# THE RECOVERY AND LIFE CYCLE ASSESSMENT OF NICKEL PARTICLES IN A MULTI-SOLENOID OPEN-GRADIENT MAGNETIC SEPARATOR

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(Received 21 August 2001; In final form 14 September 2001)

The recovery of 87% and the grade of 30% of Ni from nickel tip capacitors were achieved using a multi-solenoid open-gradient magnetic separator. A series of tests under varying conditions, such as density of pulp, magnetic flux density, pH, etc. have been studied. The experimental results of the purification tests were related to the parameters investigated. Moreover, the LCA assessment suggested that the nickel concentrate recovered by the open-gradient magnetic separator can be re-used as a raw material in pyrometallurgy, aiming to reproduce the Ni capacitor.

Keywords: Ni recovery; LCA; Open-gradient magnetic separator; Dispersant

#### 1. INTRODUCTION

In Japan, nickel tip capacitors are used in the IT industry, especially in PC and hand phone production. However, about 15–20% of the total production is imperfect and for that reason, rejected. The recycling of

ISSN 1055-6915 print: ISSN 1477-2876 online © 2002 Taylor & Francis Ltd

DOI: 10.1080/10556910290032854

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the wasted nickel tip capacitor thus becomes a very important issue. The aim of this study is to recover nickel particles from the ground waste nickel tip capacitors. For this purpose, a new type of a magnetic separator – a multi-solenoid open-gradient magnetic separator – was introduced. Recently, as the environmental issues become more sensitive, the technique called Life Cycle Assessment (LCA) that evaluates the energy and environmental impact through the entire life cycle was developed. In this frame, LCA of the magnetic separation process of nickel tip capacitor for reproduction purpose is also presented.

# 2. EXPERIMENTAL METHOD AND SAMPLE USED

# 2.1. Experimental Equipment and Method

The multi-solenoid open-gradient magnetic separator includes a tube surrounded by three separated solenoid coils with a yoke to capture magnetic particles, and a pump to feed the mixed particles into the tube (Fig. 1). This magnetic separator is suitable to capture magnetic particles smaller than  $74 \, \mu m$ .

Figure 2 depicts a schematic diagram of the multi-solenoid open-gradient magnetic separator. The apparatus has three different circulating systems of the pulp, which are used:

- to circulate the pulp through the apparatus,
- to wash and transport the non-magnetic particles and
- to transport the captured magnetic particles.

At first, the waste capacitors, ground by a ball mill, were dispersed in water and mixed by an agitator. The sample was circulated through the magnetic separator by a pump, while the magnetic valves were switched to "a" position. At the same time, the magnetic force was applied by passing the electric current through the coils. The magnetic particles created chain structures in the direction of the magnetic field. In contrast, non-magnetic particles were not affected by the magnetic field; they were transported by the fluid flow.

After 10 min of circulation, the magnetic valve B was switched to "b" position to introduce water into the apparatus tube. The water flow

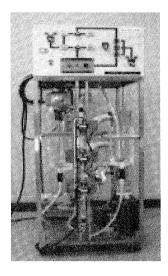


FIGURE 1 A multi-solenoid type open-gradient magnetic separator.

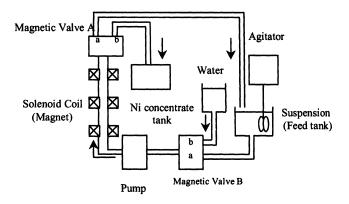


FIGURE 2 Schematic diagram of the multi-solenoid open gradient magnetic separator.

was used to wash the machine and to transport the non-magnetic materials to the feed tank. Additionally, after the magnetic valve A was switched to "b" position, and the magnetic field was also switched off, the magnetic particles were collected by water flow and transported to the Ni concentrate tank.

# 2.2. Sample and Conditions

The ground waste samples of Ni tip capacitors used in this experiment were provided by Yokohama Metal Co., Ltd. The waste nickel tip capacitors (several mm in size) are composed of many dielectric layers of barium titanate (thickness of about  $5\,\mu m$ ) and nickel inner electrodes, (thickness of about  $2\,\mu m$ ) together with two copper outer electrodes (Fig. 3).

Some representative samples of Ni tip capacitor were analyzed using scanning electron microscope (SEM) and energy dispersive X-ray spectrometer (EDS) (Fig. 4). Figure 4 illustrates layer formation of the capacitors.

The Ni tip capacitor consisted of 88% BaTiO<sub>3</sub>, 8% Ni and 4% Cu. Moreover, the particle size distribution of Ni capacitors crushed by an impact crusher is plotted in Fig. 5. It can be seen that 70 wt% of the sample has a particle size of less than 53  $\mu$ m. Hence, the ground capacitor could be processed by the new multi-solenoid open-gradient magnetic separator, which is convenient for capturing small size magnetic particles of less than 74  $\mu$ m.

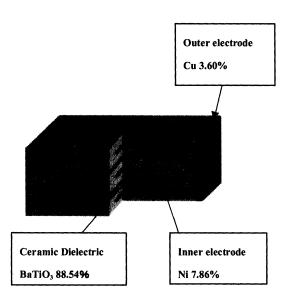


FIGURE 3 Schematic illustration of a multi-layer ceramic chip capacitor.

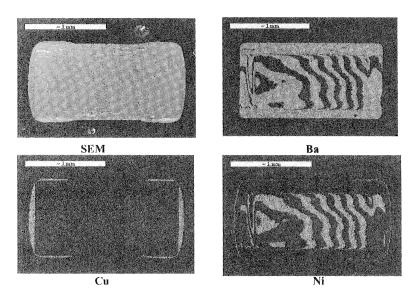


FIGURE 4 SEM photograph and EDS maps of Ni tip capacitor.

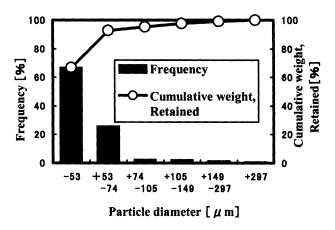


FIGURE 5 Particle size distribution of "as received" samples of Ni tip capacitors.

Furthermore, the operating conditions (i.e. the effect of the pulp density, magnetic flux density, etc.) were investigated to achieve a high grade and recovery of Ni. In order to optimize the experimental parameters, the tests were carried out at various conditions. The tests to capture fine magnetic particles were conducted with the pulp density

between 5 and 15%, grinding time 0-90 min, magnetic flux density 0.01-0.1 T, pH 6-11 (Table 1).

## 3. RESULTS AND DISCUSSIONS

# 3.1. Effect of Pulp Density

The variation of grade and recovery of nickel with the pulp density is shown in Fig. 6. The tests were conducted under the magnetic flux density of 0.03 T. Referring to the results, the maximum recovery

TABLE 1 Experimental conditions for the recovery of Ni particles from waste Ni tip capacitor using the new type of magnetic separator

Conditions	
Circulating time of the pulp	10 min
Pulp density	5–15%
Grinding time as received sample (by ball mill)	0–90 min
Magnetic flux density	0.01-0.1 T
pH	pH 6-pH 11
Dispersant	4 different types

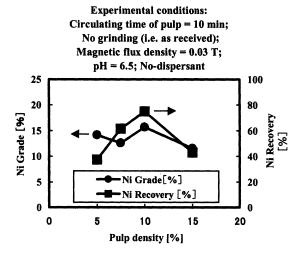


FIGURE 6 Effect of pulp concentration on the Ni grade and recovery.

and grade of the Ni components were 75.0% and 15.7% respectively, while the pulp density was 10% (Fig. 6). It was observed that at the low density of pulp the recovery of the Ni component decreased. On the other hand, at a concentration higher than 10%, the amount of magnetic particles per unit volume of the area under the effect of the magnetic field increased. This in turn caused a decrease in the grade and the recovery of the collected Ni particles. Considering the results, the pulp concentration of 10% was selected as the standard level for all subsequent experiments.

# 3.2. Effect of Grinding Time

Consequently, the variation of recovery and grade of nickel was investigated for different grinding times. Prior to all tests, the sample was ground by a ball mill. Following that the Ni particles were recovered by the magnetic separator, while the magnetic flux density of 0.03 T was applied. The relation of the Ni grade and the Ni recovery with the grinding time is shown in Fig. 7. It is clearly apparent that after 30 min of grinding, the Ni grade increased by 33.41%, whereas 24.33% Ni grade was obtained for an "as received" sample (Fig. 7).

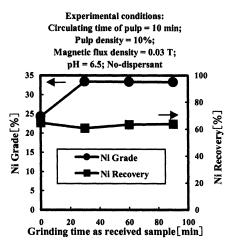


FIGURE 7 Influence of grinding time of the "as received" sample on the Ni grade and recovery (by a ball mill).

However, even when the grinding was carried out for more than  $30\,\mathrm{min}$ , the Ni grade did not change. In contrast, the recovery was not effected by changes in the grinding time. It is important to notice that the mean diameter of the sample, after  $30\,\mathrm{min}$  of grinding, was  $11.3\,\mu\mathrm{m}$ . Thus, the experimental results suggested that  $30\,\mathrm{min}$  was an appropriate time to grind the sample, prior to magnetic purification.

# 3.3. Effect of Magnetic Flux Density

Subsequently, magnetic separation was performed for a wide range of flux densities. Figure 8 shows that an increase in the magnetic flux density increased the recovery of Ni component. However, an upper limit of the magnetic flux density should be set, since the grade of Ni component decreased, while the magnetic flux density was higher than 0.04 T. This was due to the fact that Ni and BaTiO<sub>3</sub> particles created chain structures of a relatively large diameter.

A look at Fig. 8 shows that initially the recovery of nickel increased by increasing the magnetic field. So, the maximum Ni recovery of 75.82% was obtained, while the magnetic field of 0.04 T was applied. Yet, while the flux density increased further (up to 0.1 T) the Ni

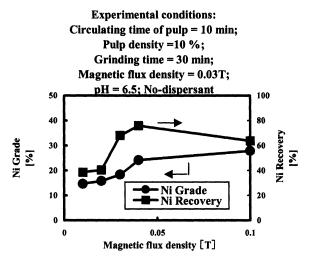


FIGURE 8 Influence of the magnetic flux density on the Ni grade and recovery.

recovery decreased. Less than 64% of nickel was recovered, while the magnetic flux density was maintained at 0.1 T. Hence, considering the results (Fig. 8), the magnetic flux density of 0.04 T was selected as the appropriate level for all subsequent experiments.

# 3.4. Effect of pH

Further, the influence of pH was investigated in order to improve the separation of the constituents of the sample. It was observed that for a wide range of pH values, from pH = 6.5 to pH = 11, the grade and the recovery of nickel were not affected (Fig. 9). Thus, taking into consideration environmental problems and equipment corrosion, a pH of 7 was selected as the standard level for all subsequent experiments.

### 3.5. Dispersants

A dispersing agent was employed during the magnetic separation of the mixed powder of nickel and BaTiO<sub>3</sub>. Subsequently, the pulp was treated with different dispersants and their effect on the purification results was investigated. Four types of dispersants were examined: sodium hexametaphosphate [(NaPO<sub>3</sub>)<sub>6</sub>], sodium pyrophosphate

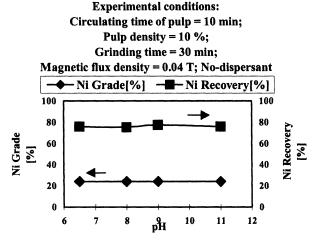
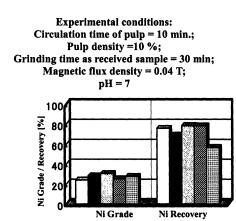


FIGURE 9 Influence of pH on the Ni grade and Ni recovery.



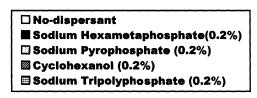


FIGURE 10 Effect of dispersants on the Ni grade and recovery.

[Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub>·10H<sub>2</sub>O], cyclohexanol [C<sub>6</sub>H<sub>11</sub>OH], sodium tripolyphosphate [Na<sub>5</sub>P<sub>3</sub>O<sub>10</sub>]. The concentration of the each dispersant was 0.2%.

The grade and the recovery of nickel, as a result of the treatment of pulp with different dispersants are depicted in Fig. 10. It can be seen that there was no significant difference between the Ni grade of the values obtained, since the Ni grade varied from 26 to 30%. In contrast, 78.50% of the component was recovered with the grade of 30.67%, while the pulp was treated with sodium pyrophosphate.

The results confirmed that the multi-solenoid open-gradient magnetic separator effectively recovered the ground Ni tip capacitor, when sodium pyrophosphate was employed.

# 4. THE LCA FOR THE MAGNETIC SEPARATION PROCESS OF NICKEL TIP CAPACITOR

Life cycle assessment (LCA) is a technique for assessing the environmental impact on a product throughout its life from the raw material

acquisition through production, use and disposal. Moreover, LCA can assist in making eco-friendly products. The LCA procedure consists of a goal and scope definition, inventory analysis, impact assessment and interpretation [1]. In addition, LCA procedure requires reliable data for inventory analysis. Consequently, LCA was carried out and the environmental impact was estimated for different possible processes of waste Ni tip capacitor. The commercial software called "NIRE-LCA Ver.2.1", developed by the National Institute for Resource and Environment of Japan, was used for calculating the environmental impacts produced by each considered process. LCA appraisal was based on three scenarios (processes):

Process 1 (Fig. 11a): Waste Ni tip capacitors are discarded (250 kg/month) and used for landfill;

Process 2 (Fig. 11b): The nickel concentrate is recovered by a gradient magnetic separator and used as a raw material in pyrometallurgy with the purpose of Ni tip capacitor reproduction.

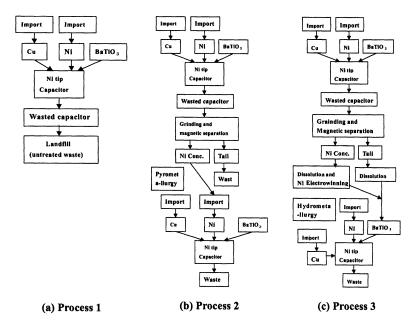


FIGURE 11 LCA flow-sheet of Process 1, Process 2 and Process 3.

	Process 1	Process 2	Process 3
Energy consumption	2190.5	3902.9	5132.6
Consumption of mineral ores	15637.3	10727.4	11208.8
Greenhouse effect	510153.0	510320.5	510333.6
Acid precipitate	4302.6	4303.2	4302.9
Water pollution	396.9	398.1	398.1
Air pollution	39769.1	39776.6	39774.3
Total amount	572449.4	569428.7	571150.3

TABLE 2 Results of LCA for each recovering process of the waste Ni tip capacitor

Process 3 (Fig. 11c): The nickel concentrate is recovered by a gradient magnetic separator and used as a raw material in hydrometallurgy with the purpose of Ni tip capacitor reproduction.

In Table 2, the results of LCA are tabulated. Referring to Table 2, Process 2 was considered as the most appropriate one. It achieved the lowest "total amount" of impact assessments as well as "energy consumption" and "consumption of the mineral ore" were relatively lower.

#### 5. CONCLUSION

The multi-solenoid open-wet gradient magnetic separator was developed to recover Ni from wasted Ni tip capacitor. It is a new circulating type magnetic separator. The separator captured the Ni particles and obtained the recovery of 78.50% and grade of 30.67%. The optimum experimental conditions were:

- 1. pulp density of 10%,
- 2. magnetic flux density of 0.04 T,
- 3. the pulp was treated with sodium pyrophosphate dispersant.

Moreover, according to LCA, the nickel concentrate recovered by the gradient magnetic separator can be used as a raw material in pyrometallurgy with the purpose of Ni tip capacitor reproduction.

# References

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