

# Failure of a Lithium-filled Target and Some Implications for Fusion Components

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In preparation for testing a Li-He heat exchanger, unexpected rapid failure of the mild steel Li preheater occurred when Li at  $\sim 400^\circ\text{C}$  flowed into the preheater then at  $\sim 200^\circ\text{C}$ . This happened before the He system was pressurized or heating with electron beams began. We attribute the failure to liquid metal embrittlement. The paper presents an analysis of the preheater plus a discussion of some implications for fusion.

Keywords: lithium, ferritic, embrittlement, cracking

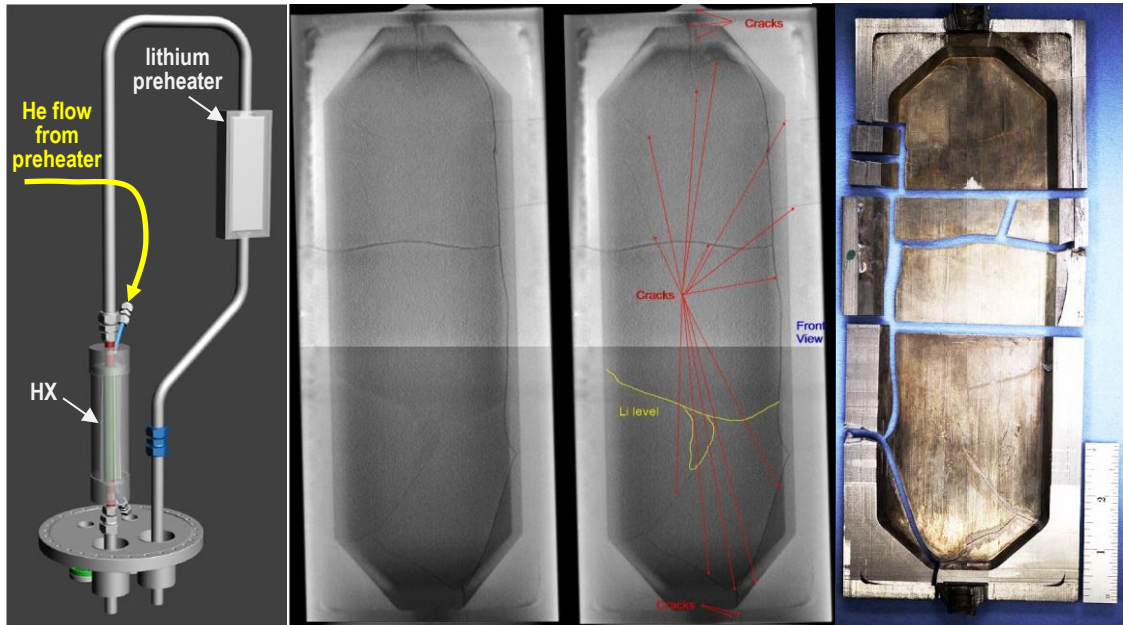


Figure 1. (a) experimental arrangement, (b) neutron radiographs of the body of the preheater from the back (left) and front (right); yellow line shows level of liquid lithium when analyzed; red lines indicate cracks, and (c) photograph of the inside of the failed unit. The separation into pieces resulted from sectioning for evaluation.

## 1. Introduction

Fusion reactors require tritium as fuel produced from lithium (Li) in blankets with liquid breeders, e.g., Li, Li-Pb or molten salts containing Li, or solid breeders, e.g.,  $\text{Li}_2\text{SiO}_4$  or  $\text{Li}_2\text{TiO}_4$ . Also, development of ways to provide liquid lithium surfaces facing the plasma is an active area of research, and Li at the edges of plasmas in NSTX and TFTR has improved performance. Fusion applications with Li are the topic of a workshop series, e.g., 3<sup>rd</sup> International Symposium on Lithium Applications in Fusion (ISLA), Frascati, October 2013[1], a book[2] and many articles, such as Ref. 3-7.

Handling and containment of liquid Li are necessary in both areas of research. This paper arose from our

consideration of possible hazards for those performing research with Li after we experienced an unanticipated rapid failure of a ferritic part exposed to liquid Li.

The major point in this paper is that unexpected rapid brittle failure occurred in ferritic steel exposed to liquid lithium. We believe the presence of thermal stress associated with the difference in temperatures between the incoming lithium and the 1018 preheater plus any residual stresses from fabrication in combination with adequate wetting by the lithium was sufficient to promote liquid metal embrittlement or LME and rapid failure of the part.

We first discuss the experimental arrangement and then LME. In the balance of the paper, we discuss some of the literature on this phenomenon and recommend more R&D to understand and mitigate the potential for LME by those who seek to systems with flowing lithium or lithium alloys in blankets and PFCs for fusion.

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## 2. Experimental arrangement

Figure 1 shows the experimental arrangement (left) and the Li preheater as well as neutron radiographs of before sectioning for metallographic examination. Ultramet, Inc. made the Li-He heat exchanger (HX) and preheater to preheat separately the helium and lithium streams going into the HX (unpublished work) for testing at Sandia using EB1200's dual electron beams[5].

The Li preheater had electrical trace to heat it above the melting point of Li and to the temperature of the liquid lithium in the connected Li loop. As the experiment neared final preparation, and before either e-beam operated, failure of the Li preheater occurred shortly after Li at about 400°C in the loop was introduced (in error) into the preheater, then at only about 200°C. Failure of the preheater occurred only a few seconds after the Li contacted the previously empty preheater.

Two 1018 mild steel plates and two pieces of nearly pure iron tubing were the materials for the preheater. Coolant channels were machined into the thicker bottom plate and an iron tube welded into each end contained the entrance and exit flows. The thinner top (lid) had a closure weld around its perimeter. Metallographic and elemental analyses at Sandia confirmed the materials were as specified, i.e., 0.15-0.20 C, 0.6-0.9 Mn, 0.15-0.30 Si and bal Fe (wt%) is consistent with 1018 steel, and the microstructure from a second unused Li preheater had the expected ferrite and pearlite regions, respectively the dark and light regions in Fig. 2. The hardness values of the pre-heater body, which averaged 86 HRB for the case and 91 HRB for the lid, indicate the material was cold rolled or formed.



Figure 3. Optical microscope images of polished and etched cross section of the lid of the second Li pre-heater.

## 3. Observations

### 3.1 Cracks

Fractography is the evaluation of features on fracture surfaces. In typical (ductile) tensile failures the fracture follows zones within the grains where the material has started to pull apart on a fine scale. The array of microvoids that coalesce appear as a “dimpled” surface and indicate local ductile movement of material. A crack that grows incrementally in each fatigue cycle, typically has striations that mark the crack growth on each step.

Typically with failures in 1018 steel the fracture surface are more ductile in appearance and more deformation is usually observed than in the case of the failed pre-heater where little ductility or deformation was found. In general, the fracture surfaces not obscured by Li reaction

products exhibited intergranular failure, i.e., the fracture path followed grain boundaries and seldom propagated into the interior of grains.

Figure 3a (top) shows a representative fracture surface. In the failure here, the very flat fracture surface that showed no shear lips indicates a large reduction in ductility in the 1018 steel, and microscopic examination revealed an intergranular fracture surface. Figure 3b

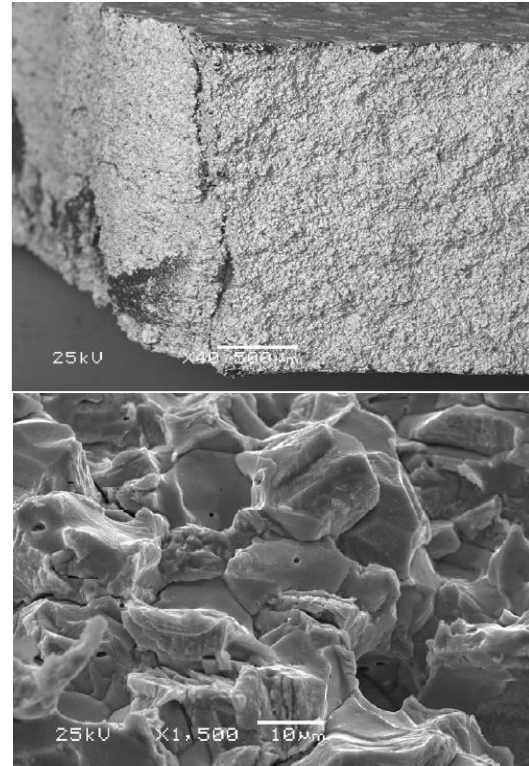


Figure 2. Representative fracture surface on lid.

(bottom) shows the surface at higher magnification and the intergranular nature of the fracture surface.

In brittle failure of the type observed for the lithium preheater, the crack growth proceeds primarily along the surfaces of grain boundaries, and a pattern of chevron-like features indicates the direction of the crack growth. By observing these features, one can backtrack to the crack origin. Our evaluation indicated a possible flaw or precrack in or near the weld (damage in this area complicated the evaluation), and weld penetration at that location was poor. We do not go into those details here but instead focus on other issues.

### 3.2 Liquid Metal Embrittlement

Many approaches in evaluating the performance limits for materials under stress can utilize the bulk mechanical properties of the materials and how these change with the environment, such as hardening under neutron irradiation. Understanding LME is challenging because the understanding depends in part on environmental features such as the wetting of the fracturing surface at the crack tip and the chemistry that can promote this as well as the stress state in this location. Basic considerations are (1) the stresses for crack propagation with LME can be significantly below the yield for the material and (2) the chemistry of wetting and the reduction in the ductility needed to propagate the crack

can enable cracks to grow very rapidly. To the degree that minor constituents on the grain boundaries or in the liquid metal are important, the situation becomes more complicated. Also, in many configurations, once a crack begins to grow the stress intensity at the crack tip may increase because of the redistribution of that portion of the load no longer carried by the failed material.

Within their grains, metals have various kinds of crystal lattices along with defects (vacancies, interstitial atoms, dislocations and voids) that disturb this structure, but the metallic bonding that holds an atom to its neighbors is strong. Grain boundaries are thin but less coherent zones where the bounding surface of one grain of coherently arranged atoms abuts another with differing orientation of the atom planes. The coherence of the grain boundaries is strong enough in ductile materials that fracture proceeds through the grains rather than along the boundaries. LME weakens the cohesion of the grain boundaries.[6]

Later we discuss available literature, primarily because we feel a researcher interested in lithium applications for fusion but not an expert in LME could compile information that, while technically accurate within the caveats given, could easily be confusing. Before proceeding, let us recap state our perspective before and after our experience with the failure of the preheater.

### 3. Conclusions

#### 3.1 Before and After Perspectives

Our experimental plan called for the lithium to be at the temperature of the preheater when it was introduced into the preheater to minimize the thermal stresses. Residual stresses might have been present in the preheater, but the system was low pressure with the lithium at ~18-20 psi above the vacuum chamber. From our experience with the lithium loop at Sandia, we recognized the need for adequate trace heating in the lines to avoid cold spots and plugging. We also knew that others in fusion working with lithium found plugging of even fairly large pipes due to high surface tension, for example, R&D on a stainless steel tube for injecting molten lithium into the vacuum chamber for a fusion experiment CDX-U.[7,8] The team had suggested this as a mechanism for vacuum sealing a system.

We anticipate component failures in our experiments and even test targets to failure. In this case our mindset was that failure in some portion of the lithium system inside the EB1200 chamber, most likely as we heated the system, would produce a dribble of lithium that would freeze on colder components, e.g., the chamber floor or wall, or the leak would plug itself but cause a rise in the pressure in the chamber that would shut down the system or at least alert the operators. What happened was a surprise.

The preheater never broke apart but, lubricated by the wetting of the fracture surfaces, streams of lithium spewed from thin cracks in streams that not only reached the vessel wall but also went up the beam line of one of the e-beams. Contact there with a ceramic insulator led to a lithium fire.

We had moved hot lithium from transfer casks, charged our lithium loop, LIMITS[9], and shot a stream of lithium in vacuum through a strong magnetic field. For the experiment with the Li-He heat exchanger, we considered many possible failure modes but did not regard either the rapid failure of the preheater due to LME nor the formation of strong lithium jets from very fine cracks and the combination of these as even remotely likely possibilities. However, history now demonstrates these were indeed possible and with an impact with high consequence. Although no one was injured in this event, there was the potential for serious injury from the energetic deflagration triggered by the preheater failure.

#### 3.2 Literature on Lithium Containment

Initially we had expected (a) stresses in the lithium preheater to be low and (b) mild steels to provide appropriate containment. Many published studies have focused on corrosion or dissolution of mild or ferritic steels by flowing liquid Li, and the literature on LME of steels has many examples, but collectively the conclusions and recommendations are by no means uniform and clear. After the excerpts below, we will comment on how we now, in retrospect after our preheater failure, approach the literature.

- (*paraphrase*) only limited corrosion, (one case) recommendation that appropriate ferrous alloys can contain liquid Li for a long service life, advised caution as the exact conditions and mechanisms of LME are not fully understood[10]
- (early paper) “In general, only low-carbon steel...is desirable materials of construction for molten lithium”[11]
- (more recent) “Ferritic steels... Lithium: the compatibility will be good enough. The susceptibility to LME will be very high”[12]
- LME of ferritic steels and of pure Fe (Armco iron) can be extreme, large reductions in ductility can occur[13]
- (*paraphrase from studies of long term exposure of mild steels to liquid Li*), a spheroidization heat treatment that converts pearlite to carbides will reduce corrosion through the dissolution of pearlite [14]

As we have continued our investigation of the literature, we noticed a trend. The more interesting papers for us were not those mentioned above and noted in the search engines we consulted, but rather those cited as references or those in the second level of references.

#### 3.3 How should we proceed?

We have reported here an unexpected failure of a mild steel component exposed to lithium that occurred at relatively low temperature and stress due to liquid metal embrittlement or LME. Our further purposes with this paper are a) to recommend study of LME for lithium and lithium-lead systems in the R&D on materials for fusion plasma facing components and blankets, and b) increase awareness of issues related to safe handling of lithium.

Fusion research has several ongoing activities in which safe handling of lithium or lithium alloys is relevant. In the US, R&D on flowing lithium systems with free

surfaces (open to vacuum) includes two systems being developed for installation in the Chinese tokamak EAST by Zakharov[15] and by Ruzik et al.[16]. Researchers at the Princeton Plasma Physics Laboratory (PPPL) have injected liquid lithium into the CDX-U plasma confinement experiment through a long tube[8] and future plans include stirring liquid Li in trays in the Lithium Tokamak Experiment (LTX) using an electron beam.[17]. The NSTX-Upgrade Team is investigating options for a follow-on experiment for the liquid lithium divertor used in NSTX[18,19] in which the surface bearing the liquid lithium could be replenished from a reservoir. Jaworski and co-workers at PPPL are investigating flowing lithium with a test stand.[20,21]

Fusion blankets with flowing lead-lithium is another area of interest and UCLA has a Li-Pb loop.[22]. Although we do not expect the lithium alloy to be as chemically active as pure Li, LME may still be a concern and should be investigated as an issue of potential high consequence. We include here a few references on LME in experiments on martensitic steels exposed to lead alloys.[23-25]

We previously have noted our concerns in approaching the literature. We have also initiated an informal exchange of information and launched this in a special session in ISLA2013. The conference report[1] includes a summary of this special session.

In our exchange of information in the US, one issue Sandia and PPPL staff have discussed informally is what safety requirements are necessary in a lab to operate a lithium loop that circulates for days. For example, how might one conduct unmanned operation with inherently safe shutdown in case of an accident? A website through which we exchange information and a future reporting of progress in the 4<sup>th</sup> International Symposium on Lithium Applications are planned.

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