STUDY OF LEAD-EXPOSED 316L STEEL USING THE "SMALL PUNCH TEST" METHOD

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Abstract

This article studies the effect of liquid lead pre-exposure on austenitic 316L steel samples using the "Small Punch Test" (SPT) method. The lead exposure and the subsequent tests in air were done at 400°C, a temperature that would be relevant for the ALFRED demonstrator. The tests were performed on a total of eighteen (18) samples considering four (4) test scenarios. The purpose of the Small Punch tests is to generate a force-deflection (F-u) record, which provides details regarding the tested material's resistance characteristics and elasto-plastic deformation. The test specimens underwent Scanning Electron Microscopy (SEM) analysis, taking into account both their surfaces prior to and following their immersion in liquid lead, as well as their fracture surfaces after the tests were concluded. To determine the material properties for every sample, MATLAB was used to process the test data.

Keywords: *Small Punch Test, Liquid lead, Austenitic 316L steel, SEM analysis*

Introduction

This work represents a continuation of studying the effect of liquid lead pre-exposure on austenitic 316L steel samples using the "Small Punch Test" (SPT) method. The lead exposure and the subsequent tests in air were done at 400°C, a temperature that would be relevant for the ALFRED (Advanced Lead-cooled Fast Reactor European Demonstrator) future NPP (Nuclear Power Plant). The "Small Punch" method has been of great scientific interest lately, being recommended as one of the test methods used to evaluate Generation IV NPP structural materials.

Small Punch tests were performed on a total of eighteen (18) samples considering four (4) test scenarios, namely: room temperature - pre-exposed samples for 500 hours; room temperature - pre-exposed samples for 1000 hours; 400°C - pre-exposed samples for 500 hours; 400°C - pre-exposed samples for 1000 hours. The purpose of the Small Punch tests is to generate a force-deflection (F-u) record, which provides details regarding the tested material's resistance characteristics and elasto-plastic deformation.

The test specimens also underwent SEM (Scanning Electron Microscopy) analysis, taking into account both their surfaces prior to and following their immersion in liquid lead, as well as their fracture surfaces after the tests were concluded. To determine the material properties for every sample, MATLAB was used to process the test data.

After a thorough analysis of all the tests, comparative graphs were plotted between the average energies for samples pre-exposed at 500h and samples pre-exposed at 1000h.

Experimental Setup

The device with which the SPT tests were performed is sketched in Fig. 1. It was developed at RATEN ICN Pitesti, and is used in conjunction with a thermo-mechanical test rig in compression mode. The construction of this device was based specifications taken from the literature [1-3] and the ASTM standard for SPT tests [4]. The specific SPT samples look like metal discs with a diameter of 10 mm and a thickness of 0.5 mm, as shown in Fig. 1. Samples were taken from 316L steel plate by mechanical punching with a device specially designed for this operation.

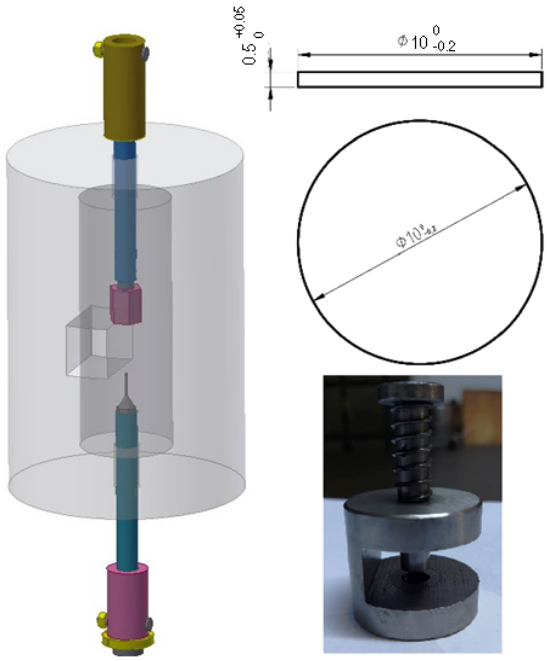


Fig. 1. Sketch of the SPT test device inserted into the furnace of the thermo-mechanical test rig.
Drawing of SPT samples made of austenitic steel 316L.
SPT sample stamping device

Prior to testing and pre-exposure in molten lead, SPT samples (see Fig. 2, upper part) were polished on one side with 4000 ppm grit abrasive paper. This is necessary in order to obtain a very flat surface and to minimize the mechanical stresses that may occur due to traces of surface machining of the sheet from which the samples were stamped. This polishing of the surface also helps to more easily identify cracks and deformations that occur during testing. Eighteen samples were processed in this way.

In the next step, the samples were introduced into a corrosion test facility in a liquid lead environment, which was configured to operate for an extended period of time under both atmospheric and temperature conditions. Exposure of the 18 samples tested was carried out in molten lead with an oxygen concentration of about 10^{-3} mass percent, at 400°C, for 500 hours (9 samples + 1 reserve sample) and for 1000 hours (9 samples + 1 reserve sample). The samples after molten lead exposure treatment are shown in Fig. 2.



Fig. 2. 316L SPT samples polished with abrasive paper with 4000 ppm grit. 316L SPT sample holder for pre-exposure in molten lead environment. SPT samples after exposure in molten lead environment

Results and discussions

A total of 18 samples were selected to perform the SPT tests in 4 test scenarios (room temperature - pre-exposed samples 500 hours, room temperature - pre-exposed samples 1000 hours, 400°C - pre-exposed samples 500 hours, 400°C - pre-exposed samples 1000 hours). The corresponding test matrix is described in Table 1.

Table 1. SPT test matrix - identification of samples and test scenarios

Sample ID	Scenarios			
	T_{room} 500 h preexposure	*T_{room} 1000 h preexposure*	*400°C 500 h preexposure*	*400°C 1000 h preexposure*
RT 500 1	x			
RT 500 2	x			
RT 500 3	x			
RT 500 4	x			
RT 1000 1		x		
RT 1000 2		x		
RT 1000 3		x		
RT 1000 4		x		
400 500 1			x	
400 500 2			x	
400 500 3			x	
400 500 4			x	
400 500 5			x	
400 1000 1				x
400 1000 2				x
400 1000 3				x
400 1000 4				x
400 1000 5				x

As shown in Fig. 2, the samples once extracted from liquid lead present deposits of solidified lead on the surface. These samples must be cleaned of deposits before being tested with a special chemical treatment designed to dissolve the lead on the surface without attacking the steel substrate.

Before analysing the resulting images, an unexposed sample was viewed in lead to discern what the surface looked like before exposure in liquid lead (as shown in Fig. 3). In order to have a baseline to determine how well they were cleaned; the samples were also observed under the electron microscope before cleaning (see Fig.3).

Lead deposits can be seen on the surface of the samples in the form of granules ranging in size from 10 µm to 0.1 µm. Already, the boundaries of the substrate steel grains can be seen, which are now visibly eroded by contact with the liquid lead. After cleaning the samples in acetic acid/oxygenated water solution (1:2), they were analysed again (see Fig. 3 bottom part).

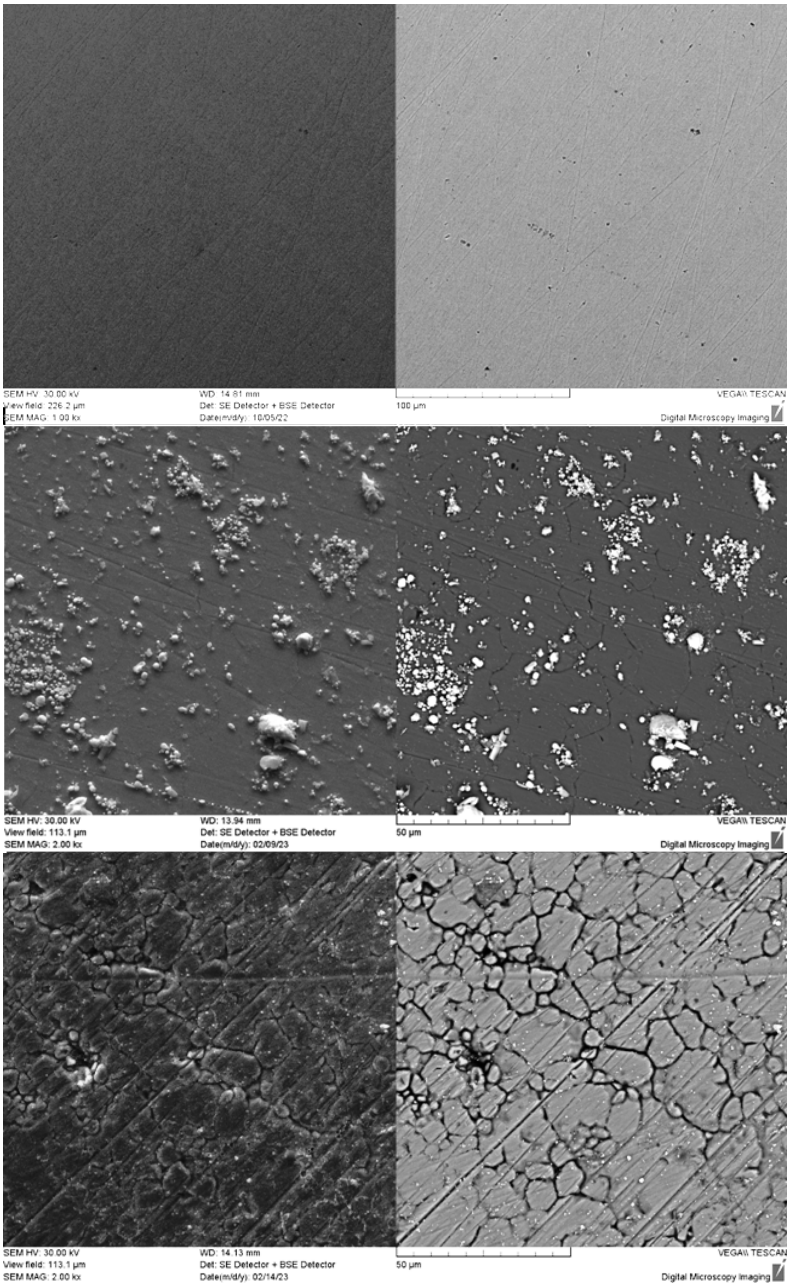


Fig. 3. Surface area of SPT samples before (TOP) and after (BOTTOM) lead exposure.
Surface of SPT samples after lead cleaning (BOTTOM)

From Fig. 3, the corrosion factor of steel in contact with lead can be seen. The metal grains on the sample's surface were reduced so that voids in the material of the size of a few microns (~ 4 µm) were created. It remains to be seen the influence of this corrosion on the SPT test results.

The SPT tests were carried out on a mechanical test rig, equipped with an oven to facilitate testing at 400°C. The tungsten carbide ball indenter head moved with a speed of 0.5 mm/min [4], the displacement of the machine's hydraulic piston (recorded as "sample deflection") and the force with which the ball pressed on the sample surface being also recorded.

The tests were performed without a strain gauge because the implementation of such a means of measurement is very difficult given the small proportions of the sample, the test fixture and the available space inside the furnace.

The graphs resulting from the tests are shown in Fig. 4, both for the scenarios considering room temperature and 400°C, temperature conditions.

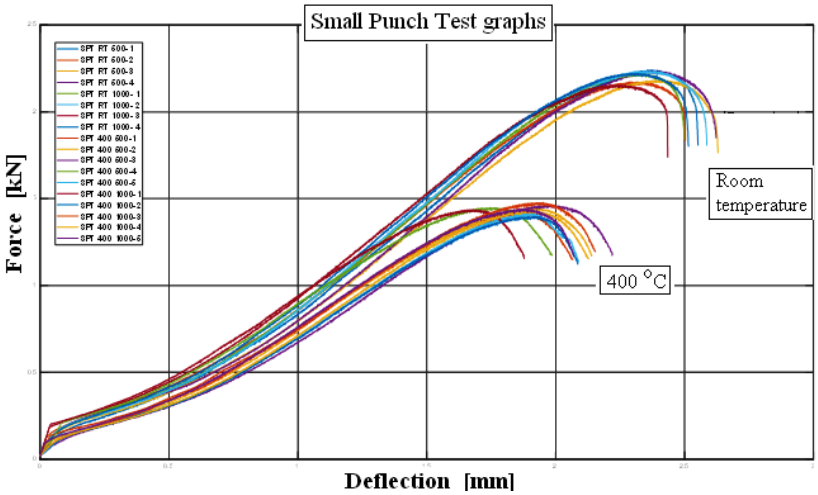


Fig. 4. Force-displacement (deflection) diagrams recorded from SPT tests for the 4 test scenarios: room temperature - pre-exposed samples 500 h; room temperature - pre-exposed samples 1000 h; 400°C - pre-exposed samples 500 h; 400°C - pre-exposed samples 1000 h

Analyzing the graphs and taking into account the studies carried out and the results obtained by other international laboratories [1-3], we can conclude that the tested material (316L steel) had a ductile behavior both at room temperature and at 400°C. The cracks on the surface of the samples are circular in shape, indicative of the ductility of the tested material, regardless of the temperature at which the test took place.

Fig.5 illustrates the break surfaces of the samples specifically selected to characterize each of the fur different scenarios, after performing the SEM analysis.

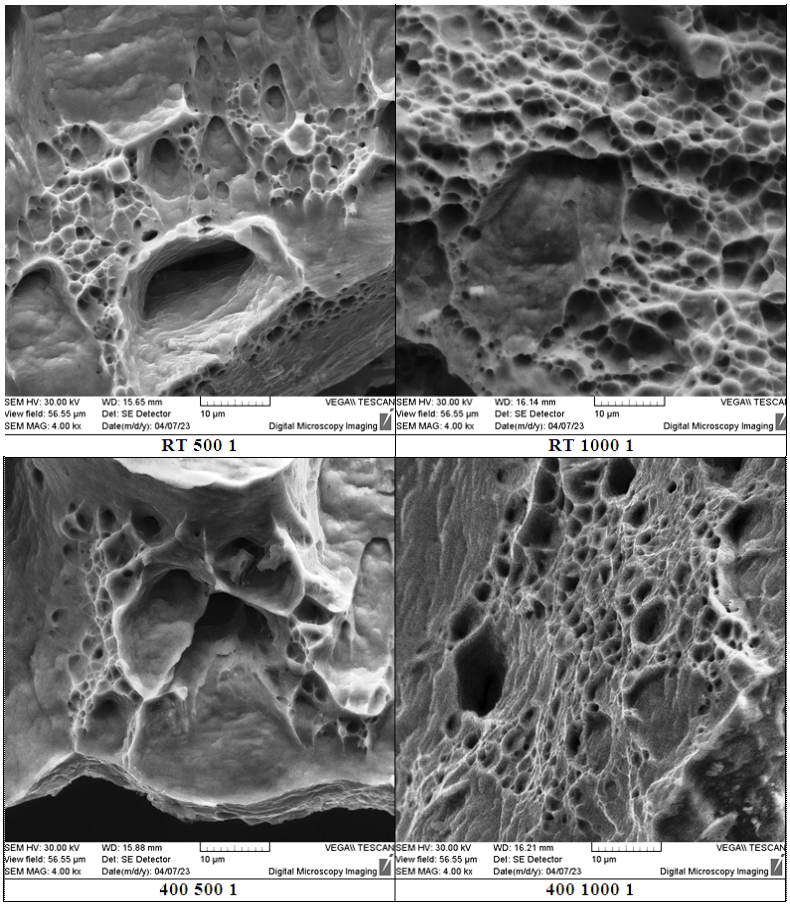


Fig. 5. SEM analysis of the break surfaces of samples selected to represent each of the 4 test scenarios

The images of the break surfaces of the samples selected to represent each of the four test scenarios highlight the ductile (cup-con) break mode. Analysing the pictures in Fig.5, together with the Force-Deflection graphs from Fig.4, we can draw as a final conclusion that the tested material (316L steel) had a ductile behaviour both at room temperature and at 400°C, whether we refer to the samples pre-exposed in molten lead for 500 hours or to those pre-exposed for 1000 hours.

As can be seen in Fig.5, the Force-Deflection curves resulting from the SPT tests are grouped in two areas (room temperature and 400°C). A clear difference between the curves of samples pre-exposed for 500h and those pre-exposed for 1000h at a given temperature is difficult to be noticed, as they are very close in terms of maximum force. For this reason, we investigated the possibility of plotting comparative graphs between the mean energies [4]. These plots are shown in Fig. 6.

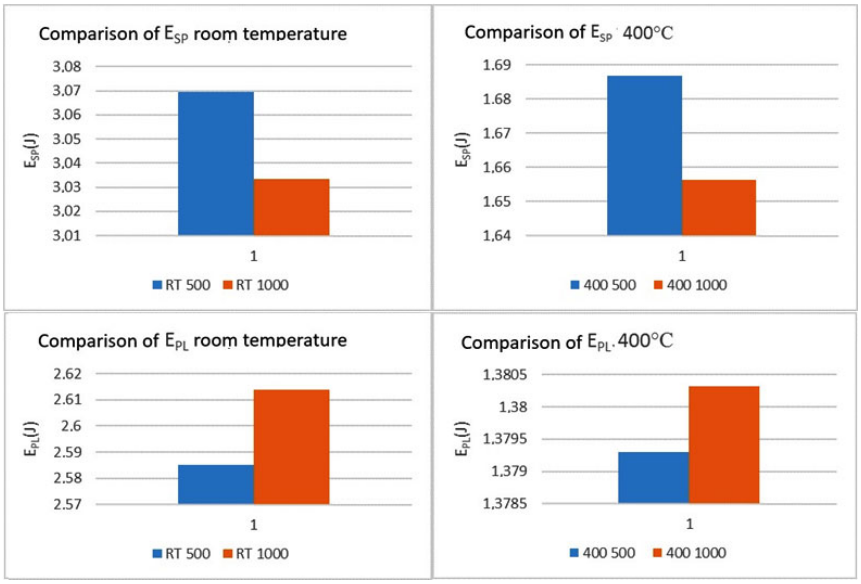


Fig. 6. Comparative plots of the average E_{SP} [4] (break energy) and E_{PL} [4] (plastic energy) energies for samples pre-exposed for 500h and those pre-exposed for 1000h, at room temperature and 400°C

Conclusions

In this study, "Small Punch Test" (SPT) method was applied on samples made of austenitic 316L steel, pre-exposed in molten lead, which were then subjected to SPT tensile tests under temperature conditions of interest to the ALFRED demonstrator. After the experimental study the following conclusions were drawn.

• The analysis of the graphs together with the microscopic analysis of the break surfaces lead to the conclusion that the tested material (316L steel) had a ductile behaviour both at room temperature and at 400°C.

• For the E_{SP} energy (the SP break energy) calculated as the area under the F-u curve up to u_f it is found that the values obtained for the samples treated in molten lead environment for 1000 h are lower than for the samples exposed for 500 h. At the same time, for the tests carried out at 400°C the values for E_{SP} energy are decreased by about 50% compared to the tests at room temperature. This may be a confirmation of embrittlement effects for tests in molten lead environment, considering the full SPT test curve (to breakage).

• If we take only the plastic flow section into account, then we can observe that the E_{PL} (plastic energy) values, calculated as the area under the F-u curve from u_e to u_m, are higher for samples exposed for 1000 h, both for tests performed in air and for tests performed at 400°C. This indicates that the embrittlement effect of molten lead does not occur during plastic deformation, but only after reaching the u_m point, when cracks already appear on the surface of the samples, which become more active, as seen from the E_{SP} energy analysis.

References

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