Effect of deformation twinning on dissolution corrosion of 316L stainless steels in contact with static liquid lead-bismuth eutectic (LBE) at 500°C

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Influence of plastic deformation on dissolution corrosion of 316L austenitic stainless steels in static, oxygen-poor liquid lead-bismuth eutectic at 500 °C

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ABSTRACT

This study addresses the effect of plastic deformation on the dissolution corrosion behaviour of 316L austenitic stainless steels. Dissolution corrosion was promoted by low oxygen conditions in liquid lead-bismuth eutectic (LBE). Specimens with controlled degree of plastic deformation (20, 40, and 60%) and a non-deformed, solution-annealed specimen were simultaneously exposed for 1000 hours at 500 °C to static LBE with low oxygen concentration ([O] < 10^-12 mass%). The corroded specimens were analysed by various material characterization techniques. All exposed specimens exhibited dissolution corrosion. The non-deformed steel showed the least dissolution attack (maximum depth: 36 μm), while the severity of attack increased with the degree of steel deformation (maximum depth in the 60% steel: 96 μm). It was, thus, concluded that increasing the amount of plastic deformation in 316L stainless steels results in higher dissolution corrosion damages for steels exposed to low-oxygen LBE conditions. Additionally, it was observed that the presence of chemical bands and δ-ferrite inclusions in the 316L steel affected its dissolution corrosion behaviour.

INTRODUCTION

The MYRRHA (Multi-purpose hYbrid Research Reactor for High-tech Applications) irradiation facility is the ESNII (European Sustainable Nuclear Industrial Initiative) lead-fast reactor (LFR) pilot plant, which aims to replace the ageing BR2 research reactor at SCK•CEN. MYRRHA is an accelerator-driven system (ADS) that will use liquid LBE as primary coolant and spallation target. The 316L austenitic stainless steel is the main candidate structural material for the MYRRHA system. Since LBE is a corrosive medium for most nuclear grade steels, understanding the factors affecting the liquid metal corrosion behaviour of the 316L steel is necessary to ensure the safe operation of the MYRRHA reactor.

Several studies have been dedicated to the investigation of the 316L steel corrosion behaviour in LBE. The effect of the steel exposure conditions, such as temperature, LBE oxygen concentration, LBE flow velocity and duration of exposure was reported by several authors. However, the studies on the effect of the steel thermomechanical treatment and microstructure on the LMC behaviour of 316L austenitic stainless steels are limited.

The overall effect of steel cold deformation on the in-service behaviour of nuclear grade stainless steels is not perfectly understood. On one hand, it was reported that cold deformation reduces the irradiation-induced swelling of austenitic stainless steels, favouring the use of cold-worked steels in ADS nuclear systems. On the other hand, corrosion experiments in LBE showed that the LMC resistance of austenitic stainless steels is reduced as result of cold working.

Tsisar et al. exposed to low-oxygen ([O] ≤ 10^-8 mass%) static LBE 1.4970 austenitic stainless steels with different thermomechanical states for 1100 and 2200 hours at 550 °C. The results of the first test (1100 hours) showed that corrosion damages became more severe as the degree of plastic deformation increased. However, in the second test (2200 hours), an as-received deformed steel showed less severe corrosion damages than a solution-annealed steel.

Rivai et al. investigated the effect of cold work on the corrosion behaviour of JPCA austenitic stainless steel after an exposure of 1000 hours at 450 °C in flowing LBE with [O] = 10^-8-10^-9 mass%. It was reported that a cold working degree of 20% limits the steel dissolution attack.

Corrosion tests performed by Kurata on JPCA and 316 steels in static LBE with high oxygen ([O] ≈ 2.8×10^-5 mass%) at 550 °C for 3000 h and low oxygen ([O] ≈ 4.2×10^-9 mass%) at 550 °C for 1000 h showed that an increase in the degree of cold working from solution-annealed to 50% results in thicker ferritization zones for both steels. However, an increase in the depth of the ferritization zone with degree of cold working at intermediate oxygen concentration ([O] ≈ 1.4×10^-7 mass%) at 500 °C for 1000 h was observed only on JPCA, while no trend was detected on 316L.

Lower corrosion rates (both oxidation and dissolution) for cold-drawn 316/316L specimens as compared to the annealed ones were reported by Johnson et al. This corrosion experiment was performed at 550 °C in non-isothermal LBE loop with [O] ≈ 3.5×10^-6 mass%. It was concluded, that cold-drawn specimens form a protective chromium oxide, while the annealed specimens under the same conditions form a thicker, less protective double-layer oxide scale, which consisted of an inner Fe-Cr oxide and an outer Fe oxide.

Since the results of the aforementioned studies on the effect of plastic deformation on the LMC behaviour of 316L or compositionally similar steels are contradictory and the oxygen concentration in LBE could be a possible reason for the observed discrepancies, it was decided to use low-oxygen conditions in this work to promote only dissolution corrosion. Thus, this work is dedicated to the study of the effect of controlled steel cold deformation and steel microstructure on...
the dissolution corrosion behaviour of 316L austenitic stainless steels exposed to low-oxygen static LBE at 500 °C for 1000 hours.

EXPERIMENTAL

Materials

The chemical composition of the tested 316L austenitic stainless steel is given in Table 1. This steel was an industrial-size 316L steel plate of 15 mm in thickness that was hot-rolled, solution-annealed at 1050-1100 °C and quenched in water.

In order to obtain specimens with different amounts of cold work, tensile specimens (Figure 1) were machined from this 316L steel plate along the rolling (L) direction. These specimens were uniaxially strained in tension to 20, 40 and 60% of plastic deformation. The stress-strain curves for all specimens are shown in Figure 2. The controlled straining of the steel specimens was performed at room temperature in air. Cylindrical corrosion specimens of 15 mm in length were machined from the one half of the tested tensile specimens, while specimens for microstructural characterisation of the non-exposed steels with different degree of cold work were machined from the other half (Figure 3). Each specimen had an internal and an external thread of 3 mm to ensure connection to each other and to the sample holder used to introduce the specimens in the LBE bath. The last specimen in the set had only an internal thread and was made from the non-deformed steel along the rolling direction. This specimen was considered as the reference material with 0% of cold-work. The corrosion specimens were ground using SiC paper up to 4000 grit, polished with diamond suspension up to 3 μm and finally cleaned with ethanol and acetone prior to their exposure to the LBE bath.

Experimental setup and testing methodology

The experimental setup is presented in Figure 3. It consists of a stainless steel autoclave with an inner alumina crucible that is inert to LBE. A heater is wrapped around the steel autoclave and the whole setup is thermally insulated to prevent heat losses. The experimental setup is placed in a glove-box, where a low-oxygen atmosphere is maintained. The temperature of the LBE bath was measured with a K-type thermocouple during the corrosion experiment. Low [O] conditions were maintained in the LBE bath by purging with Ar+5% H2 reducing gas at a flow rate of 15 ml/min. An overpressure of 150 mbar was ensured by automatic back-pressure regulators. The control of the gas flow, overpressure and heating was PLC-controlled. The LBE oxygen concentration was constantly monitored during the experiment and calculated from the output of the in-house developed and validated Bi/Bi2O3 electrochemical oxygen sensor using the following equation:

\[ E = -3.4756 \times 10^3 + 2.5217 \times 10^{-4} \cdot T - 4.3087 \times 10^{-5} \cdot T \cdot \ln \text{Co} \]  

where T is the temperature in K and Co is the LBE oxygen concentration in mass%.

Before the experiment, the liquid LBE bath was preconditioned with Ar+5% H2 gas at the targeted temperature of 500 °C. 316L steel specimens were introduced into the test setup at 500 °C when [O] was below 10-10 mass%. The low level of LBE oxygen concentration was deliberately chosen to promote dissolution corrosion.

Microstructural characterization

The microstructure of the non-exposed steels was studied by light optical microscopy (LOM) and electron backscatter diffraction (EBSD). The EBSD measurements were performed on a Nova 450 NanoSEM, FEI, with a scanning step of 5 μm at an accelerating voltage of 20 kV. Macro-hardness measurements were done on non-exposed steel specimens by means of Vickers hardness using a load of 10 kg. To determine local micro-hardness in the steel microstructure, a load of 0.05 kg was used.

After the corrosion experiment, specimens were extracted from the LBE bath, disconnected from each other and four transversal cross-sections per specimen were prepared for subsequent microstructural investigation. The segments of the exposed steel specimens were cold-mounted in resin for metallographic analysis, so as to avoid melting of the solidified LBE residues during hot mounting that would affect the interpretation of results. The embedded specimens were ground with SiC paper up to 4000 grit and further polished to mirror finish with diamond suspension up to 1 μm. The specimens were also polished with an OPS (colloidal silica) suspension, so as to impart a subtle relief on the specimen surface that facilitated the study by means of LOM. The corroded 316L specimens were analysed by LOM and scanning electron microscopy (SEM, JEOL JSM-6610LV). Chemical composition was identified by energy dispersive X-ray spectroscopy (EDS, Bruker XFlash Detector 4010) and electron probe microanalysis (EPMA, JEOL JXA-8530F). Measurements of the depth of corrosion damages and of the surface coverage by dissolution attack were performed using the AxioVision 4.8 software. Investigation of the steel microstructure on the nanoscale was done by a transmission electron microscope (TEM, JEOL ARM200F) operated at 200 kV on thin foils extracted from the corroded steel from a selected location by means of focused ion beam (FIB, FEI-DualBeam-SEM/FIB Nova 600 NanoLab). All analytical work, except from the analysis of the non-exposed steel by LOM, was done on the transversal cross-sections of the exposed steel specimens.

RESULTS

Microstructure of non-exposed 316L steels

LOM was employed to investigate the microstructure of the non-deformed steel on planes parallel and perpendicular to the rolling direction. The presence of elongated δ-ferrite inclusions (Figure 4[a] and [b]) were observed in both planes. The latter suggested that they were plate-like precipitates oriented along the rolling direction. LOM also identified a chemical banding effect, which manifested itself in alternating light and dark bands (Figure 5). The micro-hardness of the dark and light bands in the non-deformed steel was 160 HV and 148 HV, respectively. In the 60% deformed steel, the micro-hardness of the same bands was 351 HV and 325 HV, respectively. The orientation of the bands coincides with the orientation of the δ-ferrite inclusions.

EBSD results acquired from the deformed, non-exposed steels are shown in Figure 6. These results were acquired from the analysis of cross-sections perpendicular to the direction of applied tension. The microstructure of the non-deformed steel (Figure 6[a]) exhibited grains without preferred crystallographic orientation as well as the presence of annealing twins. The 20% deformed steel showed the development of a mixed {111} and {001} texture (Figure 6[b]). In the 40% and 60% deformed steels, the mixed {111} and {001} texture became stronger and deformation twins were observed (Figures 6[c] and [d]). Hardness measurements revealed an increase in macro-hardness with increasing plastic deformation: 157 HV for 0%, 240 HV for 20%, 297 HV for 40% and 331 HV for 60% cold deformation. There was no correlation found between chemical banding and grain distribution.

Results of corrosion experiment

The concentration of dissolved oxygen in the LBE bath during the exposure varied in the range between 1.2×10-12 and 1.3×10-13 mass% (Figure 7). The appreciable drop in LBE oxygen concentration in the beginning of the test resulted from the fact that the steel specimens acted as oxygen getters upon their immersion in the liquid LBE bath, drastically reducing the LBE oxygen content. The oxygen excursion after 655 hours was caused by a heater failure, upon which time the
steel specimens were extracted from the LBE bath and were temporarily stored in the glove-box with inert (Ar) atmosphere. The crucible with solidified LBE was transferred in a properly working setup and the oxygen concentration was measured again after remelting the LBE. When it was confirmed that the LBE oxygen concentration did not rise significantly in the meantime, the experiment was continued by reintroducing the steel specimens in the LBE bath at \([O] \approx 10^{11}\) mass%. The plot of Figure 7 shows the cumulative exposure conditions \(T, [O]\) from the two different parts of the corrosion test.

Representative cross-sections of corroded specimens are shown in Figure 8. All specimens exhibited LBE dissolution attack. As shown by EDS analysis (Figure 9), the dissolution-affected zone is depleted in steel alloying elements, such as Cr, Ni and Mn, and penetrated by LBE. The removal of the austenite stabilizers Ni and Mn has probably caused transformation of the austenitic steel matrix (fcc) into ferrite (bcc) in the dissolution-affected zone.\(^8,10\) The shape of the dissolution-affected zone is non-uniform and becomes even more irregular with increasing amount of plastic deformation, indicating the existence of sites of preferential LBE penetration (Figure 10). Surface coverage by dissolution attack was measured at all analysed cross-sections and it averages more than 97% for all tested specimens. Some of the cross-sections of the 20 and 60% deformed steels exhibited areas not affected by dissolution corrosion and probably protected by a thin Cr-rich oxide scale invisible by SEM.\(^6,15\,19\) The fact that the non-affected areas were observed only at 20% and 60% of cold deformation might be attributed to the stochastic character of dissolution corrosion.

The quantification of the corrosion depth was performed on SEM micrographs at 500× magnification. At least 330 random spots were measured on each of the four different cross-sections per specimen. The results of corrosion quantification are shown in Figure 11. The distribution curve of the 0% deformed specimen is shifted to the left and corresponds to lower dissolution depth values. With increasing degree of cold-work, the frequency of lower dissolution depth values decreases, the curves become flatter and shift to the right, indicating that plastic deformation increases the irregularity in the depth of the dissolution zone and leads to deeper corrosion damages. The average depth of the dissolution zone was defined as an average of all measurements obtained from four cross-sections and it is \(17 \mu m\) for 0%, \(24 \mu m\) for 20%, \(28 \mu m\) for 40% and \(35 \mu m\) for 60% cold deformation.

Since the dissolution front can advance locally quite deep into the steel, it can lead to the local steel degradation and this might be crucial for thin-walled components. Thus, knowing the maximum values of the dissolution corrosion damages is most critical and, therefore, this was chosen as a representative parameter to characterize quantitatively dissolution corrosion. Based on the four analysed cross-sections per each specimen, it was found that in the non-deformed (0%) steel the maximum corrosion depth reached \(40 \mu m\), while in the 20%, 40% and 60% deformed steel reached \(66 \mu m, 79 \mu m\) and \(96 \mu m\), respectively (Figure 12). Therefore, there is a clear trend according to which the maximum dissolution depth increases with the degree of steel plastic deformation.

**Effect of chemical banding**

In this study, it was found that the occurrence of locally-advanced dissolution attack is guided by the presence of certain bands caused by chemical segregation in the steel (Figure 13). The presence of these bands was revealed in all tested specimens, however, the impact on dissolution corrosion was more pronounced in case of the 40% and, especially, the 60% of plastic deformation. EPMA elemental mapping performed on the 60% deformed steel revealed local variations in chemical composition (Figure 14). Quantification of the steel chemical composition was done by an EPMA line scan with a distance between spots of 5 μm and presented as an average per band in Table 2. It was observed that the more severely attacked bands contain appreciably less Ni and slightly more Fe, as compared to the less attacked bands. TEM analysis of a FIB foil taken at the boundary between two different bands (Figures 13 and 15[a]) revealed that the main deformation mechanism in the Ni-poor band I was twinning. This is confirmed by a bright field image close to the \(<110>\) zone axis showing (111) twinning planes (Figure 15[b]). On the other hand, the Ni-rich band II showed dislocation tangles and low-angle tilt boundaries formed by dislocation pile-up (Figure 15[c]).

**Effect of δ-ferrite inclusions**

As already mentioned, the observed δ-ferrite steel inclusions were platelets oriented along the rolling direction. In fact, δ-ferrite forms at high temperatures (>1394 °C), but is retained at low temperatures in the steel microstructure upon solidification from the melt.\(^20\) δ-ferrite transforms into α-ferrite at low temperatures, however, the term δ-ferrite is used here to denote the origin of this secondary phase. The δ-ferrite platelets seem more resilient to the LBE dissolution attack than the steel matrix and their presence and orientation can affect the local advancement of the dissolution front. When the δ-ferrite platelets are oriented along the specimen surface, the progress of LBE dissolution attack is locally delayed (Figures 16[a] and 16[b]), since LBE does not go directly through the platelets but envelops them instead.

**DISCUSSION**

**Effect of steel plastic deformation**

Experimental results obtained in this study show a clear influence of plastic deformation on the dissolution corrosion behaviour of 316L austenitic stainless steels exposed to static liquid LBE at 500 °C, \([O] \approx 1.2 \times 10^{11}\) to \(1.3 \times 10^{13}\) mass% for 1000 hours: increasing the degree of plastic deformation from the non-deformed, solution-annealed state to 60% of plastic deformation increases the maximum depth of the dissolution-affected zone from 40 μm to 96 μm. Based on the experimental findings, the dependence of the maximum depth of the dissolution-affected zone on the degree of steel plastic deformation can be expressed by the following linear function (\(R^2 = 0.96\)):

\[
y = 1.02 \times x + 31.09
\]

where \(y\) is the depth of the dissolution-affected zone in μm and \(x\) is the degree of plastic deformation in %. It can be estimated that increasing the degree of cold-work by 1% results in an increase of the depth of the dissolution-affected zone by 1.02 μm.

It is well-established that plastic deformation of metallic materials is associated with the formation and movement of dislocations. In fcc alloys, deformation is accompanied by slip and twinning\(^21\), which leads to an increase in the amount of defects. Thus, plastic deformation leads to the distortion of the crystal lattice.\(^22\) With respect to the corrosion behaviour from the crystallographic point of view, it was found that non-ordered atoms and atoms located on the step edges of crystal lattice are more favorable to dissolve compared to the close-packed planes.\(^23\)

It has already been reported that microstructural features resulting from the steel plastic deformation facilitate the interaction between steel and liquid LBE.\(^9,10\) It is, therefore, expected that an increase in the degree of steel plastic deformation will be accompanied by a multiplication of defects providing paths of preferential LBE dissolution attack. A larger number of such defects results in deeper dissolution-affected zones within the same timeframe, which is in agreement with the herein observed increase in the dissolution rate with the degree of steel plastic deformation.
According to Yamaki et al., it was found that at 500 °C 316L dissolution kinetics is controlled by the dissolution of Ni in the liquid LBE penetrations at the interface between 316L bulk and ferrite. In the present study it was found that increase degree of cold-work results in the deeper dissolution attack. Based on the results of Yamaki and present study, it can be assumed that the plastic deformation increases the Ni dissolution rate, as it was suggested by Kurata. However, the exact nature of that phenomena is still unclear and, therefore, more detailed microstructural investigation was performed in this work.

The drop in the LBE oxygen concentration at the beginning of the experiment indicates the active oxidation of the specimens after their insertion into the LBE bath. However, oxide scales forming at such low oxygen conditions are probably defective, possibly discontinuous and are not expected to remain protective for long periods of time. It is, therefore, expected that these oxide scales will be unable to prevent for long time the intimate contact between base steel and LBE, which will soon result in the occurrence of dissolution attack.

The results of the work are in agreement with the low-[O] LBE (4.2×10^(-2) wt%) tests of Kurata, where he observed an increase of the depth of the dissolution zone with degree of plastic deformation. The Fe-Cr oxide above the dissolution zone observed in the aforementioned study was not found in this work probably because of the lower [O]. A similar trend was observed by Tsisar et al. on the corrosion behaviour of deformed 1.4970 austenitic stainless steels exposed at 550 °C for 1150 hours to low-[O] (10^(-9) to 5×10^(-11) mass%) LBE.

The herein obtained results differ from the work of Johnson et al. performed at intermediate [O], who reported the positive effect of steel cold-working on the 316/316L steel corrosion resistance due to the formation of a protective Cr-oxide layer. Formation of a double-layer oxide scale and absence of dissolution attack at 550°C in LBE with high [O] (1.2×10^(-4) mass%) for 1000 hours was reported by Kurata. However, the same work reported more severe dissolution attack for cold-worked steels exposed for a longer period of time (3000 hours). These discrepancies were related to the incubation period needed for the degradation of oxide scales and the initiation of LBE dissolution attack.

**Effect of chemical banding**

Chemical banding appears to be quite common for highly-alloyed steels. The reason of its occurrence is the segregation of steel alloying elements during solidification from the melt. The primary solid dendrites that precipitate in the melt have lower concentrations in certain alloying elements and impurities, which in turn segregate in the interdendritic regions, creating chemical inhomogeneities in the steel. Further mechanical treatment, such as rolling, aligns these elemental segregations in the direction of metal flow, resulting in the so-called chemical banding. The steel chemical composition inhomogeneities depend on the steel composition, the primary dendrite structure, time and temperature of soaking and rolling.

Chemical banding often defines the extent of property anisotropy within a steel heat. For example, Michler et al. linked differences in the hydrogen embrittlement behaviour of the same 316L steel heats to the chemical banding phenomenon. To author's best knowledge, the effect of chemical banding on the LMC behaviour of 316L stainless steels was never reported. The correlation between the depth of the dissolution-affect zone and chemical banding, as observed in this study, shows that chemical banding might also have an impact on the steel dissolution corrosion behaviour.

It was revealed that the effect of chemical banding on dissolution corrosion becomes more pronounced when the degree of plastic deformation increases. Chemical banding observed in this study revealed the variations in Ni-content (Figure 14). Bands with lower Ni-content appeared to corrode faster, compared to the bands containing more Ni. Since the steel Ni-content has an impact on the mechanical properties, i.e. lower Ni-content decreases the yield strength and leads to higher work-hardening rate, it can be speculated that bands with different chemical composition deform differently when a steel is cold worked. This speculation can be supported by TEM findings (Figure 15), which revealed that Ni-poor bands have a greater tendency towards twinning, as compared to Ni-rich bands. The difference in deformation can be explained by the influence of the Ni content on the stacking fault energy (SFE) of the material. Nickel has a tendency to increase the SFE, decreasing the probability of forming stacking faults, which are obstacles to dislocation motion and lead to work hardening during the steel plastic deformation. Thus, it can be assumed that a lower steel Ni content is more favourable for twinning, as illustrated in Figure 15(b).

Since dissolution corrosion is driven by differences in the chemical potential of highly-soluble steel alloying elements (e.g., Ni) that cause the transfer of these elements from the solid steel to the liquid metal, it might be expected that steels with higher contents of highly-soluble elements will be attacked faster than steels with lower contents of such elements. Contrary to this general perception, the present study revealed that Ni-rich bands corrode less than Ni-poor bands in the steel. Thus, it can be deduced that the corrosion rate is not solely defined by the steel chemical composition and that defects resulting from the steel thermomechanical treatment may also play a significant role in the steel corrosion behaviour. It has already been reported, for example, that dissolution corrosion is accelerated along deformation twin boundaries forming during the steel plastic deformation. Since this study showed that Ni-poor bands containing more deformation twins undergo more severe dissolution attack, it can be hypothesized that the higher density of twins in these bands accelerates locally dissolution corrosion. Therefore, in this case, microstructural defects also modify the rate of dissolution corrosion than the steel chemical composition. This hypothesis requires further verification by a more in-depth material characterisation, so as to fully understand the influence of macrochemical segregations on the steel corrosion behaviour in LBE.

**Effect of δ-ferrite inclusions**

This work revealed that δ-ferrite platelets in the microstructure of an industrial-size 316L steel heat were less affected by dissolution corrosion than the steel matrix and can locally affect the propagation of the dissolution front. The resistance of δ-ferrite to LBE dissolution attack could be associated with the significantly lower concentration of highly-soluble elements (Ni, Mn, and Cr) compared to the steel bulk. δ-ferrite plate is a more difficult to dissolve obstacle with respect to the steel matrix around it, thus delaying locally the progress of LBE dissolution attack. In order to advance, the dissolution corrosion process bypasses the δ-ferrite inclusions, eventually enveloping them (yellow arrows in Figure 16(b)). Based on the above, it is essential to know the steel microstructure in terms of the (both size and spatial) distribution and orientation of secondary steel precipitates so as to fully understand its in-service corrosion behaviour. How desirable or allowable secondary precipitates are in a steel intended for nuclear applications must be carefully evaluated, as secondary phases may act as stress concentrators (possibly detrimental in irradiation-hardened thin-walled steel components) or might be responsible for differential swelling effects, etc.

**CONCLUSIONS**

- In this study, the effect of cold deformation on the 316L austenitic stainless steel dissolution behaviour was investigated. Low-[O] LBE was used to promote dissolution corrosion in the exposed steel
specimens. Specimens with 0% (solution-annealed), 20%, 40% and 60% of plastic deformation were simultaneously exposed to static, oxygen-poisoned (O\textsubscript{2} < 10\textsuperscript{-12} mass%) LBE at 500 °C for 1000 hours.

- All specimens exhibited LBE dissolution corrosion attack. The dissolution-affected zone was depleted in Cr, Ni and Mn and probably consisted of ferritized matter penetrated by LBE. The thickness of the dissolution-affected zone was non-uniform and averaged 17 μm for 0%, 24 μm for 20%, 28 μm for 40% and 35 μm for 60% of cold deformation. The maximum depth of LBE dissolution attack significantly increased with increasing the amount of cold deformation: 40 μm for 0%, 66 μm for 20%, 79 μm for 40% and 96 μm for 60% cold deformation. Therefore, it can be concluded that, for the exposure conditions explored in this work (500 °C, 1000 hours, static LBE with [O] < 10\textsuperscript{-12} mass%), a higher degree of plastic deformation in 316L stainless steels results in more severe LBE dissolution attack.

- The heat of 316L steel used in this work appears to have chemical banding microstructure. Therefore, the effect of chemical banding on the dissolution corrosion behaviour of 316L steels was also addressed in this work. It was found that chemical banding had a greater impact on the steel dissolution corrosion behaviour for 40% and even more pronounced for 60% deformed bands. Bands with lower Ni content appeared to corrode faster, compared to bands with higher Ni content. This was herein associated with differences in the deformability of bands with appreciably different Ni contents, due to differences in the type of defects formed during the steel plastic deformation. However, this hypothesis requires further verification.

- It was also observed that δ-ferrite inclusions have an impact on the local propagation of the dissolution front. δ-ferrite inclusions tend to delay locally the advancement of the dissolution front, especially if they are oriented along the steel surface.

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REFERENCES

FIGURE CAPTIONS

FIGURE 1. Tensile specimen geometry; all dimensions are given in mm.
FIGURE 2. Stress-strain curves of tensile tests performed to 20, 40, 60% of 316L steel cold deformation.
FIGURE 3. Schematic representation of the experimental setup used for corrosion testing.
FIGURE 4. DIC LOM images of δ-ferrite inclusions in the non-deformed specimen (a) perpendicular and (b) parallel to the steel rolling direction.
FIGURE 5. Non-polarized LOM images of chemical banding (a) and corresponding etched area (b) in the non-deformed steel.
FIGURE 6. EBSD orientation maps and inverse pole figures of non-exposed steels with (a) 0%, (b) 20%, (c) 40% and (d) 60% cold deformation.
FIGURE 7. LBE oxygen concentration and temperature during the corrosion experiment.
FIGURE 8. SEM micrographs of the dissolution-affected zone in (a) 0%, (b) 20%, (c) 40% and (d) 60% cold-deformed specimens after their exposure to oxygen-poor ([O] < 10^{-12} mass%) static LBE at 500 °C for 1000 hours.
FIGURE 9. SEM image (a) and EDS line scan (b) of the dissolution-affected zone in the 60% deformed specimen exposed to oxygen-poor ([O] < 10^{-12} mass%) static LBE at 500 °C for 1000 hours.
FIGURE 10. Preferential sites for LBE dissolution attack in the (a) 60%, and (b)-(c) 40% cold deformed steel.
FIGURE 11. Distribution of the depth of the dissolution zone as a function of the degree of steel plastic deformation.
FIGURE 12. Maximum depth of dissolution attack as a function of the degree of steel plastic deformation.
FIGURE 13. Non-polarized LOM picture showing the correlation between chemical banding and dissolution corrosion damages in the 60% deformed steel.
FIGURE 14. EPMA elemental maps show the correlation of chemical bands with the shape of the dissolution-affected zone in the 60% deformed steel.
FIGURE 15. TEM results obtained from different bands: I – more corroded, Ni-poor band; II – less corroded, Ni-rich band.
FIGURE 16. DIC LOM images showing the role of δ-ferrite inclusions on the local propagation of the dissolution front.
### TABLE 1
Chemical composition of the 316L austenitic stainless steel used in this study (mass%).

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<tr>
<th></th>
<th>Fe</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Cu</th>
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<tr>
<td>Bulk</td>
<td>0.0185</td>
<td>0.67</td>
<td>1.81</td>
<td>0.032</td>
<td>0.0035</td>
<td>16.73</td>
<td>9.97</td>
<td>2.05</td>
<td>0.23</td>
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### TABLE 2
EDS results showing the chemical composition of different bands in the exposed steel specimens (mass%).

<table>
<thead>
<tr>
<th>Band</th>
<th>Fe</th>
<th>Cr</th>
<th>Ni</th>
<th>Mn</th>
<th>Si</th>
<th>Mo</th>
</tr>
</thead>
<tbody>
<tr>
<td>Less attacked</td>
<td>66.2</td>
<td>17.2</td>
<td>10.4</td>
<td>3.3</td>
<td>0.7</td>
<td>2.0</td>
</tr>
<tr>
<td>More attacked</td>
<td>68.1</td>
<td>17.4</td>
<td>8.2</td>
<td>3.4</td>
<td>0.6</td>
<td>1.8</td>
</tr>
</tbody>
</table>
FIGURE 1. Tensile specimen geometry; all dimensions are given in mm.
FIGURE 2. Stress-strain curves of tensile tests performed to 20, 40, 60% of 316L steel cold deformation.
FIGURE 3. Schematic representation of the experimental setup used for corrosion testing.

Steel autoclave

Ar+5% H₂ gas

Thermocouple

LBE

Specimens

Heater

Bi/Bi₂O₃ oxygen sensor

Alumina crucible

Thermal insulation
FIGURE 4. DIC LOM images of δ-ferrite inclusions in the non-deformed specimen (a) perpendicular and (b) parallel to the steel rolling direction.
FIGURE 5. Non-polarized LOM images of chemical banding (a) and corresponding etched area (b) in the non-deformed steel.
FIGURE 6. EBSD orientation maps and inverse pole figures of non-exposed steels with (a) 0%, (b) 20%, (c) 40% and (d) 60% cold deformation.
FIGURE 7. LBE oxygen concentration and temperature during the corrosion experiment.
FIGURE 8. SEM micrographs of the dissolution-affected zone in (a) 0%, (b) 20%, (c) 40% and (d) 60% cold-deformed specimens after their exposure to oxygen-poor ([O] <10^{-12} mass%) static LBE at 500 °C for 1000 hours.
FIGURE 9. SEM image (a) and EDS line scan (b) of the dissolution-affected zone in the 60% deformed specimen exposed to oxygen-poor ([O] < 10^{-12} mass%) static LBE at 500 °C for 1000 hours.
FIGURE 10. Preferential sites for LBE dissolution attack in the (a) 60%, and (b)-(c) 40% cold deformed steel.
FIGURE 11. Distribution of the depth of the dissolution zone as a function of the degree of steel plastic deformation.
FIGURE 12. Maximum depth of dissolution attack as a function of the degree of steel plastic deformation.
FIGURE 13. Non-polarized LOM picture showing the correlation between chemical banding and dissolution corrosion damages in the 60% deformed steel.
FIGURE 14. EPMA elemental maps show the correlation of chemical bands with the shape of the dissolution-affected zone in the 60% deformed steel.
FIGURE 15. TEM results obtained from different bands: I – more corroded, Ni-poor band; II – less corroded, Ni-rich band.
FIGURE 16. DIC LOM images showing the role of δ-ferrite inclusions on the local propagation of the dissolution front.