ACCELERATION OF MICROWAVE IRRADIATION ON THE MELTING OF FLUORIDE

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ABSTRACT

In primary aluminum industry, electrolytic process (Hall-Heroult) is the only commercially method of producing aluminum from alumina, and the fluoride is major solvent. Reduction of operation temperature of fluoride will provide an opportunity of using inert and wetted cathodes and anodes that may reduce 25% energy consumption. The melting of fluoride salt with microwave irradiation (2.45MHz, 900W) was investigated. The fluoride compound consisted of 63.66wt% AlF_3 and 36.34wt% NaF (eutectic composition) absorbs few microwave energy at room temperature; but once is preheated to above 550°C, the fluoride salt started to quickly absorb microwave energy; after preheating to 700°C, the fluoride can be molten in 2 minutes. It needs 5 minutes above 750ºC by conventional heating with same sample.

INTRODUCTION

Aluminum is one of the most versatile and sustainable materials in the global economy. The annual primary aluminum production in the world reached 24 million metric tons in 2000 [1]. But up to now, the electrolytic process (Hall-Heroult process) is the only commercially method of producing aluminum. As the Hall-Heroult process consumes large amounts of energy in melting process due to electrolysis at a high temperature (950-980°C), and generates significant carbon dioxide and perfluorocarbon emissions with the use of consumable carbon anodes, reduction of operation temperature will provide an opportunity of using inert and wetted electrodes which may reduce 25% energy consumption and significantly decrease CO_2 emission [2].

EXPERIMENTS

According to previous investigation [3, 4] on fluoride melting, the lowest melting temperature of fluoride in the NaF-AlF_3 system is 684°C and occurred at its eutectic composition (63.66% AlF_3 and 36.34% NaF by weight). The sample for this study is prepared according to this ratio, using the reagent grade chemicals. Both NaF and AlF_3 were purchased from Alfa Aesar, A Johnson Matthey Company. Each test uses 2g of the mixture.

Microwave furnace for this study was made by modifying a commercial microwave oven (Hotpoint, Model RVM1335, 2.45MHz, 900W). Schematics of the modifications are shown in Figure 1. A conventional tube furnace is placed in the microwave furnace. This is accomplished by inserting a quartz tube (50 mm in diameter), wrapped with heating coils, into the furnace. A thermocouple with metallic sheath to protect it from microwave was inserted into the glass tube, sitting directly above the sample boat by 1.5-2 cm.

The NaF and AlF_3 absorb little microwave energy at room temperature. It is necessary to preheat the fluoride to above a temperature where the dielectric constant of the materials changed to allow for microwave absorption.

The equipment is set up in the way that the preheating can be accomplished in-situ. After preheating, the conventional heating is shut off, whereas the microwave is turned on. The temperature of the sample is recorded with time. This is compared with the temperature of the sample without

microwave turned on after preheating. By comparing the Temperature-Time profiles (T-T curve) between the experiments with and without microwave, it is possible to evaluate the extent of microwave absorption by fluoride.

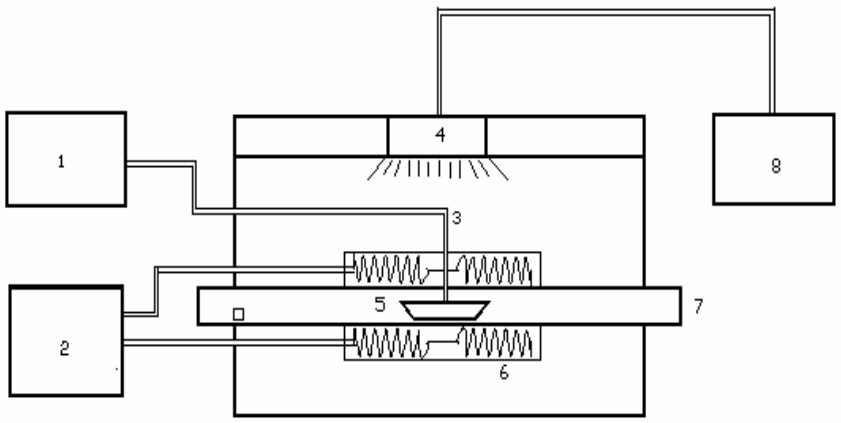

Figure 1. Sketch of the microwave furnace.
1- Thermometer; 2- Power controlling Unit; 3- Thermocouple; 4-Microwave Generator; 5- Sample Boat; 6- Conventional Heating Furnace; 7- Quartz Tube; 8- Microwave Controlling Unit

RESULTS AND DISCUSSION

After heating the fluoride sample at the desired experimental temperature for 5 minutes with conventional heating, the electric conventional heating was shut off. The temperature of the sample begins to drop and is recorded every 30 seconds. In comparison, microwave was introduced after shutting off the conventional heating. After 5 minutes of microwave irradiation, microwave was shut off and the temperature started to drop.

After completion of the experiments, sample was removed and examined. The observations were shown in Table 1. It was found that fluoride sample under conventional heating began to be hardened at 600ºC and began to be melted at 750ºC (Figure 2). On the other hand, microwave apparently accelerates the melting process. The fluoride sample begins to be hardened with microwave after conventional heating at 550ºC and partly melted with microwave after conventional heating at 650ºC. Complete melting with recrystalization was observed with 2 minutes of microwave irradiation after conventional preheating at 700ºC. Preheating to 500ºC with conventional heating followed with microwave heating does not show any changes on the physical appearance of the sample. This suggests that fluoride improves its microwave absorption capabilities at temperature above 550ºC.

It is noted that the start of microwave does not cause sudden jump of the temperature reading. The effect of microwave on the thermocouple can therefore be ignored.

It is also noted that the microwave intensity is not sufficient to maintain the sample temperature above the initial temperature achieved by the conventional preheating (Figure 3). However, microwave does contribute some extent of sample heating. Without microwave, the samples cool at a faster rate.

The duration of microwave is set at 5 minutes. After shutting off microwave, the sample cools at a faster rate as well.

Comparing the temperature-time curves with the observations shown in Table 1, it is apparent that the relation between melting phenomena and temperature reading under microwave irradiation do not agree with that under conventional heating. The conventional heating result in melting of the fluorides is at the temperature of 750ºC for over 10 minutes. On the other hand, the microwave heating is able to result in fluoride melting at a temperature as low as about 600ºC (the temperature of sample after 5 minutes microwave irradiation with 650ºC conventional preheating).

The comparison of temperature-time curves between the experiments using the ceramic boat and the metallic (steel) boat were also carried out. The cooling curve of the sample with the steel boat is parallel to that with the ceramic boat, but at 10-15°C lower than the latter. The heat loss from the steel boat is more rapid, which accelerate the cooling of the sample. However, this does not prevent the melting to occur. Since metallic boat does not absorb microwave, the melting of fluoride has to be resulted from the absorption of microwave by the fluoride.

Table 1 Physical Appearance of Fluoride Powder after Heating

Preheating temperature / ºC	With microwave on After 5minutes preheating		Conventional heating	
	5 min.	2 min.	5 min.	10 min.
750	Overflowed		Slightly sintered	7/8 molten
700	Full molten	Full molten	Harden	
650	2/3 selective molten		Harden	
600	Harden		Begin to harden	
550	Begin to harden		Loose	
500	Loose		Loose	

(1) (2)

(3)

Figure 2 Physical Appearance of Fluoride after Heating.

(1) Conventional preheating at 750ºC for 10 minutes; (2) With microwave on for 2 minutes after preheating at 650ºC, 2/3 fluoride was molten; (3) With microwave on 2 minutes after preheating at 700ºC, the fluoride was full molten and re-crystallized

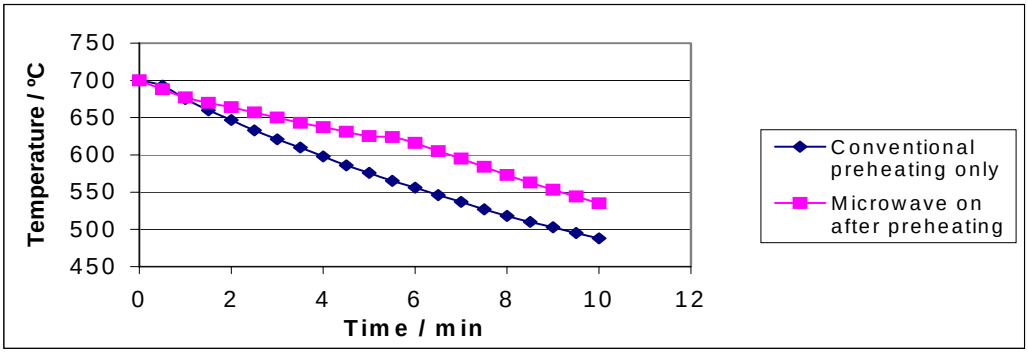

Figure 3 Cooling curve of sample after preheating at 700°C.

Since the thermocouple is not placed inside the fluoride sample, we cannot rule out the possibility that the fluoride temperature under microwave irradiation is actually higher than the reading. However, the extent of the apparent melting temperature discrepancy between the conventional heating and microwave heating is difficult to be explained solely by the position of thermocouple. Place thermocouple inside the sample is not practical. It may introduce another experimental uncertainty due to the interaction between microwave and thermocouple.

CONCLUSION

One way to reduce the energy consumption in the primary aluminum production is to reduce the operation temperature of the electrolysis. On this study it was found that microwave can be absorbed by fluoride at elevated temperatures. Microwave absorption accelerates the melting of fluorides significantly. The apparent melting temperature using microwave heating is significantly lower than that using conventional heating based on the thermocouple reading.

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