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Recovery of Metals from Sn-Ag-Cu Solder Alloy Dross

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Abstract **-** The purpose of this research work was to study the recovery of metals from dross of Sn-Ag-Cu (SAC) solder alloy by using a combined recovery process. The process was consisted of two main metal extraction processes, the primary and secondary processes. The primary process was heating and mechanical squeezing of the mixture of SAC dross and pulverized rice husk. The ground rice husk was used as an additive in the heating and extraction process. The effects of temperature and quantity of rice husk powder addition on the recovery of metal alloy were previously investigated. The experiments were performed at temperatures of 400, 450 and 500 °C. The amount of rice husk powder mixed with the SAC dross was in the range of 0–150 grams per 2 kilograms of dross. It was found that burning the mixture of dross and rice husk powder enhanced the extraction of metals from the SAC dross. The optimum recovery of metal alloy of 73.13 % was achieved by employing the heating temperature of 450 °C and the addition of rice husk powder in the amount of 50 grams per 2 kilograms of dross. In the present work, the secondary metal recovery process was employed by using an alkaline fusion of the secondary dross obtained from the primary process with sodium hydroxide (NaOH). Three mixing ratios of the secondary dross to NaOH of 1:0.5, 1:1 and 1:1.5 were studied. The fused product was leached by warm water and mechanical agitation. The undissolved metal alloy nodules were recovered from the leaching solution by decantation. The overall metal alloy recovery of 90.07 % was achieved by using the 1:1 ratio of dross to NaOH. The recovered metal alloys # 1 and #2 from the primary and secondary processes were consisted of 97.81 wt.% Sn, 1.43 wt.% Ag, 0.63 wt.% Cu and 96.51 wt.% Sn, 2.58 wt.% Ag, 0.71 wt.% Cu, respectively. The losses of metals in the fine suspended and filtrate products were 2.14 % and 7.79 %, respectively. The fine suspended product comprised 0.12 wt.% Sn, 0.20 wt.% Ag and 0.08 wt.% Cu. The filtrate product contained 4.12 wt.% Sn, while the Ag and Cu contents were lower than analytical detection limit. The recovered metal alloys could be used as a recycled material for the production of SAC solder alloy.

Keywords**:** Metal recycle, Metal recovery, Lead-free solder, SAC alloy, Dross, Rice husk.

1. Introduction

Tin–Silver–Copper (SAC) alloy is one of lead-free solder alloys which have been used as the replacement for the conventional tin-lead solder alloy in electronic packaging industry. During manufacturing process of SAC solder alloy, molten metals react with oxygen from air and form metal oxide, so called “dross” floating on top of the melt pool. Previous study mentioned that approximately 70 % of metal entrapped in the solder dross can be reclaimed (Web-1). As the price of metals, especially tin and silver, is rising, and the solder dross also consumes plenty of stocking area and inventory cost for the factory. A suitable extraction process would be beneficial for the industry if the valuable metals containing in the dross could be recovered and recycled back to the manufacturing process. Yangyuen (2001) reported the extraction of tin metal from tin dust (SnO_2) by using an alkaline leaching. The tin dust was mixed with NaOH in the ratio of 1.43:1 and roasted at 650 °C in order to convert tin oxide into a soluble product for subsequent leaching. Miskufova, *et al.* (2009) studied the leaching of aluminum dross in an alkaline solution. It was reported that a maximum extraction of about 42 % of total content of Al was recovered by leaching the dross fines with 10 % NaOH solution at 96 °C, using a ratio of solid to liquid of 1:100 for 120 min. Kaewboonthong, *et al.* (2011) has previously reported that some metals could be recovered from the SAC dross by employing a heating and mechanical squeezing technique. About 73

% of metal alloy was recovered. However, the secondary dross from the heating and squeezing process still contains some valuable metals of about 27 %. Thus, the present work was purposed to recover the remaining metals from the secondary SAC dross by using an alkaline fusion and leaching process.

2. Experimental Details

2.1. Materials

A secondary SAC dross obtained from the previous study by Kaewboonthong, *et al.* (2011) having composition of 88.90 wt.% Sn, 0.518 wt.% Cu, and 1.41 wt.% Ag and a commercial grade sodium hydroxide with composition of 96 wt.% NaOH, 0.75 wt.% $NaCO_3$ and 3.25 wt.% of inert ingredients were used in this study.

2.1. Experimental Procedure

An experimental flow diagram was shown in Fig. 1. In the previous study by Kaewboonthong, *et al.* (2011), the primary SAC dross was mixed with pulverized rice husk, heated and mechanically squeezed. The ground rice husk was used as an additive in the heating and extraction process. The effects of temperature and quantity of rice husk powder addition on the recovery of metal alloy were previously investigated. The experiments were performed at temperatures of 400, 450 and 500 °C. The amount of rice husk powder mixed with the SAC dross was in the range of 0–150 grams per 2 kilograms of dross. It was found that burning the mixture of dross and rice husk powder enhanced the extraction of metals from the SAC dross. The optimum recovery of metal alloy of 73.13 % was achieved by employing the heating temperature of 450 °C and the addition of rice husk powder in the amount of 50 grams per 2 kilograms of dross. From the primary process, metal alloy #1 and secondary dross were obtained. The secondary dross was further used in the present study. It was mixed with NaOH in weight ratios of 1:0.5, 1:1 and 1:1.5. The mixture was heated and fused in a steel pan using a natural gas torch in an exhausted hood. The fused product was cooled down in air. The free metal alloy #2 and matte were obtained. The matte was then leached with 400 ml water at temperature of 90 °C and stirred at 500 rpm for 20 min. The undissolved metal granules were separated from the leached solution by decantation. The metal granules were collected into the metal alloy # 2. The decanted solution was then filtered using filter paper. The fine suspended was dried at 105 °C. The compositions of metal alloy #1 and metal alloy #2 were analysed by an optical emission spectroscopy (OES) method. The fine suspended and filtrate solution products were analysed by using an atomic absorption spectroscopy (AAS) and an inductively coupled plasma (ICP) method, respectively.

3. Result and Discussion

From the combined recovery process (Fig.1), there were four main output products, namely metal alloy # 1 from the primary heating and squeezing process, metal alloy # 2, fine suspended, and filtrate from the secondary alkaline fusion and leaching process. Tables 1-3 are the mass balance of the output products from the processes using the ratios of secondary dross to NaOH of 1:0.5, 1:1.0, and 1:1.5, respectively. In the fusion process, the secondary SAC dross was burned with NaOH which reacted with oxides on the surface of dross and converted to into soluble matte and some metal alloy (#2) separated from the matte. However, there were some remaining fine metal granules inside the matte. The matte can be dissolved in warm water while the fine metal granules cannot. After leaching the fine metal granules were decanted. It was noticed that the amount of fine metal granules was very small compared to metal alloy # 2 product. Hence, it was collected and summed into the metal alloy #2 for a single product. It was found that an optimum recovery of metal alloy was obtained by using the ratio of dross to NaOH of 1:1.0. The recovery distribution of metal alloy #2 was increased with increasing ratio of dross to NaOH from 1:0.5 to 1:1.0. When the ratio was increased to 1:1.5 the recovery of metal alloy #2 was slightly decreased. For an overall metals recovery, the recovery of metal alloy # 1 and #2 were summed. In Table 2, it was shown that an optimum overall metals recovery was 90.07%. The chemical analysis showed that the metal alloy #1 was consisted of 97.81 wt.% Sn, 1.43 wt. % Ag, and 0.63 wt.% Cu while the metal alloy #2 comprised 96.51 wt.% Sn, 2.58 wt. % Ag, and 0.71 wt.% Cu. These products could be used as a recycling material for the production route of SAC solder alloy. In the leaching process, there were some losses of metals distributed in the fine suspended and the filtrate products in the amount of 2.14 and 7.79 %, respectively. The fine suspended comprised 0.12 wt.% Sn, 0.20 wt.% Ag and 0.08 wt.% Cu. The filtrate contained 4.12 wt. % Sn, and the Ag and Cu contents were lower than the analytical detection limit. It is suggested that the metals in the filtrate can be recovered by using a suitable electrowinning process and the fine suspended product may be recycled to the leaching process. The suggested processes are the aims for the future research in our laboratory.

4. Conclusion

The recovery of valuable metals from the dross of SAC solder alloy was studied. A combination metals extraction process was introduced. The process was consisted of two main extraction routes. Firstly, the metal alloy was recovered from the primary SAC dross by heating the dross with pulverized rice hush and followed by mechanical squeezing. Secondly, the remaining metals were recovered by using alkaline fusion of the secondary dross from the first process with NaOH and leaching the fused product with warm water with an assisted mechanical agitation. From the experimental results, it could be concluded that in the primary recovery process, an optimum recovery of 73.13 % of metals was recovered from the dross by heating the mixture of SAC dross and rice husk powder in the amount of 50 grams per 2 kilogram of dross, and heated at 450 °C. The secondary process was able to recover 16.94 % of metals from the dross by fusion the mixture of the secondary dross with NaOH at the ratio of 1:1.0 and leaching the fused product with water at 90 °C and agitated at 500 rpm for 20 min. The overall metals recovery was 90.07 % was achieved. The recovered metal alloys could be used as a recycled raw material for the production of SAC solder alloy. There were some losses in the fine suspended and the filtrate products in the leaching process in the amounts of 2.14 % and 7.79 %, respectively. It is suggested that the remaining metals in the filtrate may be further recovered by using a suitable electrowinning process and the fine suspended product may be recycled to the leaching process.

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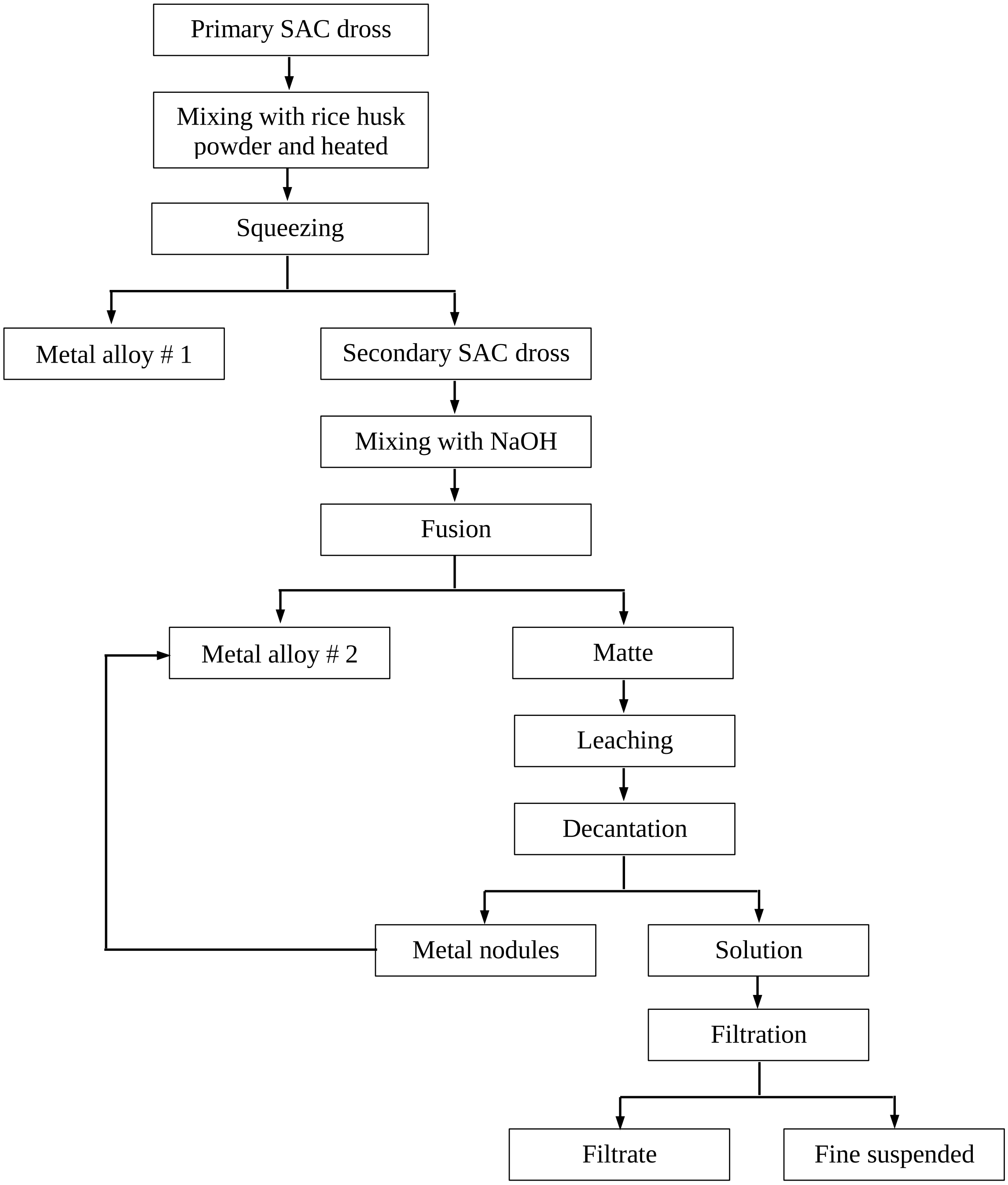

Fig. 1. Experimental flow diagram

Table. 1. Mass balance of products from the process using dross to NaOH ratio of 1:0.5

Products	% wt.	% Assay			% Distribution		
		Sn	Ag	Cu	Sn	Ag	Cu
Primary dross	100.00	94.72	1.39	0.59	-	-	-
Metal alloy #1	73.13	97.81	1.43	0.63	82.71	72.56	81.57
Metal alloy #2	14.91	96.48	2.62	0.69	16.63	27.11	18.10
Fine suspended	0.82	0.12	0.58	0.23	0.00	0.33	0.33
Filtrate	11.14	5.05	ND	ND	0.65	-	-
Reconstructed feed	100.00	-	-	-	100.00	100.00	100.00

ND means lower than the analytical detection limit.

Table. 2. Mass balance of products from the process using dross to NaOH ratio of 1:1

Products	% wt.	% Assay			% Distribution		
		Sn	Ag	Cu	Sn	Ag	Cu
Primary dross	100.00	94.72	1.39	0.59	-	-	-
Metal alloy #1	73.13	97.81	1.43	0.63	81.10	70.32	79.18
Metal alloy #2	16.94	96.51	2.58	0.71	18.54	29.39	20.53
Fine suspended	2.14	0.12	0.20	0.08	0.00	0.29	0.29
Filtrate	7.79	4.12	ND	ND	0.36	-	-
Reconstructed feed	100.00	-	-	-	100.00	100.00	100.00

ND means lower than the analytical detection limit.

Table. 3. Mass balance of products from the process using secondary to NaOH ratio of 1:1.5

Products	% wt.	% Assay			% Distribution		
		Sn	Ag	Cu	Sn	Ag	Cu
Primary dross	100.00	94.72	1.39	0.59	-	-	-
Metal alloy #1	73.13	97.81	1.43	0.63	81.47	70.57	80.23
Metal alloy #2	16.69	96.53	2.59	0.67	18.35	29.12	19.47
Fine suspended	2.89	0.06	0.16	0.06	0.00	0.31	0.30
Filtrate	7.29	2.11	ND	ND	0.18	-	-
Reconstructed feed	100.00	-	-	-	100.00	100.00	100.00

ND means lower than the analytical detection limit.