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Mechanical properties of the steel T91 in contact with lead

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ABSTRACT

The ferritic/martensitic steel T91 is among the materials selected for components to be used in the Gen IV LFR (Lead Fast Reactor) concept. Interaction of the steel with the liquid Pb has been studied for a few years. However, issues of main concern are the corrosion of T91 in Pb and its sensitivity to Liquid Metal Embrittlement, LME. The last is a phenomena usually observed at low temperature (close to the melting point of the liquid metal), over yielding point and when there is perfect wetting of the steel from the Pb. In particular, the complete absence of an intermediate oxide layer at the interface T91/liquid metal (result of accidental conditions) promotes the LME. In literature, LME is a phenomena observed mainly for the couple of materials T91/PbBi. However, preliminary studies of the interaction of T91 with Pb, under loading conditions did not show that LME could represent a problem.

The experimental cell CALLISTO was designed and manufactured in the aim of carrying out mechanical testing of materials immersed in the liquid metal. Several tensile tests were carried out in CALLISTO, with Pb. Experimental variables considered were the surface state, temperature and strain rate. No LME was observed in most of the cases. However, when specimens were notched and the wetting was induced, tests revealed the typical features of the embrittlement induced by liquid metal (LM). Results are discussed in terms of effect of the environment on the mechanical properties of the steel T91 and the brittle features observed in the fracture surface.

1 INTRODUCTION

Ferritic-martensitic steel T91 is one of the candidate materials for some of the main components of Lead-cooled Fast Reactor (LFR) and also for Accelerator Driven System (ADS). The steel would be in direct contact with the liquid lead (used as a coolant or spallation target) at different temperatures in both of these systems. Therefore, it is necessary to study the behaviour of the steel at some critical conditions (contact with liquid lead, high temperature, mechanical loading). The main aim of this work was evaluation of susceptibility of T91 to the Liquid Metal Embrittlement (LME) in liquid lead.

The oxide layer forming on the surface of the steel prevents from the direct contact of liquid lead and structural material [1]. Therefore, in order to evaluate the sensitivity to LME, it is necessary to remove the layer before the test. The presence of an oxide layer on the surface of the steel implies that the liquid metal cannot “wet” the metal. The wetting is defined by the surface tension of the liquid on the solid surface. In the case of Pb/T91, the wetting angle is high for surfaces of steel with oxide layers and low for pure unoxidised surface (the wettability is good in the second case). Two possible methods for removal of the film were proposed [2]:

- Remove oxide with the ion-beam in an ultra-high vacuum environment
- Remove oxide with a flux

For this work, the use of a flux was selected.

The SSRT (Slow Strain Rate Test) of the cylindrical notched tensile specimens then followed. The results of these tests were then used to consider whether and eventually at what conditions is the T91 steel susceptible to the LME in liquid lead. The cross-sections of few specimens were also made and analyzed with the wavelength-dispersive X-ray spectroscopy (WDX) to discover, whether the earlier removed oxide layer formed again during the tensile test or not.

2 EXPERIMENTAL

2.1 Material

The material used for all tests was the ferritic-martensitic steel T91 (Grade 91 Class 2 / S50460) according to the standard ASTM A387-Ed99, the chemical composition is in Table 1. The material had the following heat treatment: normalization at 1050°C for 15 minutes, cooling in water to the room temperature, annealing at 770°C for 45 minutes, slow cooling in air. Typical microstructure received by this heat treatment consists of laths of martensite and original austenitic grains with the average size of 20μm - according to electron backscatter diffraction (EBSD).

Table 1: Chemical composition of T91 steel

Element	Fe	C	N	P	S	Cr	Ni	Mo	Mn
Wt. %	Bal.	0,1	0,04	0,02	0,0004	8,9	0,1	0,9	0,4
Element	Si	V	Cu	Nb	Al	W	Ti	Sn	As
Wt. %	0,2	0,2	0,06	0,06	0,01	0,01	0,003	0,004	0,008

2.2 Wetting by flux

Small plates of T91 were used for the test of fluxes. Several attempts were made to select the proper procedures and the final chemical composition suitable to the couple Pb/T91. Eventually, the composition selected (Tab. 2) gave the results requested and was used for all the tensile specimens.

Table 2: Chemical composition of flux FP4201

Compound	$SnCl_2$	NH_4Cl	HCl	NaF	$ZnCl_2$	H_2O
Wt.%	3,7	13,2	4,2	0,8	65,5	12,6

2.3 Tensile tests

The tensile specimens were machined in the L-direction (Fig. 1) according to the drawing and notches were machined in the centre of these specimens.

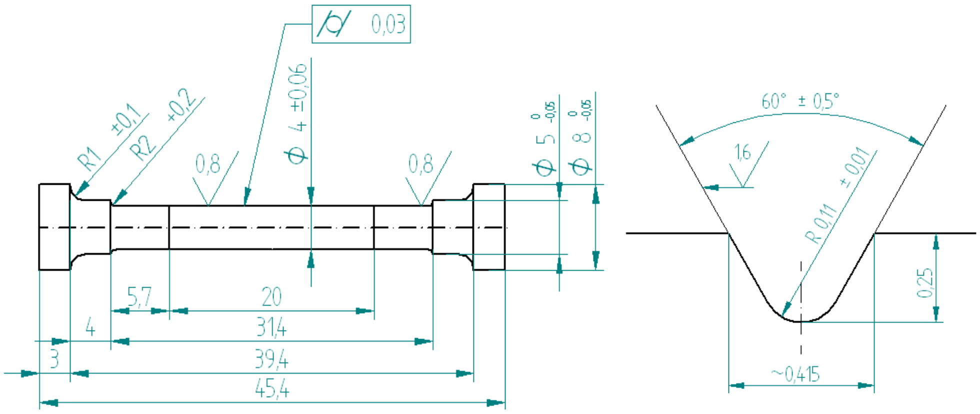

Figure 1: Drawing of used specimen and notches machined in the centre of each specimen

The SSRT (Slow Strain Rate Test) were performed with all the specimens in the cell CALLISTO, which was specially designed for tests in heavy liquid metals. The cell was assembled on a classical hydraulic loading machine, with maximum load 50kN. The tests were carried out in two different environments (air and liquid lead) at three different temperatures (350°C, 400°C and 450°C) and with two different displacement rates (72µm/h and 7200µm/h). The overview of the test parameters is presented in Table 3.

Table 3: Tensile test conditions

Specimen	B92	B87	B88	B85	B89	B33	B34	B94	B91	B93
Environment	Air	Lead	Lead	Air	Lead	Lead	Lead	Air	Lead	Lead
Displ. rate (µm/h.)	7200	7200	72	7200	7200	72	72	72	72	7200
Temp. (°C)	350			400				450		

Prior to the test, the surface of the specimens tested in liquid lead was treated with the flux FP4201 and covered with a thin layer of solid lead to ensure proper wetting especially in the area of the notch.

During loading, a mixture of Ar-6%H_2 gas was bubbled through the liquid lead to prevent the recreation of oxide film on the surface of the specimens during the test by reducing the oxygen content in the liquid metal. After test in Pb, specimens were cleaned from the lead residues in the mix of CH_3CH_2OH, H_2O_2 and CH_3COOH in 1:1:1 ratio. Fracture surfaces of all specimens were observed with a scanning electron microscope (SEM). Cross-sections of two unclean specimens was analysed with WDX line scan.

3 RESULTS

3.1 Test of fluxes

The best result from the first series of the tests was observed after the usage of the flux named ActivaTec 1000. However, even though the wetting angle was for this flux the lowest, the lead stayed in the form of the droplet at the surface of the steel which meant that the oxide layer was still present. Therefore another test was made with the flux FP4201. The result with this flux was much better, because the lead spread on the whole surface of the specimen.

After the solidification of lead the specimen was cut and the cross section was observed at the SEM and analysed with the line scan analysis (Fig. 2). The analyses did not detect any oxygen enrichment at the interface steel/Pb and this confirmed the effectiveness of the flux to induce wetting.

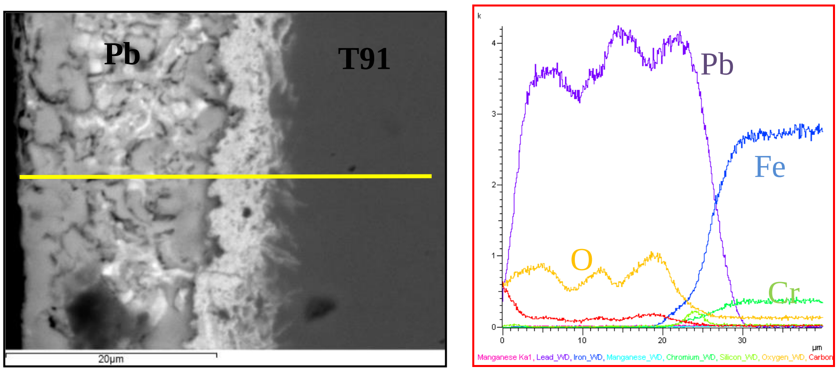

Figure 2: Analysis of cross section (T91/lead) after FP4201 was used

3.2 Tensile tests

The values of time, temperature, load and displacement of the machine grips were measured at each point. It would be very difficult to calculate values of stress and strain for the notched specimens during the test, therefore only load and displacement data were used to create the plots.

The load-displacement plot (Fig.3) summarizes behavior of all the specimens and it is possible to compare tests carried out in air and lead at different temperatures. The values of yield point and ultimate tensile strength (UTS) remained all the same for each temperature and environment. Moreover, concerning the elongation to rupture, for all the tensile tests at 400°C and 450°C, both in air and Pb, there were no marked differences. In general, at higher temperatures, higher displacement before rupture was observed for each environment. In particular, higher displacements before rupture were achieved in air for each temperature. However, these differences were not really relevant. On the other hand, for the test at 350°C the rupture of the specimen loaded in Pb happened at much lower displacement, the absorbed energy was therefore also much lower.

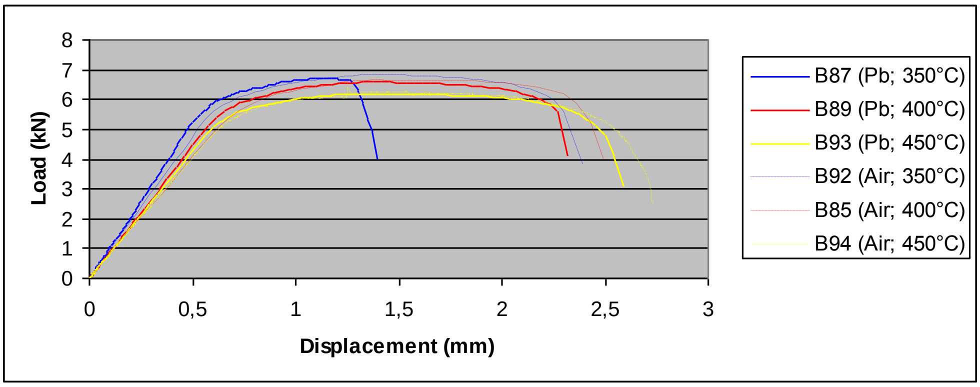

Figure 3: Comparison of tensile tests for lead and air at 350°C, 400°C and 450°C

3.2.1 WDX analysis of the specimen surfaces

Even though the oxide layer vas removed with the flux before the tests and the oxygen content was reduced by ArH_2 bubbling, the layer could be re-created during the test in liquid lead. Cross sections of two specimens (both tested in lead at 400°C, 72µm/h) were therefore prepared to analyze the interface between the steel T91 and Pb.

The results were similar for all scanned areas: At the surface area, there was increased oxygen content. However, the oxygen peak was not associated with any other oxide forming element. This suggested that some kind of oxide was present on the surface of the specimen after the test.

3.2.2 Analysis of fracture surfaces

Analysis of fracture surfaces was carried out for all the specimens after tests and removal of residual Pb. The comparison of two specimens tested at 450°C is presented in the Figure 4. The overall pictures of these fracture surfaces are very similar and also detailed pictures from all points of the surface show no difference – the fracture was ductile with typical dimples at both centre area and also edge of the fracture surface. This appearance was also observed for all the specimens tested in air. The overall picture of fracture surface of specimens tested at 400°C in liquid lead is very similar as the previous one (Fig. 5) and also the centre area and most of the edge areas showed ductile dimple fracture. However, there were also some areas at the edge which showed cleavage-like fracture, which is typical for liquid metal embrittlement.

The most significant difference, which is easily detectable even from the overall picture (Fig. 6), was for the specimens tested at 350°C in liquid lead. The cleavage-like fracture was not present only at the edge of the fracture surface as for the specimens tested at 400°C, but covered most of the fracture surface. Only small part of the surface, where the final rupture occurred, showed the ductile dimples. At 350°C in liquid lead the presence of LME was confirmed.

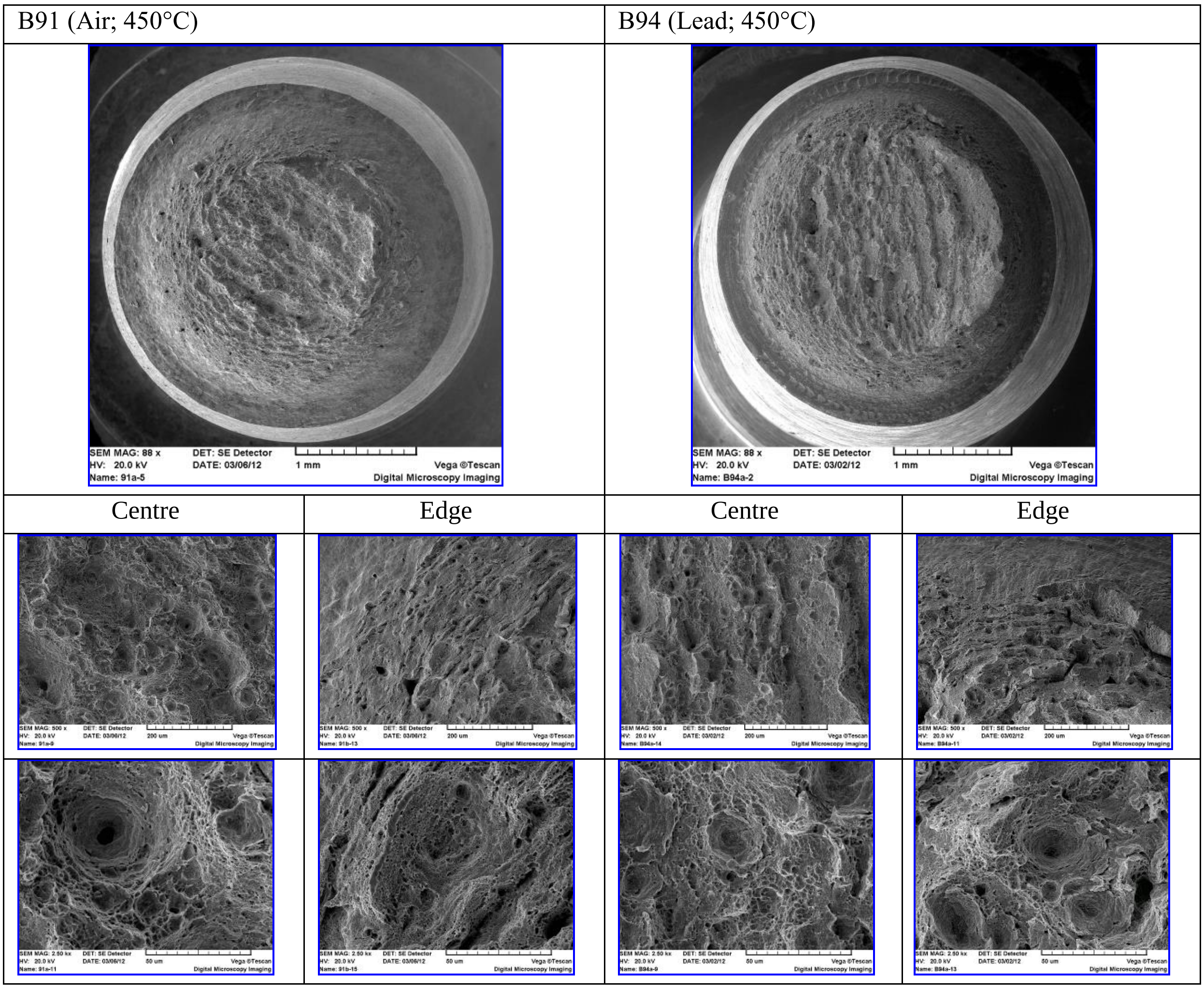

Figure 4: Fracture surfaces of specimen tested at 450°C

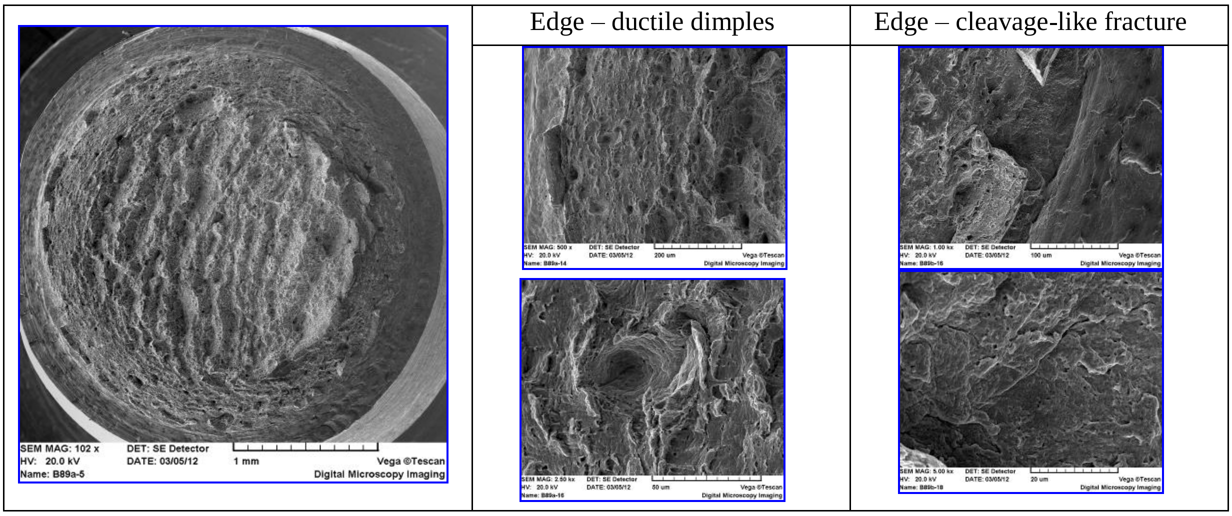

Figure 5: Fracture surfaces of B89 specimen tested at 400°C in lead

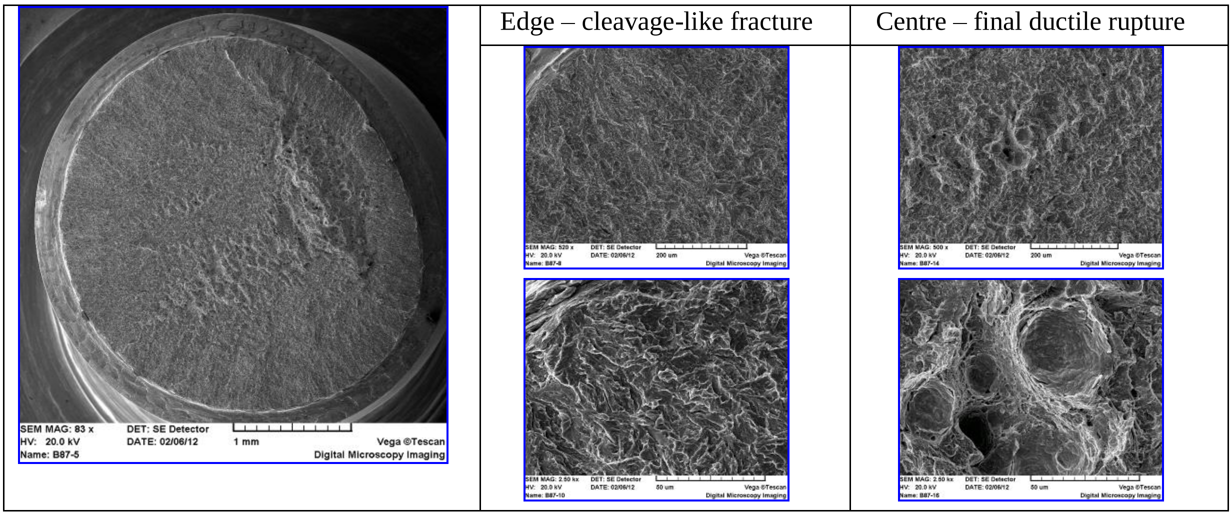

Figure 6: Fracture surfaces of B87 specimen tested at 350°C in lead

4 DISCUSSION

In order to study the susceptibility to LME of the steel T91 in liquid Pb several tests were carried out. Most of the tests, in general, do not show any sensitivity to embrittlement. However, to have a better understanding of the mechanism of interaction between Pb and T91, it was used a flux to eliminate any oxide at the surface of the steel. This process allowed the perfect contact between the two metals and therefore the interaction could better be observed. This procedure only allows to study in detail the pure interaction between the steel and the liquid metal, simulating an accidental situation rather than operating conditions of the material. During the tests, an appropriate flux (FP4201) was chosen and the surface of the specimens later tested in lead was treated with this flux. The fact that the oxide layer was removed with the flux was proven by the WDX line scan analysis on the cross-section of the T91 plate used during the test of fluxes. However, the protective layer could be recreated during the tensile test itself. For this reason another line scan was carried out on the cross section of the tensile specimens after the test (tested in lead at 400°C, 72µm/h). This analysis discovered higher content of oxygen at the area of the specimen surface. However, it was not possible to identify the composition of this oxide. It is therefore possible to assume that the detected oxides were lead-based and could be created even after the test when the cell was opened and air oxygen could reach the specimen. Another possibility is that the layer was too thin (<1µm) to detect by this method.

The embrittlement was proven for the tests at the temperature 350°C and 400°C at both tested displacement rates (72µm/h and 7200µm/h) for the notched specimens. The tests with the same conditions were already performed for the smooth specimens without notches [3] but the embrittlement did not occur. This observation was also in correspondence with other results [4], where the behaviour of variously thermal treated steel Grade 91 was studied and embrittlement was discovered only for notched specimens. This finding can mean that the liquid metal embrittlement requires tri-axial stress, which is not present at the smooth specimens. However, it is not possible to create big components with completely smooth surface. The notch therefore simulates a stress concentration in a scratch or similar localised condition in the material in operation.

The specimens tested in liquid lead at 350°C underwent quite a severe embrittlement. The specimens tested at 400°C were embrittled only in the areas near the surface of the original specimens. At 450°C the fracture was completely ductile. This observation is in correspondence with the statement, that the liquid metal environment has more effect on the mechanical resistance of structural materials, the closer it is to its melting temperature [5].

5 CONCLUSIONS

The SSRT of cylindrical notched tensile specimens made of T91 steel were performed with two different displacement rates (72µm/h and 7200µm/h) at three temperatures (350°C, 400°C and 450°C) in the environment of liquid lead and in air. The following conclusions were reached:

- The FP4201 is a suitable flux for the removal of the oxide layer formed at the surface of the T91 steel.
- The notched specimens tested in liquid lead at 350°C underwent a significant liquid metal embrittlement; most of the fracture surface consisted of the cleavage-like fracture, which is typical for LME.
- The notched specimens tested in liquid lead at 400°C also underwent a LME; the cleavage-like fracture however occurred only at the edge of the fracture surfaces.
- The embrittlement occurred independently on displacement rates for these temperatures.
- For specimens tested in liquid lead at 450°C the specimens had a ductile fracture.

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REFERENCES

[1] J. van den Bosch, R. W. Bosch, D. Sapundjiev, A. Almazouzi, “Liquid metal embrittlement susceptibility of ferritic-martensitic steel in liquid lead alloys”, Journ. of Nucl. Mat., 376, 2008, pp. 322-329

[2] T. Auger, G. Lorang, S.Guérin, J.-L. Pastol, D. Gorse, “Effect of contact conditions on embrittlement of T91 steel by lead–bismuth”, Journ. of Nucl. Mat., 335, 2004, pp. 227-231

[3] J. Klecka, Behaviour of T91 Steel in Liquid Lead Environment (Master Thesis), CVUT, Prague, 2013

[4] G. Nicaise, A. Legris, J. B. Vogt, J. Foct,” Embrittlement of the martensitic steel 91 tested in liquid lead”, Journ. of Nucl. Mat., 296, 2001, pp. 254-264

[5] S. P. Lynch, “Metal-Induced Embrittlement of Materials”, Materials Characterisation, 28, 1992, pp. 279-289