§3. Experimental Verification of Hydrogen Isotope Separation and Enrichment by Pressure Swing Adsorption Method

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A process of hydrogen isotope separation is necessary in the environmental safety treatment of exhaust gases from LHD deuterium experiments. We have been making a progress in developing a pressure swing adsorption (PSA) process for hydrogen isotope separation, using synthetic zeolite adsorbents. The results of theoretical analyses and basic experiments carried out till now have convinced us this system can show effective performance of hydrogen isotope separation in practical use. In the last fiscal year, we obtained valuable results from experiments for verification of effective hydrogen isotope separation and heavier isotope enrichment by the PSA method using the cryo-sorption apparatus of NIFS with miner improvement.

In this fiscal year, experimental series following the last fiscal year study have been carried out: the H_2-HD-D_2 three-component experiments and the experiment using synthetic zeolite 4A (SZ-4A) in addition to SZ-5A and SZ-13X.

Additional Miner-improvements of the PSA Apparatus

- Installation of dust-filters in vacuum lines for protection of electromagnetic vacuum-valves against dusts from adsorbent packed-bed.
- Shortening a sample line to reduce the response time of gas analyzing system.

Experimental Operations with the PSA Apparatus

Material and geometric conditions:

Adsorption agent: Synthetic Zeolite NaA (4A), ϕ 2 mm pellets manufactured by MERCK Co. Ltd.

Adsorption column: 21 mm inner diameter Packed-bed of SZ-4A pellets charged at 174 g.

Adsorption Operation

Mass balance of a hydrogen isotope mixture of bulk H_2 and tracers HD and D_2 in the packed-bed operated at the atmospheric pressure:

The amount of hydrogen adsorbed in the packed-bed at 77.4 K agrees with that estimated from the isotherm for bulk-H_2 on SZ-4A, within a few % error, which has been measured by a volumetric adsorption apparatus.

Also, the enrichment ratio of tracer D_2 or HD agrees with that estimated from the multi-component adsorption behavior based on isotherms in accordance with the Ideal-Solution Adsorption Theory. The enrichment ratios of tracers D_2 and HD for the adsorbed phase to the gas phase are 2 and 1.4, respectively.

Displacement Adsorption Operation

Examination of breakthrough behavior of tracers D_2 and HD in a H_2-HD-D_2 mixture flowing through the packed-bed column at 77.4 K:

Operational condition: gas flow rates of 1.0-10.0 NL/min.

From breakthrough experiments operated at the variety of gas flow rates, we obtained the characteristics of mass transfer of tracers D_2 and HD in the SZ-4A packed-bed, which were similar to those in the packed-bed of SZ-5A or SZ-13X.

Evacuating Desorption Operation

Operational conditions: evacuation rate of 600 NL/min, ultimate pressure of 140 or 93Pa for an operation period of 20 or 26 min, respectively.

The enrichment ratio of D_2 in a gas sample recovered for 20 min (reaching a pressure of 140 Pa) was 0.96, and that for 26 min (reaching a pressure of 93 Pa) was 1.24. These values are smaller than the ratio 2.0 adsorbed in the packed-bed. This disagreement suggests that the heavier component would be concentrated in the residual volume adsorbed which could not be recovered by evacuation.

Heating and Evacuating Desorption Operation

Operational conditions: evacuation rate of 600 NL/min, heating from 77.4 K up to 273 K, ultimate press. of 0.1 Pa.

By the heating and evacuating operation following the evacuation process of ultimate pressure 140 Pa, the amount of gas mixture recovered was 27 % of the total amount adsorbed. In the case of evacuation to ultimate pressure 93 Pa, the amount recovered was 17.3 % of that. From mass spectrometric analysis, it was proved that D_2 was contained at an enrichment ratio of 4.81 or 5.64 in the case of 27 % or 17.3 % residual, respectively.

These results suggest that a pressure-and-thermal swing adsorption process (PTSA) can be expected to realize a high performance system of hydrogen isotope separation and enrichment.

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