Synthesis and characterization of magnesium melting fluxes

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Keywords: magnesium melting, covering flux, decomposition temperature, surface morphology, weight loss

Abstract
Magnesium and its alloys are extremely susceptible to oxidation and fire due to their reactivity with oxygen. Therefore, special care should be taken while melting them. In this research, different types of fluxes were used to protect the molten magnesium from oxidation. These fluxes cover the molten magnesium surface and refine it. In this research, nine magnesium melting and refining fluxes were developed. Dow fluxes compositions were taken as a base to prepare five fluxes and another four fluxes were developed by varying the content of chlorides, fluorides, and oxide. The main purpose of this research is to increase the recovery of magnesium metal by melting in normal atmospheric conditions and develop a low-cost conventional melting practice. Decomposition behavior, mass change, and melting of these nine fluxes were studied by TG/DTA technique. After fusing, the surface morphology of these fluxes was studied by visual observation and scanning electron microscope. Magnesium was melted using these nine fluxes and for each flux, the weight loss of magnesium was calculated. From a thermal analysis study, it was confirmed that all the fluxes were fused before magnesium melting. Based on the flux layer which was generated during the melting and the weight loss analysis, flux 9 (250) was the best flux compared to other fluxes. The highest purity was obtained by using flux 2 with the highest tensile strength value.

1. Introduction
Magnesium alloys are widely used in numerous applications, from automotive to electronics, due to their unique characteristics. Magnesium shows high potential to substitute conventional materials such as iron and aluminum in specific applications where damping resistance is important [1–4]. Magnesium is a relatively low-cost metal with low density and machinability. All these properties make it an excellent candidate as a structural material [5, 6].

Molten magnesium has a high tendency to oxidize. So, to protect it from oxidation and burning, surface care should be taken. If the oxidation of magnesium is not controlled, a porous, non-sticky MgO layer is formed on the surface of the molten metal. This layer cannot be prevented, so it creates a passage of oxygen into magnesium melt. As a result, the liquid metal burns and forms more oxide. In addition, magnesium and its alloys evaporate easily, so the extremely fine powder will form around the cold areas of the melt. This magnesium dust easily ignites due to the high surface-to-volume ratio. Therefore, to prevent the melt from oxidation and control magnesium from evaporation is very crucial [7–10].

Magnesium has a tendency of oxidizing and contaminating with non-metallic inclusions. So, casting is done by fluxing technique or flux-less technique (in presence of gas or vacuum) [11]. Non reactive gases, such as nitrogen, argon and oxide film modifiers like sulfur hexafluoride (SF₆), sulfur dioxide (SO₂) are used in the fluxless method. Sulfur hexafluoride is an effective cover gas due to its ability to form a dense film containing magnesium oxide (MgO) and Mg fluoride (MgF₂) on the molten magnesium surface. This film prevents further oxidation and evaporation of magnesium. However, SF₆ is very expensive and an extremely powerful greenhouse gas, with a 100-year global warming potential which is estimated at 23,900 times that of carbon.
dioxide (CO₂) and hence the use of SF₆ is prohibited in many countries. Gases that are used to cover the molten magnesium surface should be continuously (fresh) supplied, and should be free from moisture. Due to the high cost and environmental pollution, the alternative fluxing technique is also preferred by many magnesium casting industries [12–15]. In the fluxing technique, each type of flux plays an important role in the magnesium melting and refining process. The main characteristics of the covering fluxes are to protect the magnesium melt against oxidation, melt before melting of magnesium, cover the surface properly, and form a dense strong film that is easily separable during pouring. As per individual characteristics, a combination of chlorides, fluorides and oxides is used as a flux that is shown in table 1 [16–22].

The main purpose of this study is to develop magnesium melting fluxes by varying the composition of chlorides, fluorides, and oxides and compare them with industrial fluxes developed by DOW Company. In this study, five industrial fluxes compositions were taken as the base and prepared another four by changing the composition. The decomposition behavior and surface morphology of nine fluxes were studied after fusing. Magnesium casting has also been prepared using nine fluxes to study weight loss and cast product quality. Following table no. 1 represents the general role of chlorides, fluorides, and oxides.

2. Experimental procedure

2.1. Flux preparation

All Fluxes were prepared by varying the amount of chlorides, fluorides, and oxides. Each chemical was preheated to 100 °C. All chemicals were ground, screened, and then hand-mixed by a glass rod for up to 5 min to get uniform distribution. The melting point of individual salts of chlorides, oxides, and fluorides are quite high compared to the melting point of magnesium metal and its alloys. Compositions of all prepared fluxes and the individual melting point of chemicals are mentioned in table 2 [20, 23–27].

2.2. Surface layer of flux on magnesium

A magnesium piece of 3 × 3 cm was cut from the magnesium ingot. The sample was kept in a small crucible and the top surface of this magnesium piece was covered with prepared flux before heating it to 640 °C in an electric resistance furnace. (Just before the melting of Mg metal.) The heated flux layer of the magnesium piece was analyzed by visual and scanning electron microscopy after cooling to room temperature. The same process is followed for all nine samples.

2.3. Magnesium casting

The magnesium ingot was covered by preheated fluxes inside the graphite crucible and melted by a resistance heating furnace. The layer of fluxes protects molten magnesium from oxidation. There was no cover gas used.

| Table 1. Characteristics of various chemicals used to synthesise the flux. |
|---|
| Chemicals | Characteristics |
| Chlorides | • The higher density of the chlorides causes the impurities to sink to the bottom of the melt as sludge. So, a combination of MgCl₂ and KCl was used to provide the low melting point eutectic. |
| | • MgCl₂ minimizes surface oxidation by creating a thin-film layer on the metal surface. |
| | • MnCl₂ is also used as an effective and economical additive in flux for the removal of iron in the production of magnesium alloys. |
| | • BaCl₂ was added to adjust the melting point. Due to its high density, encourage the flux and the flux-oxide particulates to settle at the bottom. |
| Fluorides | • Fluorides are added due to their better wettability and chemical reactivity with magnesium oxide. |
| | • The barium, strontium, and calcium fluorides provide the density required for the salt to effectively mix with the magnesium and then settle out at the bottom of the crucible by making high-density inclusions like MgF₂. |
| Oxides | • MgO absorbs the chlorides. |
| | • It also offers typical density to cover and refine the metal. |
during the melting operation. The flux layer on the molten magnesium surface was removed before pouring to avoid inclusions. The prepared liquid melt was poured into a preheated metallic mold (Cast Iron) in rod shape at 720 °C. All nine types of fluxes were tested by the same casting process. Experimental setup and final casting are shown in figure 1.

In this process, a surface study is not possible because complete melting can destroy the upper protected skin generated on liquid magnesium. The chemical composition of castings was carried out by JEOL JSM-5610LV Energy Dispersive Spectroscopy (EDS) and the data were analyzed by Oxford software. The accuracy of the instrument is 99.99 wt. %.

Table 2. Composition of Fluxes with an individual melting point.

| Sr no. | Flux no. | Composition (%) | Melting point (°C) | Sr no. | Flux no. | Composition (%) | Melting point (°C) |
|--------|----------|-----------------|--------------------|--------|----------|-----------------|--------------------|
| 1      | Flux 1 (320) | 76 MnCl₂        | 650                | 6      | Flux 6  | 45.5 MnCl₂      | 712                |
|        |          | 13 CaF₂         | 1382               |        |          | 40 KCl          | 770                |
|        |          | 11 MgO          | 2642               |        |          | 4 NaCl          | 800                |
| 2      | Flux 2 (220) | 57 KCl           | 770                |        |          | 4 CaCl₂         | 782                |
|        |          | 28 CaCl₂        | 782                |        |          | 5 CaF₂          | 1382               |
|        |          | 12.5 BaCl₂      | 959.8              |        |          | 1.5 MgO         | 2642               |
|        |          | 2.5 CaF₂        | 1382               | 7      | Flux 7  | 38.5 MgCl₂      | 712                |
| 3      | Flux 3   | 44.5 MgCl₂      | 712                |        |          | 36 KCl          | 770                |
|        |          | 38 KCl          | 770                |        |          | 4 NaCl          | 800                |
|        |          | 8 BaCl₂         | 959.8              |        |          | 4 CaCl₂         | 782                |
|        |          | 4 NaCl          | 800                |        |          | 16 CaF₂         | 1382               |
|        |          | 4 CaCl₂         | 782                | 4      | Flux 4  | 1.5 MgO         | 2642               |
|        |          | 1.5 MgO         | 2642               | 8      | Flux 5  | 45.5 MgCl₂      | 712                |
| 4      | Flux 4 (230) | 55 KCl           | 770                |        |          | 45 KCl          | 770                |
|        |          | 34 MgCl₂        | 712                |        |          | 4 NaCl          | 800                |
|        |          | 9 BaCl₂         | 959.8              |        |          | 4 CaCl₂         | 782                |
|        |          | 2 CaF₂          | 1382               |        |          | 1.5 MgO         | 2642               |
| 5      | Flux 5 (310) | 20 KCl           | 770                | 9      | Flux 9 (250) | 23 KCl      | 770              |
|        |          | 50 MgCl₂        | 712                |        |          | 72 MnCl₂       | 650                |
|        |          | 15 CaF₂         | 1382               |        |          | 2.5 BaCl₂      | 959.8              |
|        |          | 15 MgO          | 2642               |        |          | 2.5 CaF₂      | 1382                |
The hardness of the castings was measured using the FIE Micro Vickers hardness tester at 100 grams load. The averages of three measurements were reported for each system. Ultimate tensile strength and % elongation was measured by using a Monsanto-20 tensile testing machine at room temperature. The crosshead speed was adjusted to a value of 0.05 mm min$^{-1}$.

2.4. Thermogravimetric/differential thermal analysis
To understand the behavior of all nine fluxes during heating, from room temperature to till melting state, TG/DTA analysis was carried out. TG/DTA study indicates decomposition sequences, mass change and melting sequences of fluxes individually. The thermal analysis was carried out by NTEZSCH STA 449 F3 thermal analyzer system. Samples were analyzed from 23 °C to a maximum of 700 °C (973 K) with a constant heating rate of 10 °K min$^{-1}$ for each nine fluxes. Around 7–11 mg sample was taken for every analysis. Alpha alumina was used as reference material. Samples were tested in a nitrogen atmosphere as per standard procedure. The idea is to test all fluxes in air but due to presence of chlorides, oxides, or fluorides, it is not advisable to run the fluxes samples in the air. The results were analyzed for all the samples as per the thermal events exhibited by the samples.

3. Results and discussion

3.1. Thermogravimetric/differential thermal analysis of all fluxes
To melt the flux before the actual melting of solid magnesium metal, a proper combination of chemicals (salts) is very important. Not only that, but flux also had to start reducing the diffusion of oxygen on the surface of the
semisolid charge to avoid fires. According to the ternary diagram of three chlorides like MgCl₂, KCl and NaCl, melting behavior start at 385 °C–500 °C [21]. In this study, nine types of fluxes were prepared by varying the amount of MgCl₂, KCl, CaCl₂, BaCl₂, NaCl, MgO and CaF₂. Only one type of fluoride is used to improve the wettability problems of the major content of MgCl₂. By varying the amount of chemicals (salts), the melting behavior of the fluxes changed. Therefore, to understand fluxes behavior, thermal analysis has been carried out and it is reported in figure 2. Thermogravimetric/Differential Thermal Analysis graphs indicate the mass change (%) and decomposition temperature of fluxes at different stages.

As shown in figure 2(a), flux 1 decomposed at 560.7 °C temperature. DTA of Flux 1 shows three endothermic peaks at 73.7 °C, 134.6 °C, and 188.2 °C. TG graph of flux 1 shows, multistage decomposition. Around 22% of flux mass is reduced up to 250 °C due to the removal of chemically bounded water and volatile
matter present in the flux. 75.96% residual flux is present at around 700 °C. The decomposition temperature of flux 2 is 600.4 °C presented in figure 2(b). Physically bounded water present in barium chloride is released at 146.9 °C and 11.4% mass of flux is decreased at this stage. Magnesium chloride-containing fluxes like flux 3, 4, 5, 6, and 8 shows nearly a similar pattern in the DTA graph up to 250 °C temperature which is shown in figures 2(c), (d), (e), (f) and (h). Moisture present in hydrous magnesium chloride is driven off step by step in all 5 fluxes (2c, 2d, 2e, 2f, and 2h) and it is removed by the following reactions [24]:

\[
\text{MgCl}_2 \cdot 6\text{H}_2\text{O} \rightarrow \text{MgCl}_2 \cdot 4\text{H}_2\text{O} + 2\text{H}_2\text{O}; \\
\text{MgCl}_2 \cdot 4\text{H}_2\text{O} \rightarrow \text{MgCl}_2 \cdot 2\text{H}_2\text{O} + 2\text{H}_2\text{O}; \\
\text{MgCl}_2 \cdot 2\text{H}_2\text{O} \rightarrow \text{MgCl}_2 + 2\text{H}_2\text{O}
\]

(1) (2) (3)

DTA of flux 3 shows, sharp endothermic peak (melting) at 583 °C, and around 73.95% mass change occurs at this temperature. DTA of flux 4 shows two endothermic peaks at 450 °C to 540 °C which represents its melting point. Flux 5 and 6 are melted at 458.6 °C and 464.2 °C respectively. Flux 8 contains the majority of chloride and 1.5% oxide. Flux 8 shows two endothermic peaks at 436.5 °C and 634.7 °C temperatures represent its decomposition. Due to the presence of a maximum percentage of chlorides and 16% calcium fluorides, two sharp endothermic peaks are observed at 419.8 °C and 460.2 °C in flux 7 (figure 2(g)) compared to flux 8 (figure 2(h)). Flux 9 contains 72% manganese chloride, 23% potassium chloride, and a minor amount of barium chloride and calcium fluoride.

The decomposition of this flux starts at 427.5 °C and continues up to 483.5 °C (figure 2(I)). The volatile matter was removed before 200 °C in this flux. 25% to 40% mass change is observed in the TGA graph of flux 4, 5, 6, 8, and 9 up to 250 °C temperature. In the case of flux 7, 3.49% of mass reduction is observed due to the presence of more % of CaF₂ and the absence of BaCl₂ till 250 °C temperature. At around 700 °C temperature, 97.03% residual mass is present in flux 7 which is highest compared to other fluxes.

All thermal analysis results indicate that the proper covering layer of fluxes was started before the actual melting of magnesium metal. Thus, magnesium melting can be possible by using a different flux, but the amount of magnesium metal recovery varies from flux to flux. The present research work focuses on magnesium melting because once the burning of molten magnesium is controlled then alloying can be possible. Moreover, magnesium alloys like AZ series, AM series [25], Mg–Zn alloy [26], Mg–Sn alloy [27], Mg–Mn–Sn, Mg–Bi[28], Mg–Mn–Cu, Mg–Mn–Ni, etc were also prepared by using these fluxes.
3.2. Observation of surface layer of flux on magnesium

Figure 3 indicates a micrograph of fused flux layer on solid magnesium surface at 10X magnification. Figure 4 indicates a scanning electron micrograph at 100X magnification. It is observed that flux 1 generates a thick layer due to the presence of magnesium oxide. As shown in figures 3(b), (c), Flux 2 and 3 create a smooth layer on the magnesium surface. However, SEM image of them indicates the formation of small cracks (figures 4(b), (c)). Due to these cracks formation, oxygen entrapment chances are more so, it can affect the % yield of the final metal (magnesium). Flux 4 and 6 generate a porous layer along with the formation of bubbles and cracks on the surface. Flux 5 and 7 are fused on the surface and create a lumpy layer, which means some flux particles are fused and the remaining is non-fused at this stage. Flux 8 and 9 generate a smooth and adherent layer on the magnesium surface which protects the solid magnesium metal piece before actual melting.

3.3. Chemical analysis of all castings

During casting, the removal of fluxes is most important. If the flux is not removed properly from the melt surface, it forms non-metallic or intermetallic inclusions in the casting and that affects the properties of the product [12, 29]. Inclusions in magnesium and its alloys are difficult to avoid due to the magnesium and oxygen reaction in the air to form MgO and flux reactions with oxygen to form MgO [30]. This oxide inclusion may be formed during dross and sludge operations. Above 650°C temperature, oxidation of magnesium accelerates drastically [31].

To verify the presence of magnesium and other elements, a chemical analysis of all castings is carried out using EDS analysis, as shown in table 3. Chemical analysis of all castings shows that a minor amount of MgO, K, Na, Ca, or Mn inclusions is present in the casting. All inclusions are present due to the use of fluxes during melting and they are found in the form of films, clusters, or oxide particles [32, 33]. Non-metallic inclusion of MgO is found in every casting except flux 2 and 5 castings. Due to the presence of a larger amount of KCl and MgCl₂ and proper handling during casting practice, magnesium oxide in flux 2 and 5 was not found. Manganese
is observed in flux 1 and flux 9 due to the presence of MnCl₂ in both fluxes. Manganese is an effective element to control iron and other impurities and improve corrosion resistance so, the quality of magnesium casting is very good in the case of flux 1 and flux 9.

3.4. Effect of fluxes on the weight of magnesium castings
The weight loss of cast products was calculated as per formula (4).

\[
\text{Weight loss(\%)} = \frac{(\text{Weight of pure magnesium before casting–Final weight of casting}) \times 100}{\text{Weight of pure magnesium before casting}} \tag{4}
\]

Table 3. Composition of raw material and cast products.

| Material               | Flux used for casting | Mg    | Mn    | Ca    | K    | Na    | O    |
|------------------------|-----------------------|-------|-------|-------|------|-------|------|
| Raw Material (Pure Mg)  | —                     | 98.68 | 0.08  | —     | —    | —     | 1.24 |
| Mg Casting             | Flux 1 (320)          | 98.51 | 0.16  | —     | —    | —     | 1.33 |
|                        | Flux 2 (220)          | 99.75 | 0.08  | 0.01  | 0.16 | —     | —    |
|                        | Flux 3                | 97.91 | 0.03  | 0.01  | 0.05 | 0.02  | 1.98 |
|                        | Flux 4 (230)          | 98.70 | —     | 0.03  | —    | —     | 1.27 |
|                        | Flux 5 (310)          | 99.87 | 0.04  | —     | 0.09 | —     | —    |
|                        | Flux 6                | 97.86 | 0.04  | 0.04  | —    | —     | 2.06 |
|                        | Flux 7                | 97.82 | 0.08  | —     | 0.02 | —     | 2.08 |
|                        | Flux 8                | 97.48 | 0.03  | 0.16  | —    | 0.01  | 2.33 |
|                        | Flux 9 (250)          | 97.89 | 0.32  | 0.04  | —    | —     | 1.75 |

Figure 4. SEM analyses of all fused fluxes at 100X magnification.
For fluxes 1 to 5, weight loss is quite high and it is approximately around 12 wt. % while for fluxes 6 to 9 it decreases. In the case of flux 9 weight loss is only around 6%.

As per the thermal analysis result, flux 1 is decomposed at 560.7 °C but, due to the presence of magnesium oxide, a thick layer of flux was generated on the molten metal surface. It was not easily removed during casting and exposes the surface of molten magnesium to atmosphere hence oxidation is more at high temperature. So, casting developed by flux 1 has more weight loss compared to other fluxes. According to the literature, individual KCl and NaCl do not effectively break the oxide layer thus, MgCl2 and CaCl2 are added with them [21]. In flux 2, 3, 4, and 5, the maximum amount of KCl and MgCl2/CaCl2 is present and due to this around 11% to 13% weight loss occurs. Due to the proper combination of various chlorides, magnesium oxide, and/or calcium fluoride weight loss in flux 6, 7, and 8 is less which is 6.48%, 6.71%, and 8.5% respectively (figure 5). Flux 9 contains a maximum amount of MnCl2 and KCl which control intermetallic inclusions and protect magnesium melt from oxidation. It satisfies the maximum conditions required for magnesium melting as a covering flux. The surface layer generated after using flux 9 is very thin and easily separated from solid surface hence weight loss by using flux 9 is quite less compared to all other fluxes.

3.5. Effect of fluxes on the mechanical properties of magnesium castings

The hardness, ultimate tensile strength and ductility of all magnesium castings were measured as shown in figure 6. In all magnesium castings, a negligible difference is observed in hardness and ductility value. Hardness is lying between 36–40 HV and ductility value is around 8%–10%. The ultimate tensile strength of all castings is varying between 96 to 111 MPa [34].

![Figure 5. Flux vs weight losses (%).](image_url)

![Figure 6. Mechanical properties of all cast products.](image_url)
4. Conclusions

- Decomposition of all fluxes is started between 450 to 635 °C, and it protects the molten magnesium from oxidation.
- The presence of calcium fluoride reduces the decomposition temperature range and thus protects magnesium prior to actual melting by generating a thin and brittle layer at the magnesium surface.
- The addition of magnesium oxide in the flux controls the refining and the presence of inclusion into the metal.
- The combination of chloride and fluoride helps to protect the surface of magnesium metal before melting but flux 9 offers a brittle, thin, and uniform layer which ultimately gives the highest metal recovery i.e., 94%.

Acknowledgments

The authors would like to thank RCC/Dir./2017/335/26 research grant, The Maharaja Sayajirao University of Baroda for funding this research.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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