

Properties of fluxes used in molten aluminum processing

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Abstract

Molten salts play an important role in the processing of secondary aluminum and aluminum dross. Molten salts may be used in a passive role to cover the metal from oxidation or in an active role to remove inclusions, alkali metals and magnesium, and to maximize the recovery of aluminum from oxide drosses as well as during melting of recycled scrap aluminum.

The physical and chemical properties of molten salts are reviewed and analysed in terms of applications to the aluminum industry. Salt mixtures are in most cases based on either the KCl-NaCl or KCl-MgCl₂ binary systems. Additives include other chlorides, fluorides, nitrates, carbonates or sulphates. Examples of industrial use of molten salts are presented.

1. Introduction

For the treatment of molten aluminum various molten metal processing steps such as alloying, grain refining, degassing and filtration are necessary. The term fluxing is used to represent all additives to, and treatments of, molten aluminum in which chemical compounds are used. Fluxes may perform several functions such as degassing, removal of Li, Na, Ca and Mg, inclusions removal, cleaning and alloying. In addition, fluxes are also used to minimize the formation of oxides and to promote metal/oxide separation, improving the metal recovery.

2. Flux compositions and properties

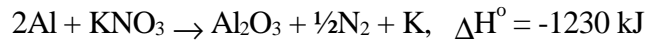
Solid fluxes are usually blends of chlorides and fluorides with additives to promote special properties. Many fluxes are based on a mixture of KCl and NaCl which forms a low temperature (665 °C) eutectic, improving the fluidity of the flux. Common ingredients are NaF and Na_3AlF_6 , and a common cover flux may contain about 47.5% NaCl, 47.5% KCl and 5% fluoride salt. Another group of cover fluxes are based on MgCl_2 -KCl which forms a low melting eutectic at 424 °C. These cover fluxes have high fluidity and form a thin layer on the melt surface. However, MgCl_2 is expensive as well as hygroscopic and is primarily used in Na-free fluxes for alloys containing more than 2 wt% Mg and which have very low limits of Na and Ca. Many ingredients are available as shown in Table 1. As seen in Table 2 these additives affect properties such as fluidity, wettability and reactivity.

NaF and KF based salts decrease the interfacial tension between the flux and the metal (Fig. 1) and between the flux and oxides (1). The reason is that with fluoride salts, the aluminum easily picks up some sodium or potassium, which both are surface active elements (2-4). Rapid reactions and a high degree of Marangoni induced flow take place when fluoride salts are brought in contact with molten aluminum. Chlorides, as well as AlF_3 and MgF_2 , exhibit this property to a much lesser extent (1-6). NaF and KF improve also the wettability which favours separation of oxide inclusions from the aluminum and metallic aluminum from the dross. Unfortunately, alkali fluoride salts lead to pickup of Na in the metal and their disposal is subject to stricter environmental regulations than for pure chloride salts.

The addition of oxygen containing compounds such as KNO_3 releases heat. The released oxygen from the decomposition of the nitrates, reacts with metallic aluminum yielding Al_2O_3 and considerable heat. This locally increases the fluidity enhancing the recovery of metallics suspended in the oxide. When using such reactive fluxes one should be aware of the possibility of explosions and splashing. Certain compounds decompose into chlorine, CO_2 or metal halide gases (AlCl_3). If they are introduced beneath the melt surface, they create bubbles that induce stirring and remove hydrogen and sodium. The most notable gas-releasing compound is hexachloroethane (C_2Cl_6) which generates Cl_2 and gaseous AlCl_3 .

Compounds that react with aluminum can be used to add certain alloying elements to the metal. NaF will add traces of Na to the melt, while K_2TiF_6 can add Ti, and KBF_4 adds B. A salt like AlF_3 , or compounds releasing chlorine remove Na, Ca, Li, Mg and Sr. Many secondary aluminum alloy producers use a NaCl-KCl based flux for oxidation protection and depend on Cl_2 or C_2Cl_6 for degassing (5-11).

Cover fluxes prevent oxidation of the molten aluminum by providing a physical barrier to oxidation. This is particularly important under highly oxidizing conditions ($T > 775\text{ }^{\circ}\text{C}$), melting of fines and chips, or making alloys containing more than 2 wt% Mg. Drossing fluxes are designed to promote separation of the aluminum oxide dross layer from the molten metal by reaction with metallic aluminum to generate heat and improve fluidity.



The liberated potassium lowers the surface-interfacial tensions and promotes coalescence of the aluminum droplets. Drossing fluxes are used to great advantage to lower the metallic content of drosses which may contain up to 60-80% free metal. Too little exothermic combustion reduces fluxing efficiency while too much flux burns excessively, creating fume and loss of metallic aluminum.

Refining fluxes contain compounds that break down and will react with sodium, magnesium, calcium, lithium and potassium to form compounds that can be removed by skimming. After a flux is used, a quiescent time for the bath is recommended to allow adequate settling of heavy inclusions or floating out of lighter-density fluxing salts and flux-wetted inclusions. A cover flux should be liquid at melt temperatures and drossing/exothermic fluxes should ignite. An exothermic wall cleaning flux is typically applied when the walls are as hot as possible to aid heating and softening of oxide buildup(12).

3. Thermodynamics of aluminum - molten salt reactions

The principal metallic impurities in molten aluminum are lithium, sodium and calcium in small concentrations (< 20 ppm) and magnesium in large concentrations (0.2 to 10%). The standard Gibbs energy of formation of several species is given in **Figure 2**(13). With a few exceptions, the thermodynamic stability decreases from the fluorides down to the sulphides in the following order:

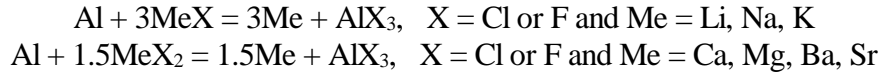


Among the exceptions are the industrially important substances Al_2O_3 and MgCl_2 . Due to the extreme stability of Al_2O_3 , only a fluoride based 'inert' electrolyte can be used in the Hall-Heroult process. Because of the low stability of MgCl_2 relative to the alkali chlorides, a NaCl-KCl-CaCl_2 based 'inert' electrolyte can be used for the electrolytic production of magnesium from MgCl_2 .

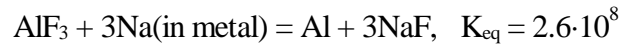
Metal chlorides with a ΔG° value more negative than that for AlCl_3 are more stable than AlCl_3 . This means that when Cl_2 is injected into aluminum containing various metallic elements, the chlorine will preferentially react with these metallic impurities. The same also applies to fluorides. Li, Na, K, Ca, Mg and Ba all form more stable chlorides and fluorides than aluminum and can therefore be removed by Cl_2 , F_2 , C_2Cl_6 or SF_6 injection. The reaction in the case of Mg is



MgCl_2 is a liquid above $712\text{ }^{\circ}\text{C}$, it is less dense than aluminum and it will tend to float to the surface. The equilibrium constants for reactions such as



are shown in **Figure 3**. Equilibrium constants much greater than one imply that the reaction proceed as written while a value much less than one, indicates that the reaction goes in reverse. Therefore, an alkali or alkali-earth chloride electrolyte has no tendency to react with aluminum while metal fluoride electrolytes are more reactive. A chloride electrolyte is therefore suitable for the refining of aluminum during Cl_2 injection since it will promote the removal of alkali/alkali-earth metal impurities while maintaining high aluminum recovery. The removal of other impurities such as Zn, Si, Fe, and Cu by chlorine or fluorine treatment is basically impossible and when fluxes contain these metals, they will contaminate the aluminum. To remove sodium or lithium from primary aluminum, the TAC process which employs the injection of AlF_3 powder into the metal, may be used.

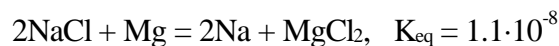


This reaction is highly favourable and sodium and/or lithium are removed from the aluminum.

4. Control of magnesium during treatment of aluminum alloys

Many useful alloys contain magnesium in quantities from 0.1 to 10%. These materials constitute a significant portion of the scrap market and are therefore available for recycling/remelting. Mill recyclers often need to produce new alloy products with lower magnesium content, and secondary smelters producing die cast and foundry ingots also need to produce low magnesium content alloys. Therefore, there is often a need to demag aluminum scrap during these remelting operations.

Based on the thermodynamic data given in **Figure 4**, it is clear that magnesium can not be removed based on exchange reactions with NaCl and KCl based fluxes alone. This is illustrated by the very low equilibrium constants for exchange reactions such as:



To determine the actual sodium content in Al-Mg alloys in equilibrium with a salt flux, we need to know the activity of the various species involved in the reaction. The molten NaCl-KCl system is nearly ideal while MgCl_2 shows a strong negative deviation from ideality. **Figure 5** shows the activity coefficient of MgCl_2 in NaCl, NaCl-KCl, and KCl melts at temperatures between 723 °C and 823 °C(23-25). The strong negative deviation is caused by the formation of MgCl_4^{2-} complexes which are stabilized by large cations with low charge such as K^+ . For use in the present analysis, the activity coefficient(γ_{MgCl_2}) of MgCl_2 in the equimolar NaCl-KCl melt is set to be 0.009. The activity coefficient of sodium in aluminum is about 426 while that of magnesium is 0.15. Using these activity coefficients, the sodium and magnesium contents in molten aluminum in equilibrium with an equimolar NaCl-KCl melt($a_{\text{NaCl}} = 0.5$), are related by:

$$Na(ppm) \approx 0.5 \times \left(\frac{wt\% Mg}{wt\% MgCl_2} \right)^{0.5}$$

Since magnesium can not be removed by chloride fluxing alone, either a reactive gas or another type of flux has to be used. In general there are three types of demagging processes; i) chlorination, ii) use of solid chlorine-containing fluxes(C_2Cl_6), and iii) the injection of AlF_3 or $NaAlF_4$ (14-21). One serious problem using chlorine gas to remove magnesium is that the demagging efficiency drops as the magnesium content in the metal falls. This may lead to emissions of $AlCl_3$ and HCl due to reactions with moisture in the air. In such cases, the use of a thin salt flux cover may trap the $AlCl_3$ gas before it is emitted into the atmosphere.



As seen in **Fig. 6(22)**, the capture efficiency of the salt increases with thickness up to about 2.5 cm.

AlF_3 , $AlCl_3$ or $SiCl_4$ additions promote the removal of magnesium from the metal. Similarly, by adding NaF and/or KF to a chloride flux, the removal of Mg from aluminum scrap is enhanced. However, the disadvantage is that this leads to contamination of the aluminum with Na and/or K as given by the following exchange reaction:

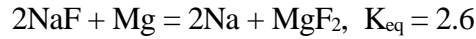
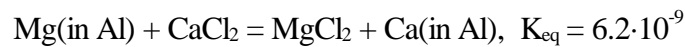


Figure 7 shows that the activity coefficient of $MgCl_2$ decreases by about 10 as 1 wt% NaF is added to equimolar $NaCl$ - KCl (26). The reason for this is that MgF_2 is much more stable than $MgCl_2$. The effect of this is that 1 wt% NaF additions to the salt flux increase the sodium content about 3 times above that with pure $NaCl$ - KCl . At higher fluoride contents, the sodium content will increase further since the activity coefficient of $MgCl_2$ decreases even more. This shows that as long as there are fluorides present in the flux and magnesium in the metal, the removal of Na is very difficult and higher concentrations of $MgCl_2$ in the flux are required.

In the case of magnesium alloys in contact with calcium compounds, the aluminum may pick up some calcium due to reactions such as

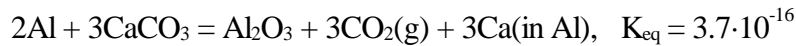


Although the equilibrium constant is small, since the activity coefficient of Ca in aluminum is very small (≈ 0.005), calcium is easily picked up by the aluminum.

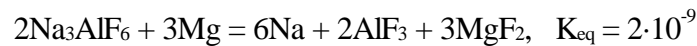
$$Ca(ppm) \approx 0.4 \times \left(\frac{wt\% Mg \times wt\% CaCl_2}{wt\% MgCl_2} \right)$$

Figure 8 shows how the sodium and calcium contents in aluminum vary with the $MgCl_2$ content of the cover flux used. These results are based on experimental tests with a Al -4.5% Mg alloy doped with sodium and calcium before the metal was treated with a 10% Cl_2 - 90% N_2 gas mixture for 30 minutes. These results are consistent with the $MgCl_2$ activity data given in **Fig. 6**. It is only after that the $MgCl_2$

content increases to 50 wt%, that it is possible to selectively remove the Na and Ca while keeping the magnesium in the alloy. When calcium carbonate is used (as flux or as caulking material), the following two reactions may cause calcium pick-up:



When using a cryolite flux for magnesium alloys, the following reaction may take place



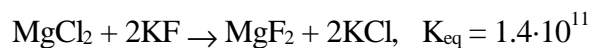
leading to pick up of sodium in the metal.

5. Inclusion removal from aluminum

Among common inclusions we find i) various refractory particles such as Al_4C_3 formed due to reactions with refractory materials, ii) oxide inclusions such as Al_2O_3 , MgO or MgAl_2O_4 films, clusters or dispersions formed during melting, alloying or metal transfer, iii) halide salt particles made up of MgCl_2 , NaCl and/or CaCl_2 formed due to poor separation of fluxing products from the metal. Figures 9 and 10 show that by the use of Cl_2 - N_2 mixtures, SiC inclusions can be removed from aluminum melts in about half an hour. As seen in Fig. 10 the inclusion removal efficiency can be enhanced by vigorously stirring the metal, allowing for decreased usage of Cl_2 gas in terms of time and concentration. Although chlorine gas is effective in removing inclusions, it has become a focus of stricter environmental regulations by government agencies(27). Experimental work over the last few years have shown that salt fluxes may successfully replace chlorine for this purpose. The injected salt flux may coat the inclusion particles, leading to i) coalescence of individual particles and ii) de-wetting of the inclusions from the aluminum. The result is that inclusions can be separated from the melt much easier.

It has been found that alumina inclusions can be removed from aluminum-magnesium alloys by holding the metal in chloride based flux with NaF or KF additions(28). As seen in Fig. 11 increasing amounts of NaF and KF increase the rate of inclusion removal. Beland et al(29) have shown on a plant scale that indeed salt flux injection has the potential to completely replace chlorine for the purpose of removing inclusions. At several Alcan installations, the so-called rotary flux injection technique is being used on a permanent basis(29).

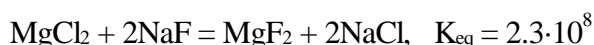
In the case of MgCl_2 - KCl based fluxes, MgCl_2 seemed to inhibit, as well as delay, the effect of the NaF and KF additions. For 50% or more MgCl_2 in the base flux, no inclusion removal was observed even with up to 10% KF additions. This can be explained by the following exchange reaction



effectively cancelling the effect of KF .

6. Recovery of aluminum droplets from a cover flux

With the use of salt fluxes we run the risk of losing metallic aluminum droplets entrained in the cover flux. With the use of some Cl_2 in the injected gas (22), or with the addition of fluorides to the salt mixture(30), as seen in Table 3 this problem disappears due to the enhanced coalescence of the fine droplets. For salt fluxes containing substantial amounts of MgCl_2 , fluoride salt additions become much less potent. As an example, for a melt with 45% MgCl_2 in KCl, a minimum of 10 wt% NaF is required to promote coalescence, as opposed to less than 1 wt% NaF in pure NaCl-KCl. The reason for this is that MgCl_2 will neutralise the alkali fluoride salts by reactions such as



7. Conclusions

From a thermodynamic point of view, metal fluorides are more stable than corresponding chlorides, oxides and sulphides. In salts, magnesium behaves irregularly. First of all, MgCl_2 is not a very stable salt as compared to alkali and other alkali earth chlorides. However, MgCl_2 forms MgCl_4^{2-} complexes, effectively stabilizing the magnesium chloride. MgF_2 is a very stable compound and when a fluoride salt is added to a chloride mixture containing magnesium, it will stabilize the magnesium in the salt.

The choice of which components to use in a flux depends on the objective(alkali removal, cleanliness, dross separation,...). For example, sodium-bearing fluoride containing fluxes should not be used with aluminum-magnesium alloys in order to avoid sodium contamination of the metal. When removing calcium from high magnesium alloys it is recommended to use a flux with around 50 wt% MgCl_2 . NaF, KF and Na_3AlF_6 additives are useful for the purposes of coalescence of small aluminum particles, recovery of aluminum from a dross flux and removal inclusions from the metal.

8. References

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Table 1. Characteristics of flux ingredients.

Chemical	Molecular Mass (g/mol)	Solid Density (g/cm ³)	Melting Point (°C)	Boiling Point (°C)
LiCl	43.39	2.068	605	1325
NaCl	58.44	2.165	801	1413
KCl	74.56	1.984	770	1500
CaCl ₂	110.99	2.15	782	1600
MgCl ₂	95.22	2.32	714	1412
AlCl ₃	133.34	2.44	190	177.8
BaCl ₂	208.25	3.92	963	1560
LiF	25.94	2.635	845	1676
NaF	41.99	2.558	993	1695
KF	58.1	2.48	858	1505
CaF ₂	78.08	3.18	1423	2500
MgF ₂	62.31		1261	2239
AlF ₃	83.98	2.882		s 1291
Na ₃ AlF ₆	209.94	2.9	1010	
LiNO ₃	68.94	2.38	264	d 600
NaNO ₃	84.99	2.261	307	d 380
KNO ₃	101.11	2.109	339	d 400
Li ₂ SO ₄	109.94	2.221	859	high
Na ₂ SO ₄	142.04		897	
K ₂ SO ₄	174.27	2.66	1069	1689
CaSO ₄	136.14	2.61	1450	high
MgSO ₄	120.37	2.66		d 1124
Li ₂ CO ₃	73.89	2.11	723	1310
Na ₂ CO ₃	105.99	2.532	851	high
K ₂ CO ₃	138.21	2.42	894	high
MgCO ₃	84.32	2.96		d 350
CaCO ₃	100.09	2.71	1339	850

s: sublimes; d: decomposes

Table 2. Properties of selected compounds used in fluxes.

Formula	Fluidity	Wetta- bility	Chemical Active	Exo- thermic	Gas Release	Element Added
AlF_3	↑		Yes			
CaCl_2	↑					
MgCl_2	↑		Yes			
MnCl_2	↑		Yes			Mn
KF	↑		Yes			K
NaF	↑		Yes			Na
NaCl	↑					
KCl	↑					
NaAlF_4			Yes			
CaF_2	↓	↑				
Na_3AlF_6	↓	↑	Yes			
Na_2SiF_6	↓	↑	Yes	Yes		
KNO_3	↑	↑	Yes	Yes	N_2 , NO_x	
C_2Cl_6			Yes		Cl_2 - AlCl_3	
K_2CO_3			Yes	Yes	CO_2	
Na_2CO_3			Yes	Yes	CO_2	
K_2TiF_6			Yes			Ti
KBF_4			Yes			B

Table 3. Coalescence ability of flux additions to NaCl-KCl at 740 °C.

5 wt % Salt	Coal-Escence	Color of flux	Gas Formed	Time (sec)	Comments
None	None	Clear	None	> 900	nothing happened
AlCl ₃	None	Clear	Small	> 900	no reaction after 2nd addition & agitation
MgCl ₂	None	slightly cloudy	Small	> 900	no reaction after 2nd addition & agitation
BaCl ₂	None	clear	Small	> 900	no reaction after 2nd addition & agitation
CaCl ₂	Poor	clear	Small	> 600	2nd addition & agitation required
LiCl	Poor	hazy, white precipitates	None	> 600	agitation required
MgF ₂	Fair	hazy	Small	11	no drop spinning & agitation required
CaF ₂	Fair	clear, grey precipitates	Small	14	no drop spinning & agitation required
AlF ₃	Good	grey/cloudy white precipitates	Small	7	droplets spun for about 1 second
LiF	Good	blue fog around droplets	Moderate	4	droplets spun for less than 1 second
Na ₃ AlF ₆	Excellent	hazy, blue fog, white precipitates	Moderate	< 1	droplets spun violently for 50 seconds
NaF	Excellent	clear, blue fog around droplet	Heavy	< 1	droplets spun violently for 17 seconds
KF	Excellent	clear, blue fog around droplet	Moderate	<1	droplets spun violently for 5 seconds

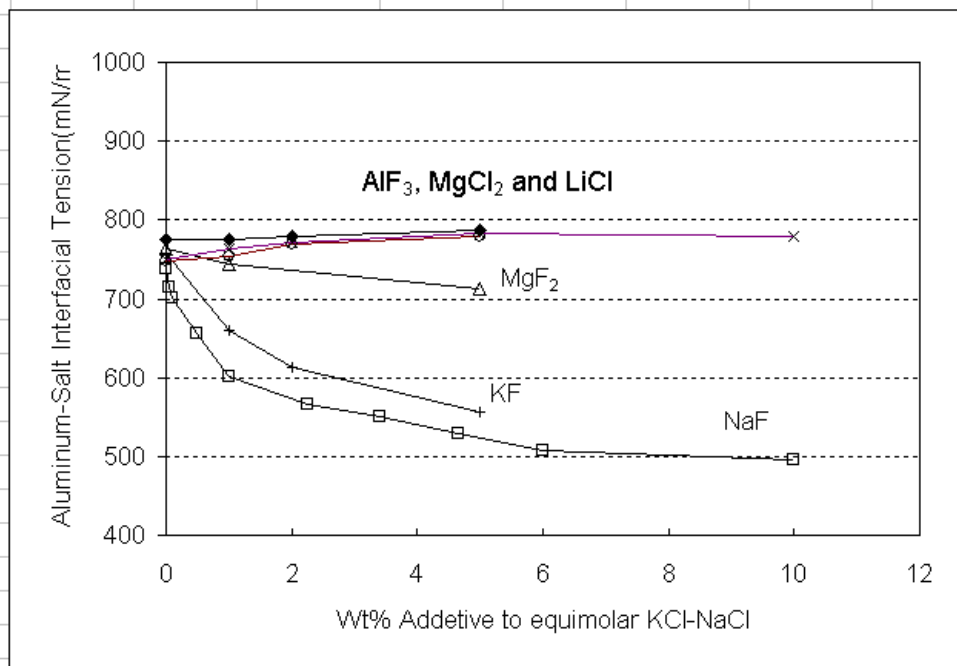


Figure 1. Aluminum-Salt Interfacial Tension

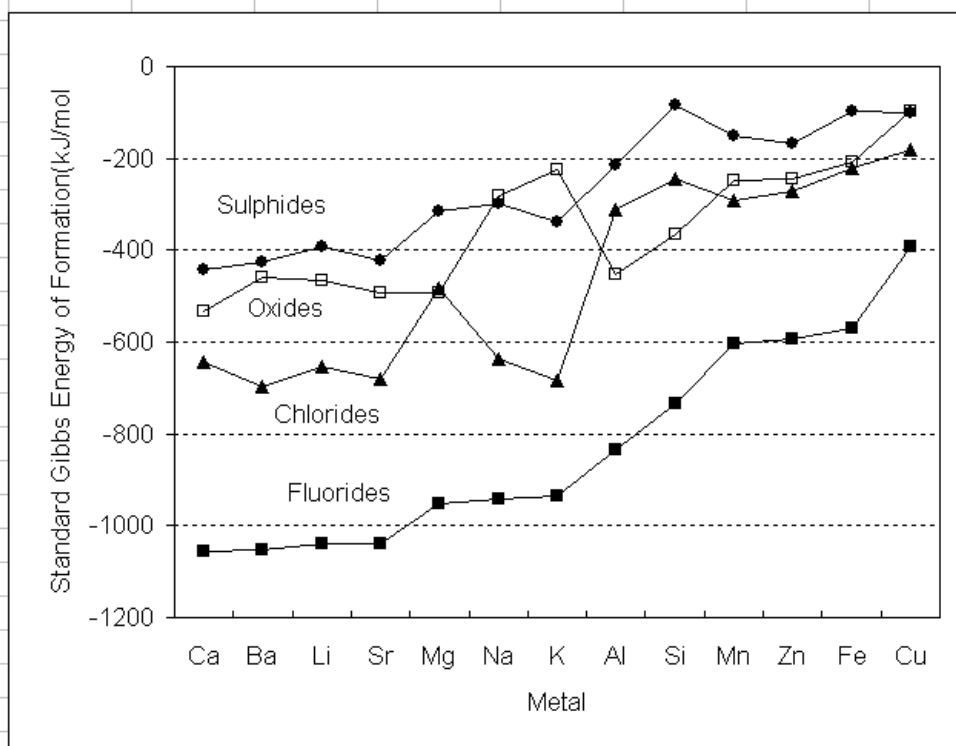


Figure 2. Standard Gibbs energy of formation of several sulphides, oxides, chlorides and fluorides. The data are given at 723 °C per mole of S, O, Cl_2 and F_2 (1).

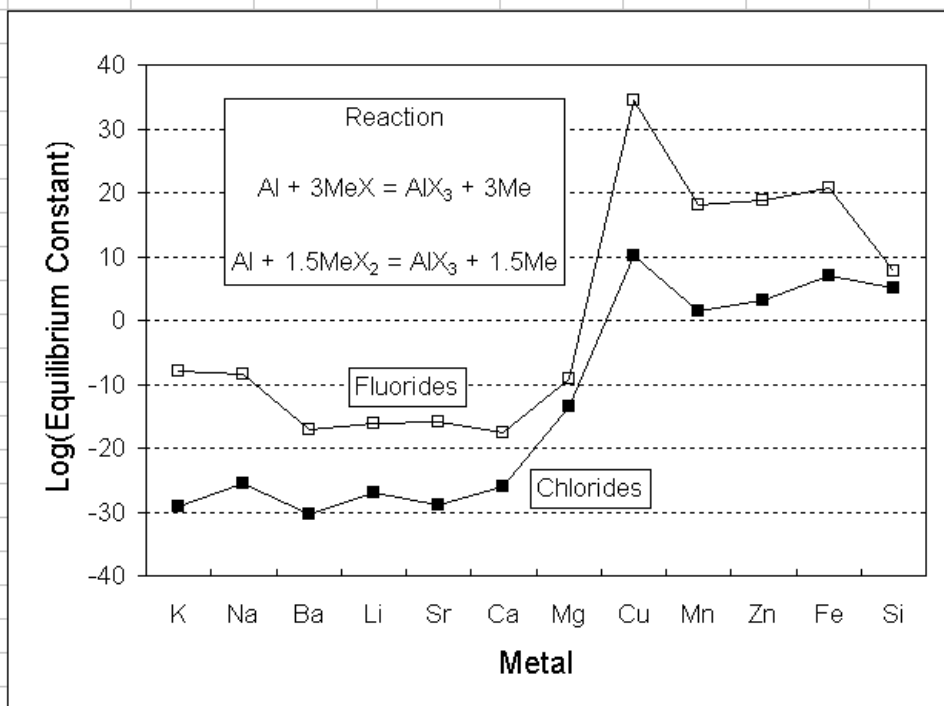


Figure 3. Exchange equilibrium between aluminum and different metal chlorides and metal fluorides at 723 °C.

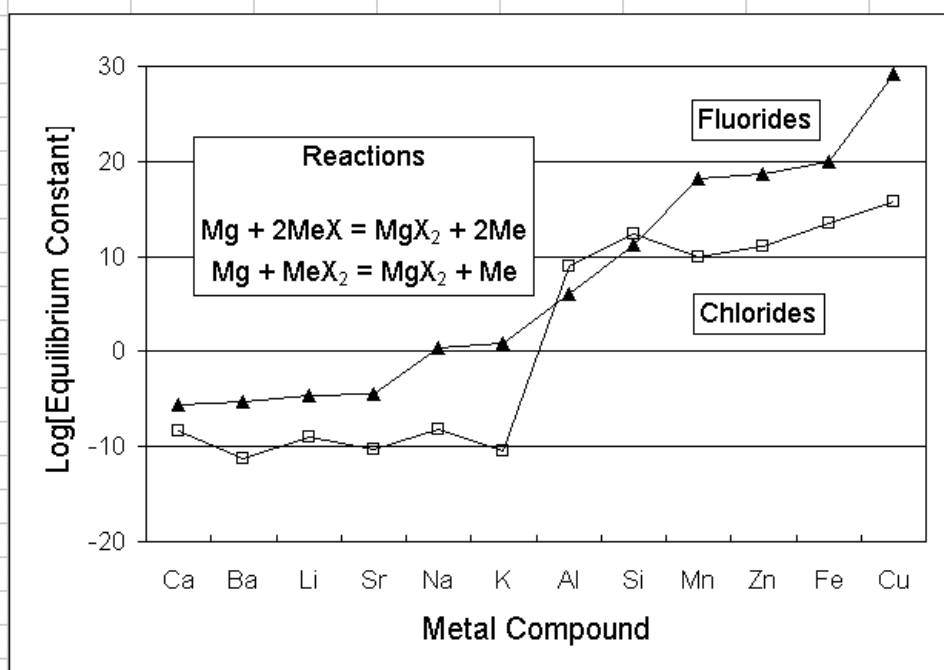


Figure 4. Exchange equilibrium between magnesium impurities in aluminum and different metal chlorides and fluorides at 723 °C.

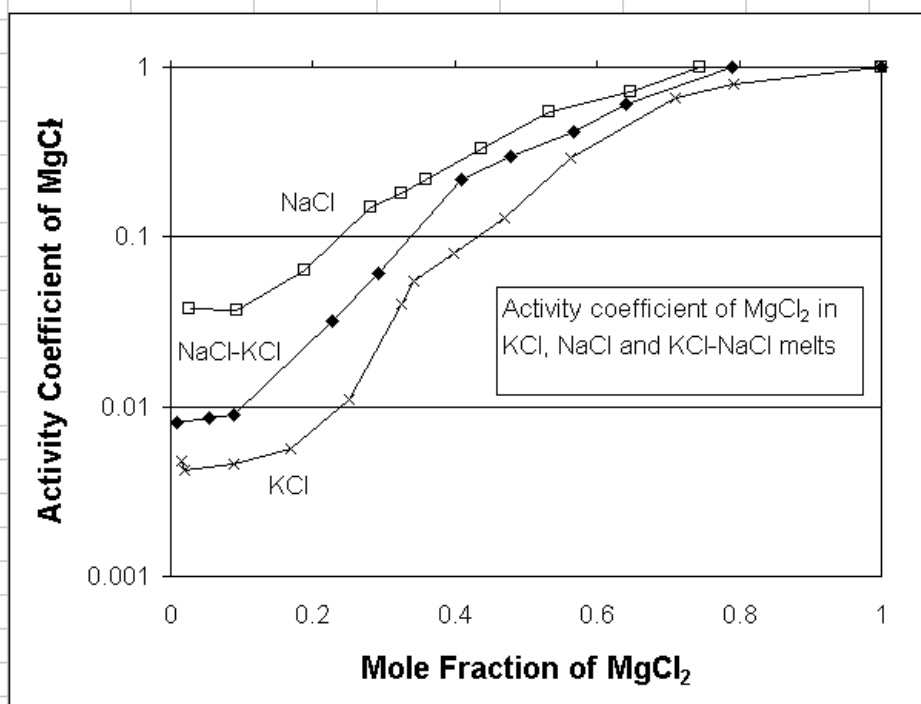


Figure 5. Activity coefficient of MgCl_2 in NaCl-KCl melts at 723 to 823 °C.

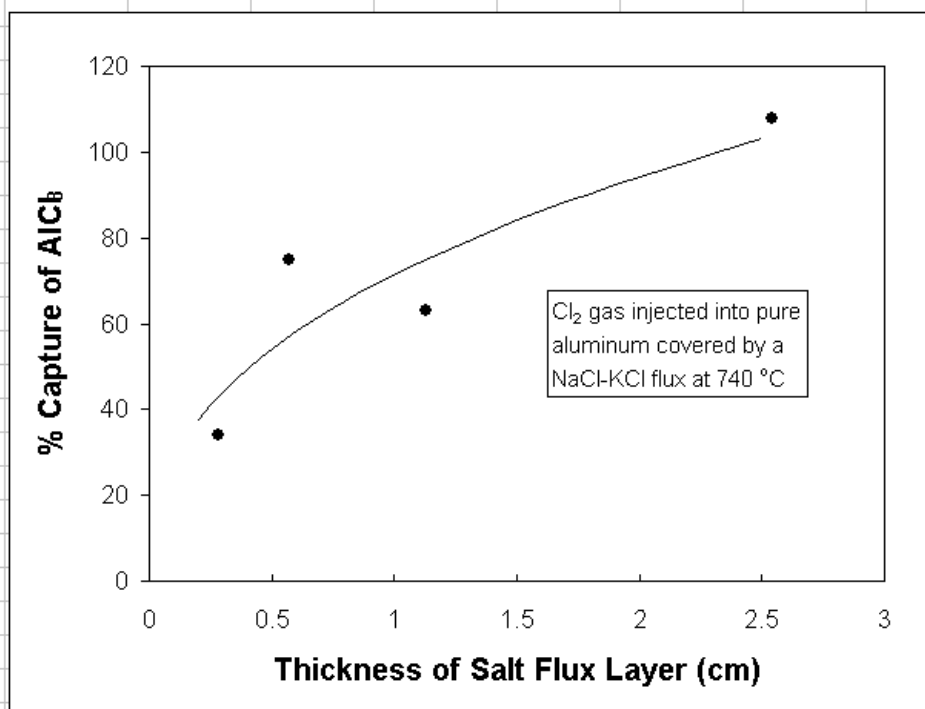


Figure 6. AlCl_3 capture efficiency versus salt flux thickness.

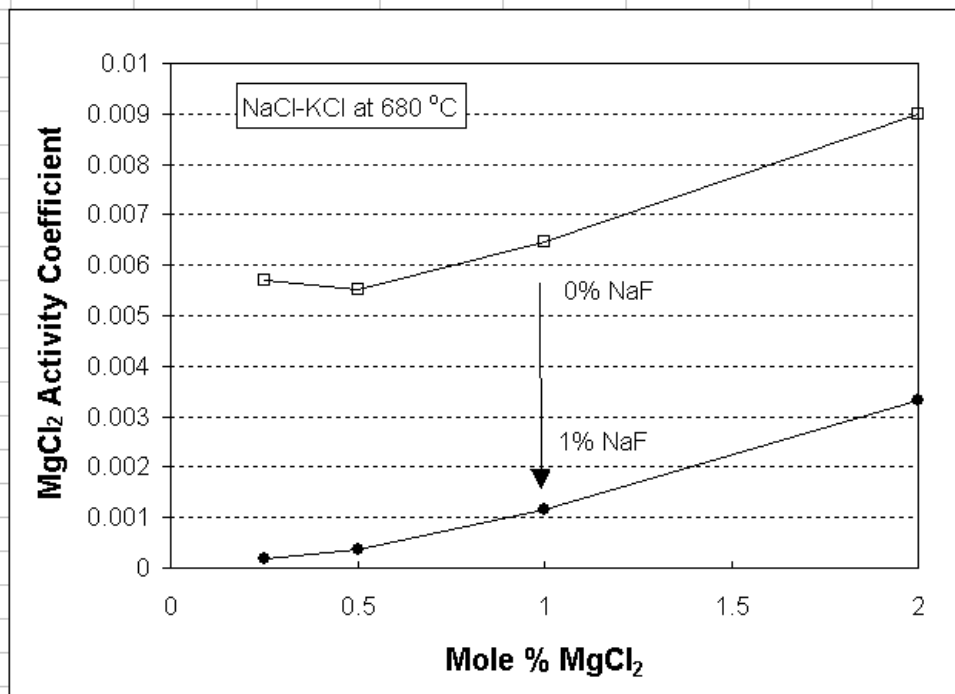


Figure 7. Activity coefficient of MgCl_2 in NaCl-KCl melts at 723 °C with NaF additions.

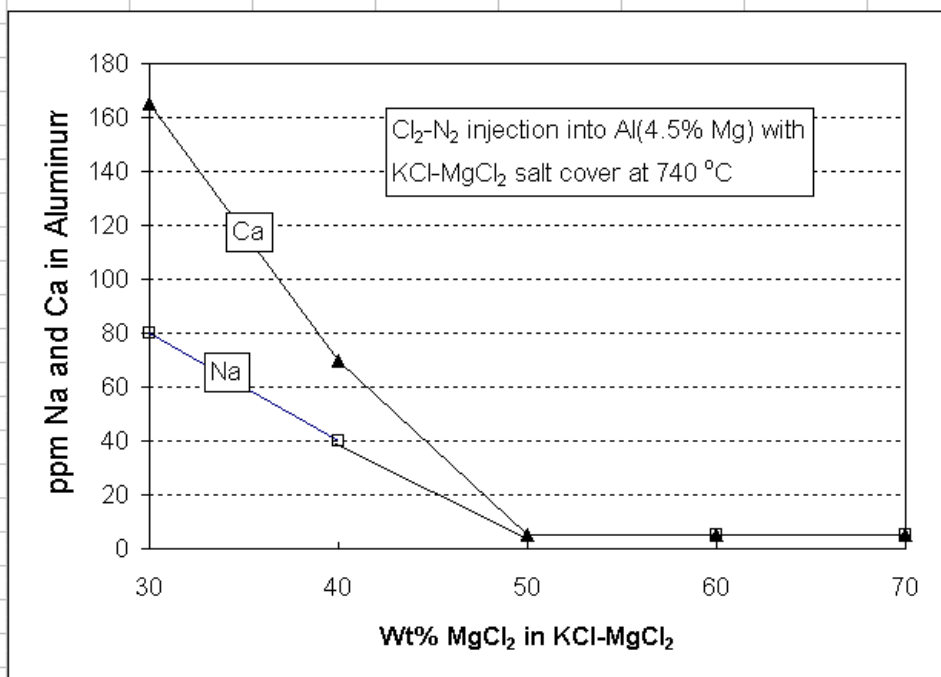


Figure 8. ppm sodium and calcium in aluminum versus MgCl₂ content in the flux.

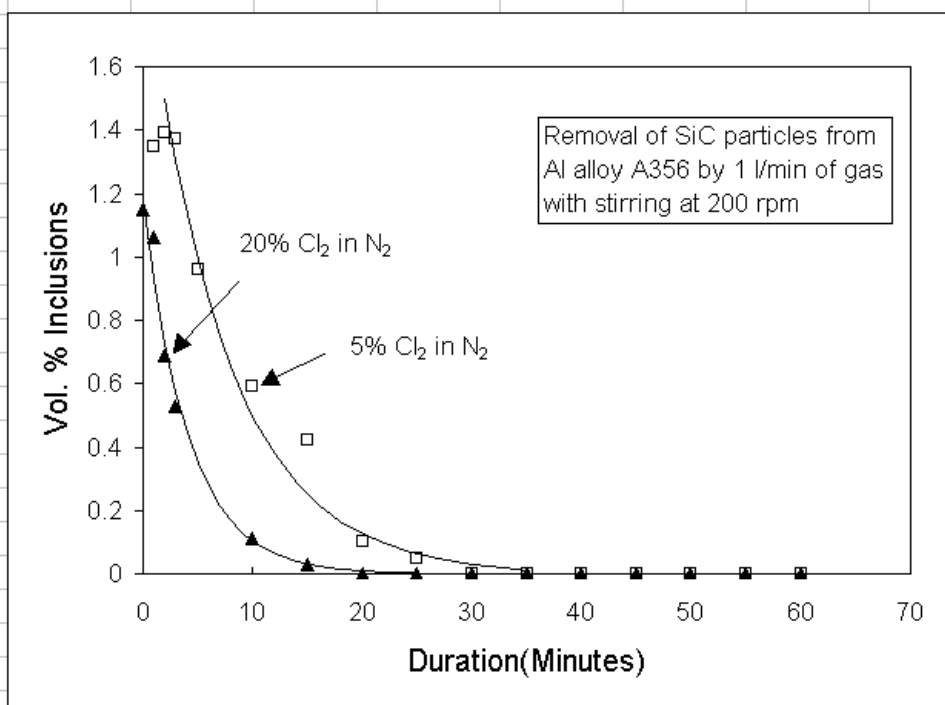


Figure 9. Percent SiC inclusions in A356 versus time for two Cl₂ contents at 200 rpm.

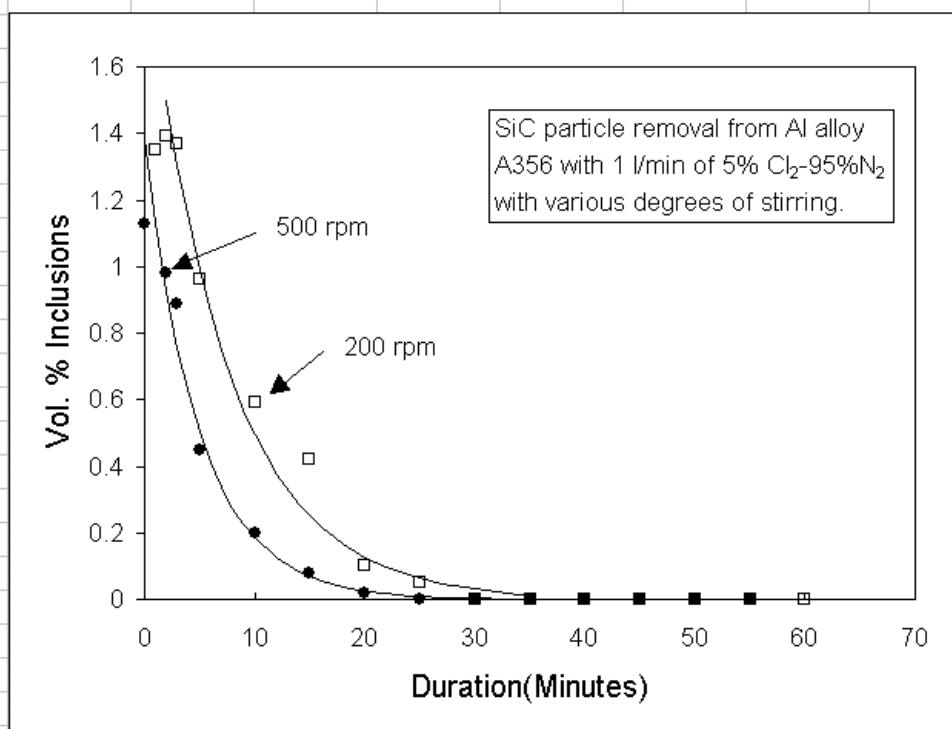


Figure 10. Percent SiC inclusions in A356 versus time for 200 and 500 rpm.

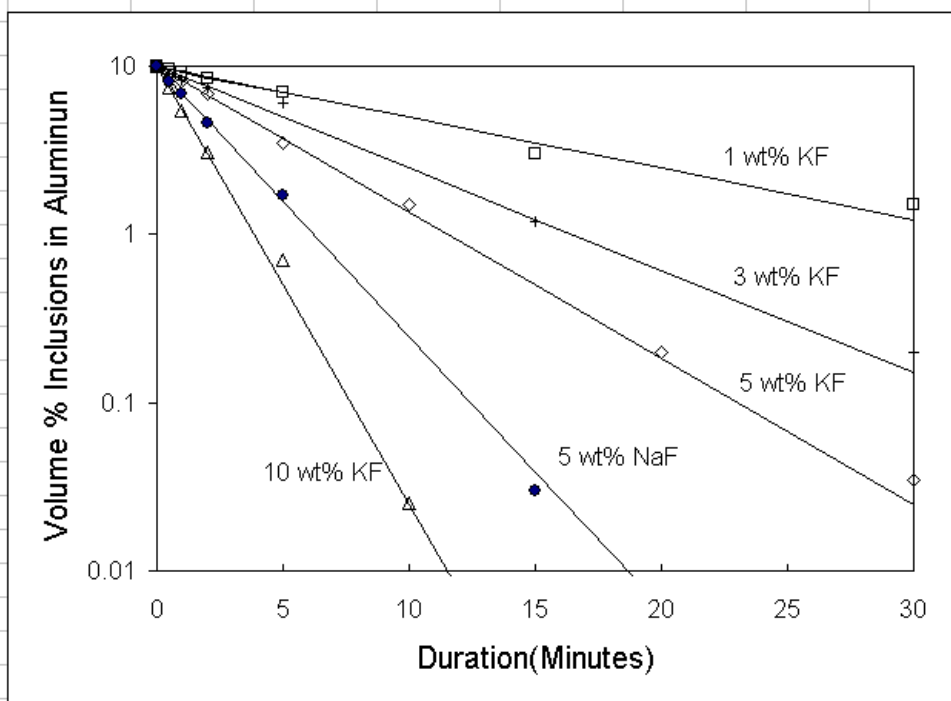


Figure 11. Percent Al_2O_3 inclusions in AA6061 during treatment with KF additions.

