# Self-adjustment of Li in Pb–17Li systems

## H. Feuerstein *, L. Hörner, S. Horn

*Forschungszentrum Karlsruhe GmbH, Postfach 3640, D-76021 Karlsruhe, Germany*

**Abstract**

With temperature gradient capsules and thermal convection loops it is shown, that self-adjustment of 15.8 at.% Li in lead–lithium systems is possible. All which is needed is a diffusion type cold trap with a solid phase. Lithium can be deposited there, it will be released if the concentration is reduced below the eutectic in case of lithium consumption. © 1998 Elsevier Science B.V. All rights reserved.

## 1. Introduction

A eutectic mixture of lead and lithium will be used as fusion reactor blanket material. During operation lithium will be consumed by burn-up, reaction with air, and evaporation.

The Pb–Li phase diagram [1] shows several intermetallic compounds and eutectic compositions. At the melting point of the interesting mixture, 234°C, a liquid phase with 15.8 at.% lithium in lead is in equilibrium with a solid phase, consisting of LiPb and Pb. In a non-isothermal Pb–Li system including a solid phase, this means the lowest temperature is below 234°C, the Li concentration in the molten phase will be always 15.8 at.%. A Li excess will deposit as LiPb at the phase boundary. An initially hypo-eutectic concentration will result in the formation of a lead phase. Either lead is deposited, or Li dissolves from the solid phase if there is enough Li.

Freeze valves in liquid metal systems have such thermal gradients from operating temperature to below 234°C.

## 2. Capsules

Freeze valves may be simulated in capsules with static mixtures in a thermal gradient. Many capsules were heated in our laboratory for different investigations [2,3]. Most of them were 420 mm long with an inner diameter of 12 mm, a typical temperature range was from 100°C to 600°C. At the end of the heating times, capsules were quenched with water to preserve the Li distribution.

Fig. 1 shows a capsule, and the lithium distribution for two initially different mixtures.

As expected, 15.8 at.% Li were found all over the range with the liquid phase. In case of an initially hyper-eutectic mixture, LiPb was deposited, in case of an initially hypo-eutectic mixture, a lead phase was formed.

## 3. Thermal convection loops

Thermal convection loops were operated for different studies [4]. All had a freeze valve before the drain tank. Some loops had also a diffusion type cold trap with a solid phase [3], usually of the same dimensions as capsules. The loops were filled with about 8 kg of a mixture with the old eutectic concentration of 17 at.% Li [5]. Consequently, LiPb was deposited in the cold trap. After a long operation time, the circulating mixture contained 15.8 at.% Li. 4.5 g of lithium were deposited, and a long LiPb plug was formed.

A new loop was built with a larger cold trap, containing 12 kg of Pb–Li mixture. One part of the cold trap was a cooler (Fig. 2). About 2 kg of the mixture were solid, and about 6 kg were circulating at temperatures between 450°C and 550°C.

Fig. 3 shows the history of the Li concentration. After 300 h 16 at.% were found. 40 g each LiPb were added three times, and 45 g each Li$_3$Pb twice (**A** (LiPb), **B** and **C** (Li$_3$Pb) in Fig. 3). The technique was described before
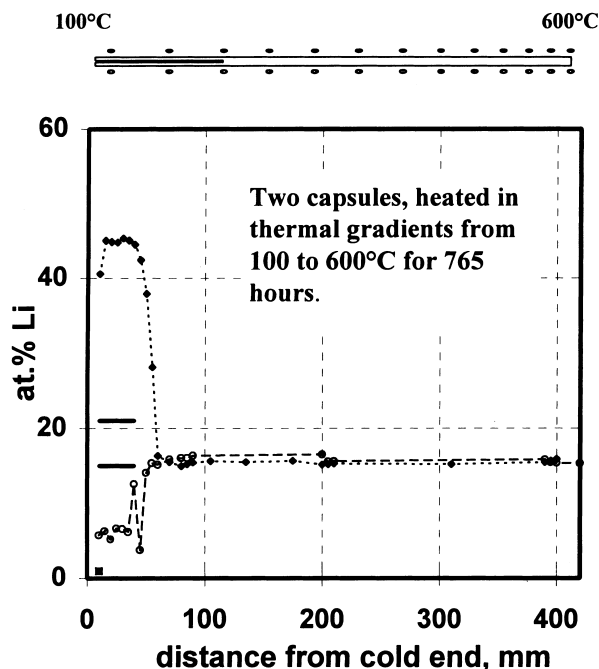
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* Corresponding author.

Fig. 1. Li distribution in two capsules, initial concentrations 15 and 21 at.% Li [2].

[6]. Altogether, 12.2 g lithium were added. Without deposition, 20 at.% Li were expected. The highest value found was 18.1 at.%, with a fast decrease to 17.5 at.%, followed by a slower deposition process, controlled by diffusion of Li in the solid phase [1].

To accelerate the Li deposition, the 234°C position in the cold trap was shifted in direction of the circulating mixture by increasing the cooling rate (**D, E, F** and **G** in Fig. 3). Lithium was deposited at different positions, 376 g or 47 cm$^3$ of LiPb were formed.

For a controlled removal of lithium, 150–170 g of lead in a crucible were submerged three times for 3–18 h (**H, I** and **K** in Fig. 3). Lithium diffused into the lead and was removed with the crucible. Complete equilibration with the circulation mixture required more than 15 h. Up to 1 g of Li could be removed with one crucible, or 1.6% of the circulating amount. This may be comparable to a blanket, in which the loss of lithium will be small too.

Because of the equilibrium concentration of 15.8 at.% Li at the phase boundary, LiPb should redissolve to adjust this concentration in the total molten mixture. Again, the solid state diffusion process is slow. An attempt was made to accelerate this by increasing the tem-

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[1] It is amazing that the deposition of Bi is much faster than those of Li [3].



steel 1.4301
12 kg Pb-Li mixture
2 kg solid in cold trap

$T_{max, loop}$ : **550°C**
$T_{min, loop}$ : **450°C**
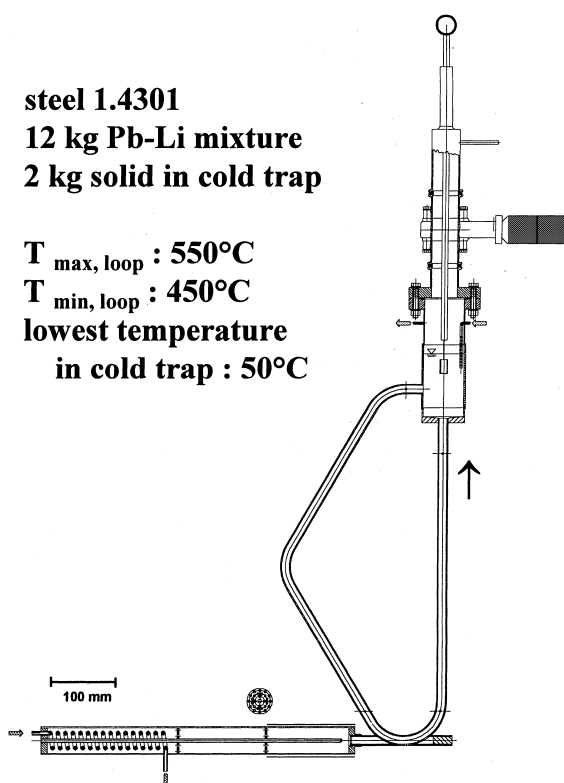lowest temperature
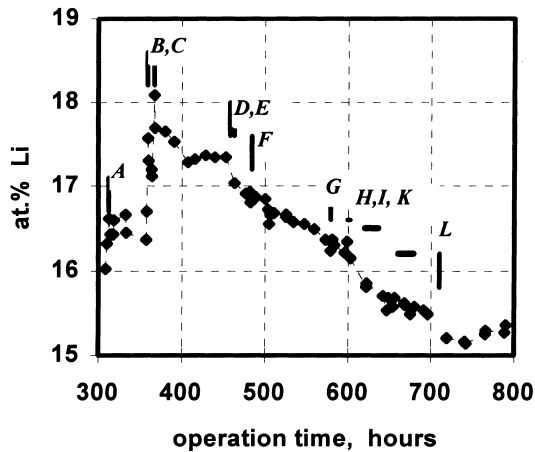in cold trap : **50°C**

Fig. 2. Thermal convection loop.

Fig. 3. History of Li concentration in a thermal convection loop. Accuracy of Li analysis ±0.05 at.%.

perature at the phase boundary by 20°C (**L** in Fig. 3). Because the deposited LiPb has a melting point of 482°C, there will be no remelting. The time was still too short to obtain equilibrium.

The experiments will be continued to prove the Li release from the cold trap, which obviously needs a longer time. Equilibration in capsules or loops always needed at least 300 h.

## 4. Summary and conclusions

The described effects were also observed in the pumped loop TRITEX [7] with 100 kg circulating Pb–Li mixture. The effects, therefore, offer the possibility for self-adjustment of the lithium concentration. All which is needed is a diffusion type cold trap with a solid phase. Lithium can be deposited there at the beginning of blanket operation. The small loss of Li by burn-up will be automatically compensated by release of Li from the phase boundary.

## References

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