THE MICROSTRUCTURAL INVESTIGATIONS OF THE LIQUID METAL EMBRITTLEMENT PHENOMENON ON 316L SPECIMENS TESTED IN THE LIQUID LEAD ENVIRONMENT

LIVIA NICOLETA STOICA, VASILE RADU, VIOREL IONESCU, ALEXANDRU IONUȚ NIȚU, ALEXANDRA GEORGIANA JINGA, DENISA TOMA, MIRELA MATEI, VALENTIN OLARU
Institute for Nuclear Research (RATEN ICN) Piteşti, Romania
e-mail: livia.stoica@nuclear.ro

Abstract

The ″liquid metal embrittlement″ phenomenon (LME) that can occur in some Generation IV reactor configurations is a complex phenomenon strongly dependent on the specific solid-liquid contact. This phenomenon can lead to the embrittlement of some metals, which are ductile in the air and can become brittle in a long contact with certain liquid metals. The paper presents the results of the investigations performed on an atlas of metallographic and SEM analyses developed on the samples resulting from the experimental tests, to highlight the fracture characteristics induced by the lead embrittlement on 316L steel. Fracture in engineering alloys can occur by a trans-granular (through the grains) or an inter-granular (along the grain boundaries) fracture path. However, regardless of the fracture path, there are essentially only four principal fracture modes: dimple rupture, cleavage, fatigue, and de-cohesive rupture, respectively. Each of these modes has a characteristic fracture surface appearance and a mechanism or mechanisms by which the fracture propagates. The investigations were conducted on the specimens tested in the air and the liquid lead, through metallographic analyses by optical microscopy and SEM analyses of fracture surfaces. The metallographic aspects and SEM analyses confirmed the propagation character of the cracking front. In principle, this can confirm the ductile, brittle or complex nature of the cross-section fracture of the samples. In this way, a complete and detailed picture of the fracture modes for the sample tested in air and lead has been obtained. Based on the investigation performed, it could be concluded that the embrittlement effect on the 316L steel tested in the liquid lead for the temperature range 350°C – 400°C cannot be neglected, especially at low deformation rates.

Keywords: *microstructural analyses, liquid metal embrittlement, Generation IV reactor, tensile test*

Introduction

The availability of energy resources, climate change, air quality and energy security plays an increasingly important role for nuclear energy in future energy sources. While current Generation II and III nuclear power plant projects provide economically acceptable electricity and public supply in many markets, further advances in the design of the nuclear power system may expand the opportunities for the use of nuclear energy. To explore these opportunities, governments, industry, and research centres around the world have begun a comprehensive analysis of the possibilities for developing next-generation nuclear power systems, known as ″Generation IV″ [1].

Liquid metal embrittlement (LME) phenomenon strongly depends on the specific solid-liquid contact, being often dramatic as lead to engineering materials failure [2]. There is a large kind of mechanisms proposed to manage LME, the subsequent definition for LME being accepted: *″LME is the loss of ductility of a normally ductile metal or metallic alloy when stressed in touch with a liquid metal that may lead to brittle fracture″*. LME is considered as a specific sort of brittle fracture case, inter-granular or trans-granular, by cleavage. Four fracture modes are mainly considered: dimple rupture, cleavage, fatigue, and de-cohesive rupture, respectively [3]. LME is among little or no penetration of the embrittling atomic species into the solid metal [4, 5]. The importance of test conditions influencing LME is somewhat well documented [6]. Variables affecting material (e.g. solid metal microstructure and chemistry), stress state (e.g., notched vs. smooth bars) and environment (including various factors that affect wetting) are affecting the behaviour appearance of LME.

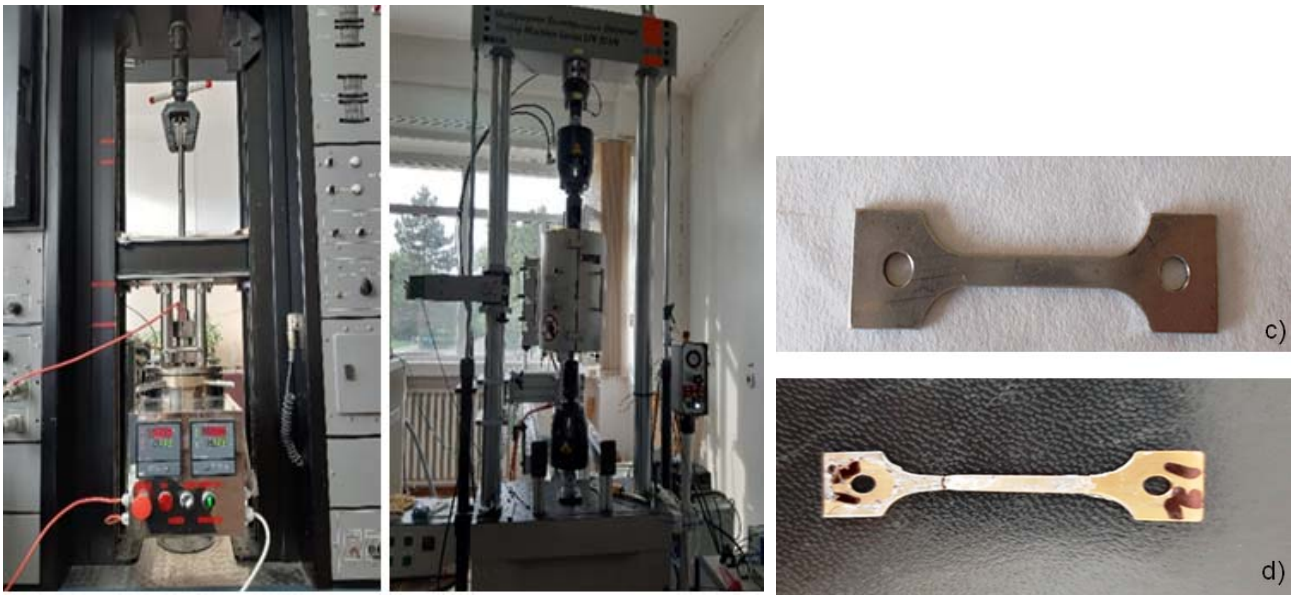
The need for LME modelling of stainless-steel systems is clear. The lack of models, in this case, is owing to the complexity of modelling a multi-component engineering alloy (in opposition to a pure material). Considering the resistance of stainless steel to LME, modelling can be instructive to the overall understanding of LME resistance but also used in practical applications associated with assessing the structural integrity of Generation IV reactor systems [7].

The paper evaluates and interprets the results obtained from a previous work focused on the experimental slow strain rate tensile tests in the air and the liquid lead using 316L specimens. Thus, metallographic analyses by optical microscopy and SEM analyses of fracture surfaces are considered and the ductile-brittle characteristics are revealed and discussed.

Experimental testing program and results

To highlight better the material behaviour under the liquid metal embrittlement, the experimental tests on the 316L stainless steel specimens used in Generation IV reactors, both in the liquid lead and the air environment, were performed.

An important facility has been designed and built up for the Instron Testing Facility (see Fig. 1a) for the liquid lead experiments. The Walter+Bai Machine (shown in Fig. 1b) allow the tensile tests in the air. Figs. 1c and 1d, respectively, present the typical specimen aspect before and after testing in the liquid lead.



a) b)

Fig. 1. a) Instron Testing Facility for SSRT in the liquid lead; b) Walter+Bai Machine in the air; c) 316L specimen before testing; d) 316L specimen after testing

To evaluate the effect of LME on the tensile behaviour of 316L steel samples, some results obtained from the air and the liquid lead tests are described in the following.

At the temperature test of 350°C and rate of deformation $5\times10^{-5}s^{-1}$ (Slow Strain Rate Testing, SSRT), Fig. 2 shows a comparison between the tensile behaviour of sample 316L in the air and liquid lead. Fig. 3 presents the mechanical stress-strain curves, obtained at the test temperature of 375ºC and strain rate of $5\times10^{-5}s^{-1}$. For the test temperature of 400ºC, at the same deformation rate of $5\times10^{-5}s^{-1}$, Fig. 4 illustrate the engineering stress-strain curves obtained in the air and the liquid lead for the 316L samples.

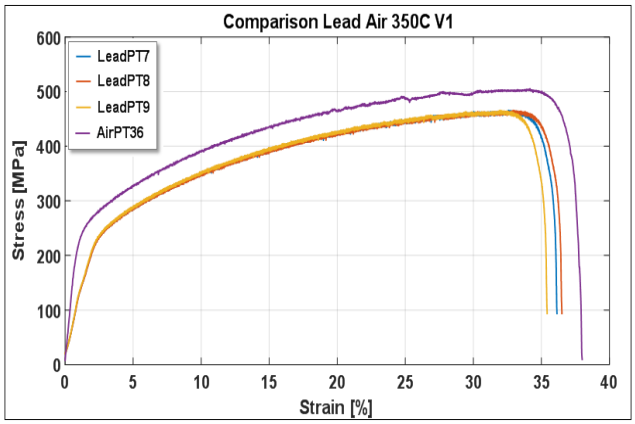


Fig. 2. Stress-strain curves in the air and lead, at 350 ºC and a strain rate of $5\times10^{-5}s^{-1}$

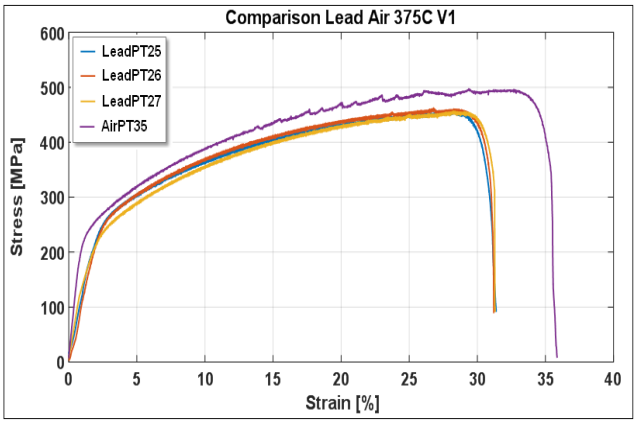


Fig. 3. Stress-strain curves in the air and lead, at 375 ºC and a strain rate of $5\times10^{-5}s^{-1}$

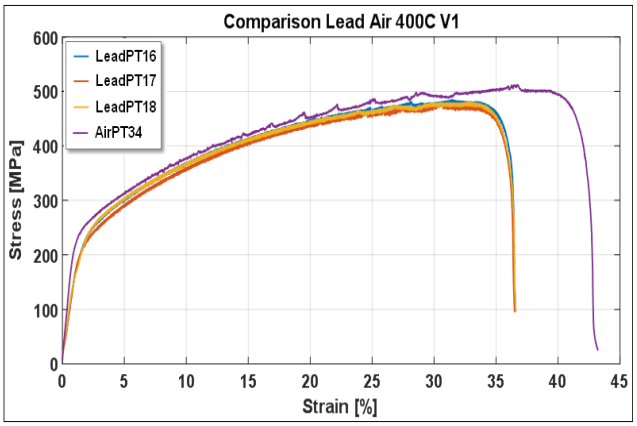


Fig. 4. Stress-strain curves in the air and lead, at 400 ºC and a strain rate of $5\times10^{-5}s^{-1}$

Following the examination of the mechanical stress-strain tensile curves obtained for the samples tested in air and liquid lead, the following statements can be made:
- It can be seen that for the temperature of 350 ºC, although the ultimate tensile strength decreases in the lead environment compared to the air, in terms of elongation at fracture, the difference is only a few per cent;
- In contrast, for temperatures of 375 ºC and 400 ºC, respectively, there is a decrease in ductility by reducing the total deformation at fracture in the liquid lead environment compared to the tests performed in the air. This means that the presence of LME cannot be neglected at these strain rates in tensile testing. At the same time, there is a decrease in ultimate tensile strength for liquid lead testing, which can also be attributed to the LME effect on 316L samples, tested in SSRT.

Study of the pattern type of fracture behaviour by optical microscopy and SEM analysis

To obtain the pattern type of fracture behaviour, the metallographic aspects of the 316L specimen fracture were analysed following the tensile tests performed in the air and in the liquid lead, respectively. In principle, this can confirm the ductile, brittle or complex nature of the cross-section fracture of the samples.

As can be seen in Fig. 5a, the cracking front of the 316L test specimen in the air at 350 ^{o}C is oriented at an angle of 45^{o} to the direction of mechanical loading and characterizes a ductile behaviour. For samples tested at the same temperature in the liquid lead (Fig. 5b), the appearance of the crack propagation front is similar, which means that the presence of LME cannot be confirmed. This is in line with the elongation at fracture, as is shown in Fig. 2.

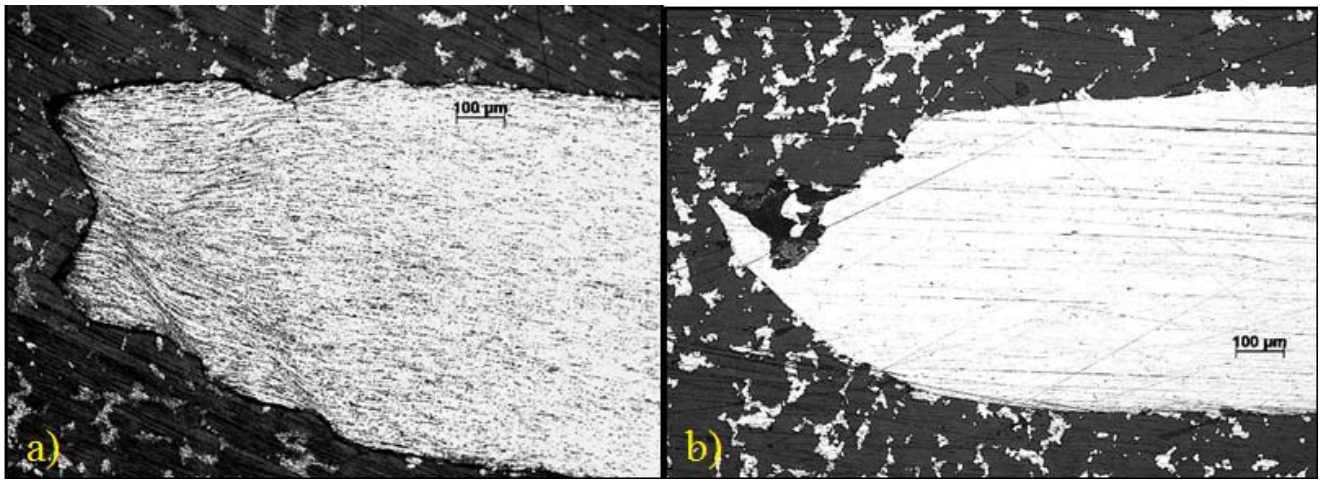


Fig. 5. Comparison of metallographic aspects of fracture fronts for samples tested at 350 °C in air (a) and lead (b)

At a temperature of 375 °C, samples were tested in the air and the same ductile behaviour can be seen in Fig. 6a. In the case of tested samples in liquid lead, at the same temperature, the fracture propagation front begins to show a brittle appearance (Fig. 6b). This brittle aspect is represented by the segment of the crack propagation front which is normal in the direction of mechanical stress. It should be noted, however, that in the liquid lead testing environment, the crack front has a mixed (ductile-brittle) character. It can be assumed that in the first part of the tensile test, the crack propagates ductile. In the final part of the test, when the LME acts on the specimen by the penetration of lead into the macroscopic crack, crack propagation acquires the brittle aspects. Thus, the presence of the LME effect shown in Fig. 6b is consistent with the mechanical behaviour in the tensile curve (see Fig. 3).

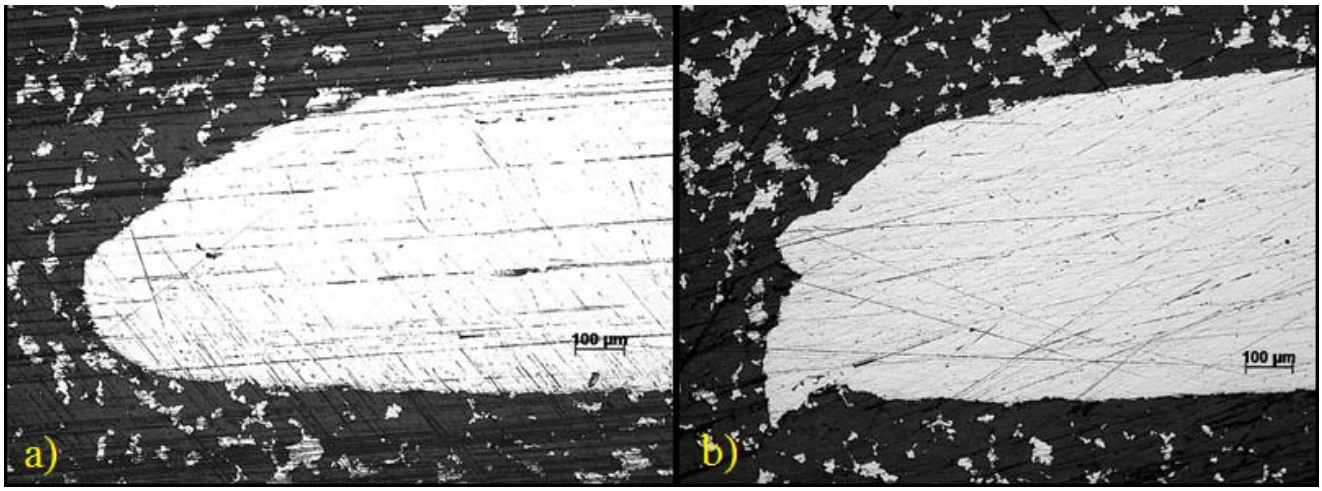


Fig. 6. Comparison of metallographic aspects of fracture fronts for samples tested at 375 °C in air (a) and lead (b)

The above mentioned features become more visible at the test temperature of 400 °C. Thus, in air testing, the ductile appearance is even more pronounced, as can be seen in Fig. 7a. For liquid lead environment tests, the influence of LME on the crack propagation path is obvious, as shown in Fig. 7b.

The crack propagation is predominantly perpendicular to the direction of the applied mechanical stress. Thus, the brittle behaviour of the fracture highlights the influence of the LME phenomenon both in the metallographic microstructural analysis and in the mechanical behaviour (see Fig. 4).

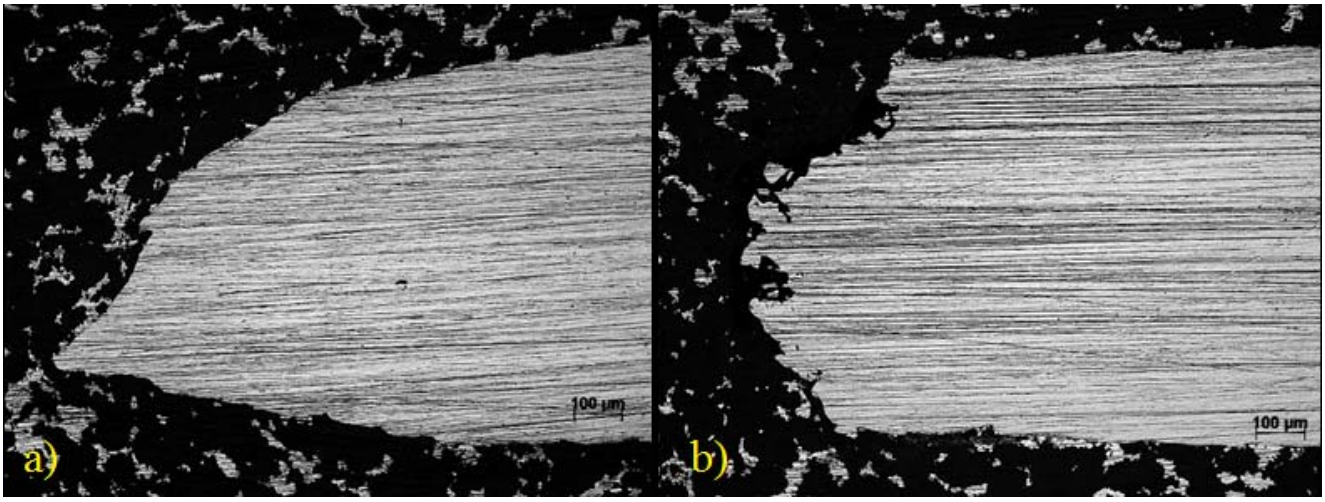


Fig. 7. Comparison of metallographic aspects of fracture fronts for samples tested at 400 °C in air (a) and lead (b)

As found in the metallographic examinations of the samples tested both in air and liquid lead environments, the following statements can be made:

- At 350 ºC, the mechanical behaviour of the samples, respectively the crack propagation is not significantly different in the lead environment compared to the air. The metallographic examination shows a ductile characteristic of crack propagation in both test environments;
- For the temperatures of 375 ºC and 400 ºC, a significant change in the character of the fracture front was observed, namely, the appearance of the features that characterize a brittle fracture. It can be assumed that this is due to the initiation of the LME mechanism during testing, especially at low strain rates (SSRT). This mention is in line with the observation of the decrease in specimen ductility by reducing the total deformation at fracture in the liquid lead environment compared to the tests performed in the air environment.

From the SEM analyses of the samples tested for the present paper, an atlas of representative images was prepared. For each sample, three depth images at x1000 magnification in three areas of the fracture surface are displayed. This way, we will have a complete and detailed picture of the samples fracture modes tested in the air and the liquid lead environments (see Figs. 8-13).

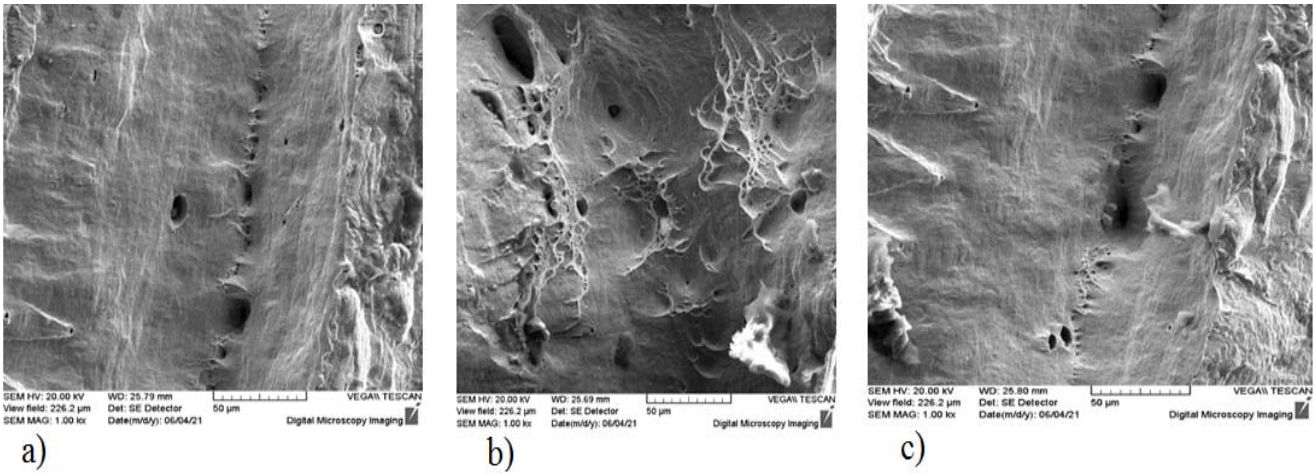


Fig. 8. SEM analyses of the specimens tested in the air at 350 ^{o}C, at a strain rate of $5\times10^{-5}s^{-1}$

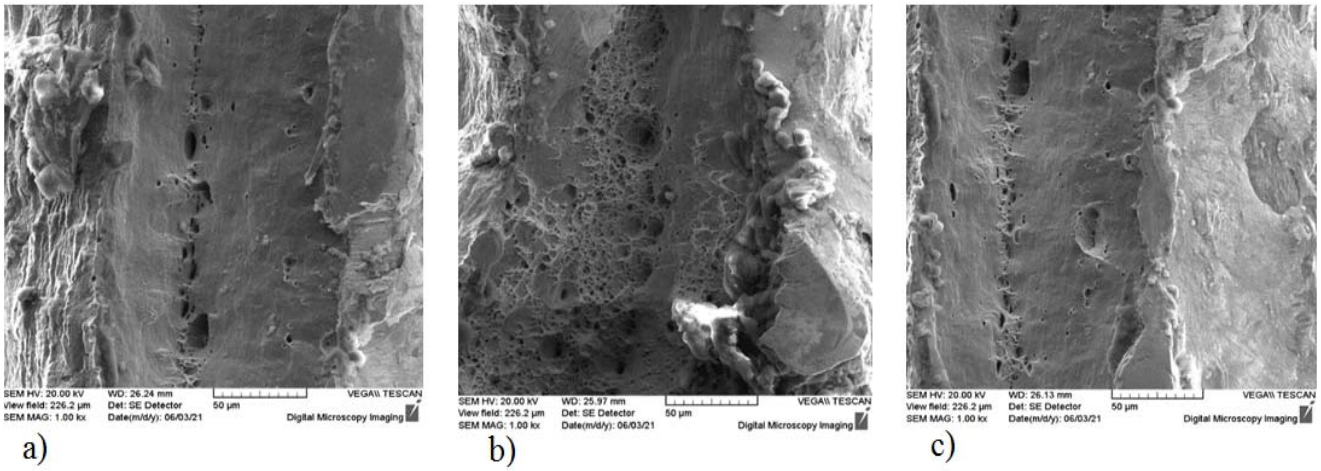


Fig. 9. SEM analyses of the specimens tested in the liquid lead at 350 ^{o}C, at a strain rate of $5\times10^{-5}s^{-1}$

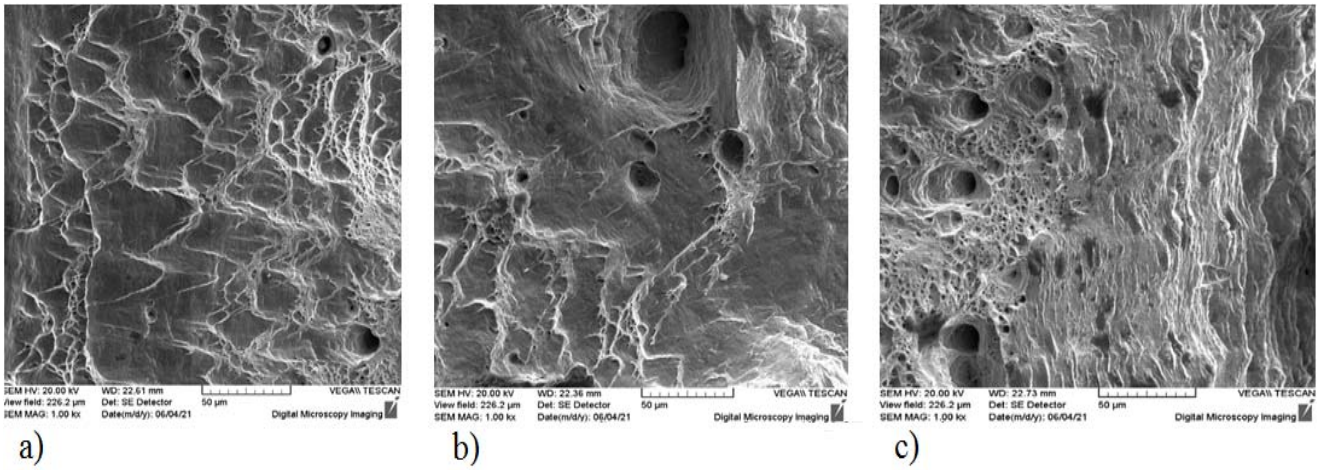


Fig. 10. SEM analyses of the specimens tested in the air at 375 ^{o}C, at a strain rate of $5\times10^{-5}s^{-1}$

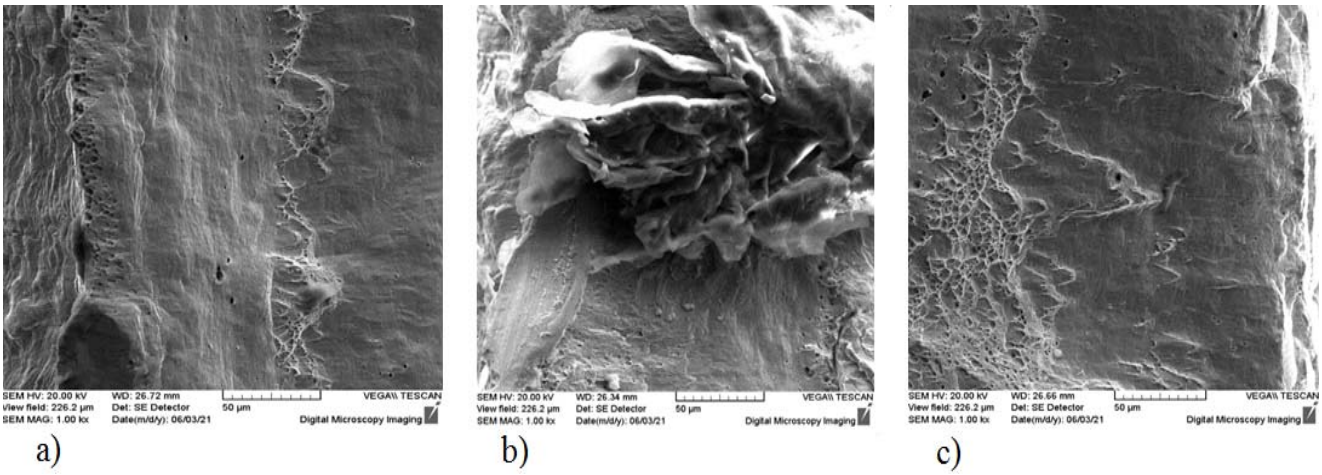


Fig. 11. SEM analyses of the specimens tested in the liquid lead at 375 ^{o}C, at a strain rate of $5\times10^{-5}s^{-1}$

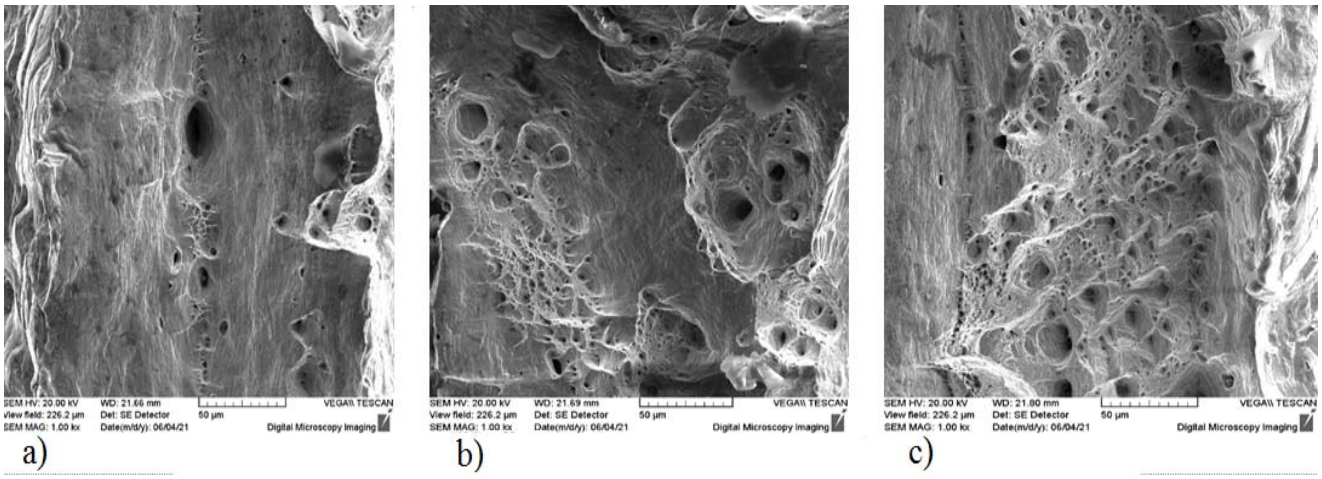


Fig. 12. SEM analyses of the specimens tested in the air at 400 ^{o}C, at a strain rate of $5\times10^{-5}s^{-1}$

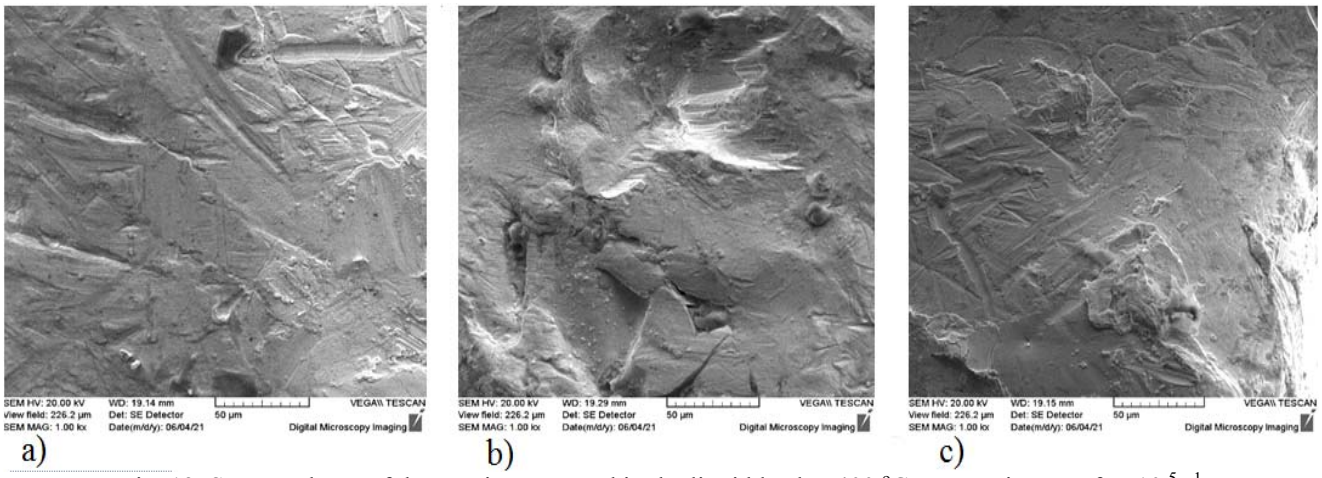


Fig. 13. SEM analyses of the specimens tested in the liquid lead at 400 ^{o}C, at a strain rate of $5\times10^{-5}s^{-1}$

SEM analyses for 350 ^{o}C

SEM analysis of the fracture surface for a sample tested in the air is shown in Fig. 8. For the lowest deformation rate (SSRT type), the microcavities (dimples) formation is noticeable in all three areas, some of them being placed inside larger cup-cone cavities. This way of cracking is typical for ductile fracture of metals.

In the case of the tests performed in the liquid lead, for the lowest deformation rate (SSRT type), the SEM analyses of the fracture surfaces (see Fig. 9) present the same morphology of the microcavities, as the tests performed in the air, namely, large cavities with walls containing microcavities (dimples). This is consistent with the metallographic analysis. Thus, it can be noted that the presence of LME due to lead environment is not highlighted by the SEM analyses either.

SEM analyses temperature of 375 ^{o}C

For tensile tests performed at this temperature in the air environment, a typical SEM surface is shown in Fig. 10 for SSRT. In this situation, for the three areas of the fracture surface observed at a magnification of x1000, the morphology of the microcavities is similar to that observed at the temperature of 350 ^{o}C. Thus, the ductile character of the specific fracture of metals is maintained.

For the tensile tests performed at the same temperature and strain rate (SSRT) in the liquid lead (see Fig. 11), the mixed ductile-brittle character appears. Thus, a part of the fracture surface has microcavities (ductile rupture) which, it is bordered by an area with a micro-cleavage aspect (see Fig. 11c). This is certainly due to the LME mechanism caused by the liquid lead during the tensile test.

SEM analyses for 400^{o}C

For tensile tests performed at this temperature in the air, a typical SEM surface is shown in Fig. 12, for the SSRT strain rate. In this situation, for the three zones of the fracture surface observed at the magnification of x1000, the morphology of the microcavities is characterized by an increase in their dimensions as well as by the multiplication of the larger cavities. This can be explained by the increased ductility of 316L steel as temperature increases.

For tensile tests performed at the same temperature and deformation rate (SSRT type) in the liquid lead environment (see Fig. 13), the fracture surface mainly shows the cleavage areas confirming the embrittlement induced by the liquid lead during the tensile test. In this case, the cleavage surfaces are quite large. SEM observations of fracture surfaces of samples tested in air and liquid lead at 400 °C are fully consistent with the metallographic observations.

Conclusions

RATEN ICN, partner in the FALCON (Fostering ALfred CONstruction) Consortium, is involved in the construction of ALFRED (Advanced Lead Fast Reactor European Demonstrator) demonstrator of LFR technology, which is based on cooling with liquid lead, and will be hosted by Romania. This is one of the arguments for starting the studies within the present paper, with the stated objective on the issue of the structural materials embrittlement specific to this type of reactor.

The paper included the metallographic and SEM analyses of the samples resulting from the experimental tests, to highlight the fracture pattern of fracture behaviour induced by the liquid lead on 316L steel.

As found, there is a significant influence of the LME effect on 316L austenitic steel samples tested in the liquid lead environment, which is particularly noticeable at the deformation rate of $5\times10^{-5}s^{-1}$ (SSRT type), for the test temperatures of 375 °C and 400 °C.

It should be noted that there are no systematic studies in the literature to assess the mechanical behaviour of 316L steel in the liquid lead environment, in the temperature range 350 °C - 400 °C. Therefore, the scientific approach of the paper is once more justified to elucidate the susceptibility of the LME phenomenon.

Further studies in the field are under development to obtain the LME phenomenon influence on the fracture mechanics parameters that characterise the 316L specimens in the liquid lead environment.

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