APPLICATION OF SOME VACUUM DISTILLATION PROCESS TO REFINE Zn FROM ALUMINUM SCRAP

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ABSTRACT: To promote aluminum scrap recycling, refining of Zn from aluminum scrap melt by some vacuum distillation process was tested. Effects of distillation conditions, as melt temperature, vacuum pressure and melt weight etc., on Zn distillation behavior were investigated theoretically and experimentally by $0.1 \sim 0.5 {\rm kg/ch}$ scale resistance-heating vacuum furnace. Several methods to increase the surface area/volume of liquid metal, as gas atomizing, mechanical atomizing and mechanical stirring were tested on $5 {\rm kg/ch}$. scale vacuum furnace in order to improve the Zn removal rate. Mechanical stirring method was more practical and easy to lower the remaining Zn content less than 0.1%.

Keywords: vacuum distillation, refining, Zn, Mechanical stirring, aluminum melts

1. INTRODUCTION

Refining of impurities such as iron, silicon and zinc from aluminum melts is important to promote aluminum scrap recycling into the wrought aluminum alloys. But, we have no commercial method for decreasing Zn content other than dilution with virgin Al or Al scrap with low Zn content. Vacuum refining, even though a well-known method to remove Zn and Mg from aluminum melts¹⁾ ~3), is not the commercial method. Vacuum refining is advantageous because it poses no environmental threat, and Zn metals are produced as byproducts which can be recycled. It is necessary to develop the economical vacuum refining process.

In this study, at first, effects of experimental conditions are investigated with theoretical and experimental approach by resistance-heating electric vacuum furnace. Then possibility of several method to increase the surface area/volume of liquid metal are experimentally evaluated for commercial method by 5kg/ch, scale vacuum furnace.

2.EXPERIMENTAL PROCEDURE

2.1 THEORETICAL APPROACH

Vapor pressure of binary Al-Zn alloys are given by vapor pressure of pure Zn ,molar fraction and activity coefficient as eq.(1)

$$P = \gamma_{Z_n}^T \cdot P_{Z_n} \cdot X_{Z_n} \tag{1}$$

where

 $\gamma_{Z_n}^T = \text{activity coefficient at temperature } T$

 P_{Zn} = vapor pressure of pure Zn

 X_{Zn} = molar fraction of Zn

Vapor pressure of pure Zn was assumed by eq.(2)

$$\log P_{Zn} = 10.0-6000/(T+273)$$
 (2)

Activity coefficient of Zn in Al at operating temperature T is calculated by eq.(3) and (4) assuming the normal solution.

$$\gamma_{\text{Zn}} = 2.161$$
 at 1000K (3)
 $T \log \gamma_{\text{Zn}}^{\text{T}} = 1000 \log(2.161)$ (4)

From eq.(1) to (4), vapor pressure of several Al-Zn alloys are calculated. When vapor pressure is replaced to the vacuum pressure, remaining Zn content is calculated as a function of temperature and vacuum pressure.

2.2 EXPERIMENTAL APPARATUS

Effects of experimental conditions as pressure, melt temperature, initial Zn content, melt weight and crucible ID(melt surface area) were investigated by resistance-heating vacuum furnace shown in Fig.1. Alloy sample, pre-melted and solidified in a 40mm-ID carbon crucible with air atmosphere, was set in a 100mm-ID carbon crucible in a 150mm-ID vacuum chamber. After evacuating to the operating pressure by rotary vacuum pump, alloy sample was heated and hold at operating temperature. After holding, the system was backfilled with N₂ gas to stop the vacuum distillation. Zn vapor was collected by water-cooled flanges. Melt temperature was measured by K-type thermocouple. Pressure in the vacuum chamber was controlled at operating pressure with Ar gas. Experimental conditions were shown in Table1.

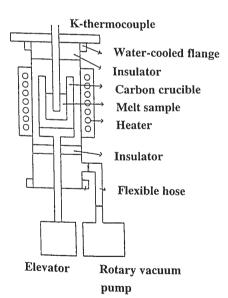


Table1 Experimental conditions				
Controlling factor	Conditions			
initial Zn content/	3			
mass%				
Vacuum pressure/Pa	20 to 50000			
Holding temperature/℃	750 to 900			
Holding time /s	0 to 1800			
Melt weight /g	mainly 300, 100,500			
Carbon crucible ID/mm	mainly 100, 40			

Fig.1 Schematic illustration of experimental apparatus

2.3 APPLICATION OF SOME METHODS

Three methods as gas atomize, mechanical atomize and mechanical stirring were tried to increase the surface/volume rate in 5 kg/ch. scale vacuum furnace. Schematic illustrations of those apparatus were shown in Fig.2. In the gas atomize or mechanical atomize methods, melted alloy sample heated to 900° C in the air atmosphere was introduced to a 300mm-ID carbon crucible heated to 900° C in a pre-evacuated vacuum chamber. In the mechanical stirring method, sample alloy was set in a 300mm-ID carbon crucible and heated in Ar gas atmosphere to the operating temperature. Then melt was stirred and evacuated to the operating pressure 10Pa. After stirring the operating time, N_2 gas was introduced to stop the vacuum distillation. Each of initial Zn content was 3mass%.

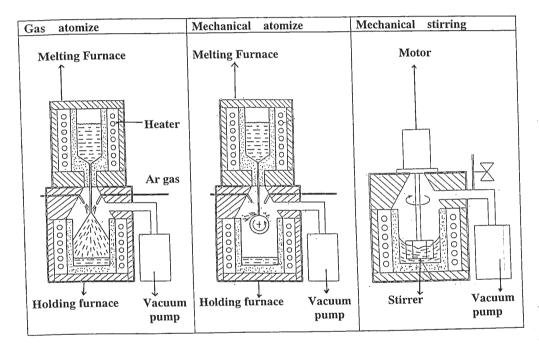


Fig.2 Schematic illustrations of several apparatus

3. RESULTS AND DISCUSSION

Calculated remaining Zn content was shown in Fig.3. Vacuum pressure had a marked effect on remained Zn content than melt temperature. In order to recycle the Al scrap into the wrought Al alloy, it was desirable to keep the remaining Zn content less than 0.1%. That was possible and practical object because it was easy to keep the melt over 750 °C in the vacuum pressure under 10Pa.

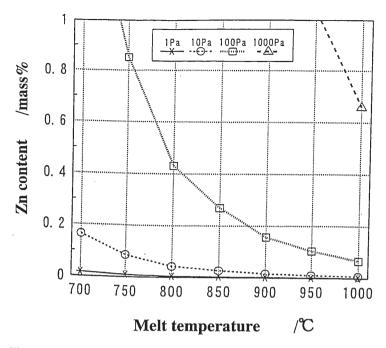


Fig.3 Calculated remaining Zn content after vacuum distillation

Typical experimental results were shown in Fig.4 and Fig.5. Zn removal rate C/Co decreased with increase of holding time, and had a tendency to be constant over 600s.

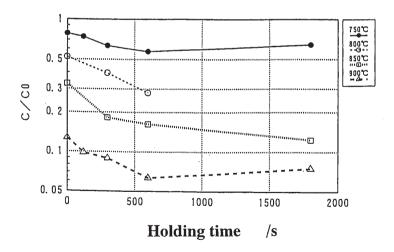


Fig.4 Zn removal behavior (Vacuum pressure:100Pa, Initial Zn content Co=3mass%,)

Influence of melt weight and crucible diameter(melt surface area) on Zn removal behavior was shown in Fig.5 and Fig.6, respectively. Melt weight has a marked influence only at the lower melt temperature.

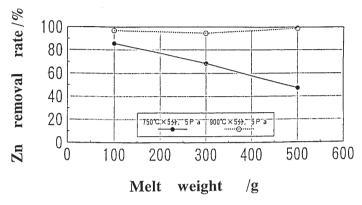


Fig.5 Effect of melt weight on Zn removal rate

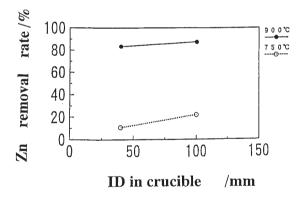


Fig.6 Effect of crucible ID(melt surface area) on Zn removal rate

Experimental results on gas atomize and mechanical atomize process is shown in Table2.

Table 2	Experimental results on gas atomize and mechanical atomize proces					,
Process	Operating	Remaining		Reaction	constant	

Process	Operating vacuum pressure	Remaining Zn content /mass%	Reaction constant / x10 ⁻⁵ ms ⁻¹
and otomica	/Pa 100	2.17	230
gas atomize mechanical	100	1.79	346
atomize	100	2.34	177
only falling	10	2.35	306
out	100	2.69	144

Zn removal behavior was improved with gas or mechanical atomize. But, it needs a tall equipment over 5m to keep a reaction time and seemed to be difficult for commercial method.

A markedly improvement on Zn removal behavior was obtained in mechanical stirring process as shown in Fig.7. Remaining Zn content was lower with increase of stirring speed, and it was easy to keep remaining Zn content less than 0.1mass% only 50s under the stirring condition 300rpm. It seemed to be the economical and commercial method.

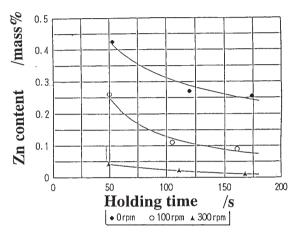


Fig.7 Effect of stirring on remaining Zn content(10Pa, 5kg/ch.)

4. CONCLUSION

Zn removal behavior from Al melt by vacuum distillation was investigated to develop Al recycling. Effects of experimental conditions were studied theoretically and experimentally. Vacuum pressure and melt temperature had a marked influence than melt weight and melt surface area. Several methods were applied to 5kg/ch. scale test and mechanical stirring had a markedly improvement on Zn removal behavior.

ACKOWLEDGMENTS

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