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### Post-test examination of a Li-Ta heat pipe exposed to H plasma in Magnum PSI

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The authors exposed a radiatively cooled, Li-filled tantalum (Ta) heat pipe (HP) to a H plasma in Magnum PSI continuously for ~2 hours. We kept the overall heat load on the inclined HP constant and varied the tilt to give peak heat fluxes of ~7.5-13 MW/m<sup>2</sup>. The peak temperature reached ~1250° C. This paper describes the post-test analysis and discusses Li HPs with materials other than Ta for fusion. A companion paper describes the experiment.

Keywords: Fusion energy, Plasma facing components, Heat pipes, lithium, refractory metals.

#### 1. Introduction

A heat pipe (HP) transports heat from its evaporator and rejects the heat at its condenser. The range of liquids inside and materials for the jackets enable a wide range of operating temperatures and uses. Lithium (Li) provides the highest performance at operating temperatures in the range of 1000-1400° C and requires refractory metals.

The work reported here is a collaboration by Sandia National Laboratories, Aavid-Thermacore Inc. (ATI), the Culham Centre for Fusion Energy (CCFE) and the Dutch Institute for Fundamental Energy Research (DIFFER). Sandia purchased an existing Tantalum (Ta) HP from ATI, who retrofitted it with a high performance porous sintered niobium (Nb) wick. Figure 1 shows the HP without the end cap. Table 1 gives details.

Component	Material	Dimensions
Jacket,	Та	OD ID L (mm) 17 15.6 197
pebble-milled surface		0.7 emissivity
Sintered wick		ID
	Nb particles	0.088-0.25 mm 55% porosity
Screen	Mo mesh	0.15mm grid
End caps Fill tube	C-103 Nb alloy Nb (1%Zr)	6mm thick
Working fluid	Li , Li ,	~5g

Table 1. Ta/Li heat pipe construction.

We exposed this HP to fusion relevant conditions (H plasma in a magnetic field) in DIFFER's linear plasma source Magnum PSI. [1,2] We kept the overall heat load on the inclined HP constant for ~2 hours and varied the tilt to get peak heat fluxes >10 MWm<sup>2</sup>. At the 30° tilt, the HP operated at ~1250° C peak in the heated zone and ~1000° C in the condenser's highly radiating zone. Our other papers give details of the test with photos and discussions of HPs and radiative coupling [3] and the near infra-red thermography (NIRT) measurements [4].



before end cap weld.

The plasma was steady

Table 2. Leak Timetable

throughout the test, but the power profiles were narrower than originally anticipated. [3] The NIRT indicated a hot spot close to the location where the axis of the H column intercepted the HP. later leaked Li. We stopped after a major Li leak occurred near that spot. Table 2 gives the time history.

0 s	plasma color changes	175	bright spots (reflections)
29	very strong radiation		indicate bulging (more Li)
49	oscillations on video	216	flow spreads down HP

			0 1 ( )
29	very strong radiation		indicate bulging (more Li)
49	oscillations on video	216	flow spreads down HP
109	nodule visible	240	flow on video cam stops
130	new shape by nodule	???	light grey coating forms
163	Li flows from primary leak over small area	254	tilt HP to horizontal

A 6-mm-diameter nodule (~0.06 g of Li) formed and a coating covered half of the area wetted by the beam. It was discernible in the video monitor while the HP was radiating strongly (white hot) with the plasma present. Li then formed wing-like structures below the nodule, Li blobs trickled away from the primary leak site, thickened and then spread to form the thin dark grey coating that covered most of the prior plasma footprint on the HP.

After the dark grey coating had formed, the wellresolved live image of the 2<sup>nd</sup> IR camera showed Li flow from what appeared to be a transverse crack as indicated by the curved and white arrows in Fig. 2. This light grey coating wetted an area ~30 mm<sup>2</sup> and thickened with a slight bulge. The description here is a recollection of the live image. The IR camera had stopped recording before this time, and the flow was not visible in the video cam.

<sup>&</sup>lt;sup>1</sup>Sandia National Laboratories is a multi-mission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525. This work was also part-funded by the RCUK Energy Programme under grant EP/I501045. DIFFER is part of the Netherlands Organisation for Scientific Research (NWO).



Figure 2. Li-Ta HP just after removal from Magnum PSI.

Other details became apparent in the post-test observations. Fig. 2 was taken on a workbench while the HP was briefly exposed to air. The nodule and light grey coating were shiny at first. Then, slowly oxidation began to reveal the grain boundaries, which are apparent in the images. The smaller coating also flowed over the dark grey coating and, we presume, was formed by the same process as the light grey coating since both overlay the dark grey coating and have closed perimeters. Adjacent to the nodule are the dark fans of a leaf-like structure.

After the post-test inspection, the HP was sealed in a bag with an argon atmosphere and later shipped to Sandia.

#### 2. Post-Test Analysis

The primary goal in the post-test analysis at Sandia was to determine the metallurgical condition of the Ta jacket at the primary (pinhole) leak site and to verify the apparent crack. Potential failure mechanisms of interest were (1) prior damage or metallurgical anomalies, (2) liquid metal embrittlement, particularly for the suspected transverse crack, and (3) hydrogen embrittlement.

#### 2.1 Preparation

At Sandia, the bagged HP was placed in an argon glove box and sectioned. Scrapings from the Li coatings were bagged under argon for high resolution x-ray diffraction analysis (XRD). Figure 3 shows a cut portion, that had  $\sim 1$  g of Li before cleaning, plus SEM and other photos from the post-test examination.

For metallographic observation, Li was cleaned from the cut pieces with water inside a fume hood. This is a strong exothermic reaction (73.2 kJ/g). Each cut piece was held in a cage of perforated SS to contain any ejected debris and placed in a "pickle jar" that held ~3 liters of water. The 1-gallon plastic jar is an approved Haz-Waste container. It sat in a catch basin to collect any splashed cleaning solution. Cleaning of the piece in Figure 3a released ~2 liters of H<sub>2</sub> (1 atm) and raised the temperature of the cleaning solution by ~13° C (estimated values). The used cleaning solution had a measured pH of 14 and required Haz-Waste disposal.

#### 2.2 Metallographic Analysis

Figure 3b shows the pinhole failure site of the primary Li leak. Splashed and mounded material overlaid the tube surface. Mounded surfaces located at A and C in Fig. 3b were ~40  $\mu$  above the tube wall. The shoulder (B) of the deep hole is ~250  $\mu$  below the tube prior surface. To the left of the hole is material that had melted and cracked.

In the microstructure of the Ta wall (Fig. 3c), the large grains indicate recrystallization. In some locations a single grain spanned the wall thickness.

Examination in an SEM of the surface under the light grey and dark grey coatings (Fig. 3d), revealed some damage (a few small holes). However, a large crack in the position indicated in Fig. 2 was not found. The surface under the coatings was like the other side of the pipe which was also hot but not but not exposed to plasma.

Figure 4 shows element maps of surfaces at the primary leak site. Oxygen (O) and sodium (Na) were significant. A small blob of ejected carbon (C) or a carbide was found but C was not widespread.



Figure 3. (a) glove box photo, coatings, no nodule; (b) primary Li leak site, 0.2x0.3 mm hole; (c) Microstructure of 0.7-mm-thick Ta wall; (d) Ta tube surface under grey coatings.



Figure 4. SEM photo of pinhole leak site and elemental maps of surface impurities.

#### 2.3 X-Ray Diffraction (XRD) Analysis of Coatings

Scrapings were double-bagged in the argon atmosphere of the glove box and then transferred to the XRD chamber. Fig. 5 shows XRD spectra of the nodule and coatings on the Li-Ta HP.

Strong (200) and (211) lines indicate that the nodule was metallic (amorphous) lithium. The coatings contained crystalline material. The dark grey coating was composed of LiOH and Li3N, as was the black deposit below the nodule. The latter had trace amounts of an unidentified phase. The light grey coating was predominantly LiOH.



2.4 Thermal Stresses and Mechanical Testing

The initial hypothesis was that the Ta jacket had become embrittled and then cracked. We performed stress analyses to look for a stress state that would promote cracking in the Ta jacket if embrittlement had occurred.

We assume the ATI testing to 1400° C annealed the Ta, the symmetry produced no bending stresses, and residual stresses would only arise from unrelaxed through-wall strains during cooling. In Magnum PSI, the mounting and minimal pressure difference caused no significant stress. Heating the top of the HP produced the stress state below:

1) overall biaxial compression in the heated Ta wall,

- 2) thermal and stress gradients through the wall, and
- bending stresses in constrained thermal expansion cause mild compression at the back of the HP and (opposing) tension in its sides.

Although the time at temperature was only 2 hours, the very high primary creep transient at 800° C for Ta [5] could relax the stress state in the hot zone of the HP. With these stresses relaxed, after the plasma heating stops, the previously heated region would shrink, go into tension, and enable the start of the suspected transverse crack.

So, the enabling condition for crack formation is confirmed, but the Ta tube did not crack. The next section further discusses the formation of the light grey coating.

Moreover, a longitudinal sliver of the Ta wall from the section under the grey coatings was bent 180° into a hairpin shape. This piece had no cracking evident in the deformed zone. Other mechanical tests were postponed.

#### 3. Discussion

The image of Li flowing from a crack (Fig. 2) to form the light grey coating was compelling. An explanation that the crack was in the dark grey coating rather than the Ta jacket is consistent with items 1-4 below.

- 1. The hot areas of the heat pipe were radiating, the HP was still operating and rapidly cooling the hot zone, and most of the heat pipe was below 800° C.
- 2. The light grey coating formed after the HP had cooled for at least 3 minutes after the plasma was shut off.
- 3. The post-test analysis did not find additional leak sites in the Ta jacket beyond the primary leak site.
- A coating of Li3N would solidify at 813° C and compress during cooling since its thermal expansion is ~2.5 times that for Ta. [5,6]

We conclude that the coating was brittle and cracked, and that Li flowed through that crack from channel(s) under the dark grey coating to form the light grey coating.

However, the explanation that a mostly Li3N coating solidified and cracked forces a second conclusion. The Li3N must have come from within the HP, as opposed to forming the nitride after the Magnum-PSI chamber was vented. The source of N is not clear. We have only the evidence of Na at the primary leak site, which suggests that some contaminants were present, and the HP's complex history of being stored for 20 years and then refitted with a porous wick that was brazed in place.

#### 4. Future Work

We hope to develop and test one or more new Li HPs with porous wicks. Our strategy is as follows.

- 1. Use commercially available high temperature jacket materials that can provide a long operating life and that ATI can load and seal.
- 2. Develop a simpler porous wick, perhaps with Additive Manufacturing (AM). Explore options for electrical insulation.
- 3. Demonstrate HP operation at 1.5 kW and 2.5 T in the upgraded Magnum-PSI; improve the test diagnostics.
- 4. Use a water-cooled sleeve around the condenser.
- 5. Operate a HP in a high power long pulse tokamak.

#### Heat Pipe Materials

Their high performance along with the temperature range for operation is why Li is the likely working fluid for HPs in PF-Cs. Many DEMO designs specify tungsten (W) as the plasma facing material in the divertor, as does ITER. Li and W are compatible. However, Li heat pipes maximize their heat transport when the evaporator operates >1400° C where pure W would recrystallize.

In our next step, we must balance high performance above 1400° C against the jacket's resistance to recyrstallization and degradation. To avoid a materials R&D project, we are looking at commercial materials, such as grain-stabilized W-Re alloys. Another option is the Nb alloy C103 used in aerospace applications. ATI can apply a silicide coating that hermetically seals the surface and has made a large non-circular C103 HP by AM.

#### HP Configurations

Figure 6 (in Ref. 1) shows a condenser-insleeve concept for a divertor target. The effective heat removal and averaged surface heat load depend on the evaporator area, as indicated in Figure 6.

If we project the evaporator area on to the top surface of the HP, 40% is reasonable for the aerial fraction



Fig. 6 HP with condenser radiating inside sleeve.

after allowances for clearance between the HP heads, etc. So, without including radiation losses from the head, the heat flux where evaporation occurs is  $\sim$ 2.5 times the array's average heat flux.

Thermal stresses are a concern in all high-performance solid-surface PFCs. For the HP arrangement above, the edge-to-center temperature gradient in the top of the HP will be a significant source of thermal stresses.

The side of the HP can also be the collector surface, as in the DIFFER test. Imagine a divertor with an array of fingers (e.g., WEST or ITER), each being the flattened wall of a square-walled loop HP<sup>1</sup>. Square tubes with rounded corners would maximize the contact surface with the plasma, and rounded rather than square corners would reduce the stresses. The estimated aerial evaporator fraction in this configuration is ~65%.

HPs can transport heat (vapor) over long distances and curved paths. A sleeve-array could be far from the heated area, and its cross-sectional shape can change along this path to the accommodate design needs in the application. Also, the condenser should not contact the sleeve. A lower condenser temperature reduces the HP's performance.

#### Heat Pipe Capabilities

The heat loads in our test were modest compared to the capability of Li HPs. ATI performance curves show input power of  $\sim$ 2800 kW at 1400° C for an emissivity of 0.5. In this regime the HP is nearly isothermal and radiates strongly along its entire length. The HP in our DIFFER test received about <sup>1</sup>/<sub>4</sub> this power and radiated strongly only in the center region.

Radiatively coupled HPs (e.g., condenser-in-sleeve) eliminate a joined vacuum boundary with a secondary heat exchanger. This is very attractive for remote maintenance, particularly for divertors and other special components that need interim replacement, e.g., shielding for RF launching structures.

#### 5.3 Concluding comments

We developed the DIFFER experiment because we believe the exploration of HPs for fusion was incomplete. Liquid metal HPs can transport high heat loads very effectively without the complication of direct plasma contact with liquid Li. Many configurations are possible including the modular replaceable unit that may offer advantages in remote maintenance.

HP technology also has its constraints. Liquid metal magneto-hydrodynamics act like a drag force on the return flow of liquid metal from the condenser to the evaporator [3,7].

The challenge of developing fusion includes advancements in fusion nuclear technology. Power exhaust and the tritium breeding cycle are two important areas where much work is needed, particularly where innovations can simplify or improve fusion designs. We believe HPs may provide useful innovations for fusion and merit further study. We plan to continue our investigations as funding permits.

#### 5. References

- [1] G. De Temmerman et al., Fus. Eng. Des. 88 (2013) 483-487
- [2] H.J.N. van Eck et al., Appl. Phys. Lett. 101 (2012) 224107
- [3] G.F. Matthews et al., "Testing of a high temperature radiatively cooled Li/Ta heat pipe in Magnum PSI," this conference
- [4] S.A. Silburn, G.F. Matthews, T.W. Morgan, R.E. Nygren, Proc. 45<sup>th</sup> Euro. Phys. Soc. Conf. on Plasma Physics, Europhysics Conf. Abs. 42A (2018) P5. 1008 http://ocs.ciemat.es/EPS2018PAP/pdf/P5.1008.pdf
- [5] A. Huq, J.W Richardson, E.R. Maxey, D. Chandra, W-M. Chien, J. Alloys & Comp. 436 (2007) 256-60
- [6] P. Hidnert, Bureau Stds J. Res. 2 (1929) 887-896
- [7] G.A. Carlson, M.A. Hoffman, Trans. ASME (Aug. 1972) 282-288

<sup>&</sup>lt;sup>1</sup> Vapor flows in one leg, fluid in the other, each optimized for its function.

Additional information for web input

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