Separation of Cobalt and Nickel from Activated Metal

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A large amount of low-level radioactive waste metal will be generated from a nuclear power plant decommissioning. Disposal of the radioactive waste must be a burden to the disposal facility, therefore reducing the disposal volume must be important issue to solve. Toward minimizing the volume of radioactive waste, the authors developed a new technique to separate cobalt and nickel elements (not nuclide) from radioactive metal, especially for activated metal, thereby to lower radioactivity concentration of the metal. Screening tests and demonstration tests were implemented. In the screening tests, three kinds of processes were compared on the performance in small-scale apparatus and the Oxygen Sparging Process was seen to have most feasible performance. In the demonstration tests, the prototype apparatus of the Oxygen Sparging Process demonstrated that it could separate cobalt from carbon steel with a decontamination factor of 100 and with an iron recovery ratio of 60% in proper process conditions.
Keywords: radioactive metal, molten metal separation, cobalt, nickel, oxygen sparging process, high temperature solvent extraction process, molten salt electro-refining process

原子力発電所の解体に伴い多量の低レベル放射性廃棄物の金属が発生する．解体に伴う放射性廃棄物の処分は LLW 処分場容量を逼迫させ，経済的にも高価であるため，放射性廃棄物量の低減は重要な課題である．このため，放射性廃棄物量の低減に向けて，低レベル放射性金属からコバルト及びニッケルを溶融分離する新規技術について，スクリーニング試験及び実証試験をおこなった．スクリーニング試験では 3 種類の分離プロセスについて性能比較した結果，優先酸化法が最も良好な性能を示した．実証試験では，優先酸化法のプロトタイプ設備などを用いて試験した結果，炭素鋼中のコバルトを DF100，鉄回収率 60%で回収できることを確認した．
Keywords: 放射性金属，金属溶融分離，コバルト，ニッケル，優先酸化法，高温溶媒抽出法，溶融塩電解法

Ⅰ Introduction

A large amount of low-level radioactive waste (LLW) metal will be generated from a nuclear power plant (NPP) decommissioning. The LLW metal is estimated approximately 25,000 ton including 5,000 ton of activated metal for 1,100 MWe Boiling Water Reactor and Pressurized Water Reactor. Disposal of the radioactive waste must be a burden to the disposal facility and environment, therefore reducing the disposal volume must be important issue to solve. Cobalt 60 (half life 5.26 y) has impact on occupational exposure at dismantling work, and nickel 63 (half life 92 y) has impact on long term safety evaluation. Therefore a technique to separate cobalt and nickel from the metal must facilitate its recycling or lower its waste category. Until now, there are many instances of chemical or physical decontamination to surface contamination such as oxide film containing cobalt and nickel, while almost no instance of decontamination to volumetrically contaminated metal such as activated metal was reported. Because cobalt and nickel are homologies with iron in the periodic table, their metallurgical separation is traditionally taken as not so economical process.

The authors have implemented demonstration tests of the molten metal separation techniques to separate cobalt and nickel from volumetrically contaminated metal. [1,2] Dry method using molten metal was focused on because of fewer amounts of waste and more compact apparatus than those of wet method. Three kinds of molten metal separation processes were selected from dry method for screening tests. The result of the screening tests showed the Oxygen Sparging Process is superior to the others. Therefore, the demonstration tests of the process were implemented using a prototype apparatus. It showed that the process met performance to lower radioactivity concentration of activated metals for facilitating radioactive metal recycling or to lower radioactive waste category from LLW to very low level radioactive waste (VLLW).

This paper summarizes the development of the molten metal separation techniques as a part of the verification tests on decommissioning technologies for commercial nuclear power plants.

Ⅱ Objectives

The objectives of the development are to demonstrate the performance of processes to remove cobalt and nickel from LLW metal and to lower the radioactive concentration. The targets on performance were set as follows.

－DF: 100 minimum
(DF is an element concentration ratio before and after processing, and DF of cobalt is displayed like DFCo.)

－Recovery rate: 60% minimum
(Recovery rate is a metal recovery rate in the process, which is given by metal weight divided by original metal weight)

－Waste rate: 40% maximum
(Waste rate is a waste generation ratio in the process, which is given by waste weight divided by original metal weight.)

These targets were decided under an assumption that LLW activated metal shall be processed to lower waste category to VLLW; DF was set as 100 minimum to increase the metal volume to be processed, recovery ratio of iron was set as 60%

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minimum to increase recycling ratio, and waste ratio was set as 40% maximum to decrease secondary waste volume.

Ⅲ Screening Tests

Two kinds of methods have been utilized for separating cobalt and nickel from iron, i.e., dry method and wet method. Wet method have been practically applied in the area of metal refinement but not appropriate for radioactive metal because it tends to generate large amount of liquid waste, while dry method must be feasible because of small amount of secondary waste and compact apparatus. Therefore authors reviewed dry method and selected 3 kinds of processes for screening tests; the High Temperature Solvent Extraction Process, the Oxygen Sparging Process and the Molten Salt Electro-refining Process.

Melt, sludge, salt and metals in these tests were sampled and measured to quantify concentration of iron, cobalt and nickel by ICP (Inductively Coupled Plasma Spectrometry) and others.

1 High Temperature Solvent Extraction Process

The High Temperature Solvent Extraction Process is a process to melt radioactive metal such as carbon steel or stainless steel together with solvent and to separate cobalt and nickel from iron utilizing the difference of distribution ratio of solute elements between the iron phase and the solvent phase.

Tin-lead alloy (Sn+Pb) was used as a solvent because it forms 2 liquid phases coexistence system with stainless steel and carbon steel. Carbon and silicon were used as extractors because of oxygen affinity difference between separation elements and recovery metal. This process may need to repeat to improve separation performance (Fig.1). In actual full scale apparatus, separation between metal and solvent needs tilting the crucible, while in the screening tests, separation was made after metal cooling down. The solvent used in the extraction step can be used repeatedly.

(1) Test Method

A test specimen and the solvent (Sn+Pb) were melted in the vacuum induction-melting furnace (30kW, 3kHz) under atmospheric pressure with argon atmosphere. Test condition and apparatus is shown in Table 1 and Fig.2 respectively. Test specimens of carbon steel and stainless steel added by 1% cobalt were prepared for the tests.

In the extraction step, carbon was added as an extractant to the saturation of iron into melt of a test specimen and the solvent alloy (Sn+Pb). They are melted at about 1300 ℃ to raise distribution ratio of the separation element. Carbon crucible was used in order that a melting crucible could maintain an extract phase at carbon saturation. For carbon steel specimen, the melt were kept at 1400 ℃ for 2 hours for homogenization, and after holding at 1300℃ for 15 minutes, and turned off the circuit to cool down. For stainless steel specimen, the melt was kept at 1450℃ for 15 minutes in consideration of its melting point rise by contained chromium, then kept at 1400℃ for 20 minutes and cooled down. Samples were taken from the extractant phase (Fe+C) and the solvent phase (Sn+Pb+Ni+Co) for analysis.

In the reverse extraction step, silicon was added as an extractant by 33% to form a extractant phase (Fe+Si+Ni+Co).

(2) Test Result

In the tests to vary the solvent (Sn+Pb) composition, the optimum value for separation performance was found the composition (Sn 52%+Pb 48%). In the tests to vary (Sn＋Pb)/Fe ratio, the optimum value for recovery performance was found the ratio (Sn+Pb)/Fe 30.

Using the optimum values gained above, performance tests

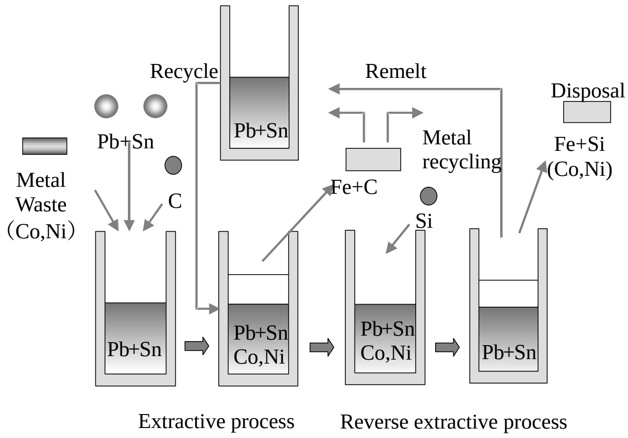

Fig.1 Flow diagram of high temperature extraction process

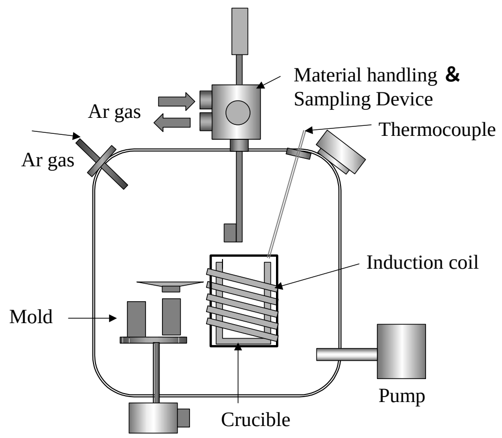

Fig. 2 Schematic drawing of high temperature extraction process

Table 1 Screening test condition for high temperature extraction process

Metal Processed	Test Specimen,Weight kg	Solvent Lead %	(Sn+Pb)/Fe	Co Addition % to Solvent	Extraction Temp. ℃	Atmospheric Gas, MPa
Stainless Steel	SUS304,.3	Sn+Pb, 40-55	10-30	1	1,450-1,400	Ar: 0.1013
Carbon Steel	STPG370, 3	Sn+Pb, 40-55	10-30	1	1,400-1,300	Ar: 0.1013

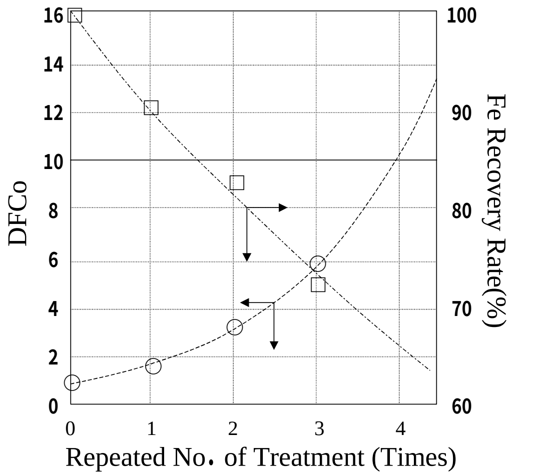

Fig. 3 DFCo, Fe recovery rate and repeated number of high temperature extraction process

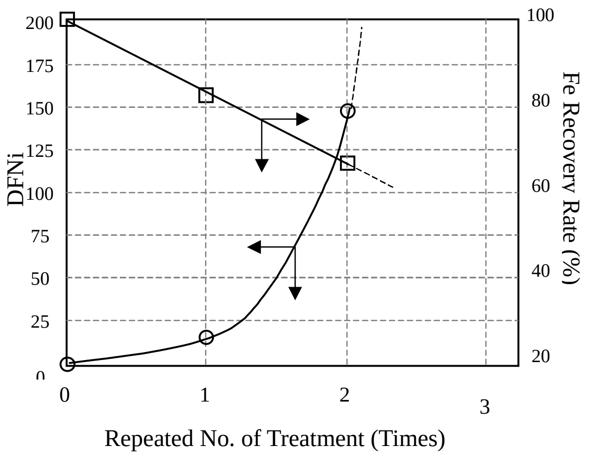

Fig. 4 DFNi, Fe recovery rate and repeated number of high temperature extraction process

on carbon steel and stainless steel were implemented and found that DFCo was approximately 1.3 and DFNi was approximately 10. Because the process does not meet the targets by one cycle, the effect of cycle repetition was calculated using the DF above. As the result, for carbon steel DFCo10 and Fe recovery rate 60% were obtained by four times of repetition (Fig.3). For stainless steel, DFNi 100 and Fe recovery rate 60% were obtained by twice of repetition (Fig.4). Plots of repletion number 2 and 3 in these figures show calculated values.

2 Oxygen Sparging Method

The Oxygen Sparging Process is a process to separate iron from cobalt and nickel under melting condition utilizing the difference of oxygen affinities between metals. The standard free energy for oxidation differs approximately 100 J/g・molO_2 between iron and cobalt (Fig.5). The process consists of three steps (Fig.6). The first is the oxidation step in which oxygen gas is sparged into molten carbon steel or stainless steel to oxidize chromium and iron to form a slag phase with a flux prior to nickel and cobalt which are left in metal phase. The second is the pre-reduction step in which the slight amount of nickel and

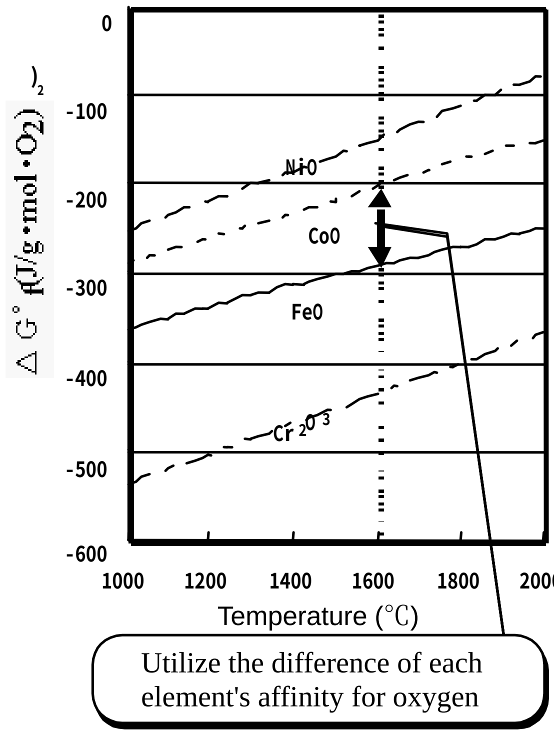

Fig. 5 Principal of oxygen sparging process

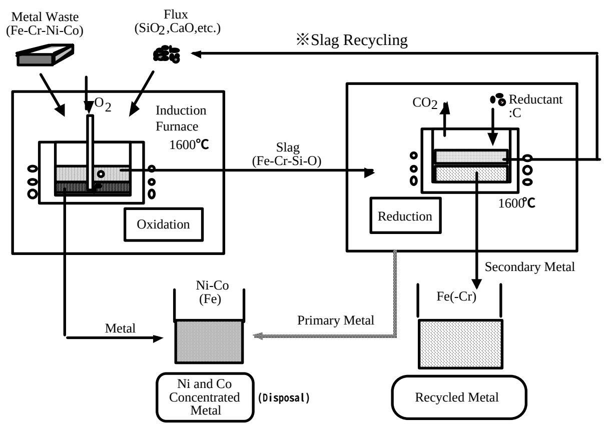

Fig. 6 Concept of oxygen sparging process

cobalt contained in the oxidation slag are reduced by carbon and settled into the metal. The third is the post-reduction step in which chromium and iron in the slag is reduced by additional carbon and they are recovered as a metal. These double reduction steps enable the effective separation of metals.

The slag in the post-reduction step will be a waste if it is not reused. Therefore it is important that the slag can be reused as a flux in the oxidation step to minimize the amount of the waste.

(1) Test Method

An induction furnace (21 kW) with triple crucibles, i.e., a magnesia crucible (2,500 cm^3), a graphite crucible heated by induction and an alumina holder were prepared for the tests. The experimental apparatus are shown in Fig.7. Test specimens

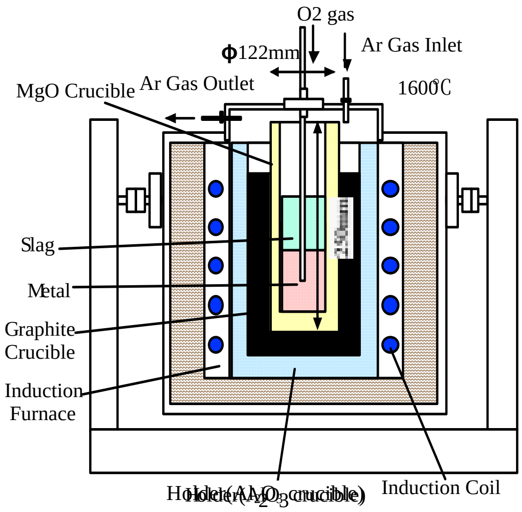

Fig. 7 Schematic drawing of oxygen sparging process

were prepared to add 3.4 % of cobalt to stainless steel and 0.03-0.1 % of cobalt to carbon steel. Each step of the process was tested separately, using test specimens (3kg) under the test condition shown in Table 2.

In the oxidation step, the molten slag attacks the magnesia crucible strongly, therefore the oxidation was implemented in 4 stages with changing the crucible for each stage.

(2) Test Result

In the tests to vary the flux ($MgO+SiO_2$) composition, the composition (MgO 35%+ SiO_2 65%) was found to minimize the slag attack to the crucible. The slag of this flux composition can dissolve FeO in the range of 5-90 % for ($MgO+SiO_2$) without a big change of slag temperature, therefore the slag is very stable for the change of FeO content in the slag.

In the carbon steel test on a scale of 3 kg metal, DFCo 110 and the iron recovery ratio 72% were seen as shown in Fig.8, meeting the target. Even on a larger scale (20 kg metal) test, similar results were obtained. In the stainless steel tests, DFCo 41, DFNi 56 and the iron recovery ratio 62% were seen as shown in Fig.8. The reason why DFCo and DFNi of stainless steel are low comparing with those of carbon steel is that high concentration of chromium makes the slag viscosity higher. This problem could be solved by separating chromium concentrated slag before the oxidation step, because oxygen affinity of chromium is much higher than that of iron to oxidize chromium prior to iron.

3 Molten Salt Electro-refining Process

The Molten Salt Electro-refining Process is a process to electrolyze waste metal as an anode in molten salt. Radioactive metal containing cobalt and nickel is electrolyzed to preferentially dissolve iron, i.e., relatively base metal from the anode and recover it at the cathode as shown in Fig.9. In this process, cobalt and nickel leave from the anode as slime or as

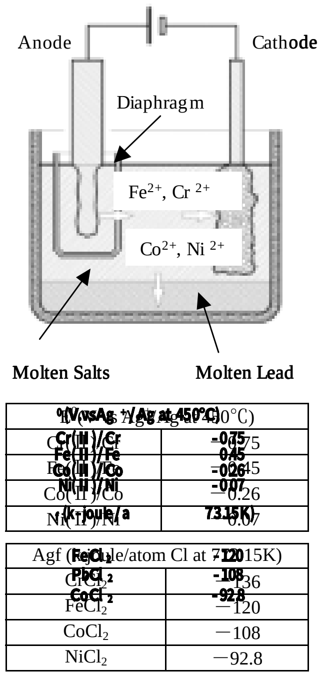

Fig. 9 Diagram of molten salt electro-refining process

Table 2 Screening test condition for oxygen sparging process

Test Specimen, Weight kg	Temperature ℃	Oxidation Ratio in Residual Metal %	Base Ratio in Slag, O/SiO_2	Element Added wt%
Stainless Steel, 3	1,600	Fe:15-20	1.1	Co:3.4
Carbon Steel, 3	1,600	Fe:15-20	0.5-1.0	Co:0.03-0.1

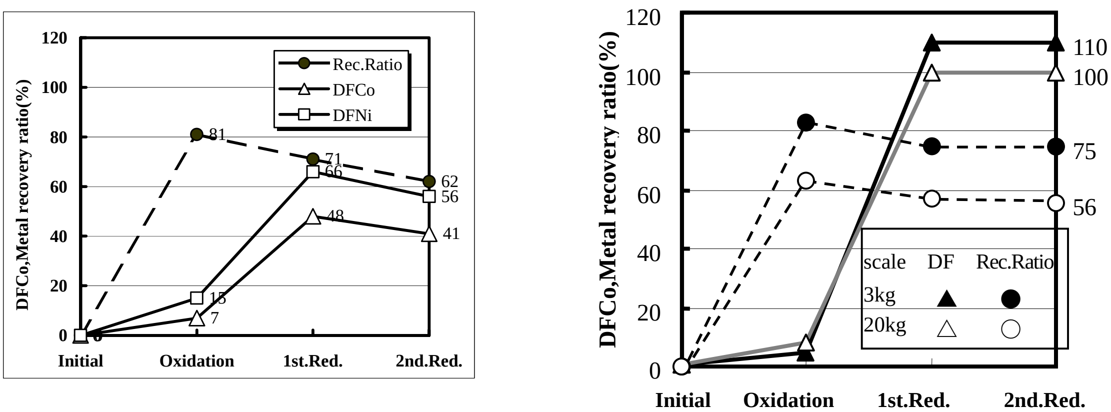

Fig. 8 DFCo, Fe recovery rate of oxygen sparging process screening tests
left: carbon steel, right: stainless steel

Table 3 Screening test condition for molten salt electro-refining process/ diaphragm method

Metal Processed	Run No.	Shape of Test Specimen, Composition, wt%	Salt used, kg	Fe Conc. Average, %	Diaphragm	Current Density, A/cm^2
Stainless Steel SUS304	6	One-piece, Cr17.2, Ni7.9, Co1.1	3.3	1.0	Quartz Filter	0.05-0.4
Carbon Steel STPG370	5	One-piece, Co1.0	3.3	1.0	Quartz Filter	0.05-0.2

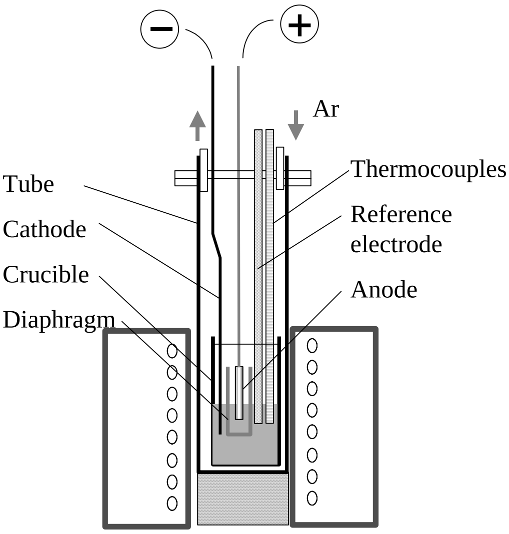

Fig. 10 Schematic drawing of molten salt electro-refining process/ the diaphragm method

ion and tend to contaminate recovery metals by deposition at the anode. In order to protect cobalt and nickel from anode deposition, two methods were taken into account. One is the diaphragm method to constitute a diaphragm barrier between cathode and anode to collect slime. The other is the lead extraction method to extract cobalt and nickel left from anode by liquid lead at the bottom of the molten salt utilizing the difference of the oxidation-reduction potential.

Although combined use of both the methods is available, each one was separately tested on their performance.

(1) Test Method

In the diaphragm method tests, 1,100g of $LiCl\text{-}KCl\text{-}FeCl_3$ at 500℃ were loaded into to a vertical electrolysis cell made of Pyrex as shown in Fig.10 and Table 3. $FeCl_3$ was added as supporting electrolyte. Cobalt (not radioactive element) was added by 1% to prepare test specimens of stainless steel or carbon steel used as an anode. A bar of stainless steel with a diameter of 5mm was used as cathode. As a reference electrode to measure electric potential at anode, AgCl/LiCl-KCl was used. The diaphragm for collecting slimes is made of quartz filter. The reactor (crucible) loaded with the molten salt was heated at 500℃ under argon atmosphere and test specimen metals were electrolyzed with constant current.

In the lead extraction method tests, 1,800-2,900g of $LiCl\text{-}NaCl\text{-}FeCl_2$ was loaded as shown in Fig.11 and Table 4. Test specimens of carbon steel or stainless steel added with cobalt 1% was used as anode, and a stainless steel bar (10mm ϕ) was used as a cathode. As a reference electrode to measure electric potential at anode, AgCl/LiCl-KCl was also used. Molten lead was loaded at the bottom of the cell to extract nickel ion and cobalt ion. The reactor (crucible) loaded with the molten salt was heated at 500℃ under argon atmosphere and sample metals were electrolyzed with constant current.

(2) Test Result

In the diaphragm method tests, DFCo 100 and DFNi 700 for stainless steel were seen at current density of 0.2 A/cm^2 and DFCo 10 for carbon steel was seen at current density of 0.05 A/cm^2 (Fig.12). In both cases iron recovery rate was 60 %. Because DF value fell when there is no diaphragm, it is assumed that the quartz filter diaphragm is effective to prevent the slime from moving to the cathode.

In the lead extraction method tests, agitating the boundary surface between the molten salt and lead as well as setting flow guide plate was effective to improve DF of cobalt and nickel. DFCo 15 and iron recovery rate of 60 % were seen for carbon steel. Since stainless steel performance was almost same as that for carbon steel, slime that supposed to be nickel origin deposited on the anode and raised the anode potential. Therefore the anode

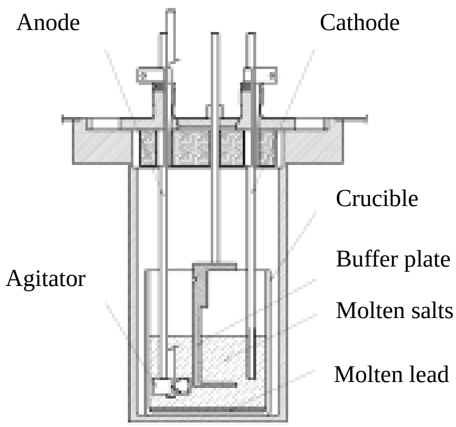

Fig. 11 Schematic drawing of molten salt electro-refining process/ the lead extraction method

Table 4 Screening test condition for molten salt electro-refining process/ lead extraction method

Metal Processed	Run No.	Shape of Test Specimen, Composition, wt%	Salt used, kg	Fe Conc. Average, wt%	Agitation	Current Density, A/cm^2
Stainless Steel SUS304	2	One-piece, Cr18.4, Ni8.5, Co1.0	1.8-2.9	1.0	Yes	0.2
Carbon Steel STPG370	10	One-piece, Co0.1-1.0	1.8-2.9	1.0	Yes	0.15-0.45

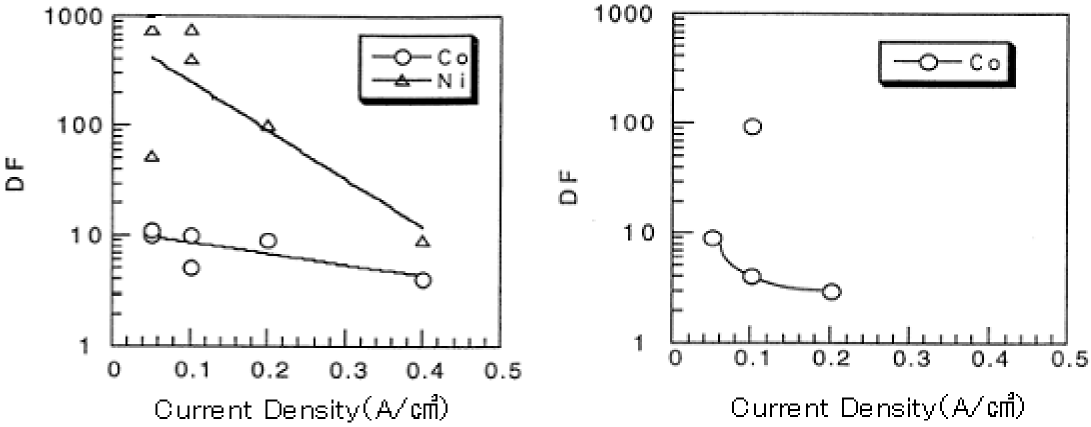

Fig. 12 DF and current density of molten salt electro-refining process
left: stainless steel, right: carbon steel

surface needed to be periodically cleaned.

From the result above, it is estimated that the process combining the both methods, i.e., diaphragm and lead extraction, can achieve DFCo 150 and iron recovery rate of 60 % by multiplying both of DFs above and appears to meet the target. However, issues still remain on the handling of toxic lead at high temperature and heavy apparatus due to double facilities of both methods.

4 Summary of the Screening Tests

The screening tests on the three processes clarified following facts.

(1) The High Temperature Solvent Extraction Process has superior separation performance on nickel. It was estimated twice cycle repetition to both of stainless steel and carbon steel can achieve DFNi 100 with iron recovery rate 60%. But one on cobalt was not so good as estimated DFCo 2 in the twice cycle repetition to both of stainless steel and carbon steel with iron recovery rate 60%.

(2) The Oxygen Sparging Method has DFCo 100, DFNi 100 with iron recovery rate 60% for carbon steel, but DFCo 40, DFNi 50 with iron recovery rate 60% for stainless steel.

(3) Regarding the Molten Salt Extraction Method, the Diaphragm method has DFCo 10, DFNi 700 with iron recovery rate 80%, and the Lead Extraction method has DFCo 15 with iron recovery rate 70%, which did not achieve the target value in single method. Although the combined use of both methods was estimated to achieve DFCo 150 with iron recovery rate 60%, issues must be remained unsolved for the combined use such as large sized total facilities and lead handling at high temperature.

Ⅳ Demonstration Tests on the Oxygen Sparging Process

As a result of the screening tests, the Oxygen Sparging Process was found to separate cobalt and nickel with superior DF and iron recovery, and was, therefore, selected for demonstration tests. Since the process showed excellent performance for carbon steel, major demonstration tests were implemented using carbon steel. The demonstration tests consist of three kinds of tests on the process. First is the one through operation test to confirm process operability in the real process steps for carbon steel, second is the radioisotope test to confirm process performance with radioactive element for carbon steel, and third is the small amount elements tests.

1 One Through Operation Tests

This one through operation test aims to demonstrate that the prototype process can sequentially operate in one through, confirming the target performance of DF and iron recovery rate under actual conditions. In the screening tests, all tests were implemented in a step-by-step basis. In the demonstration tests, the metal and slag were not cooled during the process operation.

(1) Test Method

Ten (10) kg of carbon steel and 1% cobalt, which was added to quantify the separation ratio of cobalt precisely, were melted in a induction furnace, and then poured into a preheated ladle (0.1 m^3, Inconel with an alumina stump) as shown in Table 5. In the oxidation step, the oxygen gas was sparged (0.15 m^3/min) into the ladle. After the oxidation, the slag was poured into an electric furnace by tilting and separated from the metal. In the pre-reduction and post-reduction steps, the proper amount of carbon was added to the slag in the furnace to recover the cobalt and iron.

Table 5 Demonstration test condition for oxygen sparging process

Name of Test	Test Specimen, Weight kg	Temperature ℃	Oxidation Ratio in Residual Metal %	Base Ratio in Slag, MgO/SiO_2	Element Added %
One-through	CS, 10	1,600	Fe:15-25	0.5	Co:1
Radioisotope	CS, SUS, 0.5	1,600	Fe:15-25	0.5	Co:0.01, Co60

Note: OT; One Through Test, RI; Radioisotope Test, R.R; Recovery Rate

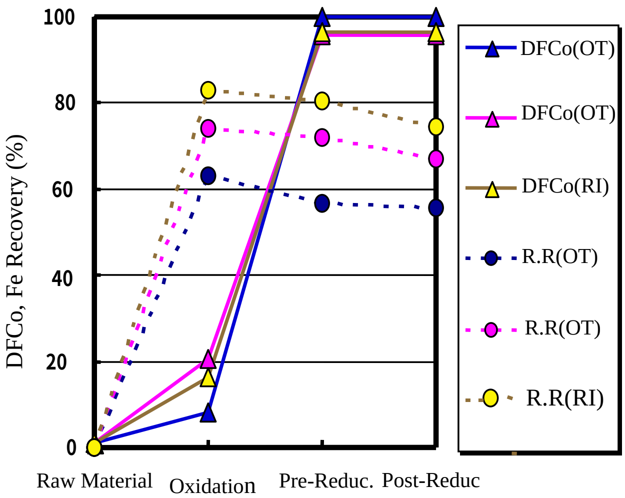

Fig.13 DFCo and Fe recovery rate of oxygen sparging process demonstration tests

(2) Test Results

It was seen that under actual operating conditions the prototype process could achieve a DF of almost 100 for cobalt and an iron recovery ratio of approximately 70% (Fig.13), meeting the target values. Once the specimen was melted, external heat input was not needed at the oxidation stage, because the iron oxidation reaction is exothermic, covering the heat loss of the apparatus. This means that actual size apparatus could be simplified. No severe attack to the ladle wall was observed because the surface of alumina stump was covered by solidified slag (self-coating).

2 Radioactive Isotope Tests

Since the Oxygen Sparging Process is a process of element separation, all tests described above were implemented by non-radioactive elements. It is, however, important to confirm the separation performance with radioactive elements. Considering the separation performance would not depend on radioactive concentration, very low level Co60 was prepared for the test specimen.

(1) Test Method

Cobalt 11.5 mg was sealed in a vacuum quartz cell. Two cells were irradiated in a high neutron flux reactor (the Japan Materials Testing Reactor) for 10 minutes. For the test specimen, 500 g of carbon steel was mixed with the irradiated cobalt to prepare approximately 1000 Bq/g as shown in Table 5. The test specimen was oxidized and reduced according to its process procedure for tracing Co60 behavior. A germanium detector measured radioactivity distribution between the slag and the metal. The tests were implemented twice under an identical condition.

(2) Test Results

It was seen that the process achieved a DFCo of almost 100 (92 for run 1, 96 for run 2) and an iron recovery ratio of approximately 75% (Fig.13). It was confirmed that separation performance of radioactive element would be same as that gained by non-radioactive element.

3 Minor Elements Tests

The tramp elements tests and the trace elements tests were implemented to investigate behavior of minor elements that could exist in waste metals.

(1) Test Method

For the tramp elements tests, test specimens were prepared by adding 5 g of each tramp element such as aluminum, cupper, lead, and zinc into 500 g of carbon steel or stainless steel respectively, and melted in a crucible for the oxidation step. These tests were implemented by same procedure as that described above.

For the trace elements tests, test specimens were prepared by adding one or some of metal elements such as niobium 5g, cesium 1g, manganese 5g, strontium 5g into 500 g of carbon steel, and melt in a crucible for the oxidation step. These tests were implemented by same procedure as that described above.

(2) Test Results

In both of the tramp elements tests using carbon steel and stainless steel, white fume was observed during heat-up and at the first stage of the oxidation step. The white fume was detected as oxidized zinc vaporized in air. Almost all of zinc, lead and aluminum were vaporized during heating. Cupper behaved as same as nickel throughout the oxidation and reduction step.

In the trace elements tests, all of cesium and strontium went out into slag or gas during the first stage of oxidation. Niobium and manganese went into slag, as same as chromium. The result above was shown in the Fig.14. It was confirmed that all elements have different behavior with each other and the Oxygen Sparging Process was confirmed to have good separation performance.

Ⅴ Plant Applicability

1 Study of Process Design

Based on the process data obtained in the tests above, process design was studied for actual application to LLW waste metal arising from a commercial NPP decommissioning (Table6).

2 Effect of the Process on Cost

The cost saving of the process depends on conditions of the object of waste metal and the waste disposal site. The volume of LLW arising from the decommissioning and the disposal cost must be the key factors. According to a trial calculation under an assumption that LLW metal from 10 NPP decommissioning is processed in one facility of the Oxygen Sparging Process, the adoption of the process could save approximately 10% compared with simple melting option.

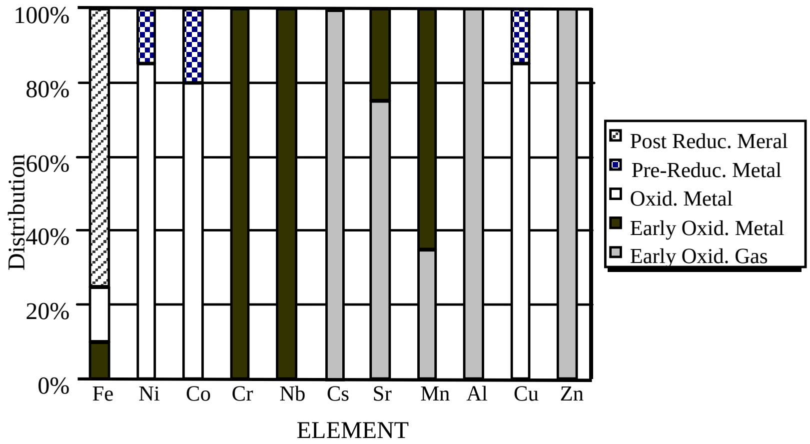

Fig. 14 Small element behavior in oxygen sparging process

Table 6 Optimum conditions for oxygen sparging process

Step in process	Item	Condition
Oxidation	Temperature ℃	1600
	Oxygen Amount%	80-90 to oxidize iron in equivalent
	Refractory	Erosion protection slag coating Magnesia refractory brick & stump
Pre-Reduction	Temperature ℃	1600
	Carbon %	0.04 of oxidized slug in equivalent
Post-Reduction	Temperature	1600℃
	Carbon %	1.5 of oxidized slug in equivalent

When radioactive metal can be recycled for such application as block, plate or sheet in the nuclear industry, the process might save more than the case above. Metal can be below clearance level and released freely when the process is applied to VLLW metal, but it is not so economical, because the disposal cost for VLLW is not so expensive comparing with that for LLW.

Ⅵ Conclusions

The screening tests and the demonstration tests were implemented on the molten metal separation technique to separate cobalt and nickel in a volumetrically contaminated metal such as activated metal. The results are as follows.

(1) As the result of the screening tests on three processes such as the High Temperature Solvent Extraction Process, the Oxygen Sparging Process and the Molten Salt Electrolysis Process, the Oxygen Sparging Process was found to separate cobalt and nickel with superior separation performance to the others.

(2) In the demonstration test of the Oxygen Sparging Process using 10 kg of carbon steel test specimens, it could separate cobalt from steel by approximately DF 100 and provide an iron recovery ratio of 60%. This performance was confirmed by the radioisotope test.

(3) Although the effect of this process on the cost savings depends on the conditions of the object of waste metal and the waste disposal site, a trial calculation showed that the process for decommissioning LLW metals of several NPPs could save the metallic LLW disposal cost.

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