

# Recovery of Hydrogen Isotopes by Liquid-Gas Contactor from $\text{Li}_{17}\text{Pb}_{83}$ Blanket

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(Received: 11 September 2014 / Accepted: 19 January 2015)

Rates of  $\text{H}_2$  transfer between  $\text{Ar}+\text{H}_2$  gas bubbles and fluidized  $\text{Li}_{17}\text{Pb}_{83}$  (Li-Pb) are experimentally and analytically determined.  $\text{Ar}+\text{H}_2$  gas bubbles directly contact with liquid Li-Pb in a SS-304 vessel. The diameter of bubbles and terminal rising velocity are estimated from balance between several forces including surface tension, gravity, inertia force and drag ones. Then, variations of the  $\text{H}_2$  concentration in a bubble contacting with Li-Pb are estimated in terms of the  $\text{H}_2$  capacity in the bubble and diffusion rate through Li-Pb interface. It is found that  $\text{H}_2$  transfer between bubbles and liquid is limited by H diffusion in the Li-Pb boundary layer. Variations of the  $\text{H}_2$  concentration with time are determined experimentally, and the results are extended to the design of a liquid-gas contactor for tritium recovery from a Li-Pb blanket flow in a commercial fusion reactor.

**Keywords:** Li-Pb eutectic alloy, tritium, breeder, liquid-gas contactor, hydrogen diffusion, recovery

## 1. Introduction

A eutectic alloy of  $\text{Li}_{17}\text{Pb}_{83}$  (Li-Pb) is a promising material for a liquid blanket of a fusion reactor, which makes it possible to constitute a high-performance blanket system producing heat for electric power generation and tritium for the next generation fuel at the same time effectively [1,2]. Fluidized eutectic Li-Pb alloy is expected so as to act as a dual coolant along with He flow and tritium breeder to produce the next-cycle D-T fuel. In addition, Li-Pb works as a protect shield from strong neutron beam and gamma ray along with an energy convertor from nuclear energy through heat finally to electricity. The inlet and outlet temperatures are designed within allowable ones with use of reduced activation steel such as F82H or EUROFER. It is expected that material durability is kept in good condition even under the harsh operating conditions of temperature and neutron/gamma irradiation. Efficient recovery of heat and tritium outside of the reactor chamber is the most important issue. The tritium recovery issue has been investigated experimentally and analytically in our previous works [3-6].

One of the most important physical properties to design a liquid blanket system of a fusion reactor is diffusivity and solubility of tritium in breeders. Although several researches were presented for the solubility, diffusivity and isotopic exchange of H isotopes in static Li-Pb [7-12], there seems to be scattering in solubility in

especial. This may be because differences in chemical conditions of Li-Pb and simultaneous absorption of other gaseous species affect the solubility and diffusivity in Li-Pb [13-15]. There are also several researches on recovery of tritium from fluidized breeder under conditions resembling to an actual blanket system [16-22]. These methods are classified into two. The first candidate method to recover tritium continuously from a fluidized Li-Pb loop will be a permeation window system. Some basic experiments related with the H permeation window were presented previously [16-20]. As another way, a dispersed liquid contact or a liquid-bubble contactor tower was also investigated experimentally [21,22], where droplets and liquid Li-Pb flow counter-currently in a packed tower. However there is less experimental information on H isotope recovery from fluidized Li-Pb by a bubble column or liquid-gas direct contactor.

In order to obtain basic mass-transfer parameters for tritium recovery from the Li-Pb blanket, a bubbling tower for direct contact between  $\text{Ar}+\text{H}_2$  and Li-Pb or simulant fluid is set up in our laboratory, and experiment of  $\text{H}_2$  absorption/desorption is performed. The tritium recovery system is closely connected with not only the physical properties of solubility and diffusivity but also fluidized conditions of Li-Pb such as the bubble rising velocity, surface tension, viscous force and so on. All those parameters are related with the tritium recovery rate, and the total design for the blanket system is presented.

Li-Pb of 3.3 L in volume is made up in a SS-304 vessel after its surfaces are cleaned up in the experiment.

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After Li-Pb homogeneity is confirmed, bubbles of an Ar-H<sub>2</sub> mixture are supplied into a fresh Li-Pb or conversely bubbles of Ar are done to Li-Pb pre-saturated with H isotopes. These experimental results are analyzed using material balance equations between gas bubbles and Li-Pb. Results are compared among different conditions and are extended to a design of a tritium recovery system of a liquid blanket. The present collaboration research is performed as one of the ITER-BA activities.

## 2. Analysis

### 2.1. Formation and movement of bubbles in liquid

Transfer of hydrogen isotope from gas bubbles to surrounding liquid or vice versa is analyzed based on not only material balance and mass-transfer rate equations but also a momentum equation to determine the movement of a gas bubble. The present analysis is deeply related with the review study on bubble formation [23,24] and Kondo's analysis for Li-Pb [21], where a similar momentum transfer and mass-transfer analysis is carried out. Description below outlines the above previous researches.

When gas bubbles are blown through an I-type nozzle into liquid bulk with a constant (low) flow rate,  $Q$  [m<sup>3</sup>/s], a relation between the I-type nozzle diameter,  $D$  [m], and the diameter of bubble,  $d$  [m], is written as follows:

$$\pi\sigma D = (\rho_L - \rho_G) \frac{\pi d^3}{6} g \quad \text{for low } Q \quad (1),$$

where the buoyancy force on the left-hand side is balanced with the surface tension on the right-hand one, and it is assumed that the direction of gas bubbles blown through the nozzle is right angle to the nozzle surface. In addition,  $\sigma$  [N/m] is a surface tension,  $\rho_L$  and  $\rho_G$  [kg/m<sup>3</sup>] are the densities of liquid and gas and  $g$  [m/s<sup>2</sup>] is gravity.

When the bubble flow rate becomes high, on the contrary, the effect of inertial force should be taken into consideration. Then the bubble diameter,  $d$  [m], and the total flow rate  $Q$  of gas blown into liquid is related by the following equation:

$$\frac{(\alpha\rho_L + \rho_G)}{3\pi d^2} Q^2 = (\rho_L - \rho_G) \frac{\pi d^3}{6} g \quad \text{for high } Q \quad (2),$$

where the buoyancy force is balanced with the inertial force on the left-hand side. Bubbles formed in Li-Pb bulk volume rise in the upward direction, and  $\alpha$  is defined as a dimensionless constant to express the effect of virtual mass of surrounding liquid which is estimated as  $\alpha=11/16$  [23].

A gas bubble ejected from a nozzle is accelerated by the buoyancy force initially, and then it approaches to a terminal velocity due to resistance of viscous force. The distance approaching to the terminal velocity is estimated 3.9 mm in the present condition. Its value is much smaller than the distance between the nozzle and liquid surface,  $L$  [m]. Therefore, it can be assumed here that the bubble rises always at the terminal velocity,  $u$  [m/s]. The terminal velocity  $u$  is estimated from the following equation:

$$\frac{C_D}{2} \rho_L u^2 \frac{\pi d^2}{4} = (\rho_L - \rho_G) \frac{\pi d^3}{6} g \quad (3),$$

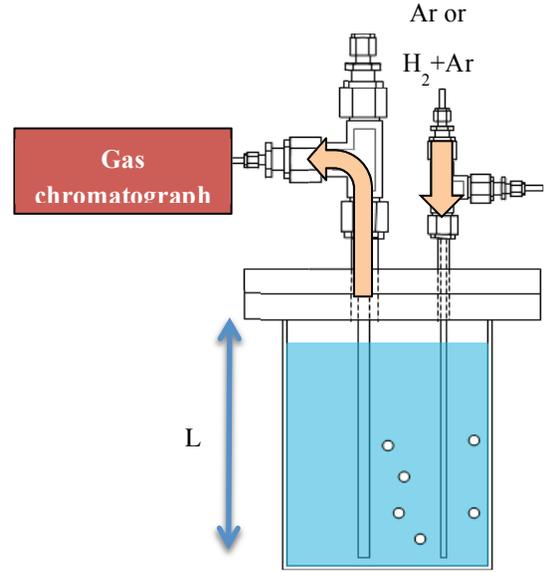


Fig. 1 Schematic figure of LiPb-gas bubble contactor

where  $C_D$  [-] is a drag coefficient, and the buoyancy force is balanced with the viscous drag force on the left-hand side. The  $C_D$  value is correlated in terms of the kinematic viscosity of liquid,  $\nu_L$  [m<sup>2</sup>/s], or the dimensionless  $Re$  number, which is defined as  $Re=ud/\nu_L$ . When  $Re<10$  in the Stokes region,  $C_D$  is equal to  $C_D=24/Re$ , where fluid convection inside gas bubbles or liquid is ignored. When the convection is taken into consideration, the following Hadamard-Rybczynski equation [23] for  $C_D$  holds:

$$C_D = \frac{8(2\mu_L + 3\mu_G)}{Re(\mu_L + \mu_G)} \quad (4).$$

On the contrary when  $Re>10^3$  in the Newton region, the constant relation of  $C_D=0.44$  holds. In the middle  $Re$  region, experimental data are summarized to an empirical correlation such as  $C_D=(0.55+4.8/Re^{0.5})^2$ . The  $C_D$  value is approximated by  $24/Re$  in the present experimental condition of  $D=4.35$ mm. Consequently, the bubble terminal rising velocity is determined from Eqs. (1) or (2) and (3) as a function of  $\rho_L$ ,  $\rho_G$ ,  $\nu_L$ ,  $\sigma$  and  $D$ .

Relation between the total flow rate of gas bubbles blown into liquid,  $Q$  [m<sup>3</sup>/s], and the number of bubbles present in the Li-Pb vessel,  $N$  [-], are estimated by the following equation when the Li-Pb depth is  $L$  [m]:

$$Q = \frac{\pi d^3}{6} N \frac{u}{L} \quad (5).$$

Since the total surface of the spherical gas bubbles,  $S$  [m<sup>2</sup>], is described as  $S=\pi d^2 N$ , the total gas-liquid surface present in the Li-Pb vessel is expressed by the equation:

$$S = \frac{6LQ}{ud} \quad (6).$$

The expression of Eq. (6) means that the total surface area of bubbles in the Li-Pb vessel,  $S$  [m<sup>2</sup>], is determined by the depth of the liquid Li-Pb vessel,  $L$ , and the total gas flow rate,  $Q$ . If  $Q$  is low,  $S$  becomes a function of the nozzle diameter,  $D$ , straightforwardly. The relating physical properties are viscosity of liquid,  $\mu_L$  [Pas], and the surface tension between liquid and gas,  $\sigma$  [N/m].

## 2.2. H transfer between bubbles and liquid

As seen schematically in Fig. 1, gas bubbles ejected from a nozzle may move upward independently when  $N$  is small. H absorption into Li-Pb or conversely H desorption from it to bubbles proceeds in the vessel independently. In order to constitute a rate equation for H transfer from Li-Pb in vessel to bubbles or vice versa is deduced under the following assumptions:

- (i) Since Li-Pb is well mixed by bubble movement, the H concentration dissolved in the Li-Pb bulk (its volume  $V_{LiPb}$  [m<sup>3</sup>]) is assumed uniform except for a concentration boundary layer formed near interface between Li-Pb and spherical bubbles.
- (ii) Association or decomposition of bubbles ejected in Li-Pb are neglected. The number  $N$  is unchanged during gas absorption/desorption operation.
- (iii) H<sub>2</sub> concentration in bubbles is uniform due to inside gas convection.
- (iv) Variations of the H<sub>2</sub> concentration in the top open space (volume  $V_s$ ) with time are taken into consideration.
- (v) The movement of gas bubbles is taken into consideration in the rate of H<sub>2</sub> transfer from Li-Pb into the open space but direct desorption of H<sub>2</sub> from the Li-Pb surfaces is ignored.

When the effect of flow inside the liquid or gas phase is ignored, the one-dimensional transient diffusion equation for the H isotope concentration,  $c_L$  [mol/m<sup>3</sup>], in the liquid boundary layer is described as follows:

$$\frac{\partial c_L}{\partial t} = \frac{D_L}{r^2} \frac{\partial}{\partial r} \left( r^2 \frac{\partial c_L}{\partial r} \right) \quad \text{for } r > a \text{ region} \quad (7).$$

The initial and boundary conditions for the H absorption operation in the liquid are given by the equations:

$$t = 0 \quad c_L = c_{L,\infty} \quad (8),$$

$$r = a \quad c_L = c_{L,s} \quad (9),$$

$$r = \infty \quad c_L = c_{L,\infty} \quad (10).$$

The solution to Eq. (7) under the initial and boundary conditions of Eqs. (8)-(10) is obtained as follows:

$$\frac{c_{L,s} - c_L}{c_{L,s} - c_{L,\infty}} = 1 - \frac{a(r-a)}{2\sqrt{\pi D_L t}} \int_0^{\tau} \frac{1}{t^{2/3}} \exp\left[-\frac{(r-a)^2}{4D_L t}\right] dt \quad (11).$$

The H mass flux from liquid to gas,  $j_H$  [mol-H/m<sup>2</sup>s], per unit surface area of one bubble is obtained from the H concentration profile of Eq. (11) as follows:

$$j_H = -D_L \left. \frac{\partial c_L}{\partial r} \right|_{r=a} = (c_{L,s} - c_{L,\infty}) \left( \frac{D_L}{a} + \frac{\sqrt{D_L}}{\sqrt{\pi t}} \right) \quad (12).$$

In the case of H desorption from Li-Pb to bubbles, the order in the driving-force term on the right-hand side of Eq. (12) is interchanged. When the effect of convective flow in the liquid or gas phase is taken into consideration, the first term on the right-hand side of Eq. (12) can be replaced by the following equation:

$$j_H = (c_{L,s} - c_{L,\infty}) \left( k_m + \frac{\sqrt{D_L}}{\sqrt{\pi t}} \right) \quad (13)$$

where  $k_m$  [m/s] is a mass-transfer coefficient and is a function of not only temperature but also the terminal

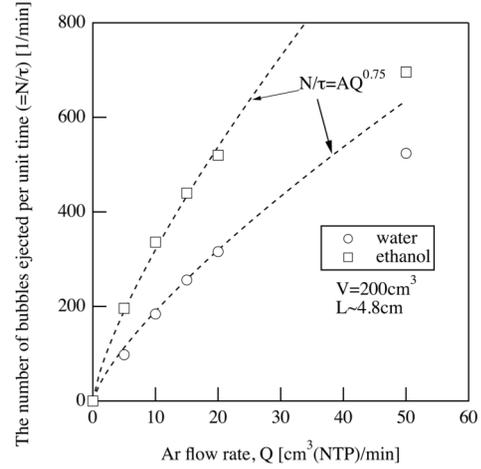


Fig. 2 Preliminary experiment on number density of bubbles,  $N$ , using simulant liquid, where  $\tau$  is a residence time.

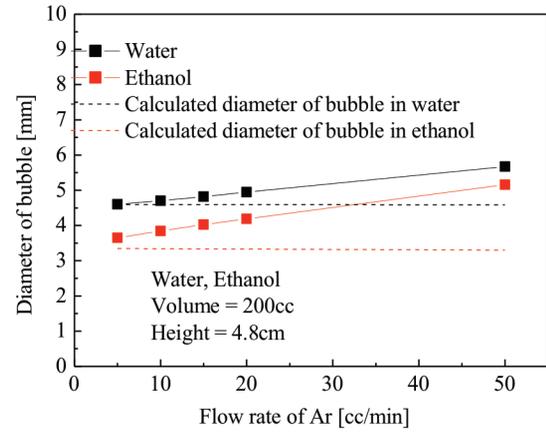


Fig. 3 Comparison between measurement and estimation of bubble diameter

velocity,  $u$ . The second term on the right-hand side becomes small when the contact time is long. Since the total H transfer rate between Li-Pb and bubbles is calculated as  $j_H S$ , the H balance in the Li-Pb bulk layer is described as follows:

$$V_L \frac{dc_{L,\infty}}{dt} = k_m S (c_{L,s} - c_{L,\infty}) \quad (14).$$

It is not simple extension to derive Eq. (14) from Eq. (13). This is because the bulk concentration  $c_{L,\infty}$  is assumed to be constant before the derivation of Eq. (12). The overall H<sub>2</sub> transfer rate between bubbles and Li-Pb is described in terms of the mass-transfer coefficient,  $k_m$ . Its driving force is expressed by the difference in the H concentration between surface and bulk changing with time.

The H<sub>2</sub> balance equation in the top open space is described as follows:

$$\frac{V_s}{RT} \frac{dp_{H_2,s}}{dt} = \frac{Q}{RT} (p_{H_2,bubble} - p_{H_2,s}) \quad (15).$$

The equilibrium relation for H<sub>2</sub> is held between gas in

bubbles and Li-Pb interface:

$$c_{L,S} = K_L \sqrt{p_{\text{H}_2, \text{bubble}}} \quad (16),$$

where  $K_L$  [ $\text{mol}\cdot\text{H}/\text{m}^3\text{Pa}^{0.5}$ ] is the Sieverts' constant for atomic H solution in Li-Pb.

### 3. Experimental

#### 3.1 Observation of bubble diameter

Bubble diameter  $d$  is a key parameter to predict rates of H transfer between bubbles and liquid. However, when the experimental apparatus has been set up in our laboratory, the inside cannot be observed from the outside. Therefore, another experimental apparatus made of glass vessel filled with water or ethanol is set up before the Li-Pb experiment. The reason why water and ethanol are selected here is that the bubble diameter is deeply related with the  $\sigma/\rho_L$  value judging from Eq. (1). Its value calculated from Eq. (1) for the Li-Pb and Ar system is  $d=3.9$  mm and is between water-Ar and ethanol-Ar as seen Fig. 3. The SS-304 tube of  $D=2.175$ mm in inner diameter is inserted in the liquid and a constant flow rate of Ar gas of  $Q$  is supplied through the tube to liquid. The number of gas bubbles,  $N$ , formed is counted by visual observation using a high-speed video-camera. Fig. 2 shows an example of the number of bubbles ejected from a nozzle as a function of  $Q$ . The  $N$  value increases almost linearly under a small  $Q$  condition. With the increase of  $Q$ , the  $N$  value deviates from the linear relation. The average diameter of gas bubbles formed in liquid bulk  $d$  is calculated from  $Q$  and  $N$ . The results are shown in Fig. 3. The estimated  $d$  value is almost coincident with Eq. (1) in case of small  $Q$  condition.

#### 3.2 Make-up of Li-Pb eutectic alloy

At first, specified amounts of plate-type solid Li rods (218.07g) of 99.9 % and Pb plates (27.752 kg) of 99.9 % were put in order separately in a SS-304 vessel of 3.3 L in volume, and the whole was heated up to around 500°C with evacuating the vessel by a turbo-molecular pump. After confirming the melted condition of Li and Pb, the system was slowly cooled down below the expected melting temperature. Variations of temperature during cooling with time are shown in Fig. 4. Heat radiation rate is balanced with solidification heat at the melting temperature at 235°C, where a steady-state temperature condition comes out. Cycles of heating (melting) and cooling (solidifying) were repeated to confirm whole homogeneity of the liquid metal alloy. Temperature is maintained at constant during the H absorption or desorption operation, where the whole Li-Pb layer is in liquid state.

#### 3.3 Experiment of H absorption in or desorption from Li-Pb

An experimental apparatus was set up, which is composed of a gas supply section to feed an Ar or Ar+H<sub>2</sub>

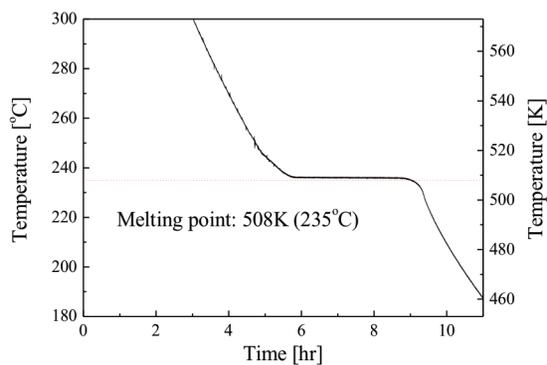


Fig. 4 Cooling curve of Li-Pb including solidifying

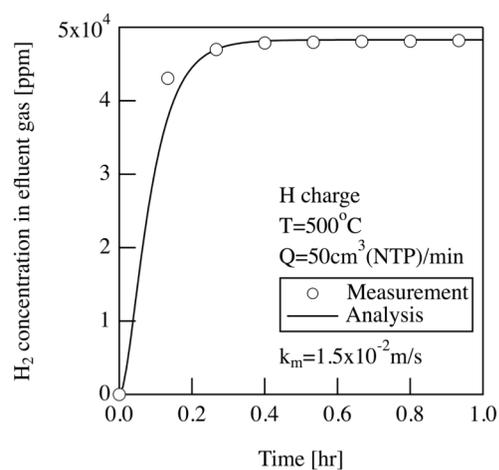


Fig. 5 H<sub>2</sub> concentration history for H absorption experiment

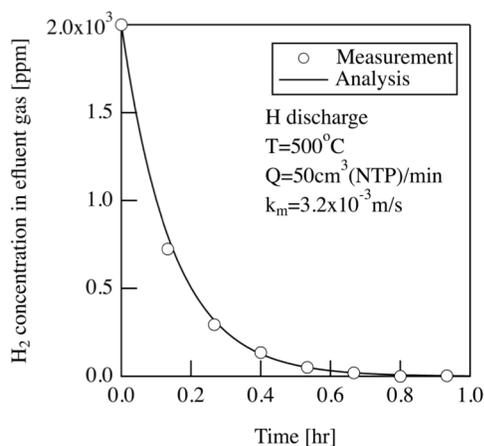


Fig. 6 H<sub>2</sub> concentration history for H desorption experiment

mixture into the liquid Li-Pb vessel, the Li-Pb vessel made of SS-304 with a volume of 3.3 L, electric heaters, temperature controllers and gas chromatography to determine the H<sub>2</sub> concentration in gaseous phase. After confirming the condition where the whole Li-Pb in the vessel is maintained at constant temperature between 400-600°C, an Ar-H<sub>2</sub> mixture is introduced under a constant flow rate, which ranges between 10 – 100

cm<sup>3</sup>(NTP)/min. The H<sub>2</sub> partial pressure in gas introduced into Li-Pb is 5.0 kPa or 50.0 kPa in the H absorption experiment. After supplying the H<sub>2</sub>-Ar gas mixture to the Li-Pb vessel sufficiently, the gas line is changed to pure Ar line. The outlet H<sub>2</sub> concentration is determined in a similar way to the H absorption experiment.

#### 4. Results and Discussion

An example of the experimental results for H charge into Li-Pb is shown in Fig. 5, which conditions are 500°C and  $Q=50\text{cm}^3(\text{NTP})/\text{min}$ . As seen in the figure, close agreement is obtained between experiment and calculation by selecting a proper  $k_m$  value as a unique parameter. In a similar way to the absorption, the H desorption process is also well simulated by the same analysis as seen in Fig. 6. This may be because the rate-determining step is H diffusion in liquid Li-Pb, which is a linear system. Several experiments are repeated at 500°C. The unique  $k_m$  value was determined per each H absorption/desorption run.

The values of the mass-transfer coefficient at 500°C along with other experimental results at 600°C [18,21] are summarized in Fig. 7. When several experiments of absorption and desorption are repeated at 500°C, each run of absorption or desorption was reproduced by a unique value of  $k_m$  in the range from zero to each saturation value. The  $k_m$  value is  $1\text{-}3 \times 10^{-2}$  m/s for the H absorption run and  $1\text{-}4 \times 10^{-4}$  m/s for the desorption one. It is noticed that different  $k_m$  values were obtained between H absorption and desorption runs.

One reason to explain the different  $k_m$  values between H absorption and desorption runs is the effect of surface reaction. The surface reaction process included for the H absorption is decomposition of a H<sub>2</sub> molecule to two H atoms and that for the H desorption is recombination of two H atoms to a H<sub>2</sub> molecule. The decomposition proceeds at relatively higher pressure and recombination does at lower pressure. The two processes are nonlinear. Therefore the  $k_m$  value may become different between absorption and desorption, when the process of nonlinear reaction affects the overall process.

Another reason is the effect of Ar absorption into Li-Pb during the H absorption/desorption experiment [15]. In the present study, only the H<sub>2</sub> concentration is detected in the effluent mixture by means of gas chromatography, which uses Ar carrier and a TCD detector. Therefore the concentration of Ar or the total flow rate is not detected. Ar may be present as a single molecular form in liquid Li-Pb, which is different from atomic H absorption in liquid Li-Pb [4,9,14]. Therefore, the process may proceed independently. The effect of Ar simultaneous absorption in Li-Pb may be less than expected. However, further experiment will be necessary to clarify the different  $k_m$  values between H absorption and desorption. In addition, experiment of different temperature conditions is

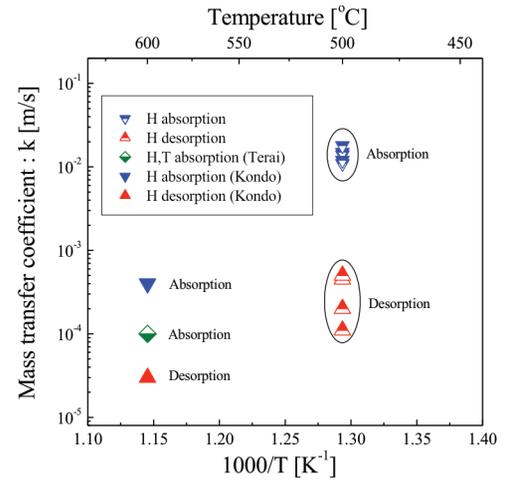


Fig. 7 Correlation of mass-transfer coefficient of H absorption or desorption in/out Li-Pb

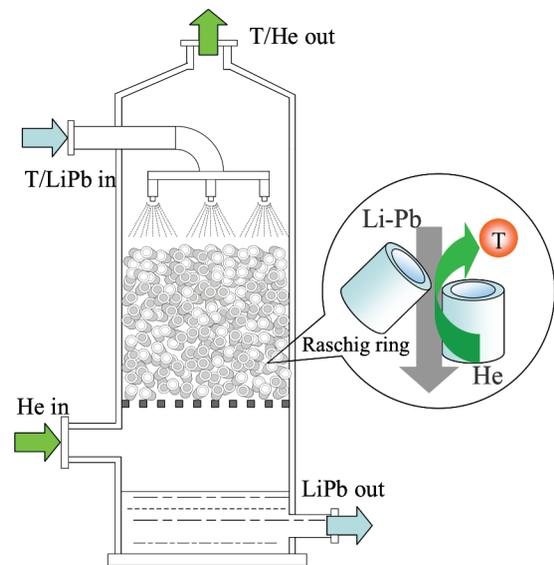


Fig. 8 LiPb/He counter-current flow contactor for T recovery

necessary to compare with previous data [18,21] and to clarify the reasons why the different  $k_m$  values appear in the H absorption/desorption runs.

Now, we continue the experiment for wider different temperatures or flow conditions after some modification of the experimental apparatus. In the near future, these results will be presented. In addition, we initiated a design work for LiPb/inert gas direct contactor for T recovery from a Li-Pb blanket loop as seen in Fig. 8. Fig. 9 shows the McCabe-Thiele diagram for the LiPb/He extraction tower designed here. The flow rate and column height are determined based on diagram evaluation.

#### 5. Conclusions

Recovery rates of H from the liquid Li-Pb blanket by means of direct contact between Ar-H<sub>2</sub> (Ar) and Li-Pb eutectic alloy determined experimentally were fitted well

by analysis, which was deduced based on balances among surface tension, inertia, buoyancy and drag forces. It was found that the bubble diameter and the number of bubbles formed in Li-Pb or simulant fluid are estimated well by the analysis. In addition, H absorption/desorption analysis is given, and the results were correlated well in terms of a single mass-transfer coefficient. The effluent  $\text{H}_2$  concentration from the Li-Pb vessel was well simulated by the diffusion-limited analytical solution. Difference in  $k_m$  values between absorption and desorption was discussed based on effects of reaction rate process on interface between gas bubbles and Li-Pb and Ar absorption. The present study is carried out under the collaboration work of the ITER-BA study between JAEA and Kyushu University.

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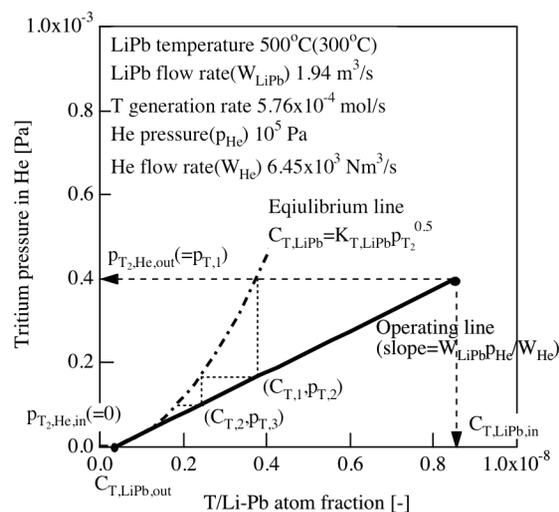


Fig. 9 McCabe-Thiele diagram for LiPb/He counter-current extraction tower

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