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Investigation of Martensite Transformation in 316L Stainless Steel

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Abstract

A large number of parameters affect martensite formation in steel, such as composition, applied stress, plastic strain and temperature. Low cycle fatigue tests on 316L austenitic stainless steel have been carried out with different strain amplitudes at room temperature and cryogenic temperatures. The influences of deformation and temperature have been investigated by carrying out in-situ and ex-situ neutron diffraction measurements in the ENGIN-X diffractometer at ISIS (UK) to analyze the volume fraction and strain in the martensite phase. Increasing plastic deformation and decreasing temperature were shown to promote martensite transformation and increase the hardness of the material. The influence of the appearance of martensite due to deformation and temperature will be discussed.

Keywords: Martensite transofrmation, Stainless Steel, Neutron diffraction, Low cyle fatigue

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1. Introduction

Austenitic stainless steels make up over 70% of the total stainless steel production worldwide. They contain a minimum of 16% chromium, and sufficient nickel and/or manganese to retain an austenitic structure at all temperatures. Chromium creates a protection against oxidation and prevents corrosion in humid conditions. The crystallographic structure of austenite is a face centred cubic (fcc) unit cell characterized by 12 slip systems (3 directions on 4 slip planes). Plastic deformation occurs by slip parallel to the closest packed plane {111} and in the direction <110> where the shortest distance separates two atoms. Martensite transformation represents the phase change from austenite to martensite that may form by twinning or slip, with both deformation modes associated with shear. Martensite transformation can be visualized as a shear of the parent phase into the product phase that conserves an invariant habit-plane in the lattice structure. This transformation is diffusionless, meaning that the chemical composition of the austenite (parent phase) and martensite (product phase) are identical. The displacive transformation does not require long-range diffusion of atoms, but rather occurs by a form of cooperative, "military" movement of many atoms that results in a change in the crystal structure. These movements are small, usually less than the interatomic distances, and the atoms maintain their relative disposition. However, this movement of atoms can give rise to three different crystal structures of martensite: Body-centred tetragonal structure (bct) defined as α'martensite, Body-centred cubic (bcc) defined as α'martensite and Hexagonal close-packing (h.c.p) defined as ε martensite. In steels, the carbon atoms play an important role in deformation of lattice on bct relative to bcc structure. The carbon atoms seats in interstitial sites, it can induce a distortion in the lattice. The martensite tends to be tetragonal in Fe-C steel with high carbon rate and cubic for stainless steels with low carbon rate. In Fe-alloys, the ε martensite present phase is mainly due to low stacking fault energy in close–packed plane and tends to proceed the α' formation [1]. For this reason, the ε phase is often described as metastable intermediate phase. Dash and Otte [2] showed that ε phase is not necessarily an intermediate phase but rather a consequence of low stacking fault energy. Generally for the indirect transformation, the formation of α'martensite occurs at the intersection of two ε plates, but might also appears at intersection between one ε plate and one slipping plane or by intersection of a ε plate with twin [3]. According to the Burgers and Bogers model [4] with the double-shear theory to induce fcc to bcc transformation, the nucleation site at the intersection of two shear-bands is a favourable site for the initiation of martensite growth.

A number of parameters exert great influence on the martensite formation, such as steel composition, applied stress, plastic strain and temperature. The stability of the austenitic phase depends on the composition of stainless steel - low content of Nickel, Chromium or Carbon reduce the stability of austenite and increase the martensite start temperature. The deformation induced by the applied load can supply the necessary strain energy to assist martensite formation, and also introduce defects to promote the nucleation of martensitic grains. Further reduction of temperature provides a large chemical driving force that promotes martensite formation. Deformation-induced martensite formation is a thermo-mechanical phenomenon, whereby the transformation of the austenite phase into martensite occurs above the martensite start temperature due to deformation assistance. The crystallographic structure of martensite is not dense and can have many more slip planes (up to 48 slip planes). Heat is required in order to activate the slip system. For low temperature and sometimes at room temperature, the lack of slip systems can render the material brittle. This paper reports the results of the investigation of martensite formation as a function of strain and temperature history using neutron diffraction.

Neutron diffraction is an attractive non-destructive method for measurement internal stress and phase composition in polycrystalline materials. In-situ neutron diffraction tests have been performed on ENGIN-X diffractometer at the ISIS spallation neutron source in Rutherford Appleton Laboratory (UK) to try and observe the formation of martensite during low cycle fatigue at room temperature and at cryogenic temperatures. ENGIN-X is the only diffractometer in the world that can perform in-situ fatigue tests at cryogenic temperature. Low cycle fatigue tests at room temperature and cryogenic temperatures together with neutron diffraction have been used to correlate the influence of temperature and strain amplitude on the formation of martensite.

2. Experimental setup

The material used in this project was 316L austenite stainless steel having the following chemical composition in weight percent by mass: Ni 10.02%, Cr 16.72%, Si 0.43%, Mn 1.75%, C 0.017%, Mo 2.04%, Cu 0.56%, N 0.0514% and Fe-balance. In-situ low cycle fatigue tests (LCF) with 1.1% strain amplitude were carried out at ambient and cryogenic temperatures using the time-of-flight strain scanner ENGIN-X [5] at the ISIS spallation neutron source. The specimen had a gauge length of 14mm, diameter of 8mm and threaded ends on both sides. All the low cycle fatigue tests have been performed with triangular waveform and a strain rate of 2.2×10^{-3} s^{-1} to allow easy comparison of the results. A 100kN Instron stress-rig was placed horizontally with an angle of 45° to the incident neutron beam and two detector banks has fixed scattering angles of ±90° to the incident neutron beam. A cryostat was mounted on the stress-rig to provide cooling for the samples required for the cryogenic LCF tests. Diffraction patterns parallel and perpendicular to the loading axis were collected simultaneously.

Fracture surfaces of the different specimens after LCF tests with different strain amplitude have also been measured with neutron diffraction to determine the amount of martensite present. Since there is a direct relationship between stress and martensite formation, the fracture region should contain the highest level of martensite.

The diffraction patterns obtained were analysed using the in-house Open Genie software, which invokes the General Structure Analysis Software (GSAS). From the data, the presence of austenite and martensite phase could be identified. Lattice strains can be extracted from the shift of recorded diffraction peaks using single peak fitting analysis.

3. Results and Discussion

3.1 Stress/strain influence on martensite formation

3.1.1 In-situ neutron measurement with low cycle fatigue at 1.1% strain amplitude at room temperature

Low cycle fatigue test at room temperature has been carried out in strain-controlled at $\Delta\varepsilon_t/2 = 1.1\%$ strain amplitude, where the sample has undergone a plastic deformation during each cycle to deform by the total strain of +1.1% in tension and -1.1% in compression.
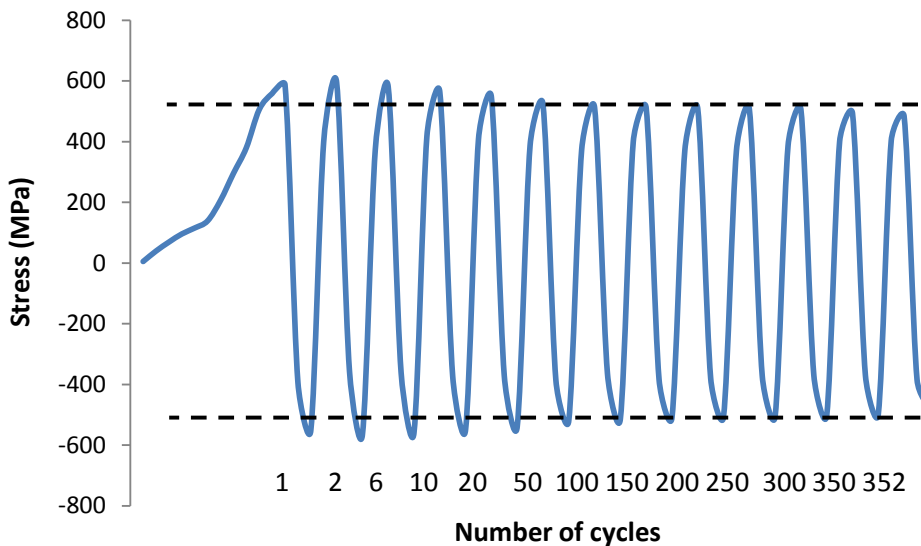

Fig. 1 Stress evolution for low cycle fatigue test at room temperature with $\Delta\varepsilon t/2 = 1.1\%$ strain amplitude

Fig. 1 depicts the stress evolution with the number of cycles during the test. Up to the 150th cycle, the stress evolution appears to correspond to softening. Similar behaviour of stainless steel has been observed in the literature

[6-8]. It has been reported that cyclic softening is associated with the annihilation of dislocations during plastic deformation. Whilst initially the monotonic plastic deformation causes dislocation nucleation, during subsequent reverse cycling the rate of dislocation nucleation competes with the interaction between dislocations already present in the material that may lead to dislocation density reduction due to rearrangement, dislocations escaping to the sample surface, and annihilation. Reduction in dislocation density also reduces flow stress, causing apparent softening [9]. However, the curve shows a change in the trend of stress evolution between 150th to 350th cycle where a reduction in the rate of softening occurs. This stage of very moderate softening produces a plateau of nearly constant stress that persists until at the 350th cycle the stress starts to decrease rapidly until failure.

Fig. 2 shows the diffraction patterns for sample prior to deformation and during 200th cycle. As indicated in Fig. 2b, the beginning of martensite formation is apparent in the sample. The martensite possesses a bcc structure, so that the (110), (200) and (211) diffracting planes produce the most intense peaks. The 200th cycle seems to correspond approximately to the stabilisation of the cyclic stress, of the moderate softening stage, as indicated in Fig. 1. The fact that martensite was not detected at 150th cycle could be due to the amount of martensite being below the detection threshold (<1%). Even at the 200th cycle, the low intensity of the (110) peak means that the amount of α'martensite formed is still very low.
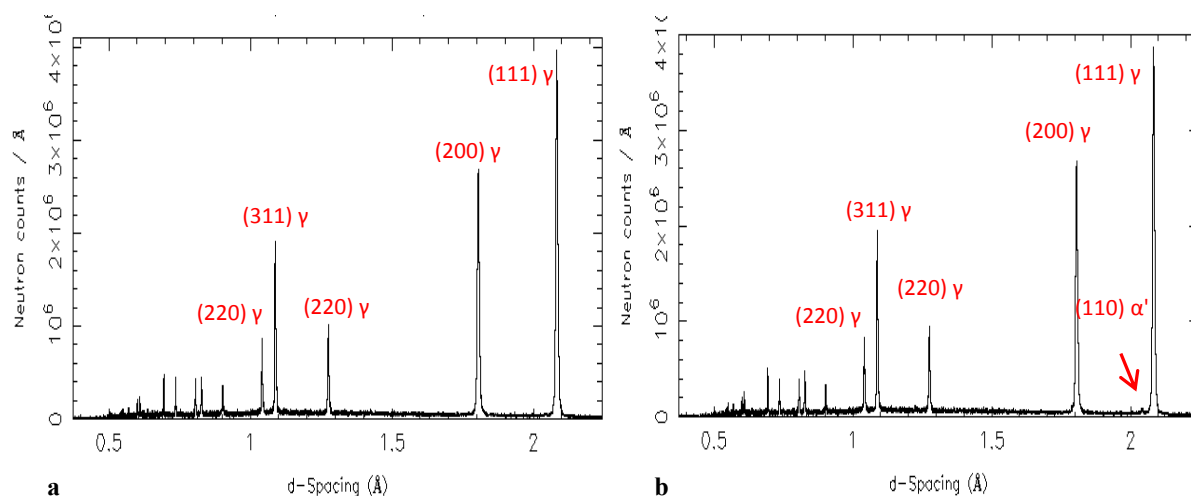

Fig.2. Diffraction patterns of 316L stainless steel: (a) no deformation (b) at 200th cycle

In the literature [6-8] martensite formation has been shown to lead to substantial cyclic hardening of the material and to a decrease in its fatigue life. No hardening has been observed on our test. It is due to the fact that the amount of martensite is too small to produce hardening. However, when martensite has been observed around the 200th cycle, the softening rate was reduced, as shown in Fig. 1.

The structure of sample was analysed by neutron diffraction throughout the low cycle fatigue test. In Fig. 3 the lattice strains are plotted as a function of the number of cycles, with values taken being similar to those in Fig. 1. Since the lattice planes are oriented differently, the load sharing between them is non-uniform, so that the material displays strain anisotropy. The elastic strain parallel to the deformation direction produced at each cycle follows the same trend as the stress. The strain range is reduced for the (111) reflection because the material has the highest stiffness is the direction of this plane's normal.
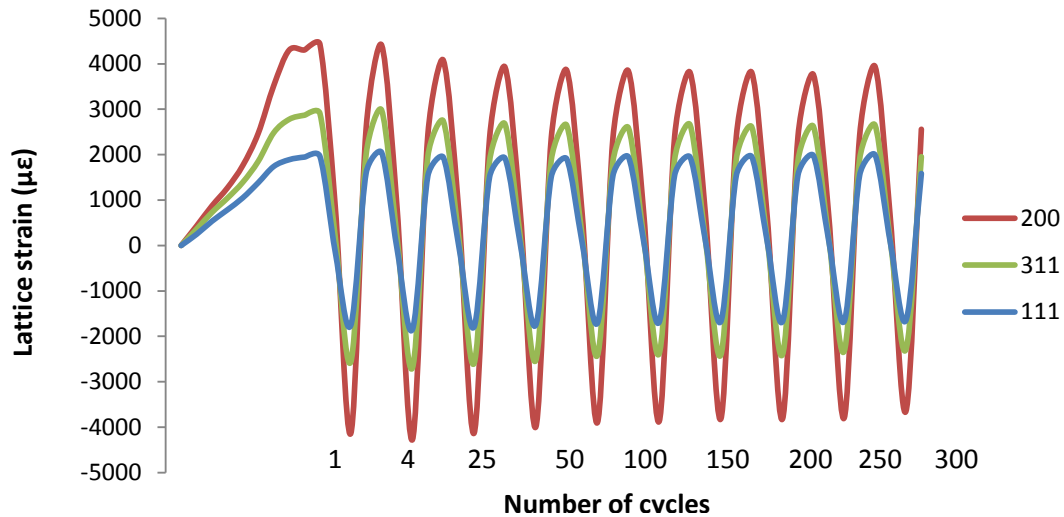
Fig. 3 Variation of the lattice strain in a low cycle fatigue test at room temperature with the $\Delta\varepsilon_t/2 = 1.1\%$ strain amplitude.

3.1.2 Low cycle fatigue with different strain amplitude

Seven low cycle fatigue tests we conducted with strain amplitude at $\Delta\varepsilon_t/2 = 0.5\%$, 0.7%, 0.9%, 1.1%, 1.3%, 1.5% and 2%. Fig. 4 shows the maximum macroscopic loading stress ($\Delta\sigma/2$) plotted versus the number of cycles on a semi-logarithmic diagram. In this figure, three different types of response can be observed. Samples deformed with the strain amplitude of 0.5% to 1.1% ($\Delta\varepsilon_t/2$) exhibit softening throughout the test. Between the strain amplitude of 1.3% and 1.5%, the softening tends to saturate. At the strain amplitude of 2%, no softening occurs, whilst significant secondary hardening is also observed. It is also interesting to note that the 1.3% strain amplitude test exhibits stress amplitude higher than the 1.5% strain amplitude test for the first ten cycles. It was found that this was due to the particular sample having a higher Young's modulus than other samples, found to be equal to 193GPa for this sample.

Neutron measurements were carried out on the fracture surfaces of these seven samples after fatigue at different strain amplitude. The fracture area was expected to have larger amounts of martensite comparing to other positions within the sample. Martensite phase is harder, but also more fragile; consequently, the heterogeneous plastic deformation may promote the formation of martensite at this location. Fig. 5 shows the intensity evolution of the martensite peak (110) α' with the fatigue strain amplitude. It is clear that the (110) α' peak intensity increases with the strain amplitude. The intensity of the peak correlates positively with the volume fraction of martensite at the fracture surface. It can be concluded that the quantity of martensite increases with the strain amplitude but remains well below the amount of the austenite.

In the literature [6-8], the effect of martensite formation has been shown to lead to substantial cyclic hardening of the material. The diffraction measurements have shown that the quantity of martensite increases with the cyclic strain amplitude. Although no real cyclic hardening is observed in fatigue tests with the strain amplitude less than 2%, the tests with the strain amplitude of 0.5% to 1.5% have shown reduced softening. The decrease in the softening slope is attributed to the progressive appearance of martensite. However, the amount of martensite is too low to produce hardening. In the in-situ fatigue test with the strain amplitude of 1.1%, when martensite was revealed by neutron diffraction during the test, the softening rate was changed and only a slight softening occurs (Fig. 1). These results are consistent and demonstrate that martensite formation evolves with the increase of strain amplitude and increases the hardness of the material.
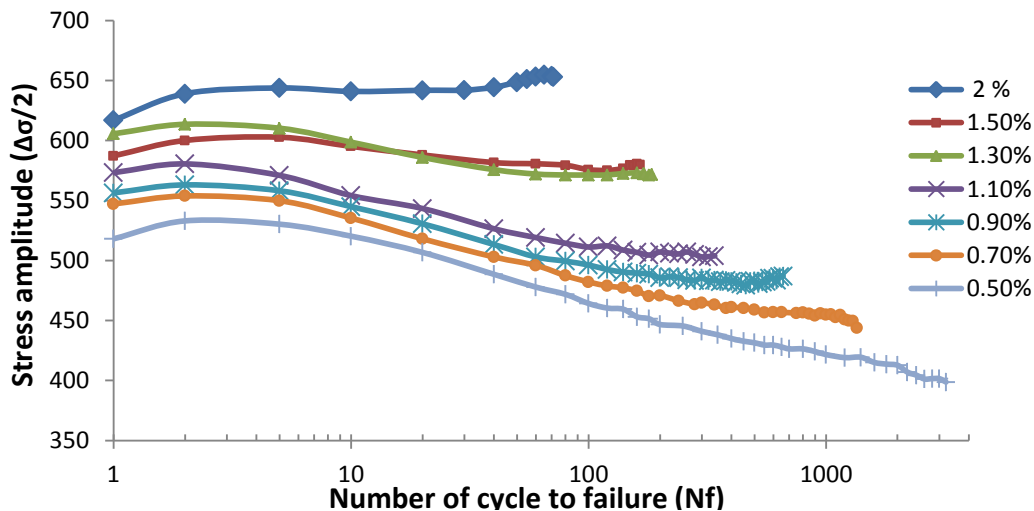
Fig. 4. Stress evolution (Δσ/2) in function of cycle life (Nf)
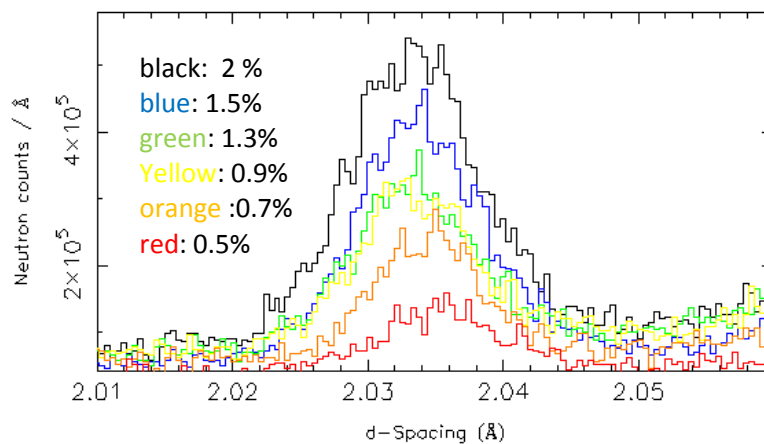
Fig. 5. Intensity evolution of the martensite peak (110) α' with different strain amplitudes

3.2   The influence of temperature on the martensite formation

3.2.1 In-situ neutron measurement with low cycle fatigue at the 1.1% strain amplitude at -170°C

Temperature is the most important parameter that influences the activation of martensite transformation. Temperature is known to promote the martensite transformation by increasing the chemical driving force. In-situ neutron measurement was performed at -170°C to evaluate the influence of temperature on fatigue behaviour. In contrast to room temperature, the phenomenon of softening didn't occur and hardening occurred from the first cycle. The hardening slope was more pronounced between cycle numbers 10 and 100, as shown in Fig. 6. The maximum stress increases much to higher values than in the room temperature tests, and the increase is more rapid.
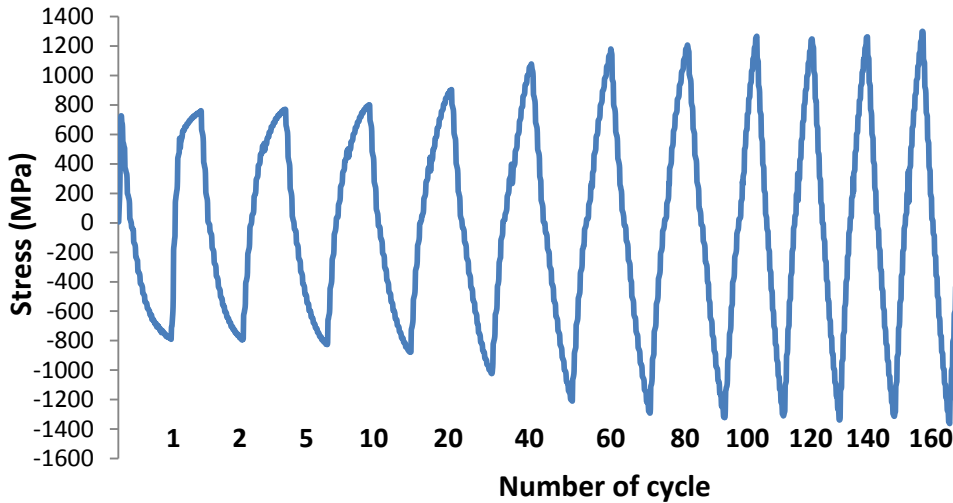
Fig. 6. Stress evolution for low cycle fatigue test at -170°C with Δεt/2 = 1.1% strain amplitude

The increased samples stress is evidently associated with the martensite formation inside the material. The diffraction pattern in Fig. 7 shows that at cycle 10, α'martensite is already present in large quantities and that also the presence of hexagonal martensite must be noted. Hexagonal martensite appears when the stacking fault energy is low enough. According to Remy and Pineau [10], the stacking fault energy decreases with decreasing temperature which explains the appearance of ε martensite at low temperature.
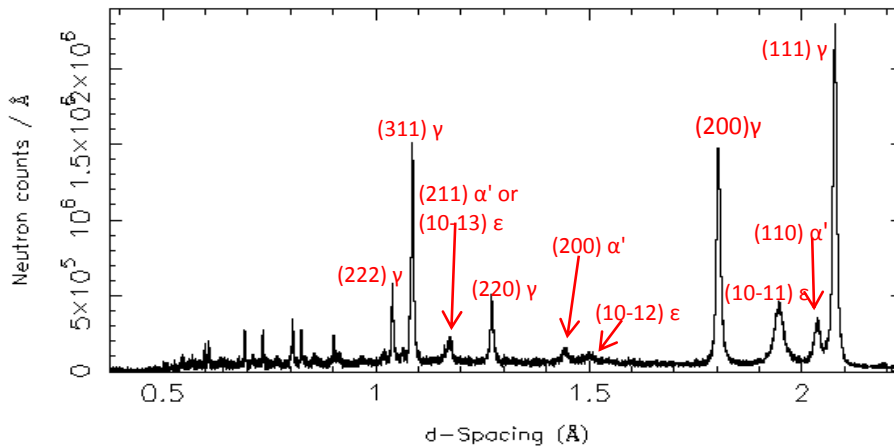

Fig.7. Diffraction pattern of 316L stainless steel during fatigue cycle 10 at -170°C

At cycle 100 in Fig. 8, the intensity of peaks of austenite becomes strongly reduced, whilst the (110), (200) and (211) peaks of α'martensite grow. In agreement with the curve for stress vs cycle number, the martensite volume fraction (peak intensity) increases slowly after cycle 100, and this causes a reduction of stress slope in Fig. 6. Between cycles 100 and 150, martensite formation begins to stabilise since a large quantity of austenite has already been transformed. The maximum tensile stress reached is around 1250 MPa after 100 cycles of deformation. However, the increase of sample stress is not the only consequence of martensite transformation: plastic strain is also affected by.

Fig. 9 depicts the stress-strain curve, which shows a decrease of plasticity with the number of cycles. The correlation between martensite and reduction of ductility are readily seen by comparing the increase of martensite

intensity peak with the stress-strain curve. At cycle 2, the α'martensite is not yet present in sample and the reduction of the austenite peak intensity is essentially due to the appearance of hexagonal martensite. The plasticity is not affected and the material does not undergo hardening. The ε martensite starts to be well detected by the detector at +0.9% of deformation and does not grow after the cycle 10, as shown in Fig. 10. At cycle 10, amount of ε martensite remains greater than the α'martensite and there is little change in plastic strain. Between cycle numbers 10 and 50, the austenite peak intensity (111) decreases quickly and it is compensated by the formation of α'martensite.

During the first cycle the partitioning of the total strain is partitioned into plastic strain of 1.34% and the elastic strain of 0.86%. After several cycles and the formation of significant amounts of α'martensite, the plastic strain is reduced to 0.52% and elastic strain to 1.68% at cycle 100. In conclusion, the plastic strain of austenite is reduced during the experiment and is compensated by the elastic strain with the increasing amount of α'martensite. The α'martensite is more brittle than austenite due to its bcc lattice structure. This result shows the reduction of ductility that ultimately leads to the ductile to brittle transition of 316L stainless steel by reducing of its ability to undergo plastic deformation.
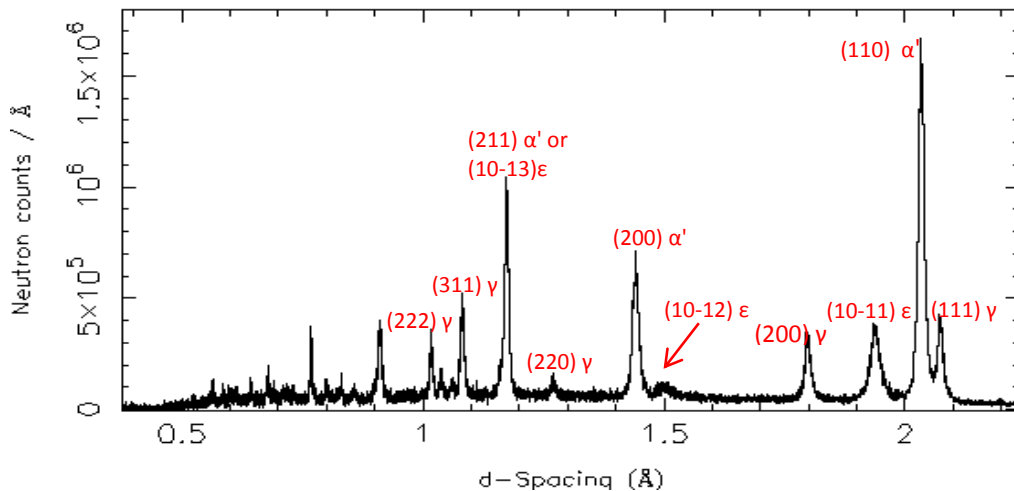

Fig. 8. Diffraction pattern of 316L stainless steel during fatigue cycle 100 at -170°C
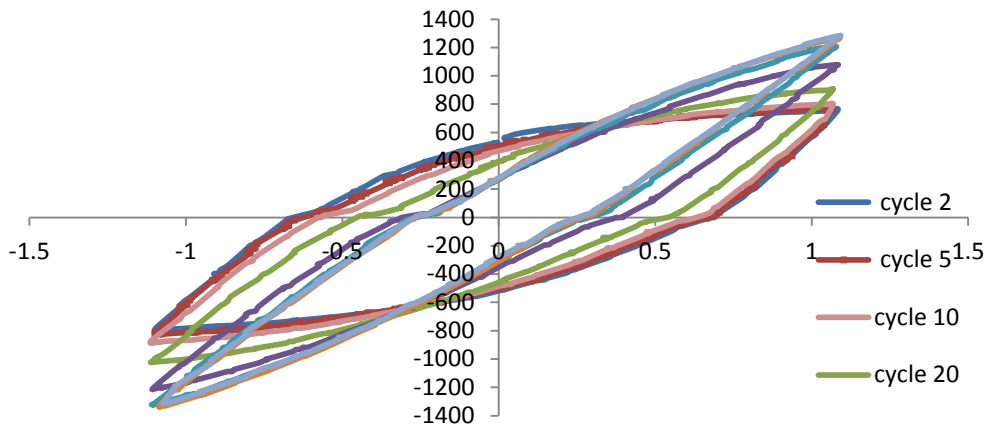

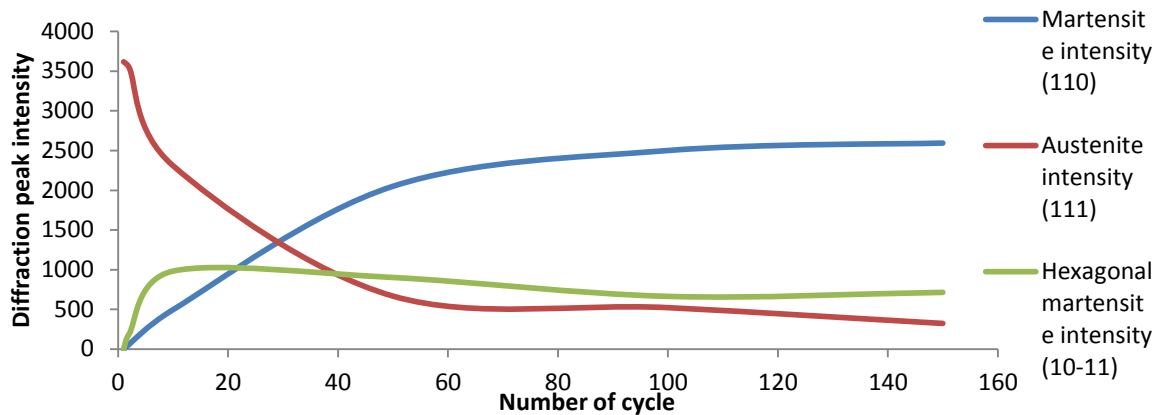Fig. 9. Stress versus strain during low cycle fatigue test at -170°C

Fig. 10. Evolution of intensity of diffraction peak for each phase (martensite, hexagonal martensite and austenite) during low cycle fatigue test at -170°C

3.2.2 *Low cycle fatigue with different temperatures*

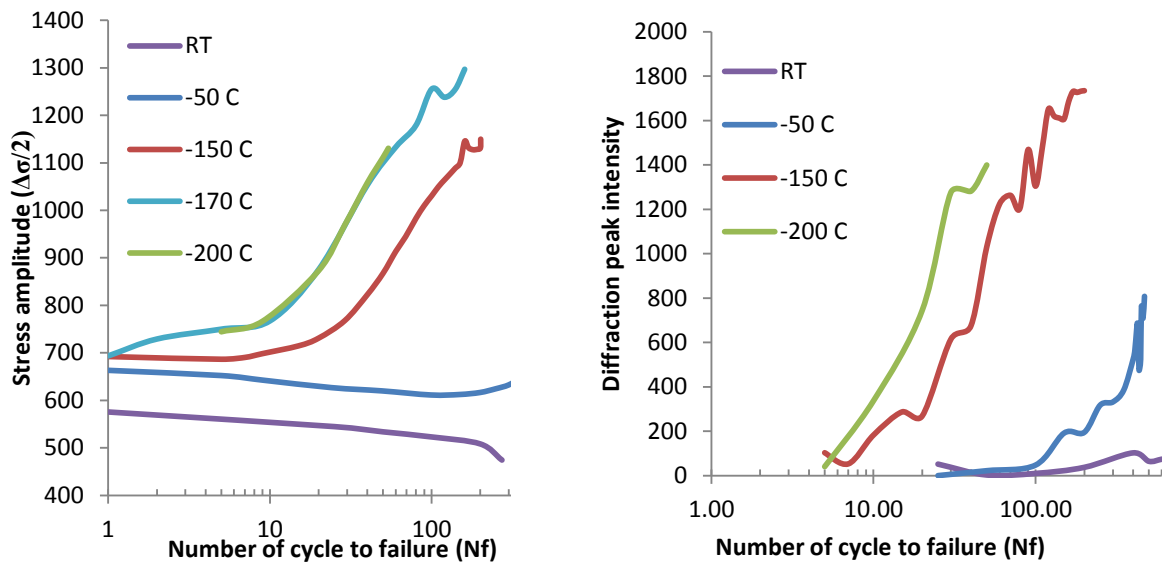Fig. 11. (a) Stress evolution (Δσ/2) (b) Intensity evolution in function of cycle life (Nf) at room temperature and cryogenic temperatures

Temperature is the most important parameter affecting the formation of the martensite phase. Koistinen and Marburger [11] demonstrate that the fraction of formed martensite is directly related to temperature. Fatigue tests have been carried out at ambient temperature, -50°C, -150°C, -170°C and -200°C. Fig. 12(a) shows the evolution of the maximum stress through the fatigue cycles at different test temperatures. It is obvious that low temperature deformation promotes strain hardening. The room temperature test shows stress softening throughout the entire experiment. The tests at -50°C show stress softening until the 100th cycle, at which point strain hardening appears as the result of the creation of martensite. The tests at -150°C, -170°C and -200°C show strain hardening from the beginning of the experiment throughout the whole fatigue cycle life. These are the temperatures at which the formation of martensite is thermally favoured, as shown by Kaufmann and Cohen [12]. The curves for -170°C and -200°C overlap perfectly, which shows that the hardening of the material cannot be increased further. Thus, the rate

of formation of martensite probably reaches its maximum after -170 °C. Between -150°C and -170°C there must exist a temperature beyond which the martensite volume fraction cannot be increased any further. Fig. 12 shows the intensity evolution of the martensite peak through the fatigue cycles for tests at different temperatures. The trend of the intensity evolution corresponds to the strain hardening, as shown in Fig. 11**.**

4. Conclusion

Different low cycle fatigue tests on the samples of 316L austenitic stainless steel have been carried out as a function of strain amplitude and temperature. Martensitic transformation was the main cause of the variation in behaviour of this material under fatigue testing and was observed both at room and cryogenic temperatures. In-situ and ex-situ measurements were carried out by neutron diffraction in the ENGIN-X diffractometer at ISIS to analyse martensite phase in the material.

Low cycle fatigue tests at room temperature with strain-controlled amplitudes of $\Delta\varepsilon_t/2 = 0.5\%$, 0.7%, 0.9%, 1.1%, 1.3%, 1.5% and 2% showed that martensite formation evolves with the increase of strain amplitude. The amount of martensite is small (<3% for test with strain amplitude of 0.5% and <10% for test with strain amplitude 2%). Samples deformed at the 0.5% to 1.1% strain amplitude ($\Delta\varepsilon_t/2$) exhibit softening throughout the test. Between 1.3% and 1.5% strain, softening tends to saturate and at 2% softening does not occur and secondary hardening is observed. Although there is no real hardening observed for tests with strain amplitude less than 2%, the decrease of softening observed was attributed to the appearance of martensite.

Temperature is known to promote the martensitic transformation by increasing the chemical driving force. After the low cycle fatigue tests at cryogenic temperatures, the material contains three phases, austenite, α'martensite and ε martensite. During the fatigue test, the α'martensite appears very rapidly in large quantity (greater than the austenite phase). The rate of strain hardening is large and the maximum stress before failure reaches 1300MPa for the test at -170°C (maximum stress is 800MPa at the first cycle). Hexagonal martensite is also detected but the concentration does not grow after the initial deformation, although it may promote the transformation. The structural transformation from fcc to bcc influences the ductility of austenite and results in decreasing the plastic deformation capability of the material. There also seems to be a temperature below which the formation of martensite cannot be increased by further temperature reduction. This temperature is found to lie between -150°C and -170°C for this material.

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