LCF BEHAVIOUR OF 301LN STEEL: COARSE-GRAINED VS. UFG-BIMODAL STRUCTURE

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ABSTRACT

Low-cycle fatigue (LCF) behaviour of metastable austenitic 301LN steel with different grain sizes – coarse-grained (14 µm) and UFG (1.4 µm) with a grain bimodality – produced by reversion annealing (RA) was investigated. Symmetrical push-pull LCF tests were conducted on flat sheet specimens at room temperature with constant strain rate of 2×10^{-3} s^{-1} and constant total strain amplitude ranging from 0.4% to 0.8%. After completion of fatigue tests a ferritescope was adopted for quantitative assessment of volume fraction of deformation induced martensite (DIM). Microstructural changes, distribution and morphology of DIM in the volume of material were characterized at different scales by colour etching, TEM and EBSD techniques. Experimental data on microstructural changes are confronted with the stress-strain response and with the chemical heterogeneity present in the material.

KEYWORDS

Low cycle fatigue, 301LN austenitic stainless steel, ultrafine-grained (UFG) microstructure, reversion annealing, deformation induced martensite

INTRODUCTION

Grain size refinement leading to considerably enhanced mechanical properties of metallic materials represents a viable topic in the material research for many decades. A great attention has been devoted in the past to ultrafine-grained (UFG) materials prepared by various severe plastic deformation (SPD) methods with the most frequent representatives ECAP and HPT (for the review see e.g. [1]). The SPD refinement generally results in an improvement of strength characteristics under monotonic and high-cycle fatigue (HCF) loading conditions. Nevertheless, due to the instability of UFG structure it has also a detrimental effect on the fatigue characteristics obtained during strain controlled, i.e. low-cycle fatigue (LCF) tests [1].

An alternative and very promising method for grain refinement and thus strengthening of metastable austenitic steels represents so called reversion annealing (RA) [2]. It has been repeatedly shown that controlled annealing of cold-rolled austenitic steels containing significant fraction of deformation induced martensite (DIM) results in an excellent combination of tensile strength and elongation due to efficient grain refinement up to submicron scale [2] (for a recent review see also [3]). Positive effect of grain refinement via RA on high-cycle fatigue (HCF) behaviour has been documented for AISI 304 steel [4] and more recently also for 301LN sheet steel fatigued in fully reversed bending [5] or in load

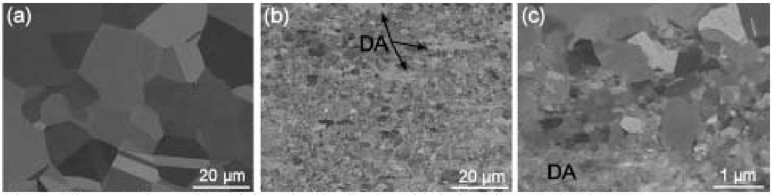

Fig. 1: Microstructure of CG (a) and UFG (b,c) 301LN steel (ECCI, SEM–FEG).

controlled axial tests with the stress ratio R = 0.1 [6]. Even though the LCF behaviour of this steel is of great importance for potential industrial applications, it has been studied very rarely so far [7].

The present work represents a continuation of our systematic studies on LCF behaviour of 301LN steel with UFG structure produced by RA. Some preliminary results on the cyclic stress-strain response and the magnetic phase content after cyclic straining has been reported recently for 301LN steel with two different grain sizes [7]. Here the main attention is devoted to the characterization of microstructural changes and the destabilization of the initially fully austenitic structure in the volume of cyclically strained 301LN steel. Experimental data obtained by various microscopic techniques are related to the cyclic stress-strain response and confronted with the chemical heterogeneity present in the material.

EXPERIMENTAL

A commercial metastable austenitic 301LN steel having the chemical composition (in wt.%) 0.017C, 0.52Si, 1.29Mn,17.3Cr, 6.5Ni, 0.2Cu, 0.15Mo, 0.15N, rest Fe was subjected to the following thermo-mechanical treatment at the University of Oulu, Finland. Sheet material with initial fully austenitic structure was cold rolled (CR) with the reduction of 63% and 72% to the thickness of 2 mm and 1.5 mm, respectively. Reversion annealing of the CR 2 mm and 1.5 mm thick sheets at 1000 °C/200 s and 800 °C/1 s, respectively, resulted in two considerably different grain sizes (cf. Fig. 1a and 1b). The coarse-grained (CG) structure with equiaxed grains containing typical annealing twins with the average grain size of 14 µm is apparent from Fig. 1a. Annealing at 800 °C/1 s resulted in the ultrafine-grained (UFG) structure with the average grain size of 1.4 µm (Fig. 1b). The grain size in UFG structure across the sheet thickness is not, however, fully uniform and moreover, locally, only partially recrystallized grains can be detected within the steel structure. Both features, i.e. the presence of grain bimodality and deformed austenite (DA) in the UFG structure are apparent from Fig. 1b and 1c. From the processed sheet material the flat “dog-bone” shape specimens having the gauge width and length of 8 mm and 10 mm, respectively, were cut perpendicular to the rolling direction. The central part of specimens was carefully polished mechanically and electrolytically. For more details on the specimen geometry including the size of RA zone see [7].

Low cycle fatigue tests were conducted at room temperature in air in tension-compression under strain control with constant total strain amplitude ε_{at} ranging from 0.4% to 0.9%. The strain rate of 2×10^{-3} s^{-1} was kept constant during all fatigue tests. Strain was recorded and controlled with an MTS extensometer having the gauge length of 8 mm. Due to fully reversed loading ($R_\varepsilon = -1$) a special anti-buckling fixtures were adopted to avoid specimen bending. To reduce possible friction Teflon sheets 0.5 mm in thickness were inserted between the fixtures and the specimen surface.

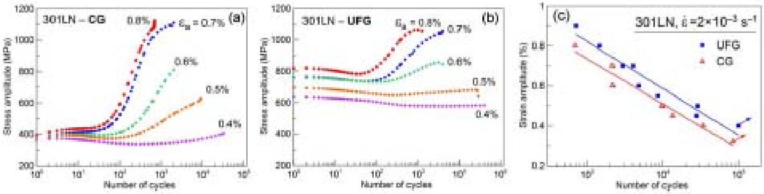

Fig. 2: Cyclic hardening/softening curves (a,b) and fatigue life curves (c) of CG and UFG 301LN steel cyclically strained with various constant total strain amplitudes.

The gauge parts of testing specimens after completion of LCF tests were sectioned by spark erosion across the specimen thickness perpendicular to the stress axis (i.e. parallel to the rolling direction) and carefully polished mechanically and electrolytically. The microstructure on the prepared longitudinal cross sections was documented at different scales by two techniques: Beraha II color etching and EBSD (electron backscatter diffraction) technique in high-resolution SEM–FEG (LYRA 3 XMU from TESCAN and FEI Verios 460L). Detail characterization of structural changes in the central part of specimens was performed using transmission electron microscopy (TEM) with a Philips CM12 or a JEOL JEM-2100F. Thin foils were prepared by standard twin jet electropolishing technique from thin sheets taken from cuts parallel to the stress axis with the cut plane inclined 30° to the specimen surface. Local chemical analyses were performed in the line scan mode using EDS (energy dispersive spectroscopy).

RESULTS

Cyclic stress-strain response and fatigue life

Cyclic hardening/softening curves and fatigue life curves for both CG and UFG states of 301LN steel are shown in Fig. 2. Cyclic straining of UFG material (Fig. 2b) results in initial slight cyclic softening followed by cyclic hardening with the exception of the lowest strain amplitude of 0.4% at which tendency to saturation can be observed. CG material (Fig. 2a) exhibits a short initial stage of cyclic hardening followed by tendency to mild softening for low strain amplitudes ($\varepsilon_{at} < 0.65\%$) or saturation for high strain amplitudes. At approximately the same number of cycles for each strain amplitude (cf. Fig. 2a and 2b) rapid cyclic hardening stage follows. Comparison of the cyclic stress-strain response of both materials shows that the initial values of stress amplitude differ considerably which is in agreement with almost two times higher yield stress of UFG material in comparison with CG counterpart [7]. Nevertheless, due to rapid cyclic hardening of the CG material the stress amplitudes are with the exception of the lowest strain amplitude almost identical.

Fatigue life curves for both CG and UFG materials plotted as the dependence of the applied strain amplitude vs. number of cycles to fracture are shown in Fig. 2c. The slopes of fatigue life curves are for both materials nearly identical. The results clearly show higher fatigue life in case of UFG material.

Destabilization of austenitic structure and DIM formation

Fig. 3 shows overall distribution of DIM across the whole longitudinal cross-section of the gauge part of 301LN flat specimens fatigued to the end of fatigue life with ε_{at} = 0.5%. Color etching (Beraha II) reveals that at macroscopic scale the DIM (dark features in Fig. 3) is not

Fig. 3: Distribution of DIM across the sheet full-thickness of 301LN steel as revealed by color etching for (a) UFG and (b) CG state, both after cyclic straining with ε_{at} = 0.5% until the end of fatigue life. (Beraha II, OM, longitudinal cross section).

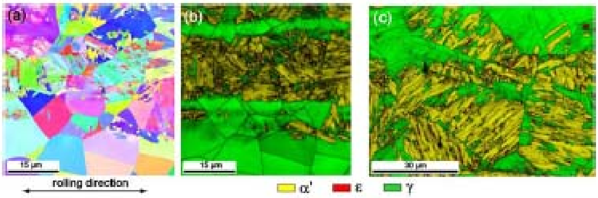

Fig. 4: Microstructure of CG 301LN steel cycled with ε_{at} = 0.5% up to the end of fatigue life as revealed by EBSD on the longitudinal sheet cross section in its central part (a,b) and outside the central part (c). Stress axis is perpendicular to the plane of micrographs.

distributed homogeneously across the plate thickness. The central part of high DIM density can be distinguished in both UFG and CG material. The DIM is noticeably arranged into more or less compact bands running parallel to the rolling direction. Outer layers bounding the central part exhibit much lower density of DIM with a more complex morphology of DIM distribution within the austenite matrix.

Figs 4a and 4b show the result of EBSD mapping (step size of 80 nm) of an area selected in the central part of fatigued CG 301LN steel and Fig. 5c in the outer layer with a lower DIM density (cf. Fig. 3b). In the central part the DIM is concentrated into bands of different thickness running across the structure, irrespective of individual grain orientations – cf. the inverse pole figure and phase map shown in Fig. 4a and 4b, respectively. The internal structure of DIM areas is not homogeneous but consists of numerous sub-structural units – plates and packets or colonies of α'-martensite accompanied by a small fraction of ε-martensite phase and locally by a small islands of still not-transformed austenite. Similar EBSD results on the DIM distribution have been recently reported also in UFG material [9].

TEM examinations generally revealed a great variability in microstructure features in individuals grains of fatigued CG and UFG 301LN steel – some of them are documented in Fig. 5. In addition to dislocation tangles or numerous layers of ε-martensite or stacking faults various forms of α'-martensite were detected in CG material: colony of α'-martensite (Fig. 5a), embryos of α'-martensite at the intersection of ε-martensite plates (Fig. 5b), α'-martensite growing within individual ε-martensite platelets (Fig. 5c) or α'-martensite formation at the grain boundary in the place of impact of ε-martensite platelets (Fig 5d). In the case of

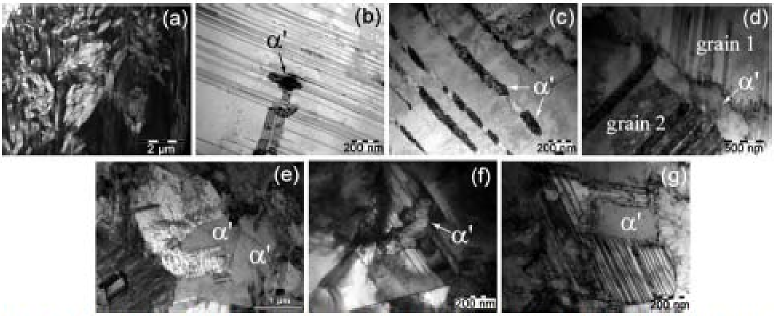

Fig. 5: Morphology of DIM as revealed by TEM in CG (a–d) and UFG (e–g) 301LN steel fatigued to the end of fatigue life with $\varepsilon_a = 0.4\%$(a,b,e,f) and $\varepsilon_a = 0.6\%$(c,d,g).

fatigued UFG material the DIM was detected in the form of small islands within individual grains (see Fig. 5f) or in the compact areas filling partially or fully initially austenitic grains (Figs. 5e and 5g).

DISCUSSION

The experimental study on the destabilization of the initially fully austenitic structure and DIM formation during cyclic straining of 301LN steel with two different grain sizes brought two important results: (i) DIM is not distributed homogeneously across the thickness of flat specimens and furthermore (ii) there is a great variability in both the mechanisms of DIM formation and morphology of DIM in the volume of the material.

Both the above important results can be rationally reconciled with our recent studies on the stability of various products forms of wrought 304- [8,9] and 316-type [8] austenitic stainless steels. Both studies demonstrated convincingly that these Cr–Ni austenitic steels are never fully chemically homogeneous and furthermore due to the specific solidification behavior and contemporary continuous casting practice the most metastable area of any wrought full-cross-section semi-product is always its central part. Applying the same philosophy in the present case, i.e. performing the local chemical analysis across the DIM bands shown in Fig. 4b it can be checked whether the above statements are valid also for 301LN steel. Fig. 6b

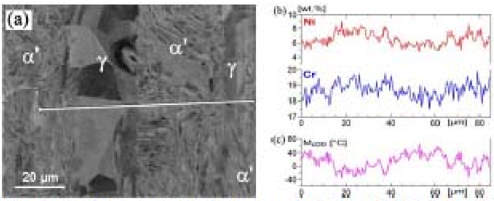

Fig. 6: (a) Microstructure of CG 301LN steel as revealed by ECCI in the central part of the flat specimen cycled with $\varepsilon_{at} = 0.5\%$ up to the end of fatigue life. (b) Segregation profiles of Cr and Ni obtained by EDS along the white line indicated in (a). (c) Variations in the characteristic temperature M_{d30} calculated using Pickering's equation [8].

shows characteristic local variations in chromium and nickel determined by line scan EDS analysis along the line position of which is indicated in Fig. 6a. The adoption of this data together with data obtained on other elements – Mn, Si, Cu, Mo (unfortunately local variations for two very strong austenite stabilizers C and N could not be established and thus only their nominal content was considered) into empirical Pickering's equation, clear and significant local fluctuations of the characteristic temperature M_{d30} are indicated (cf. Figs. 6a and 6c). These characteristic local chemical variations are also very probably responsible for the presence of heavily deformed and partially recrystallized grains in UFG material (see Fig. 1c) obtained by RA at 800 °C for 1 s [8]. Finally, since the stacking fault energy (SFE) of austenite depends primarily on the chemical composition the presence of diverse mechanisms of DIM formation documented by TEM can be explained.

In summary, the present study on the destabilization of 301LN steel with two different grain sizes pointed out the importance of characterization of microstructural changes at different scales. Only combinations of various techniques can yield more coherent view on the structure of cyclically strained metastable austenitic steel. Since the cyclic stress-strain response in axial LCF fatigue tests reflects the microstructure changes in the whole volume of material the evaluation of DIM formation should be performed not only on the sheet surface but also across its thickness.

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REFERENCES

[1] Estrin, Y., Vinogradov, A.: Extreme grain refinement by severe plastic deformation: A wealth of challenging science, Acta Mater. 61 (2013) pp. 782–817.
[2] Karjalainen, L.P., Taulavuoiri, T., Sellman, M., Kyröläinen A., Some strengthening methods for austenitic stainless steels, Steel Res. Int. 79 (2008) pp. 404–412.
[3] Järvenpää, A., Jaskari, M., Man, J., Karjalainen, L.P.: Austenite stability in reversion-treated structures of a 301LN steel under tensile loading. Mater. Charact. 127 (2017) pp. 12–26.
[4] Di Schino, A., Kenny, J.M.: Grain size dependence of the fatigue behaviour of a ultrafine-grained AISI 304 stainless steel, Mater. Lett. 57 (2003) pp. 3182–3185.
[5] Hamada, A.S., Karjalainen, L.P., Venkata Surya, P.K.C., Misra, R.D.K.: Fatigue behavior of ultrafine-grained and coarse-grained Cr–Ni austenitic stainless steels, Mater. Sci. Eng. A 528 (2011) pp. 3890–3896.
[6] Järvepää, A., Karjalainen, L.P., Jaskari, M.: Effect of grain size on fatigue behavior of type 301LN stainless steel, Int J Fatigue 65 (2014) 93–98.
[7] Chlupová, A., Man, J., Kuběna, I., Polák, J., Karjalainen, L.P.: LCF behaviour of ultrafine grained 301LN stainless steel, Proc. Eng. 74 (2014) pp. 147–150.
[8] Man, J., Kuběna, I., Smaga, M., Man, O., Järvenpää, A., Weidner, A., Chlup, Z., Polák, J.: Microstructural changes during deformation of AISI 300 grade austenitic stainless steels: Impact of chemical heterogeneity, Proc. Struct. Integr. 2 (2016) pp. 2299–2306.
[9] Man, J., Smaga, M., Kuběna, I., Eifler, D., Polák, J.: Effect of metallurgical variables on the austenite stability in fatigued AISI 304 type steels, Eng. Fract. Mech. (2017), submitted for publication

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