Cyclic plasticity and internal dislocation structure in two-phase alloy

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Abstract. Austenitic-ferritic stainless steel has been cycled in symmetrical push-pull regime with constant strain rate and different strain amplitudes. Hysteresis loops were recorded and analysed. The plot of the second derivative of the half-loop vs. relative strain shows one peak for low amplitude straining corresponding to the cyclic deformation of softer phase, austenite. With increasing strain amplitude second peak appears and corresponds to the cyclic deformation of the harder phase, ferrite. Transmission electron microscopy study revealed parallel bands in austenite and initial virgin dislocation structure in ferrite at low amplitude while cell structure in austenite and secondary wall structure in ferrite at high amplitude.

1. Introduction
Cyclic straining results in the modification of the internal structure of the material and the changes of its cyclic stress-strain response follow. The relation of the cyclic stress-strain response and internal dislocation structure has been studied extensively in single-phase materials [1] where the dislocation structure determines the internal stress amplitude at particular strain amplitude. In two-phase alloys in which one or both phases are deformed plastically the dislocation structure starts to develop always in the softer phase [2] and with increasing strain amplitude in both phases. Generally, the parallel arrangement of phases and also of individual microvolumes can be supposed and therefore the statistical theory [1] can be applied for the description of the hysteresis loop.

In this contribution the hysteresis loop and the internal dislocation structure in two-phase austenitic-ferritic duplex steel have been studied in cycling with different strain amplitudes at room temperature and the relation of probability density function of the internal critical stresses to dislocation arrangement in both phases is demonstrated.

2. Experimental
Austenitic-ferritic SAF 2507 type duplex stainless steel was supplied by Sandvik, Sweden as rods of 30 mm in diameter. The chemical composition (in wt. %) was: 0.02 C, 23.0 Cr, 7.0 Ni, 3.8 Mo and 0.27 N, the rest Fe. The structure of the steel was formed by the islands of austenite elongated in the rolling direction embedded in a ferritic matrix. The volume fraction of austenite was 53%, the volume fraction of ferrite was 47%. Cylindrical specimens had the gauge length 12 mm and the diameter 8 mm. They were cycled in a computer controlled electrohydraulic testing machine using longitudinal extensometer having the gage length of 8 mm with constant strain rate 2.5x10⁻³ s⁻¹. Hysteresis loops with high number of data points were continually recorded and stored for further analysis. The second
derivatives of the tensile or compressive half-loops were obtained using smoothing and numerical derivative procedures.

Thin foils for the transmission electron microscopy were prepared using standard procedure from sections taken parallel to the specimen axis. They were studied in a transmission electron microscope STEM Philips CM-12 operating at 120 kV using a double tilt holder and a MegaView II digital camera.

3. Results and discussion

Cyclic straining of austenitic-ferritic duplex steel with constant strain amplitudes results in a short initial hardening followed by long term softening with a tendency to saturation [3]. Figure 1 shows saturated tensile hysteresis half-loops in relative coordinates. In this representation the details of the shape of the loop are not evident and the shape of the half-loops is approximately determined by the shape of the half-loop with the highest strain amplitude. The shape of the loop and its evolution in cyclic straining can be found by plotting the second derivative of the half-loop (divided by the half of the square of the effective elastic modulus $E_{eff}$) vs. the relative strain or vs. fictive stress (relative strain multiplied by the half of the elastic modulus). $E_{eff}$ is the effective elastic modulus evaluated for the tensile hysteresis half-loop from the slope of the half-loop where the second derivative reaches the minimum. This plot can be used for the determination of the effective stress and for the probability density distribution of the internal critical stresses [1].

![Figure 1. Tensile half-loops in relative coordinates.](image)

![Figure 2a. Evolution of the second derivative of the tensile half-loop with the number of cycles (a) $\varepsilon_a = 2.5 \times 10^{-3}$, ($\varepsilon_{ap} = 2 \times 10^{-4}$).](image)
of the strain rate due to imperfection in the cylinder and appears at relative strain corresponding to zero stress in all plots. No appreciable changes of both peaks during cyclic straining appeared.

Dislocation structure in a specimen cycled with low strain amplitude shows figure 3. The typical dislocation structure corresponding to cyclic loading is apparent only in austenitic grains. Dislocation structure in ferritic grains corresponds to the non-deformed state. It is typical for all grains observed. Dislocations in the austenite lie in parallel bands, forming pile-ups. This is a typical dislocation arrangement provided one slip system is activated and cross-slip is difficult. Low stacking fault energy is a reason for the splitting of the perfect dislocation into the two Shockley partials in the primary slip plane. In the ferritic grains, low density of randomly oriented dislocations is found (see figure 3). The

Figure 2. Evolution of the second derivative of the tensile half-loop with the number of cycles (b) $\varepsilon_a = 3.5 \times 10^{-3}$, ($\varepsilon_{ap} = 7.2 \times 10^{-4}$), (c) $\varepsilon_a = 1 \times 10^{-2}$, ($\varepsilon_{ap} = 6.4 \times 10^{-3}$).

Figure 3. Dislocation structure in specimen cycled with constant plastic strain amplitude $1 \times 10^{-4}$ for $5 \times 10^6$ cycles.

Figure 4. Dislocation structure in specimen cycled with constant plastic strain amplitude $2 \times 10^{-3}$ to fracture ($N_f = 10140$ cycles).
Figure 5. Dislocation structure in specimen cycled with constant plastic strain amplitude $1 \times 10^{-2}$ to fracture ($N_f = 925$ cycles), (a) austenitic grain, (b) ferritic grain.

dislocation density and arrangement is the same as in the virgin material. The applied stress is thus not high enough to induce a notable dislocation activity in majority of ferritic grains. This is in agreement with the analysis of the loop shape in low amplitude loading.

With increasing strain amplitude characteristic dislocation structure is formed both in austenitic and in ferritic grains. In austenitic grains parallel bands become thicker and their density increases until secondary slip is activated. In narrow twins ladder-like structure can be formed (see e.g. [4]). The dislocation structure in the ferritic grains is composed of vein-like matrix and well defined walls. Two or more slip systems are activated; the walls are produced by the interaction of the dislocations from two of most active slip systems. Screw dislocation segments are present in the channels. Characteristic dislocation structure in specimen cycled with the high strain amplitude shows figure 5. Characteristic cell dislocation structure in austenite was produced by multiple slip (figure 5a). Dislocations are concentrated to the cell walls and individual cells are disoriented by a small angle. In the ferritic grain (figure 5b) cells are formed in the lower left corner while secondary walls dominate the majority of the grain.

Due to much higher effective stress in the ferrite the second peak of the second derivative is shifted to higher fictive stress and both peaks of the probability density function of the internal critical stresses are well separated. The effective stresses 120 MPa and 340 MPa were evaluated for austenite and ferrite respectively. The maxima of the critical internal stresses of elementary volumes in the probability density plot with the highest occurrence correspond to 180 MPa in austenite and 290 MPa in ferrite. The major part of the cyclic stress in duplex steel for all plastic strain amplitudes is thus carried by ferritic phase. The dislocation structures of both phases vary appreciably with applied plastic strain amplitudes in agreement with the changes of the probability density function of the internal critical stresses.

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