



# Liquid metal embrittlement (LME) susceptibility of the 8–9% Cr martensitic steels F82H-mod., OPTIFER IVb and their simulated welded structures in liquid Pb–17Li

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## Abstract

This paper describes the results of uniaxial tensile tests on two low activation steels F82H-mod. and OPTIFER IVb. The tests were performed under vacuum and in liquid Pb–17Li at temperatures of 250°C and 400°C at a constant displacement rate of 0.1 mm min<sup>-1</sup>, which corresponded to an initial strain rate of 1.1 × 10<sup>-4</sup> s<sup>-1</sup>. Neither F82H-mod. nor OPTIFER IVb exhibited a liquid metal embrittlement (LME) susceptibility in the tempered fully martensitic state. However, tests of the heat affected zone (HAZ) specimens indicated an LME susceptibility for both steels tested at 250°C in Pb–17Li, while increasing the test temperature to 400°C led to the recovery in ductility. The effect of post-weld heat-treatment (PWHT) of the HAZ specimens of 750°C/1 h for F82H-mod. and 730°C/3 h for OPTIFER IVb was sufficient to regain the majority of their original mechanical properties. © 2000 Elsevier Science B.V. All rights reserved.

## 1. Introduction

The development of reduced-activation ferritic and martensitic steels (hereafter, denoted as RAFs), which combine good mechanical properties with reduced activation characteristics, is one of the challenges facing fusion research and development. The production of RAFs has followed a substitutional approach in which molybdenum and niobium (the primary producers of long-lived activity) have been replaced by additions of tungsten and tantalum. The status of RAFs has recently been reviewed, with particular attention given to the mechanical and irradiation properties of F82H-mod. and OPTIFER [1]. However, little or nothing is known about their mechanical properties in the candidate liquid breeder Pb–17Li.

Previously reported work on some conventional 11% and 12% CrMoNb martensitic steels [2,3] and some 8–11% CrWV low activation steels [4] has shown that although a simulated welding (i.e. heat affected zones, HAZs) of the steels were subject to liquid metal

embrittlement (LME), appropriate post-weld heat-treatment (PWHT) greatly reduced the embrittling effect. A similar effect was also observed for TIG welded MANET II following a PWHT [5].

LME manifests itself as a loss of ductility of a metal or alloy when under tension and in contact with a specific liquid metal. In most cases the effect occurs just above the melting point of the embrittling liquid metal, in that when the testing temperature is increased the ductility of the test specimen is regained. Although no one theory of LME fits all the systems, good background reading into the proposed mechanisms of embrittlement is contained within the available reviews [6–8]. In this paper, we describe the preliminary results of tensile tests at 250°C and 400°C on F82H-mod. and OPTIFER IVb steels in Pb–17Li and under vacuum. Comparison with existing data for MANET and alternative RAFs are also made.

## 2. Experimental

### 2.1. Materials

The chemical compositions of the F82H-mod. (produced by Nippon Kokan Company, NKK) and

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Table 1  
Chemical composition of the F82H-mod. and OPTIFER IVb steels

Steel	Fe	Cr	W	Mn	V	Ta	C	N	Si
F82H-mod.	Bal.	7.66	2.00	0.16	0.16	0.02	0.09	0.005	0.11
OPTIFER IVb	Bal.	8.3	1.4	0.34	0.22	0.06	0.12	0.03	

OPTIFER IVb (charge 986635, produced by Saarstahl GmbH) are shown in Table 1.

The tensile test specimens (15 mm gauge length, 3 mm diameter) were fabricated by Andalò Gianni S.r.l. for ENEA Brasimone. A post-fabrication vacuum heat-treatment was given to all the specimens to produce an optimised fully martensitic structure. In the case of F82H-mod. (1040°C/30 min fast cool, 750°C/1 h) whereas the OPTIFER IVb utilised a lower austenisation temperature (950°C/30 min fast cool, 730°C/3 h) [9,10].

To simulate a welding operation some specimens (protected from oxidation by an outer stainless steel container) were heat-treated (1300°C/3 min, water quenched). All subsequent heat-treatments were carried out in a vacuum furnace or in liquid Pb–17Li in an argon filled glove box. The Pb–17Li alloy used in this study had a lithium content of  $0.625 \pm 0.09$  wt% and came from a batch of semi-industrial production, described previously [2].

## 2.2. Apparatus and testing procedure

The apparatus used in this study, which has previously been described in detail [11], basically consisted of a carousel which enabled the sequential testing of up to a maximum of six specimens under either vacuum or liquid Pb–17Li at temperatures up to 400°C. To determine the LME susceptibility of F82H-mod. and OPTIFER IVb, tests were performed on various specimens having three distinct heat treatments:

- |                             |  |
|-----------------------------|--|
| (A) ‘Normal heat treatment’ | F82H-mod. (1040°C/30 min + 750°C/1 h);<br>OPTIFER (950°C/30 min + 730°C/3 h) |
| (B) ‘Heat affected zone’    | (1300°C/3 min, water quenched)   |
| (C) HAZ + PWHT              | (B) + F82H-mod. (750°C/1 h);<br>OPTIFER IVb 730°C/3 h)                       |

These three types of heat treated specimens were then tested under vacuum and liquid Pb–17Li at 250°C and 400°C. As the phenomena of LME requires an intimate contact between the specimen and the liquid metal, all of the specimens tested in Pb–17Li were pre-wetted. The pre-wetting was achieved by heating the specimens in Pb–17Li (500°C/15 h) in an argon-filled glove box,

which enabled optical inspection of the specimens, to ensure complete wetting, prior to insertion in the carousel. As the wetting procedure effectively added another thermal treatment, an equivalent heat-treatment (500°C/15 h, vacuum) was performed for those specimens to be tested under vacuum.

## 3. Results

Generally two tensile specimens of each type of heat-treatment were tested under each specific environmental condition of temperature, liquid metal or vacuum. The results of the F82H-mod. and OPTIFER IVb steels are shown in Figs. 1–4, in which the extent of plastic strain at rupture is plotted against 0.2% yield strength.

It can be clearly seen from the results of the F82H-mod. steel that an embrittling effect of liquid Pb–17Li occurred for the HAZ specimens at 250°C (Fig. 1). Whereas, tests at 400°C show an increased ductility of the HAZ specimens when tested in liquid Pb–17Li compared to tests under vacuum. (Fig. 2).

The observed recovery in ductility on increasing temperature from 250°C to 400°C is commonly observed

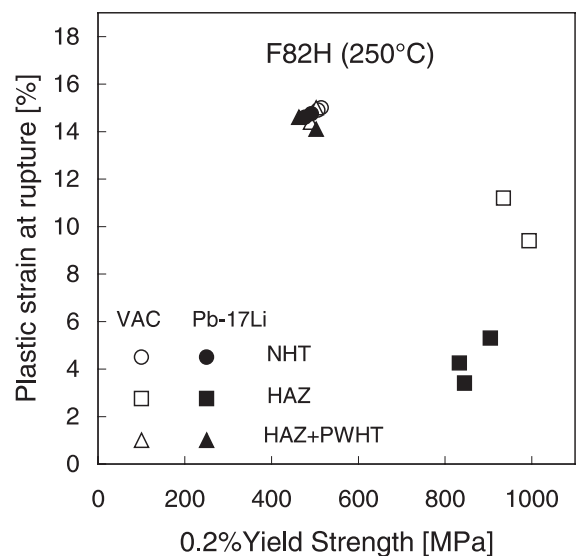


Fig. 1. Plastic strain at rupture plotted against 0.2% yield strength for F82H-mod. specimens tested at 250°C.

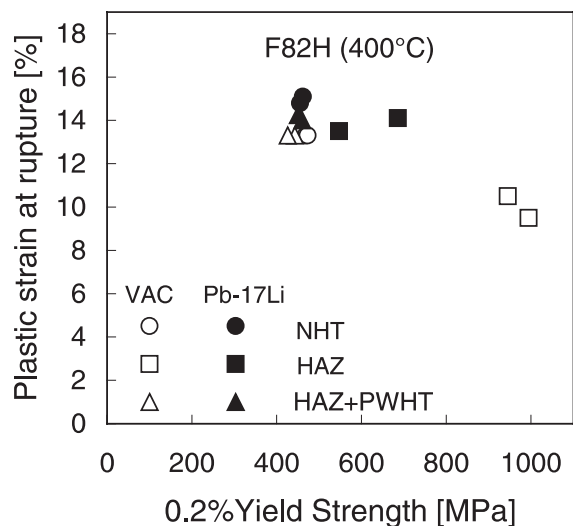


Fig. 2. Plastic strain at rupture plotted against 0.2% yield strength for F82H-mod. specimens tested at 400°C.

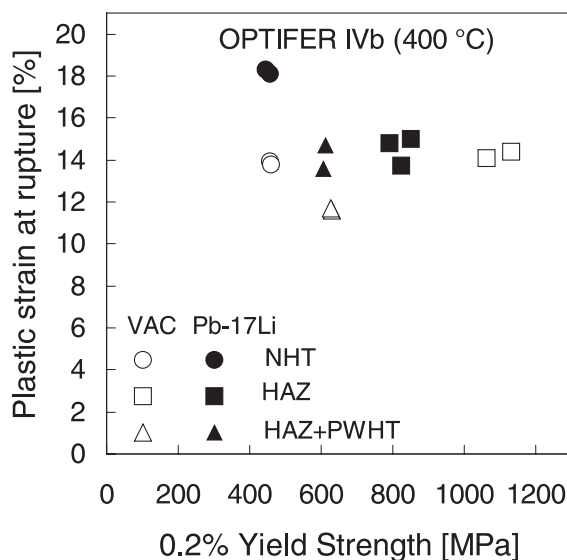


Fig. 4. Plastic strain at rupture plotted against 0.2% yield strength for OPTIFER IVb specimens tested at 400°C.

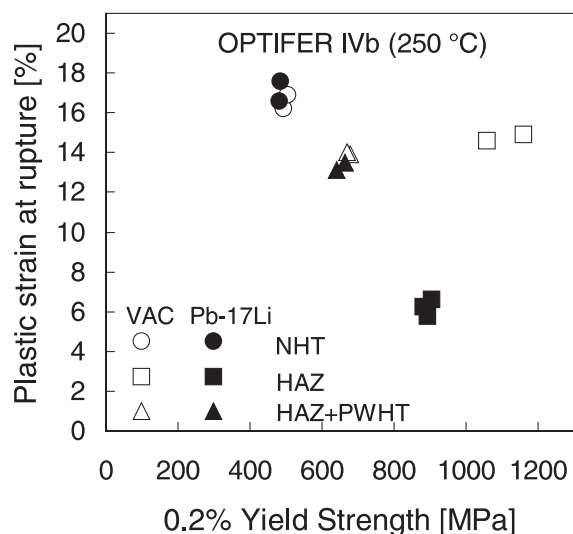


Fig. 3. Plastic strain at rupture plotted against 0.2% yield strength for OPTIFER IVb specimens tested at 250°C.

in nearly all LME systems, in that the embrittling effect usually occurs over a small, well-defined temperature range just above the melting point of the embrittling liquid metal [6–8]. Similar values of 0.2% yield strength and plastic strain at rupture were obtained for both the NHT and HAZ + PWHT specimens tested in vacuum and liquid Pb–17Li. This indicates that the F82H-mod. PWHT of 750°C/1 h was successful in modifying the properties of the HAZ to those exhibited by the NHT specimens. It should be noted that the values obtained

from the HAZ + PWHT specimens generally had a lower value of UTS ( $\approx 20$  MPa), in comparison to the NHT specimens, which exhibited almost identical values for plastic strain at rupture (Table 2).

SEM fractographic analysis of the F82H-mod. HAZ specimens tested under Pb–17Li at 250°C showed a mixed fracture surface of both brittle intergranular and ductile dimpled zones. Whereas, all of the other specimens exhibited only a fully dimpled fracture surface.

The results of the OPTIFER IVb specimens (Figs. 3 and 4) indicate a broadly similar behaviour to F82H-mod. in that the HAZ specimens tested at 250°C in Pb–17Li exhibited an embrittlement (Fig. 3) which disappeared on testing at 400°C (Fig. 4).

However, unlike the results for the F82H-mod. specimens there was a larger difference observed between the OPTIFER IVb NHT and HAZ + PWHT specimens. At both 250°C and 400°C, the NHT specimens exhibited a lower value ( $\approx 140$  MPa) of UTS and an increased ductility (2–4% greater plastic strain at rupture) compared to the values of the HAZ + PWHT specimens (Table 2). This indicated that although the PWHT was sufficient to avoid an LME effect it did not completely recover the original mechanical properties of the steel.

Fractographic analysis of the tested OPTIFER IVb specimens indicated an almost identical behaviour to that shown by the F82H-mod. specimens. The HAZ specimens tested at 250°C under liquid Pb–17Li showed a mixed brittle and ductile fracture. Whereas, the NHT and HAZ + PWHT specimens showed only ductile fracture for tests under liquid Pb–17Li and vacuum.

Table 2

Average values of UTS and plastic strain at rupture for F82H-mod. and OPTIFER IVb in comparison to MANET and four alternative low activation steels

Steel	Heat treatment	UTS (MPa)				Plastic strain at rupture (%)			
		250°C		400°C		250°C		400°C	
		Pb–17Li	Vacuum	Pb–17Li	Vacuum	Pb–17Li	Vacuum	Pb–17Li	Vacuum
F82H-mod.	NHT	546	559	488	502	14.7	15.0	15.0	13.2
	HAZ	899	1012	609	1066	5.7	9.1	13.4	8.2
	HAZ + PWHT	516	532	463	491	14.5	14.7	14.2	13.3
OPTIFER IVb	NHT	540	569	497	500	17.1	16.6	18.2	13.9
	HAZ	994	1275	901	1274	6.2	14.8	14.5	14.2
	HAZ + PWHT	705	717	632	661	13.3	14.0	14.2	11.8
LA7TaLN Ref. [4]	NHT	655	707	575	569	14.1	15.5	16.6	15.8
	HAZ	1190	1320	966	1310	5.8	10.8	13.5	11.2
	HAZ + PWHT	688	700	688	595	14.3	13.6	16.7	15.7
LA12TaLC Ref. [4]	NHT	521	595	488	500	16.3	15.6	17.0	14.0
	HAZ	1030	1140	847	1120	4.5	12.8	15.2	13.7
	HAZ + PWHT	580	600	524	536	13.7	13.9	13.3	12.9
LA12TaLN Ref. [4]	NHT	573	662	498	520	16.2	13.7	17.7	14.3
	HAZ	1150	1320	935	1300	1.7	14.2	12.9	11.4
	HAZ + PWHT	571	589	474	509	14.0	13.3	13.4	13.2
LA13Ta Ref. [4]	NHT	600	627	–	–	16.9	16.2	–	–
	HAZ	1300	1420	1270	1490	1.0	13.7	3.9	13.9
	HAZ + PWHT	771	–	707	–	9.0	–	8.4	–
MANET Ref. [2]	NHT	707	693	604	721	16.1	16.1	19.1	13.6
	HAZ	1210	1370	1390	1350	0.4	9.4	11.8	9.0
	HAZ + PWHT	717	734	653	708	13.7	14.4	17.8	11.2

#### 4. Conclusions

The results of these tests show that neither F82H-mod. nor OPTIFER IVb exhibited a LME susceptibility in the tempered fully martensitic state. However, the HAZ specimens of both steels exhibited an LME when tested in Pb–17Li at 250°C but the ductility of the steels returned when tested at 400°C. The use of a PWHT for F82H-mod. (750°C/1 h) and OPTIFER IVb (730°C/3 h) was very effective in regaining the majority of the properties exhibited by the NHT specimens.

Comparison can also be made of the values of the residual plastic strain at rupture for the HAZ specimens of F82H-mod. and OPTIFER IVb under liquid Pb–17Li at 250°C with previously investigated steels [2,4] shown in Table 2. This shows that both F82H-mod. and OPTIFER IVb retain 5.7% and 6.2% of plastic strain at rupture, respectively, which is similar to LA7TaLN (5.8%), slightly greater than LA12TaLC (4.5%) but much greater than LA12TaLN (1.7%), LA13Ta (1.0%) or MANET (0.4%).

The retention of a significant amount of plastic strain at rupture for the HAZ specimens is an added advantage of F82H-mod. and OPTIFER IVb over MANET as it confers an additional safety margin.

It is worthwhile to note that the temperatures of the PWHT used in this and previous studies [2–5] have generally been the same as the specific tempering temperatures and times advised for each of the steels. Care should be taken to observe these temperatures and times, as two previous studies on MANET [2] and HT-9 [3] indicate that reducing the temperature by 75°C or 150°C greatly reduces the effectiveness of the PWHT.

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