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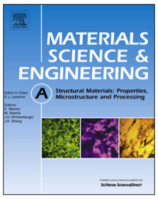

Assessment of dislocation density in asymmetrically cyclic loaded non-conventional stainless steel using X-ray diffraction profile analysis

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ABSTRACT

Asymmetric stress-controlled fatigue i.e., ratcheting behaviour of a non-conventional stainless steel X12CrMnNiN17-7-5 has been investigated with varying mean stresses, stress amplitudes and number of cycles at room temperature using a servo hydraulic universal testing machine. The X-ray diffraction profile analysis using the modified Williamson–Hall equation has been carried out in order to estimate the dislocation densities in the specimens subjected to ratcheting deformation. Increase in strain accumulation has been explained by the increase in dislocation densities in the ratcheted specimens and a correlation between the strain produced by ratcheting deformation and the estimated dislocation density has been established.

1. Introduction

Stainless steels are the candidate material for applications in chemical, process and power generation industries owing to its excellent combination of mechanical properties, oxidation and corrosion resistance under monotonic and cyclic loading conditions [1]. Amongst the numerous grades of stainless steels, the austenitic stainless steels are class of materials having face centred cubic lattice structure which is stable from room temperature to the melting point. X12CrMnNiN17-7-5 (ISO/TR 15510) is a non-conventional special grade of austenitic stainless steel that was developed to conserve Ni and is potentially used for making components such as trim, wheel covers, flat conveyer chains, railroad passenger car bodies etc. The steel is used in architectural applications such as doors, panels, window and frame. In such applications, deformation of the components is primarily dictated by various kinds of fatigue like high and low cycle fatigue, asymmetric stress-controlled fatigue etc. The asymmetric stress-controlled fatigue (also known as ratcheting) takes place when components deform under the influence of positive or negative mean stress (σ_m) by accumulating positive or negative plastic strain (ϵ_p) in the structure, respectively. Therefore, it is a critical issue and emphasis must be laid to understand the ratcheting behaviour of the X12CrMnNiN17-7-5 stainless steel.

It is established that stainless steels undergo phase transformation upon deformation [2,3] and the same is expected to take place in this steel as well. The ratcheting deformation of materials takes place owing to the variations in microstructures and the dislocation density in it during the course of load application. However, the number of literature relating the microstructural variations and the dislocation density with the strain accumulation during ratcheting deformation is limited. In addition, the ratcheting behaviour varies with the applied mean stresses (σ_m), stress amplitudes (σ_a) and number of cycles (N) since the microstructures and the dislocation densities in the specimens change with these. Therefore, the estimation of dislocation densities in the tested specimens in each condition would be of immense importance to provide a clear picture of the extent of deformation induced in the specimens.

Dislocation density in cyclically deformed materials can be measured by direct methods like Transmission Electron Microscopy (TEM) and indirect methods like X-ray or neutron diffraction [4–6]. The direct methods reveal the microstructural information in an extremely small area of specimen, whereas the indirect methods reveal the average data over a relatively large area exposed to irradiation. The preparation of specimen for TEM study is difficult and complicated. In addition, the thinness of TEM specimen sometimes results in a low dislocation density [7]. On the contrary, the specimen preparation for studying X-ray diffraction is easy and less time consuming. In addition, the tests can be performed over a bulk specimen that is suitable for estimating dislocation density of a material more precisely. Accordingly, X-ray diffraction profile analysis is used for various specimens such as

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bulk single and polycrystalline materials; polycrystalline powders and single phase materials to reveal various information such as dislocation density, dislocation character and crystal size [8–10].

In the present investigation, asymmetric stress-controlled fatigue (ratcheting) behaviour of a non-conventional stainless steel has been studied with varying mean stresses (σ_m), stress amplitudes (σ_a) and number of cycles (N). The X-ray diffraction profile analysis has been carried out in order to estimate the dislocation densities in the specimens subjected to ratcheting deformation and a correlation between the strain produced by ratcheting and the estimated dislocation density has been established.

2. Experimental procedure

The material used in the present investigation is a commercially pure non-conventional austenitic stainless steel which is known as X12CrMnNiN17-7-5 according to ISO/TR 15510:1997 [11]. The material was procured in the form of a round bar having diameter of 16 mm. The past history of the steel was not known and therefore, it was required to remove any residual stresses present in it. The steel was further subjected to solution annealing that helped in dissolving carbide phases at high temperature. The solution annealing of the steel was carried out by heating the steel at 960 °C for 1 h followed by water quenching. The specimens for microstructural analysis were mechanically polished up to 0.25 μm surface finish and they were electro polished using a solution of 20% perchloric acid and 80% acetic acid in ice-cooled atmosphere. The polished specimens were etched with aqua regia [a solution of 75% HCl and 25% HNO_3]. The microstructural examination was carried out using optical microscopy and Scanning Electron Microscopy (SEM).

All the specimens were tested under stress-controlled (stress rate = 50 MPa/S) fatigue at room temperature using a servo hydraulic universal testing machine of capacity ± 100 kN (Model: INSTRON, 8800R). Tests were carried out under different combinations of mean stresses (σ_m), stress amplitudes (σ_a) and number of cycles (N) as shown in Table 1. At least 200 data points were collected per cycle during each fatigue test.

The XRD profiles were taken using PAN analytical X-ray diffractometer (Model: DY-1656) with Cu Kα ($\lambda = 1.5418$ Å) radiation operating at 40 kV and 40 mA at a scan speed of 2°/min. The modified Williamson–Hall equation was used to estimate the dislocation densities in the specimens subjected to ratcheting deformation.

3. Results and discussion

3.1. Microstructure

A typical micrograph of the stainless steel under investigation is shown in Fig. 1. It is evident from the figure that the steel possesses equi-axed austenite grains (confirmed by XRD and the pattern is reported in Section 3.3). The grain size was measured using linear intercept method as per ASTM E112 standard and the same was 65 ± 4.3 μm.

Table 1
Test matrix used for all ratcheting experiments (same for 50 and 100 cycles of loading).

Mean stress (σ_m), MPa	Stress amplitude (σ_a), MPa
100	400 450 500
150	400 450 500
200	400 450 500

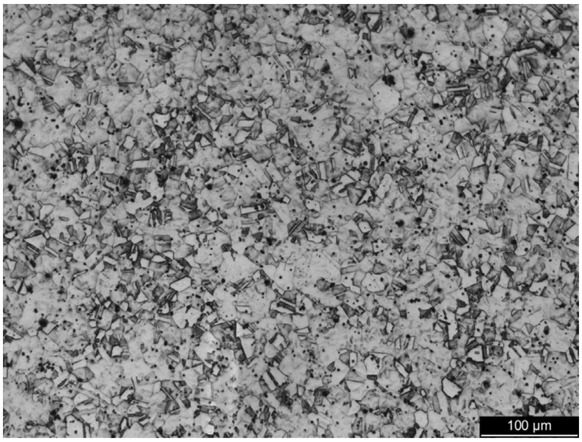

Fig. 1. A typical optical micrograph of the investigated non-conventional stainless steel.

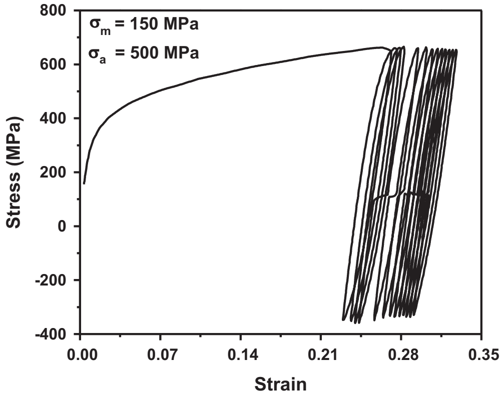

Fig. 2. A typical set of hysteresis loops generated during ratcheting deformation for $\sigma_m - \sigma_a$ combination of 150 and 500 MPa.

3.2. Accumulation of ratcheting strain

The accumulation of ratcheting strain attains a saturation plateau after about 100 cycles of loading for stainless steels [12]. Therefore, the first set of ratcheting tests was carried out up to 100 cycles of loading and another set of tests was carried out up to 50 cycles of loading for comparison. Typical set of hysteresis loops produced during ratcheting deformation is shown in Fig. 2 for $\sigma_m - \sigma_a$ combination of 150 and 500 MPa. It is evident from the figure that the accumulation of ratcheting strain produces unclosed hysteresis loops which shift towards positive plastic strain with increase in number of cycles, as the applied mean stress is positive, as expected. It is available in literature that hysteresis loops shift towards negative plastic strain when the applied mean stress is negative [13]. The variation of ratcheting strain with mean stresses (σ_m), stress amplitudes (σ_a) and number of cycles (N) employed in the present investigation is shown in Fig. 3(a and b). It can be seen from these figures that accumulation of ratcheting strain increases with increase in mean stress (σ_m) at constant stress amplitude (σ_a) and number of cycles (N); with increase in stress amplitude (σ_a) at constant mean stress (σ_m) and number of cycles (N) as well as with increase in number of cycles (N) at a constant mean stress (σ_m) and stress amplitude (σ_a). When σ_m increases at a constant σ_a total zone of fatigue cycling