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Effect of Sodium-Containing Fluxes on the Residual Sodium Content and Distribution in Al–Mg Alloys

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Abstract: To get an insight into the residual sodium content of Al–Mg alloys, three types of sodium-containing fluxes were introduced into the melt with different magnesium content. The increment, existing form and distribution of sodium in Al–Mg alloys with different magnesium content were analyzed. The results show that the influence of different sodium salts on the increment of sodium in Al–10Mg alloy is significantly different. The NaF raised the sodium level in Al–10Mg alloy to the highest extent, Na₃AlF₆ coming second, and NaCl did not have an obvious influence. The magnesium element in the aluminum melt was found to be the key factor leading to the increment of sodium level. After the salt fluxing treatment of aluminum alloy with different magnesium content, this increment would be proportional to the content of magnesium. EDS mapping indicates the sodium introduced by the fluxes was distributed in the form of Na-rich particles in Al–Mg alloys and preferentially located near Al₃Mg₂(β) phase in the situation of high magnesium content.

Keywords: Al–Mg alloy; sodium-containing fluxes; residual sodium content; distribution; sodium-induced brittleness

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1. Introduction

Oxide inclusions are usually considered as one of the fundamental defects detrimental to the performances of aluminum alloys. To date, the most widely accepted and frequently adopted method to remove oxide inclusions from molten aluminum is fluxing treatment. Most salt fluxes are based on the NaCl–KCl or the KCl–MgCl₂ binary systems. Additives include other chlorides, fluorides, nitrates, carbonates or sulphates [1]. In the process of fluxing treatment, the salt fluxes are injected into the melt to absorb the inclusions, then float to the surface of the molten aluminum. When the salt fluxes are brought in contact with molten aluminum, the aluminum will pick up some impurities due to the exchange reactions between the fluxes and the melt [1].

Al–Mg alloy ingots with magnesium content higher than 2 wt.% often suffer from brittle fracture during the deformation process at high temperatures (200–400 °C) due to the high sodium content (>10 ppm) [2–4]. In the production process of this type of aluminum alloy, it is necessary to control the sodium content to avoid high-temperature brittleness [5]. To avoid introducing the sodium in the smelting process, factories always choose more expensive sodium-free fluxes instead of sodium-containing fluxes in the production of Al–Mg alloys (especially for those with high magnesium content), thereby increasing production costs. However, Tremblay [6] found that the sodium content was less than 3 ppm in the Al–5Mg alloys fluxed with fluxes containing NaCl. It was implied that not all sodium components in the fluxes will significantly increase the sodium content

of the alloys. At present, NaCl, NaF and Na_3AlF_6 are among the most widely used sodium salts in the production of fluxes. NaCl is the basic component of the NaCl-KCl system fluxes, while NaF and Na_3AlF_6 are considered to be effective components for the removal of nonmetallic oxide inclusions [7,8], and they are widely used in the smelting of recycled aluminum. In order to further clarify the principle of composition design of flux for Al-Mg alloy, it is necessary to study the influence of different sodium-containing fluxes on the residual sodium content of aluminum alloys with high magnesium content.

Some achievements have been made in the study of the existing form and distribution of sodium and the mechanism of high-temperature brittleness in Al-Mg alloys. Ransley [4] suggested that magnesium dissolves in the matrix in Al-Mg alloys with magnesium content less than 2 wt.%, while sodium exists as a ternary compound (AlSiNa); Once the magnesium content is increased to a higher level, magnesium will form Mg_2Si phase as a result of incorporation with the impurity silicon in the alloy, while sodium exists in the form of free atoms. However, the existing form of sodium in Al-Mg alloys with different magnesium contents have not been confirmed experimentally. Horikawa [9] proposed that the elongation of Al-Mg alloys with low magnesium content is not affected by sodium. When the magnesium content is higher than 2 wt.%, the alloys with trace sodium gradually become brittle. This phenomenon was attributed to the trace sodium segregate on the grain boundary to form a sodium film, thereby leading to a decrease in the strength of the grain boundary during high-temperature deformation. However, no sodium film was detected on the grain boundaries in that research. Lynch [10] denied the distribution of sodium in Al-Mg alloys as mentioned earlier. He considered that sodium probably clustered as particles instead of films, but it was a hypothesis. In addition, Feng [11] suggested that sodium introduced by flux is enriched on the grain boundary of Al-Cu alloy, reducing the bonding force between the matrix and CuAl_2 phase. Sweet [12] observed that sodium exists as discrete particles on the grain boundary in Al-Li alloy.

Thus far, the existing form and distribution of sodium in Al-Mg alloys have still been controversial, and there are few studies on the effect of sodium salt in fluxes on the residual sodium content in Al-Mg alloys. In this paper, the influence of different sodium salt components and the addition volume of fluxes on the content of residual sodium in Al-Mg alloy was studied, and the existing form and distribution of sodium in Al-Mg alloy were analyzed. The present study aims to provide an experimental basis for the design of appropriate salt fluxes used for Al-Mg alloys. Furthermore, it intends to shed a light on ways to avoid high-temperature brittleness.

2. Materials and Methods

The raw materials used in the experiment were commercial Al and Mg ingots, both with a purity of 99.7%. Al-Mg alloys with different magnesium contents were prepared according to the designed ratio. All input materials for preparing the fluxes used in the present experiment were chemically pure. They were mixed and melted by a certain ratio, and subsequently broken into fine particles after cooling. The compositions of fluxes are shown in Table 1.

Table 1. The component of salt fluxes.

Flux Name	Component
NK	50 wt.%NaCl + 50 wt.%KCl
NK- Na_3AlF_6	47.5 wt.%NaCl + 47.5 wt.%KCl + 5 wt.% Na_3AlF_6
NK-NaF	47.5 wt.%NaCl + 47.5 wt.%KCl + 5 wt.%NaF

The prepared Al-Mg alloys were melted and held at 740 °C in a resistance furnace, and the fluxes were added into the melts at the amount of 0.3, 1.5, 5, and 10 wt.% of the alloys, respectively. The melt was held for 15 min after fluxing and the dross was skimmed before pouring into the steel mold.

The samples for analysis were cut along the longitudinal section at the center plane of the as-cast ingot and followed by mechanical polishing. During the preparation, Anhydrous ethanol is used as a cleaning and cooling medium; the samples must not be contaminated by water, because the sodium will react with the water. The chemical composition of the Al–Mg alloy ingots was analyzed by SPECTROMAXx spark spectrometer (Spectro, Kleve, Germany). A Hitachi-SU8220 scanning electron microscope (Hitachi High-Tech Corporation, Tokyo, Japan) integrating with a flat-Quad EDX (SEM, Hitachi High-Tech Corporation, Tokyo, Japan) was used to analyze the morphology and composition of the samples; the acceleration voltage used was 5 KV.

3. Results and Discussions

3.1. Effect of Different Sodium-Containing Fluxes on the Residual Sodium Content in Al–10Mg Alloys

As can be seen from Figure 1, all fluxes cause an increase in the sodium content in Al–10Mg alloy after refining. However, there is a divergence upon the extent of increment for different fluxes. The initial sodium content in Al–10Mg alloy was 2.8 ppm, which increased to 7.9 ppm after 0.3 wt.% NK flux was added. When the addition amount was increased to 10 wt.%, the sodium content reached 18.2 ppm. When the flux was changed to NK–NaF, the sodium in the Al–10Mg increased remarkably. Even though at an addition of 0.3 wt.%, the sodium level in the alloy soared to 35.1 ppm, significantly exceeding the critical sodium content (10 ppm) that would cause high-temperature brittleness. When the added amount of NK–NaF flux reached 10 wt.%, the sodium content in the Al–10Mg alloy dramatically increased up to 264 ppm, approximately 15 times larger than that of melt refined by NK flux under the same conditions. The NK–Na₃AlF₆ flux posed a modest influence on the sodium content, which increased steadily over the addition from 0.3 to 1.5 wt.% and then stayed unchanged, similar to NK flux.

Figure 1. Effect of different sodium-containing fluxes on the residual content of sodium in Al–10Mg alloy.

The reason for the increase in sodium content in the alloy can be ascribed to the chemical reaction between the sodium component of the flux and alloying elements in the alloy. NaCl can react with Al and Mg in the melt to produce sodium [13], and the equilibrium constants of reactions at 740 °C are, respectively, 6.08×10^{-24} and 1.01×10^{-8} [14]. It can be seen that the increase in sodium was mainly caused by the reaction of Mg and NaCl because the smaller the reaction equilibrium constant is, the less the amount of sodium introduced is. Tremblay [6] used, respectively, 6.7 wt.% NaCl and 1 wt.% (NaCl–MgCl₂) flux to refine Al–5Mg. After refining, the sodium content of the alloy was less than 3 ppm,

which was lower than that of the Al–10Mg alloy added with 0.3 wt.% NK flux in our experiment. We suggest that the higher magnesium content was responsible for the higher sodium increment since the high concentration of reactant proceeded the reaction in the forward direction. Furthermore, MgCl_2 can effectively remove alkali metals and alkali earth metals in the aluminum melt [15]. Hence, a small amount of sodium produced by the reaction of NaCl and Mg could be removed by MgCl_2 . However, KCl in NK flux could not effectively remove the sodium. As a consequence, the Al–5Mg alloy in Tremblay's experiment had a lower residual sodium content than the present study. During the aluminum alloy smelting process, the amount of flux used is generally in the range from 0.05 to 0.5 wt.%, while the accumulative amount of flux is sometimes multiplied to 1–15 wt.% in the waste aluminum recycling process due to the different types of input materials and treatment processes [16,17]. The sodium content in the Al–10Mg alloy fluxed by 0.3 wt.% NK flux is lower than that which causes high-temperature brittleness. NaCl can induce a slight amount of sodium increase in Al–Mg alloy with a high magnesium concentration, but it is not enough to cause high-temperature brittleness under the normal addition amount of flux in primary aluminum production.

NaF can also react with Al and Mg in the melt, and the equilibrium constants of reactions at 740 °C are 6.95×10^{-9} and 3.59, respectively. Compared to the NaCl, it is clear that the reaction between NaF and Mg is much easier, which indicates a higher increment of sodium. When using NK– Na_3AlF_6 flux, the sodium content in the Al–10Mg alloy is significantly higher than that of NK flux. The reason is that Na_3AlF_6 would partially ionize into NaF and AlF_3 in the molten state at high temperature [18], and then NaF reacted with Mg to form free sodium. Therefore, NaF is the key factor causing the increase in sodium in Al–Mg alloy. The NaF and Na_3AlF_6 in the flux will significantly increase the sodium content of the alloy during the refining of Al–Mg alloy or the recycling of Al–Mg alloy. From this perspective, these two sodium salts should be avoided in the refining of alloys that have a limitation on sodium content, especially in the refining of Al alloys with high magnesium content in high-temperature brittleness is prone to occur.

3.2. Effect of Mg Content on the Residual Content of Sodium in Al–Mg Alloys

As Figure 2 shows, the sodium content in all alloys increases with the addition of NK–NaF flux increasing. Nevertheless, there is a significant difference in increment. At the same addition level, the sodium content in CP–Al (99.7% purity) is the lowest, whereas the sodium content in Al–10Mg alloy is the highest. When the addition of flux increased from 0.3 to 10 wt.%, the sodium content in CP–Al increased from 1.4 to 31.4 ppm. When 0.3 wt.% NK–NaF flux was added, the sodium content of Al–10Mg alloy was 35.1 ppm, which was 1.4 and 4.1 times that of Al–5Mg alloy and Al–2Mg alloy, respectively. When the addition of flux was increased to 10 wt.%, the sodium content of Al–2Mg, Al–5Mg and Al–10Mg alloy increased to 113.8, 172.1 and 264 ppm, respectively.

The sodium increment in CP–Al after refining with NK–NaF flux is much smaller than Al–Mg alloys, as the sodium increment in CP–Al was introduced by the reaction between NaF and Al, whose equilibrium constant is much smaller than that between NaF and Mg. As shown in Figure 3, the residual sodium increases with the flux addition while the magnesium content decreases sharply, which further indicates that the increase in sodium in Al–2Mg alloy was mainly due to the reaction between NaF and Mg. Therefore, fluxes containing NaF reduce the magnesium content whilst they increase the sodium content during the recycling of Al–Mg alloys.

Figure 2. Effect of NK–NaF flux on the residual content of sodium in Al–Mg alloys.

Figure 3. Effect of NK–NaF flux on the contents of sodium and magnesium in Al–2Mg alloy.

At the same addition of flux, the sodium increment of Al–10Mg alloy was the largest, followed by Al–5Mg alloy, and Al–2Mg alloy was the smallest. The higher the magnesium content is, the higher the sodium increment in Al–Mg alloy. This is mainly due to the high concentration of magnesium facilitating the reaction toward the direction of sodium formation. The calculated phase diagram of Al–Mg–Na ternary alloy showed that the solubility of sodium in the alloy increases with the increase in magnesium content in Al–Mg alloys [19]. In other words, the sodium introduced by the reaction was more likely to be dissolved in the alloy without gasification. Therefore, the Al–Mg alloy with higher magnesium content is more likely to accommodate sodium, and hence make the greater increment of sodium possible.

3.3. The Distribution of Residual Sodium in Al–Mg Alloys

Figure 4 shows the distribution of Na, Mg and Si elements in the as-cast Al–2Mg alloy which was treated by 10 wt.% NK–NaF flux. It shows that the impurity element silicon was not completely dissolved in the Al–2Mg but formed as Si-rich particle. Due to the nonequilibrium solidification, most of the Mg stayed in solid solution during solidification, while a minority of Mg exist in the form of Mg-rich particles (as indicated by the arrow).

It is worth noting that sodium aggregates and distributes in the form of Na-rich particles. However, the nature of Na-rich particles cannot be drawn from the EDS mapping.

Figure 4. Elemental distribution of Na, Mg and Si in as-cast Al–2Mg alloy (a) Na, Mg and Si; (b) Mg; (c) Na; (d) Si.

The solubility of Na in liquid Al was found to be 0.115 at. % at 1023 K and the maximum solubility of Na in solid Al to be less than 0.003 at. % [20]. Na will enrich in the front of the solid–liquid interface during the solidification of the alloy. When the concentration of Na in the residual liquid phase is higher than 0.115 at. %, the Na droplet will repel out of the residual liquid phase because the concentration of Na in the residual liquid phase is higher than the solubility of Na in liquid Al. Liquid sodium is immiscible with respect to liquid aluminum [20]. Due to the interfacial tension between the liquid aluminum and liquid sodium, the repelled sodium exists in the form of droplets in order to minimize the interfacial energy. Whether the sodium droplets are entrapped in the matrix depends on the size of the droplets and the growth rate of solid aluminum. The smaller the Na droplet is and the faster the solid grows, the easier it is for the droplet to attach to the solid. When the solid grows further, the Na droplet will be engulfed by the matrix. Such droplets are uniformly distributed in the matrix. During solidification the droplets are driven to preferentially collide in the residual liquid phase, and they have a large chance to coalesce and coarsen for further decrease in the interfacial energy. The big droplets remain in the liquid phase until the temperature is lowered enough [21]. Under the experimental conditions of this work, the temperature gradient of the alloy is large and the growth rate of solid phases is fast. So many small Na droplets, once formed, are uniformly distributed in the matrix and a few big Na droplets enrich in the last part of solidifying liquid.

Figure 5 shows the distribution of Na, Mg and Si elements in the as-cast Al–5Mg alloy which was treated by 10 wt.% NK–NaF flux. All the regions showing high Mg content overlap with the regions enriching in Si and the trace impurity silicon and alloying element magnesium formed Mg_2Si compound in the Al–5Mg. Therefore, the regions containing very high Mg content are Mg_2Si compounds and the regions that enriched less Mg are

the $\text{Al}_3\text{Mg}_2(\beta)$ phase. Compared to Al–2Mg alloy, $\text{Al}_3\text{Mg}_2(\beta)$ phase precipitated during solidification in Al–5Mg alloy. Meanwhile, sodium exists in the form of Na-rich particles in the Al–5Mg alloy as the same as that of Al–2Mg, and it was positioned near the $\text{Al}_3\text{Mg}_2(\beta)$ phase. Horikawa [9] found that silicon could inhibit the high-temperature brittleness of Al–Mg alloys with high magnesium content. That sodium could be attracted to the edge of Mg_2Si in the presence of silicon reduces the amount of sodium at the grain boundary. However, in the production of wrought Al–Mg alloys, the addition of silicon is rarely used. It is therefore suggested to avoid sodium increment of the alloy by a cautious selection and use of salt flux.

Figure 5. Elemental distribution of Na, Mg and Si in as-cast Al–5Mg alloy (a) Na, Mg and Si; (b) Mg; (c) Na; (d) Si.

Figure 6 shows the distribution of Na, Mg and Si elements in the as-cast Al–10Mg alloy with an addition of 10 wt.% NK–NaF flux. It can be seen that there was more $\text{Al}_3\text{Mg}_2(\beta)$ phase precipitated during solidification in Al–10Mg alloy. Similar to Al–5Mg alloy, the impurity element silicon was in the form of Si-rich particle in Al–10Mg alloy, whilst the sodium exists in the form of Na-rich particles beside the $\text{Al}_3\text{Mg}_2(\beta)$ phase. Different from Al–5Mg alloy, besides the large red spots shown in Figure 6, there are some small red spots scattered in the Al–10Mg alloy, which indicates that possibly a part of sodium is distributed in Al–10Mg alloy in the form of fine particles. It can be seen from Figure 7 that these fine sodium particles are located at the edge of the $\text{Al}_3\text{Mg}_2(\beta)$ phase. The EDS spectrum clearly shows Na, Mg and Al in the particle, and the Na content is 16 wt.%.

Figure 6. Elemental distribution of Na, Mg and Si in as-cast Al-10Mg alloy (a) Na, Mg and Si; (b) Mg; (c) Na; (d) Si.

Figure 7. (a) SEM shows the Na and $\text{Al}_3\text{Mg}_2(\beta)$ phase; (b) the EDS result corresponding to the arrow.

As mentioned above, the Al-Mg alloy with higher Mg content picked up more Na. However, comparing Figures 4–6, we can see fewer Na-rich particles on the higher Mg content samples. That means that the higher the Mg content is, the higher content of Na is in solid solution in the matrix.

In summary, in Al-2Mg alloy, sodium aggregates in the form of Na-rich particles. Whilst in Al-5Mg alloy and Al-10Mg alloy, sodium aggregates mainly in the form of Na-rich particles near $\text{Al}_3\text{Mg}_2(\beta)$ phase. Furthermore, some small Na-rich particles are found distributed at the edges of the $\text{Al}_3\text{Mg}_2(\beta)$ phase in Al-10Mg alloy. Those findings are to some extent contradictory to the traditional theory that sodium segregates at the grain boundary and forms sodium film. The mechanism of high-temperature brittleness induced by the Na-rich particles is still unclear, and there is much work that needs to be done in the future.

4. Conclusions

(1) When Al–10Mg melt was treated by 0.3 wt.% (the dosage generally used in industry) of fluxes with sodium component of NaCl, Na₃AlF₆ and NaF, respectively, the corresponding residual sodium content of the alloy is 7.9, 22.7 and 35.1 ppm. NaF and Na₃AlF₆ more easily cause the increase in sodium in Al–Mg alloys, and they should be avoided in the refining of Al–Mg alloys with strict sodium content. In comparison, NaCl does not cause a significant increase in sodium in Al–Mg alloys.

(2) A sodium component in the flux, along with magnesium in the alloy, can cause increment of residual sodium content in alloys. The higher the magnesium content in the alloy, the more easily sodium is introduced and the higher the sodium increment. When 10 wt.% of NK–NaF flux was added, the sodium content of Al–2Mg, Al–5Mg and Al–10Mg alloy was 113.8, 172.1 and 264 ppm, respectively.

(3) The sodium introduced by the fluxes presents as Na-rich particles in the Al–Mg alloy, and mainly distributes near the Al₃Mg₂(β) phase in the Al–Mg alloy with a high magnesium content.

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