interface preserve for a maximum period of 96h and illustrates the significance of sorting Cr particles as reinforcement, being noble for Mg-based composites.

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