Recovery of Nd and Dy from Magnet Scrap by Utilizing Molten Salt

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Nd–Fe–B magnet

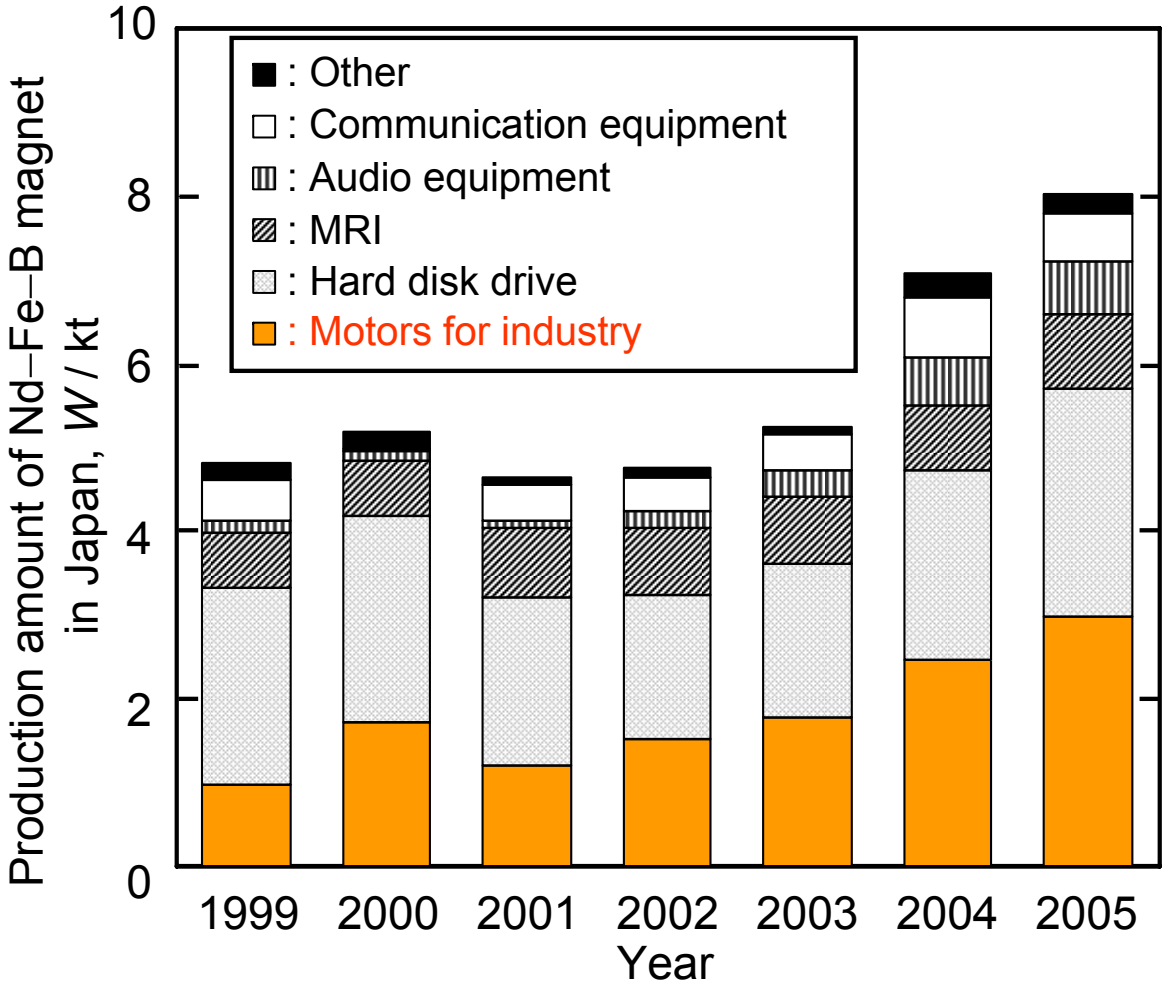


Fig. Production amount of Nd–Fe–B magnet in Japan.

Nd–Fe–B rare earth magnet is the strongest permanent magnet.

- Applications:
  - Hard Disk Drive (HDD)
  - **Motors for**
    - **air conditioner**
    - **generator**
    - **electric vehicle**

★ Large amount of magnet scrap will be produced in the future.

Supply of REE

- Worlds' 97 % supply is dominated by China.
- Prices of REE are escalating especially for Dy.
- Dy is very scarce and limited.

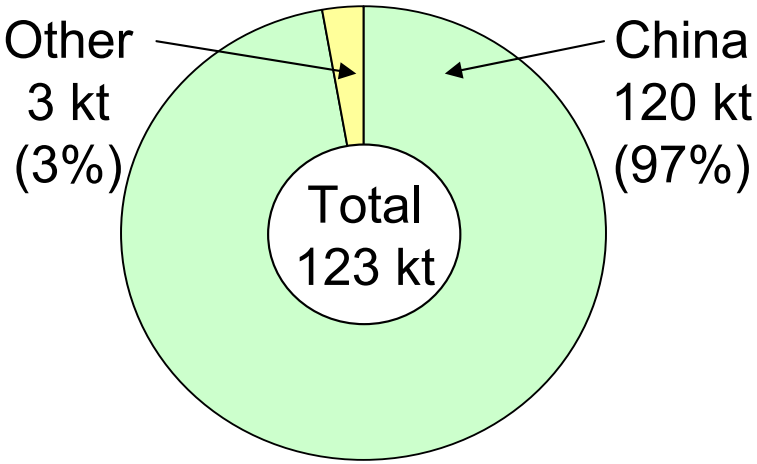


Fig. World share in supply of REE in 2006.
(USGS Mineral Commodity Summaries (2007))

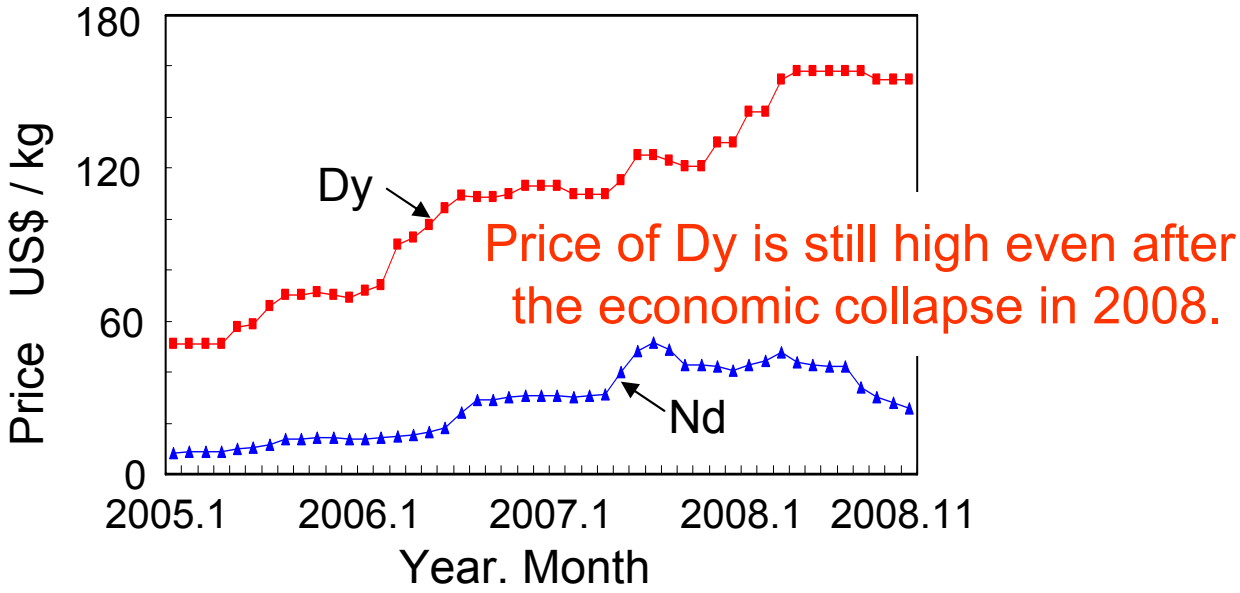
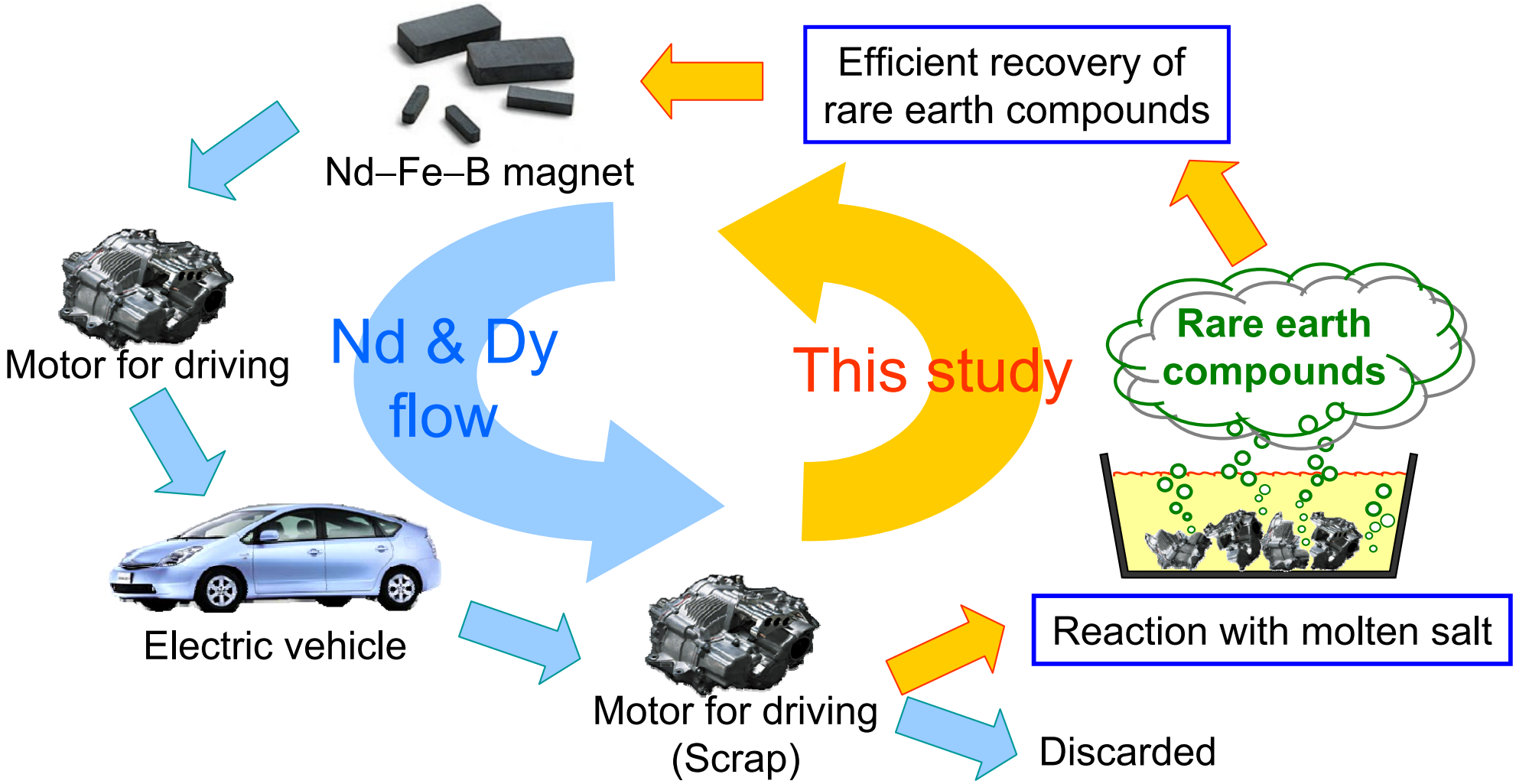


Fig. Change in prices of Nd and Dy.
(Industrial rare metals (2007): Arum Ltd.)

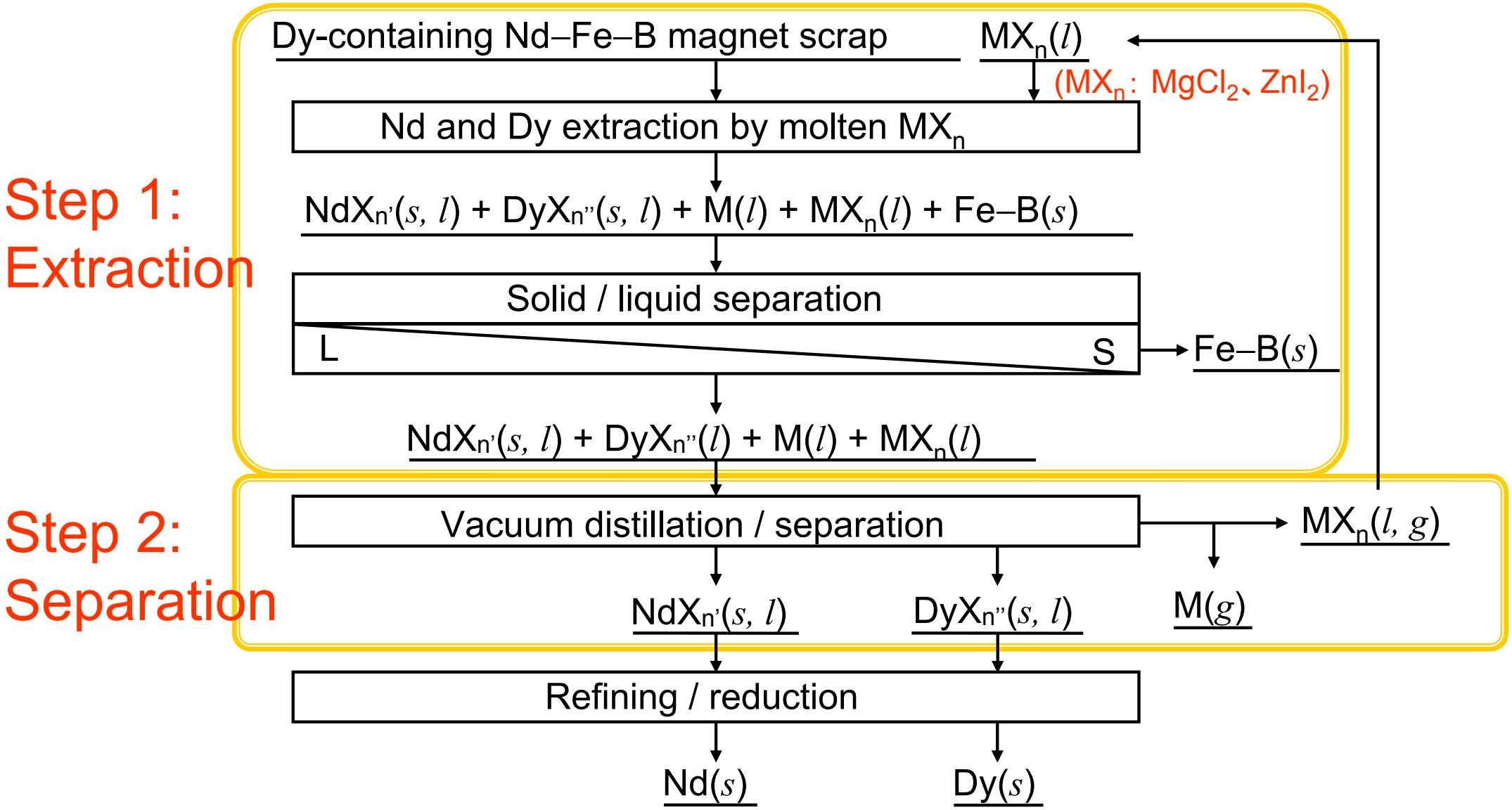
Development of new recycling process is important

Aim of this study



Development of effective recovery process
by utilizing molten salt as a rare earth extracting agent

Flowchart of recycling of the magnet scrap



Experimental procedure (extraction)

- Selective extraction of Nd and Dy by immersing the magnet alloy into molten salt

Extracting agent	$MgCl_2$	ZnI_2
Reaction temp.	1273 K	740 K
Reaction time	3 ~ 12 h	12 h

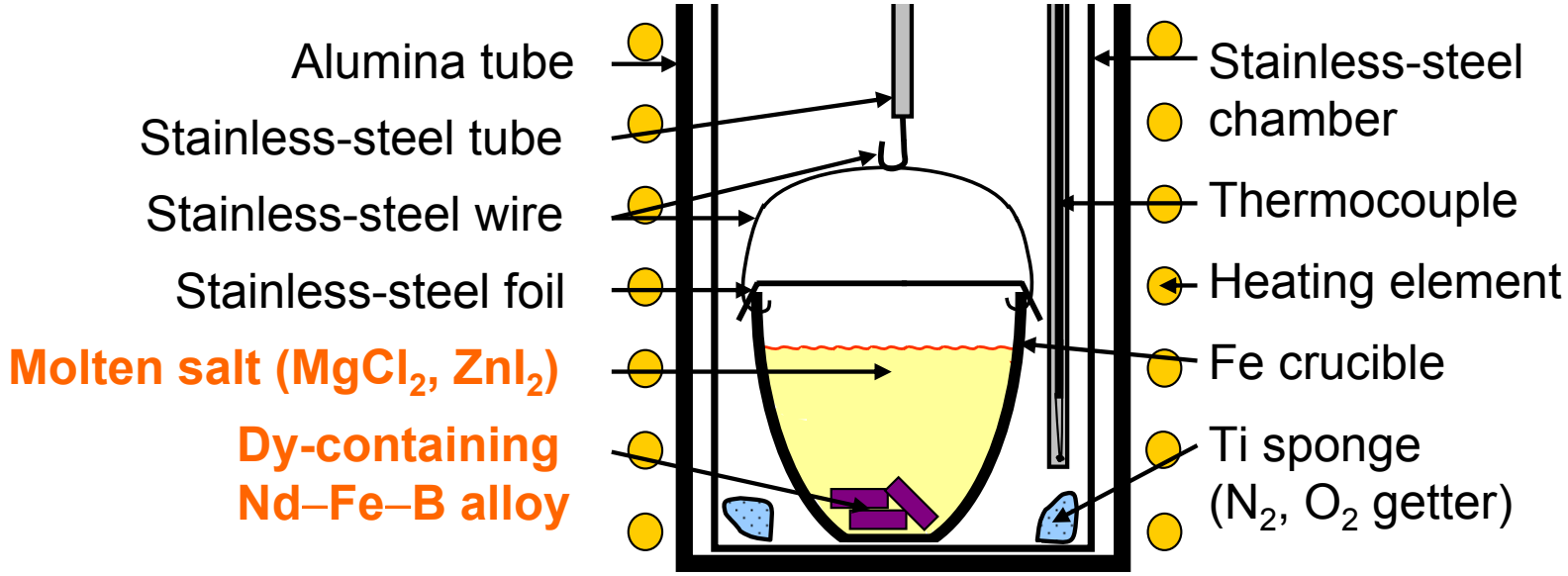


Fig. Schematic illustration of experimental setup for the extraction experiments.

Experimental procedure (separation)

- Separation and recovery of rare earth compounds by vacuum distillation

Extracting agent	$MgCl_2$	ZnI_2
Reaction temp.	1273 K	1073 K
Reaction time	6 h	1 h

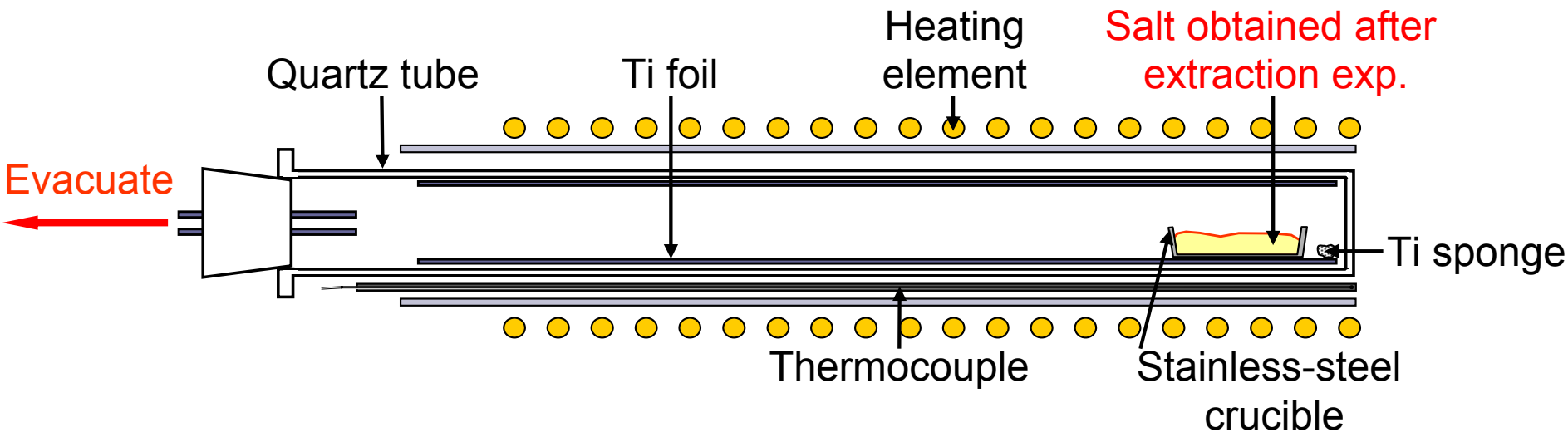


Fig. Schematic illustration of experimental setup for the separation experiments.

Development of novel recovery process

- Recovery of Nd and Dy by utilizing molten $MgCl_2$.
- Recovery of Nd and Dy by utilizing molten ZnI_2.

$$2\ Nd\ (\text{in magnet alloy}, s) + 3\ MgCl_2(l) \rightarrow 2\ NdCl_3(l) + 3\ Mg(l)$$

$$Dy\ (\text{in magnet alloy}, s) + MgCl_2(l) \rightarrow DyCl_2(l) + Mg(l)$$

Results (extraction)

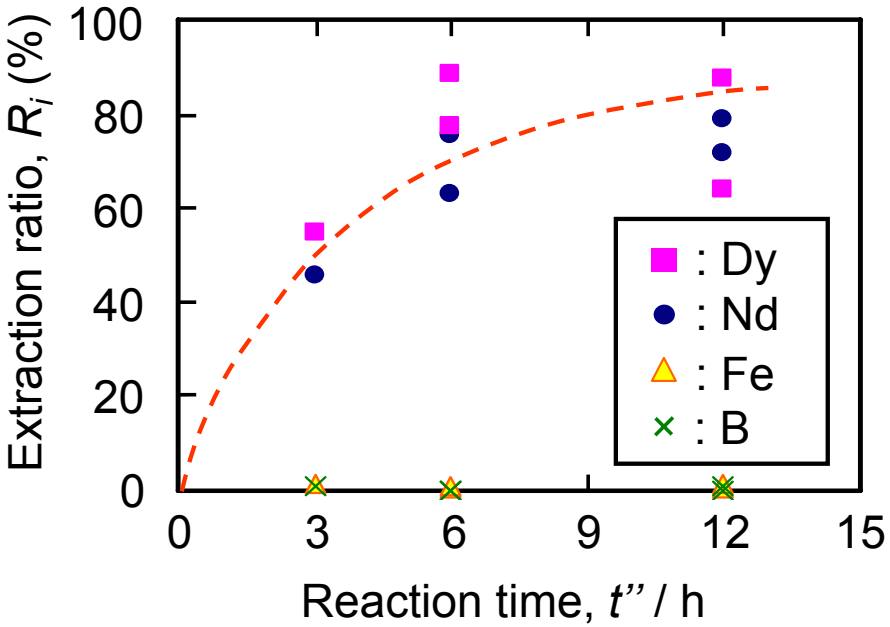
Composition analysis:

Extraction ratio,

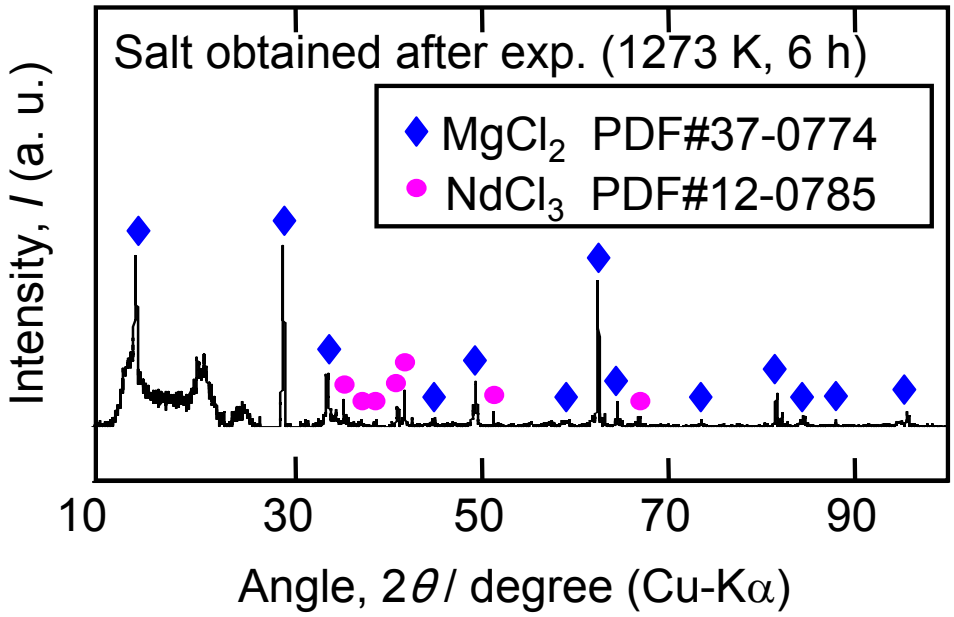
$$R_i = \frac{w_{i,\ \mathrm{salt}}}{w_{i,\ \mathrm{Nd\text{–}Fe\text{–}B}}} \times 100\ (\%)$$

$w_{i,\ \mathrm{salt}}$: mass of element i in salt after exp.

$w_{i,\ \mathrm{Nd\text{–}Fe\text{–}B}}$: initial mass of element i in alloy



XRD analysis:



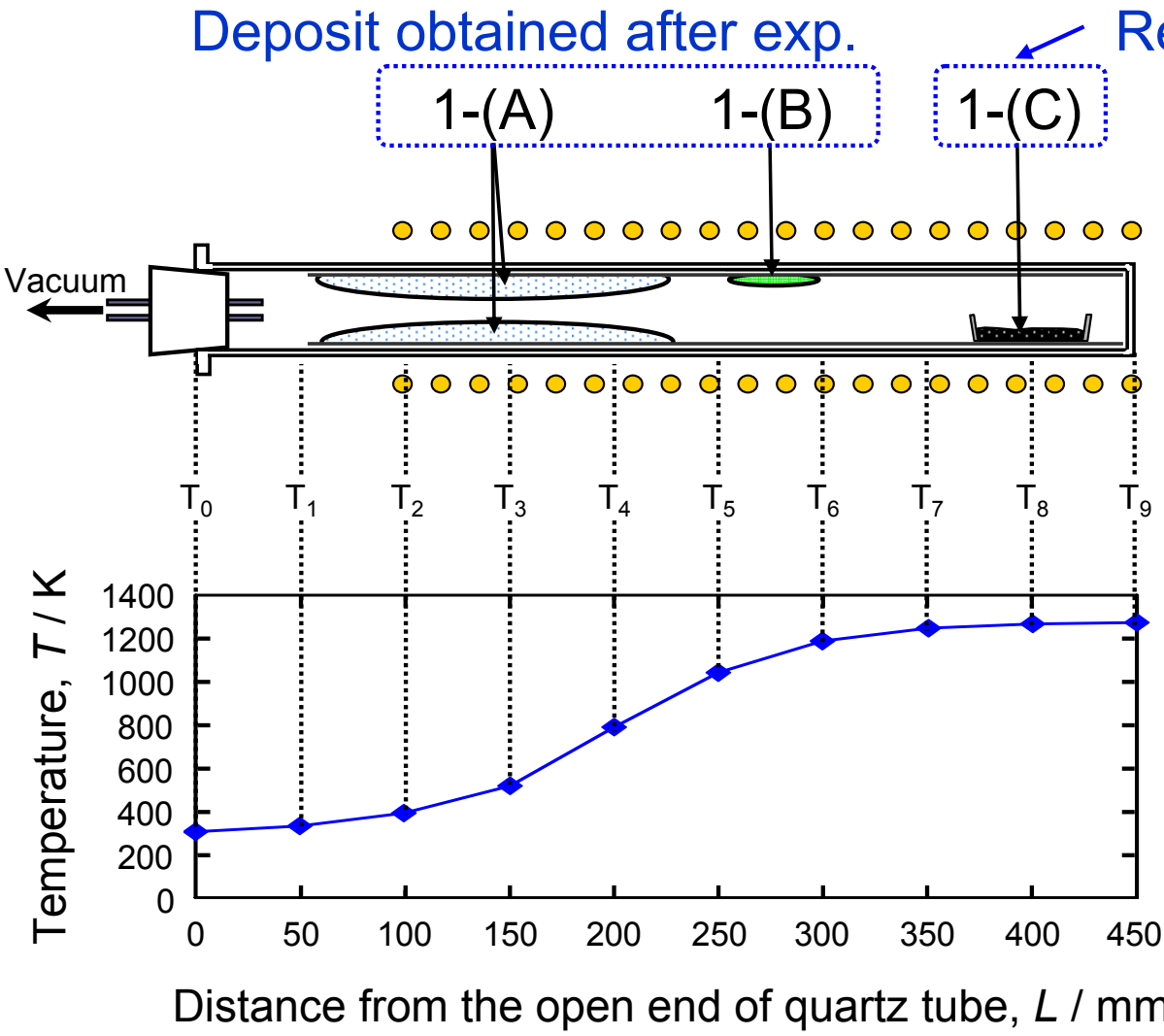
$$2\ \mathrm{Nd}(s) + 3\ \mathrm{MgCl_2}(l) \rightarrow 2\ \mathrm{NdCl_3}(l) + 3\ \mathrm{Mg}(l)$$

$$\Delta G^{\circ} = -151.7\ \mathrm{kJ\ at\ 1273\ K}$$

Nd and Dy were selectively extracted into molten $MgCl_2$.

Results (vacuum distillation)

- Deposition of samples after vacuum distillation



Sample	: Temperature during exp.
1-(A)	: 500 K ~ 900 K
1-(B)	: 1050 K
1-(C)	: 1273 K

Results (vacuum distillation)

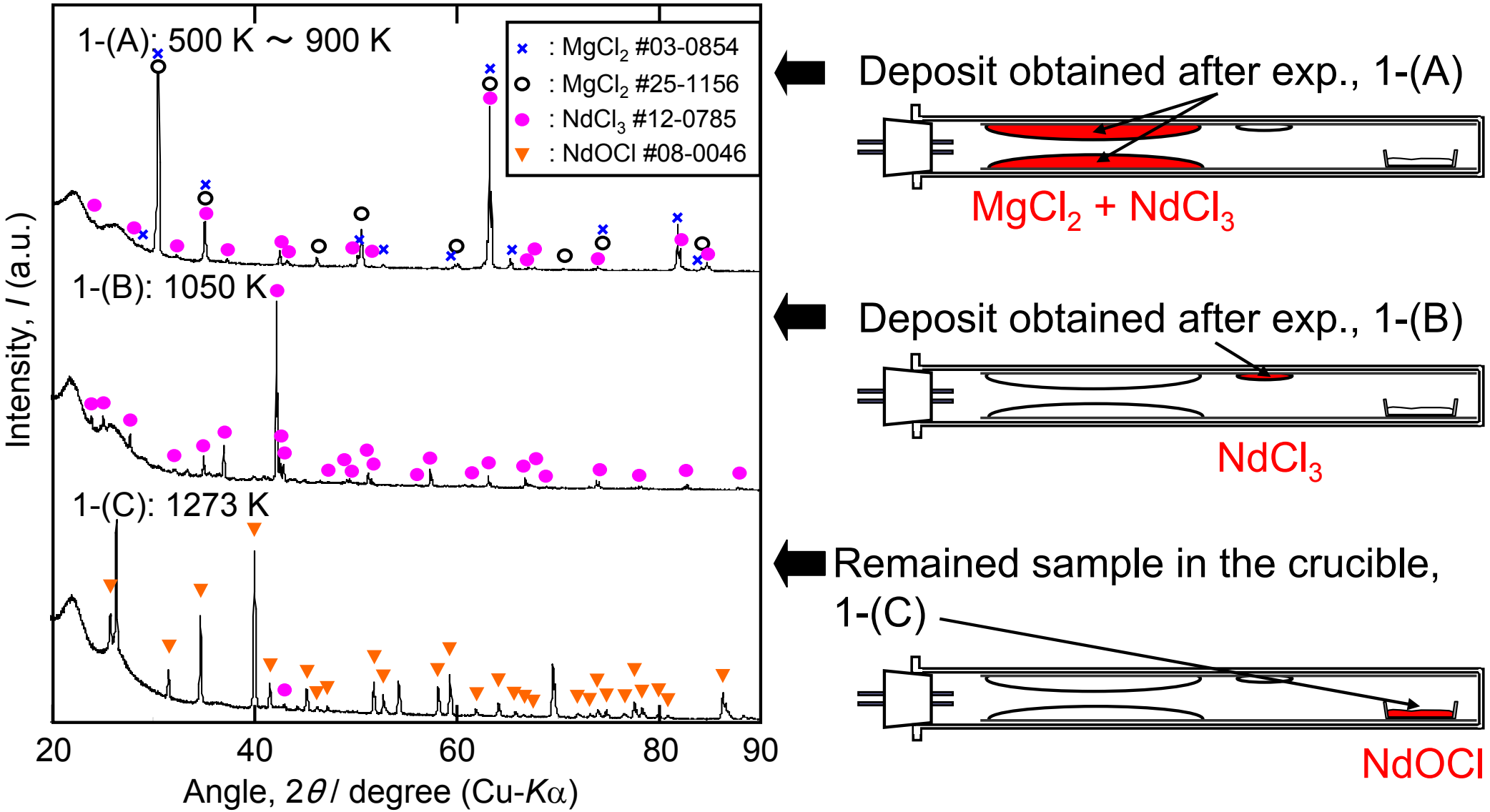
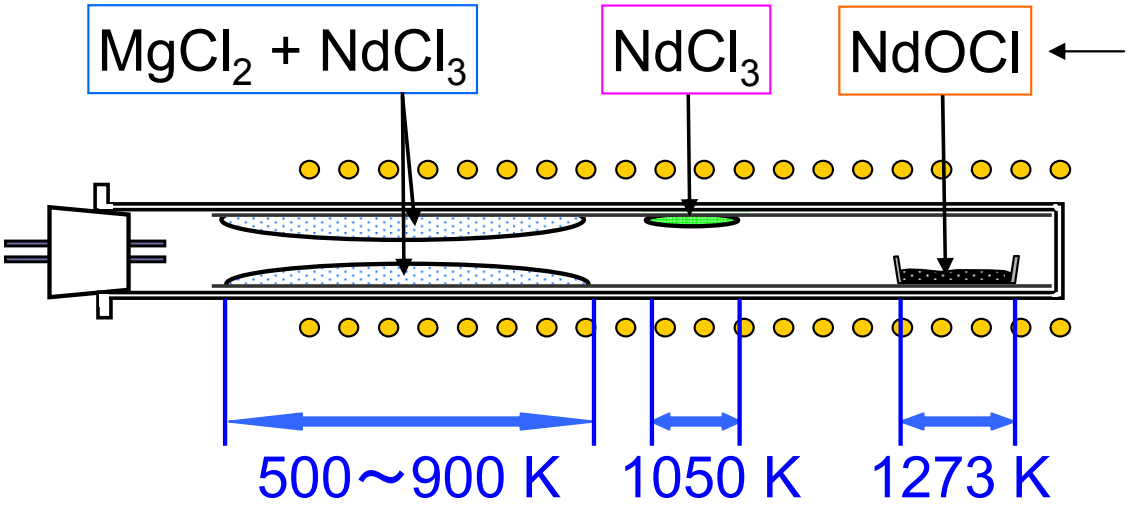


Fig. XRD patterns of the samples obtained after vacuum distillation.

Summary of recovery exp. by utilizing $MgCl_2$

- In the extraction step:
  - Selective extraction of Nd and Dy was experimentally demonstrated.
- In the separation step:
  - $MgCl_2$ was removed from mixed salt.
  - $NdCl_3$ were recovered by gas phase transportation.



Generation of NdOCl is probably due to the hydration of the salt during handling.

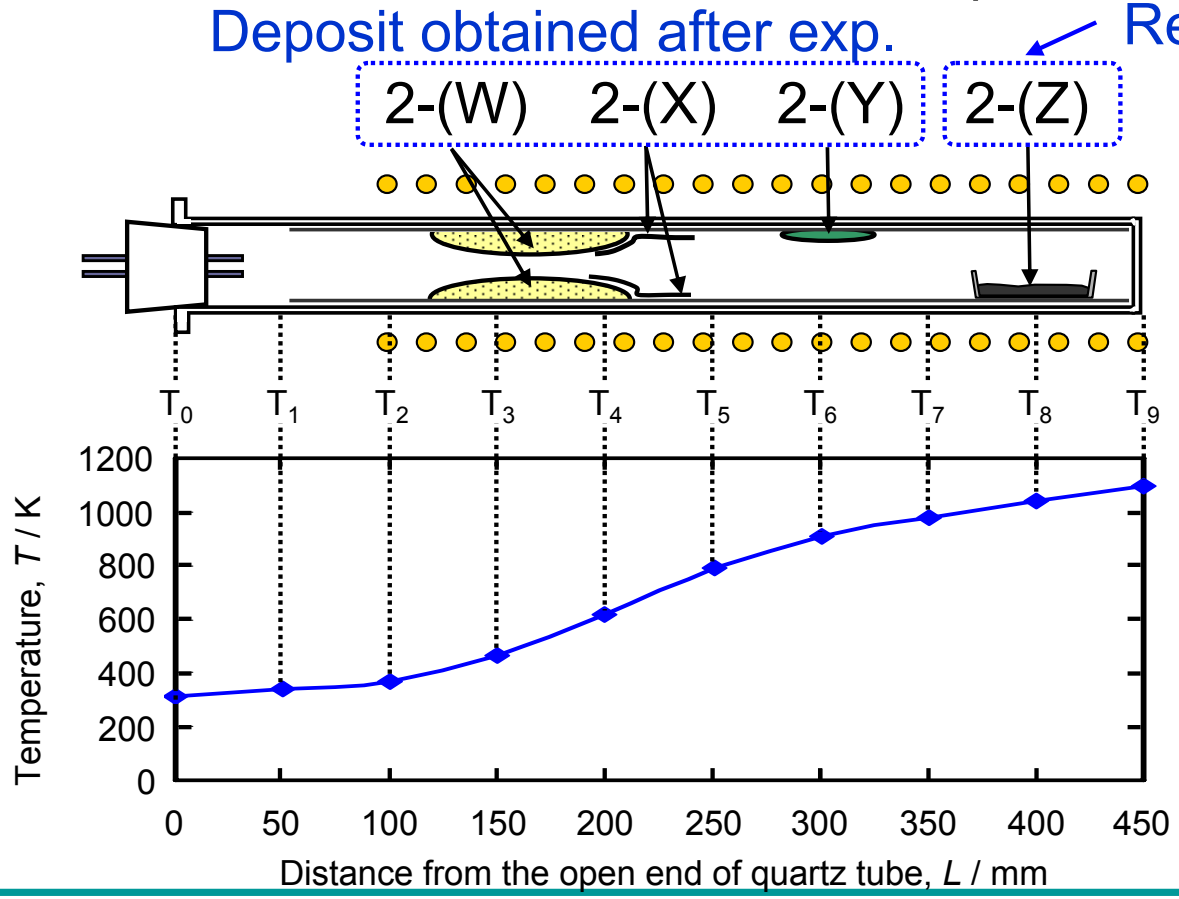
Development of novel recovery process

- Recovery of Nd and Dy by utilizing molten $MgCl_2$.
- Recovery of Nd and Dy by utilizing molten ZnI_2.

$$2\ Nd\ (\text{in magnet alloy}, s) + 3\ ZnI_2(l) \rightarrow 2\ NdI_3(l) + 3\ Zn(l)$$
$$2\ Dy\ (\text{in magnet alloy}, s) + 3\ ZnI_2(l) \rightarrow 2\ DyI_3(l) + 3\ Zn(l)$$

Results (vacuum distillation)

- Small pieces of the magnet alloy were used in the extraction experiment. (740 K, 12 h)
- Substance obtained after extraction exp. was distilled under vacuum.(1073 K、1 h)



Sample	: Temperature during exp.
2-(W)	: 410 K ～ 640 K
2-(X)	: 500 K ～ 700 K
2-(Y)	: 920 K
2-(Z)	: 1073 K

Results (vacuum distillation)

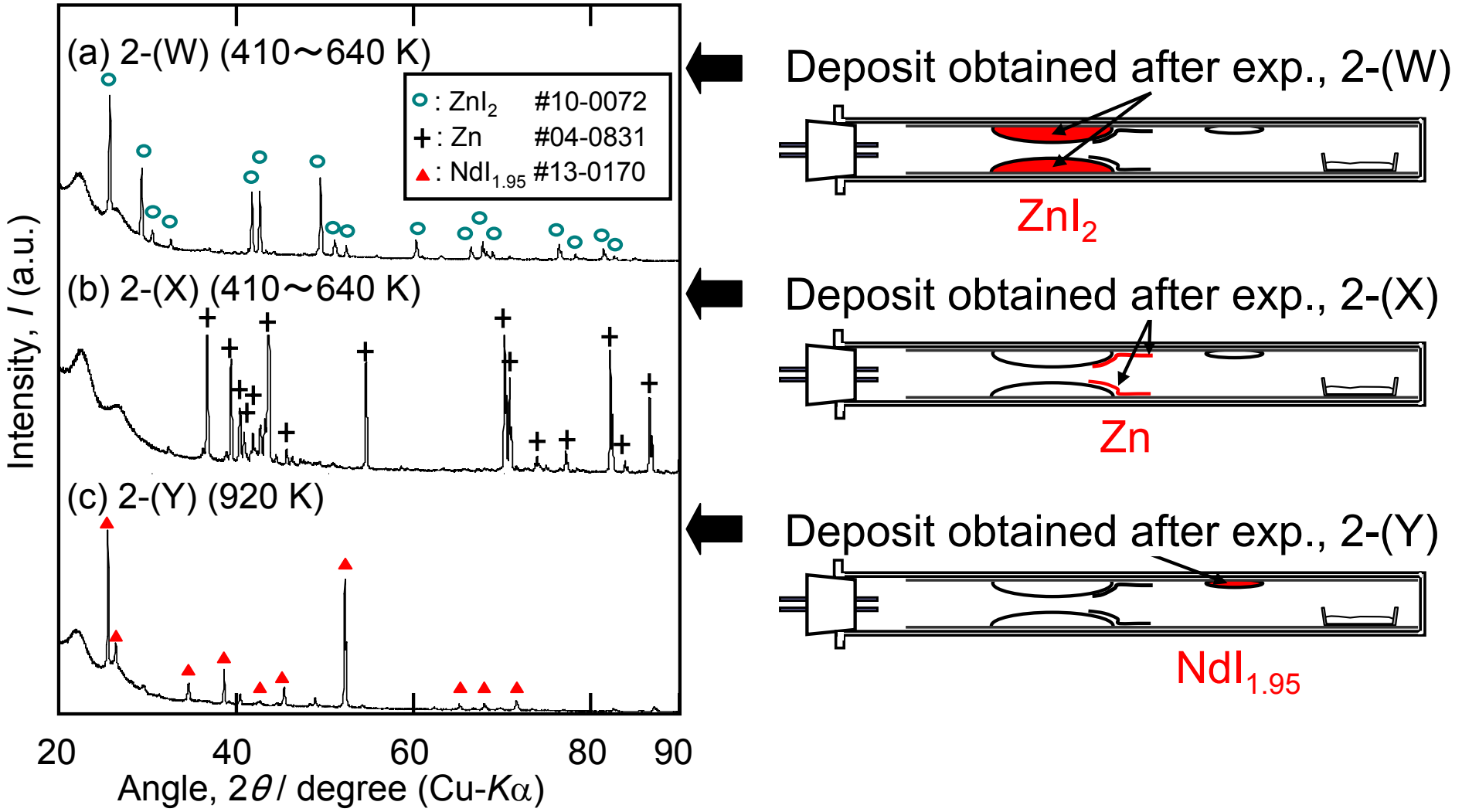
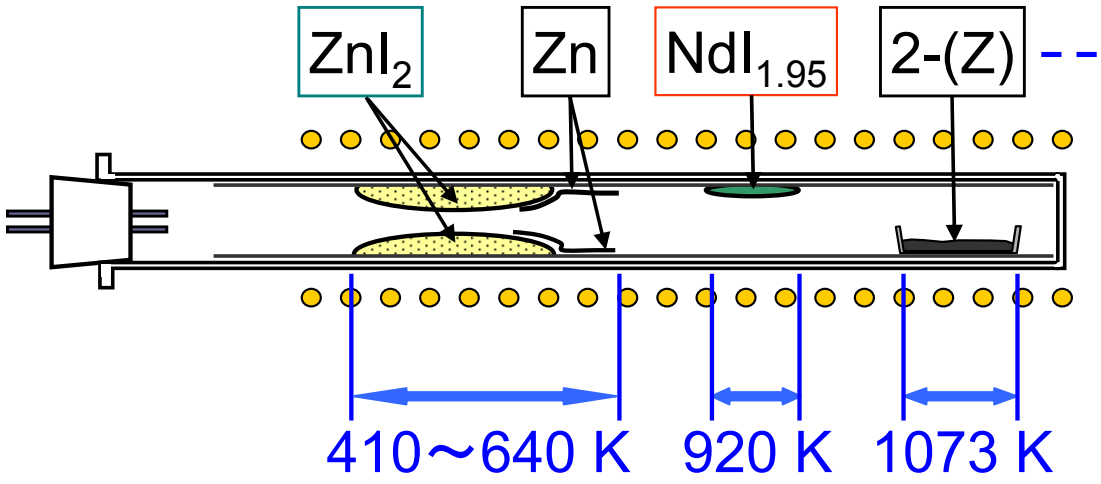


Fig. XRD patterns of the samples obtained after vacuum distillation.

Summary of recovery exp. by utilizing ZnI_2



Obtained residue 2-(Z)

Leaching by distilled water

Dissolved portion

Residue after leaching

NdI_x, DyI_x

Fe-B

$2\ Nd(s) + 3\ ZnI_2(l) \rightarrow 2\ NdI_3(s) + 3\ Zn(l)\ \Delta G^\circ = -472.0$ kJ at 740 K

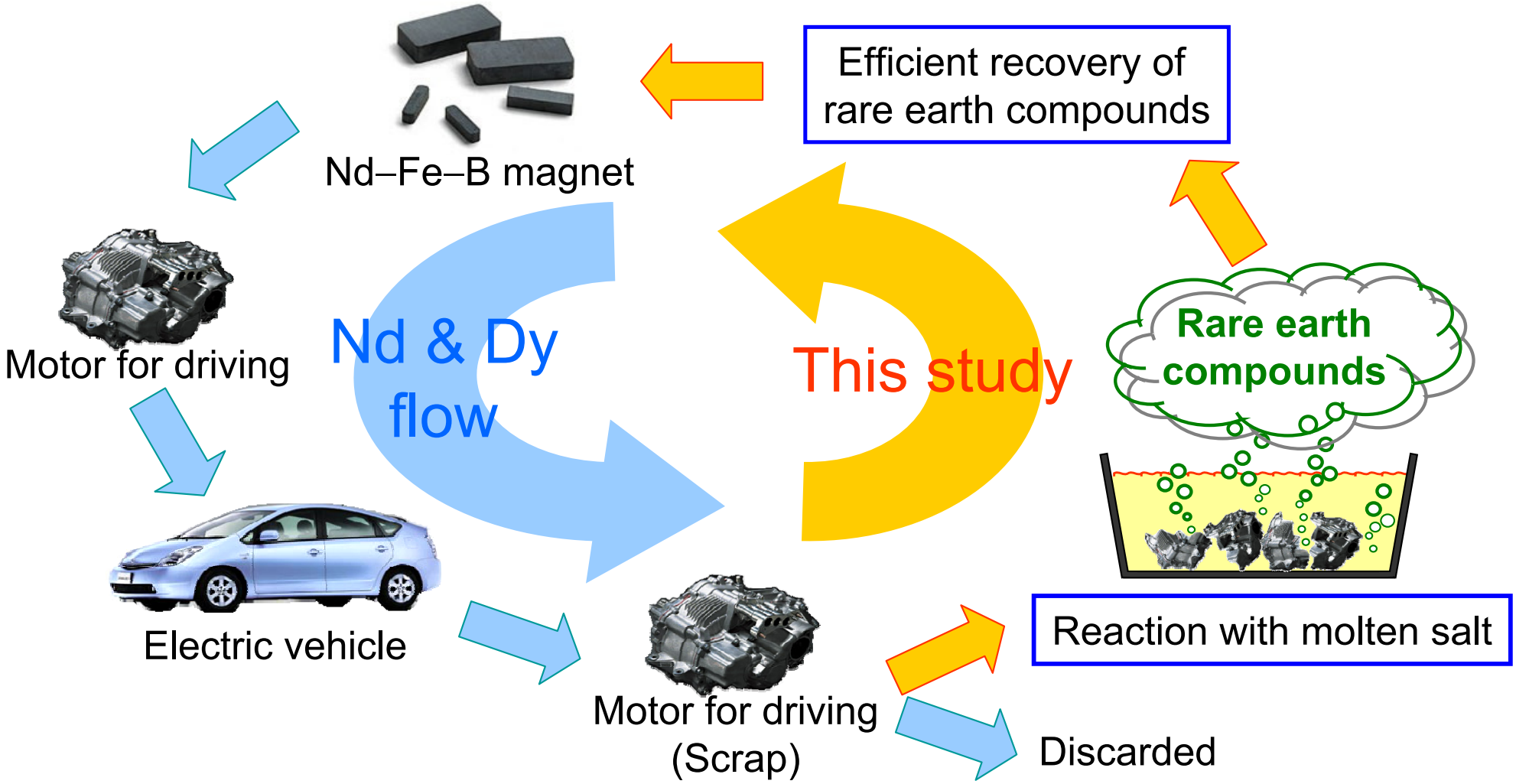
- Selective extraction of Nd and Dy by ZnI_2 is feasible.
- $NdI_{1.95}$ was recovered by gas phase transportation.

Conclusion

- Recovery of Nd and Dy from magnet alloy by utilizing molten $MgCl_2$ or ZnI_2 was investigated.
- Extraction experiment
  - By utilizing $MgCl_2$ and ZnI_2, selective extraction of Nd and Dy was experimentally verified.
- Separation experiment
  - Excess extracting agent was removed by vacuum distillation.
  - Gas phase transportation of rare earth compounds was experimentally verified.

Effective recovery process can be established by utilizing molten salt.

Aim of this study



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Travel expense of the author (Sakae Shirayama) was supported by the grant from the Foundation for the Promotion of Industrial Science (FPIS).

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