GRAIN REFINEMENT EFFECT ON FATIGUE PROPERTIES OF AUSTENITIC STAINLESS STEEL WITH DEFORMATION INDUCED MARTENSITE FORMATION

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Abstract

Fatigue properties of metastable austenitic 301LN steel with coarse and ultrafine-grained microstructures were investigated at ambient temperature and the effect of grain refinement was evaluated. Two different microstructural states of material were introduced by different annealing conditions during special thermo-mechanical treatment. Fatigue life curves were obtained and compared for both coarse-grained and ultra-fine-grained states of austenitic stainless steel. Magnetic measurements before, during and after cyclic loading were performed to reveal structural changes, i.e. formation of deformation-induced martensite. Relationship between microstructure refinement, phase composition and fatigue properties was discussed.

Keywords:

Fatigue, 301LN austenitic stainless steel, grain refinement, reversion annealing, phase transformation

1. INTRODUCTION

Reversion annealing (RA) is method suitable for grain refinement of metastable austenitic steels. This thermo-mechanical treatment (TMT) is based on phase transformation when originally coarse grained (CG) austenitic structure is cold rolled to obtain fine deformation induced martensitic structure which is during annealing reverted back to austenite, now with ultrafine-grained (UFG) microstructure. Material in CG state is widely used in many industrial applications and exhibits excellent plasticity and deformation characteristics, nevertheless yield stress and strength of such a material is relatively low. RA is able to refine grain size, increase strength and preserve plasticity of resulting material [1, 2]. Effect of grain refinement on tensile properties (e.g. [3–5]) or high-cycle fatigue [6–9] has been already documented, nevertheless low cycle fatigue (LCF) properties, which are of great importance for structural design, were not systematically studied so far.

The aim of this work is to present results of recent studies on fatigue behavior of 301LN steel with two different grain sizes prepared by RA. Phase transformation from initially fully austenitic structure (paramagnetic) to martensite (ferromagnetic) was observed using measurement of magnetic properties before and after cyclic loading. Changes in mechanical response of both materials were related to the phase content.

2. EXPERIMENTS

2.1 Material and specimens

Metastable austenitic AISI 301LN steel with chemical composition in wt.%: 0.017 C, 0.52 Si, 1.29 Mn, 17.3 Cr, 6.5 Ni, 0.2 Cu, 0.15 Mo, 0.15 N, rest Fe, was subjected to the TMT at the University of Oulu, Finland [10]. Two different annealing conditions resulted in two grain sizes, determined by the linear intercept method. Annealing at 1000 °C/200 s resulted in the coarse grained structure (CG) with average grain size of 14 μm. Equiaxed austenitic grains contain typical annealing twins (see **Fig. 1a**). Annealing at and 800 °C/1 s produced approximately 10 times finer austenitic microstructure more or less uniform with average grain size of 1.4 μm. Some individual bigger grains (about 20 μm, see **Fig. 1b**) were present. The flat dog-bone specimens (see **Fig. 1c**) with gauge length 10 mm were machined from a sheet material and their surface was mechanically grinded and polished. Microstructure was characterized using scanning electron microscope Tescan LYRA 3 XMU. To visualize grain boundaries the chemical etching was performed.

2.2 Mechanical properties measurement

Monotonic loading i.e. results from tensile tests and Vickers hardness measurement HV10 were reported earlier [11]. Application of special TMT leads to a desirable combination of high strength (over 1000 MPa) and high ductility (over 60%) in case of the UFG 301LN steel – see **Table 1**. Vickers hardness measurement (HV 10) was performed for both materials on samples in three different states. The most significant difference in HV10 values were observed in annealed state. Increase of hardness for fully austenitic material in case of UFG steel is caused by the microstructure refinement and reinforcing effect of grain boundaries. Hardness after tensile test in the vicinity of fracture for both CG and UFG materials indicates approximately the same amount of deformation induced martensite (about 55% of ferrite).

Table 1. Tensile properties and results of Vickers hardness measurement HV10 of 301LN steel after TMT.

	Ultimate tensile stress	Yield strength	Total elongation	HV10 cold rolled	HV10 annealed	HV10 after tensile test
UFG	1056 MPa	685 MPa	60%	549	313	495
CG	890 MPa	300 MPa	75%	522	196	478

LCF tests were conducted in air at room temperature, using a servo-hydraulic testing system MTS 810. The tests were executed under total strain control with different levels of total strain amplitude ranging from 0.4 % to 0.9 %, with constant strain rate of $2\times10^{-3}\,s^{-1}$. Strain was controlled and recorded using MTS extensometer having the gauge length of 8 mm. Water cooled hydraulic grips were used to keep constant temperature of the specimen. For fully reversed loading (R = –1) a special anti-buckling fixtures were adopted (see **Fig. 1d**) to avoid specimen buckling. Teflon inserts were applied to reduce friction between the fixtures and specimen.

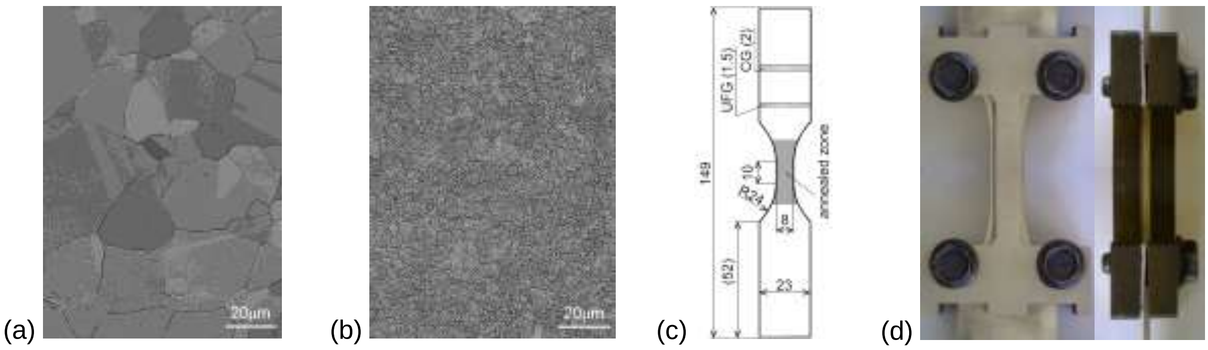


Fig. 1: (a, b) Microstructure of CG and UFG material after TMT showing fully austenitic microstructure, equiaxed grains and typical annealing twins in case of CG material, (c) shape of the flat dog-bone specimen with dimensions in mm, (d) special anti-buckling fixtures.

2.3 Magnetic measurements

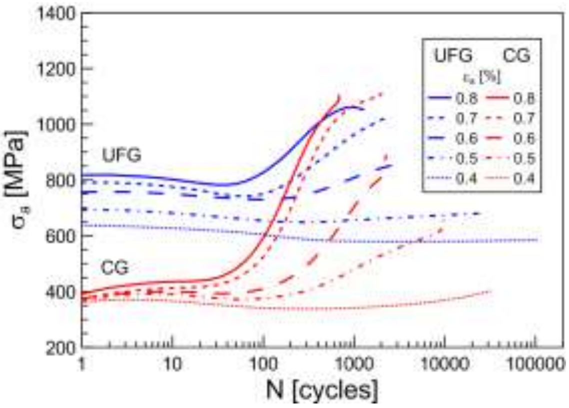
Magnetic induction data, as an indicator of the amount of deformation induced α´martensite, were measured using Fischer Feritscope FMP 30. Magnetic phase fraction was indicated in vol. % of ferrite. In literature a linear correlation between vol.% of ferrite and vol.% of α´ martensite is given only up to 60% of α´ martensite. Assuming higher martensite fractions developed during loading the α´martensite fraction was thus indicated as vol.% of ferrite. Several correction factors were taken into account because of the shape and thickness of measured specimens. Amount of magnetic phase was measured before loading and a fraction of magnetic phase along the specimen length was evaluated after the test termination.

3. RESULTS AND DISCUSSION

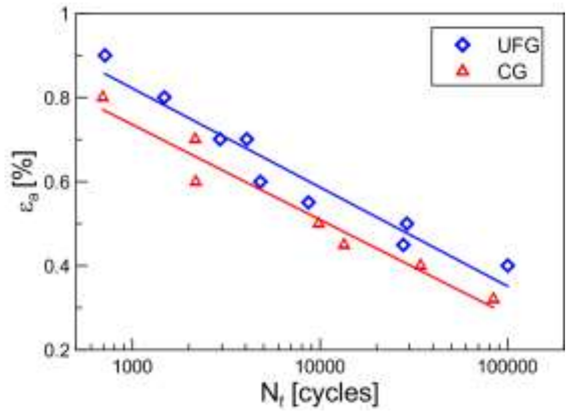
3.1 Cyclic deformation behavior

Cyclic hardening/softening curves and fatigue life curves for both CG and UFG states of austenitic stainless steel were measured. They are shown in **Fig. 2**. Cyclic straining of UFG material (Fig. 2a) results in slight initial cyclic softening followed by hardening. CG material exhibits slight cyclic hardening followed by a tendency to softening. At approximately the same number of cycles for each strain amplitude rapid cyclic hardening stage follows. Comparison of cyclic hardening/softening curves of both materials shows that initial values of stress amplitude differ considerably. It is in agreement with almost two times higher yield stress of UFG material in comparison with CG counterpart. Nevertheless due to rapid cyclic hardening of the CG material the stress amplitudes of both materials before fracture are almost identical.

Fig. 2b shows fatigue life curves for both UFG and CG materials plotted as the dependence of the applied strain amplitude vs. number of cycles to fracture. The results clearly show higher fatigue life in case of UFG material.



(a)

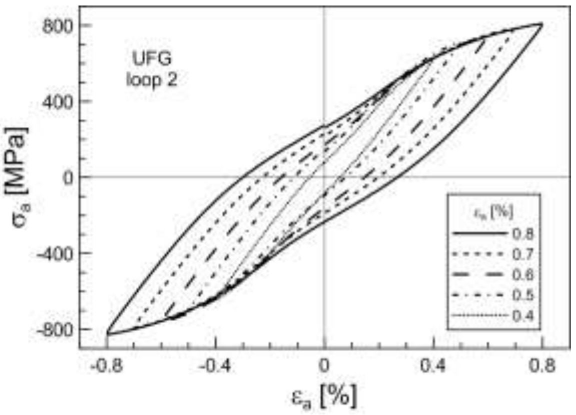


(b)

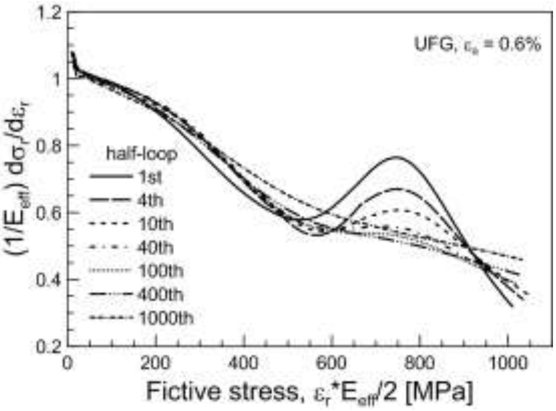
Fig. 2: (a) Plot of the stress amplitude σ_a vs. number of cycles N of AISI 301LN cycled with different ε_a for CG and UFG materials. (b) Fatigue life curves for UFG and CG materials.

During the fatigue tests stress-strain dependence within the cycle, i.e. the hysteresis loop was recorded. CG material exhibits usual shape of hysteresis loop, however in case of UFG material initial cycles of fatigue loading exhibited some irregularities demonstrated by constriction of the loop and by the nonmonotonic first derivative of both tensile and compression half-loops. This behavior was present in all tests i.e. for various levels of applied strain amplitude. **Fig. 3a** shows the first closed hysteresis loops for five different strain amplitudes. **Fig. 3b** shows the evolution of the first derivative of the tensile half-loop during cycling. The relative strain ε_r, relative stress σ_r and effective elastic modulus E_{eff} are used for its representation (for details see [12]).

It is evident from the stress-strain plot that hysteresis loop is constricted in the region of low applied stresses for both tensile and compression parts of loop, i.e. the constriction starts in the area when the stress has changed direction. With increasing number of elapsed cycles during fatigue loading the constriction disappears and later (in tens of cycles) the shape of hysteresis loop becomes normal. In the plot of the first derivative (see **Fig 3b**) the constriction appears as a peak and valley. It becomes less pronounced with increasing number of cycles. Approximately at 10% of fatigue life the constriction effect of the hysteresis loop shape is not perceptible.



(a)



(b)

Fig. 3: (a) Hysteresis loops of the first closed hysteresis loop at various levels of applied strain amplitude for UFG material. (b) Plot of the first derivative showing the disappearance of the constriction of the loop with increasing number of cycles.

Plastic deformation in meta-stable austenitic stainless steels is accompanied by a deformation-induced solid state phase transformation of paramagnetic γ austenite (f.c.c. phase) to ferromagnetic α' martensite (b.c.c. phase). Ferromagnetic materials have a structure that is divided into domains, each of which is a region of uniform magnetic polarization. When an external magnetic field is applied, the boundaries between the domains shift, the domains rotate and it is accompanied by the change of the dimensions of the material. The changes of the dimension can be induced also due to the reverse magnetostrictive effect, or Villari effect. Magnetic domain alignment can be caused by the application of the external stress. As a result magnetic field arises and positive strain is induced when tensile stress is applied and negative strain is induced when compressive stress is applied. Since in cyclic straining the sign of the stress is changed in each cycle the constriction of the hysteresis loop results provided the domains can reorient freely in agreement with the direction of the magnetic field. This effect has been observed in cyclically deformed nickel single and polycrystals [13, 14] and also in ferritic stainless steel [12].

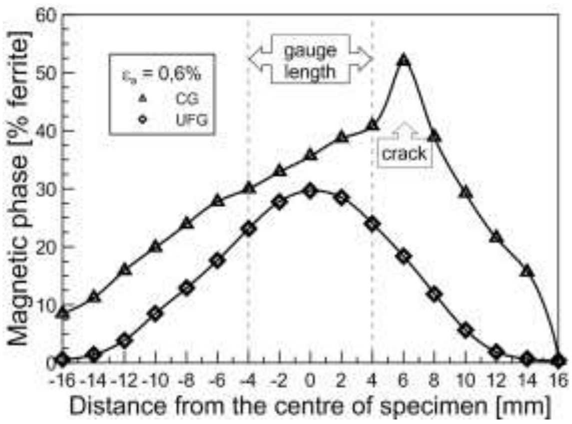
Magnetic Feritscope measurements can provide us with the comprehensive information about the plastic-strain-induced martensite fraction in metastable austenitic steels and about the change of the magnetic field due to the application of the elastic strain. Unfortunately the changes of the magnetic field due to Villari effect are too small and can be detected only by a very sensitive instrument.

In case of UFG steel it seems that the constriction of hysteresis loop can be related to the reverse magneto-elastic effect owing to the grain refinement effect. However, either some fraction of magnetic phase must be present in the material in the initial stage before cycling or magnetic phase is produced in the first tensile quarter-cycle. Magnetic domains in residual magnetic phase can reorient easily with alternating stress, which in annealed material is anticipated. Since we have observed that in further cyclic loading during which the faction of martensite increases the constriction gradually disappears, the presence of residual annealed magnetic phase is more probable. The presence of a small fraction of magnetic phase in UFG material before cycling is also supported by the fact that annealing time of the martensite during reversion was only 1 s and some residual tempered martensite could remain in the annealed austenitic structure.

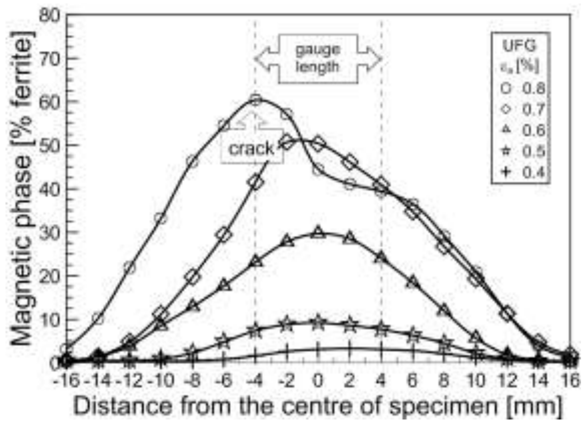
In case of UFG 301 steel the hysteresis loop constriction tied to the Villari effect diminishes and/or disappears completely with increasing number of cycles, i.e. with increasing martensite fraction similarly to the case of cycled nickel [13,14].

3.2 Martensite volume fraction after test termination

The fraction of magnetic phase in the gauge length of both materials before fatigue loading as determined by Ferritscope was close to zero, i.e. nominally fully austenitic structure after TMT was confirmed. After the test termination a profile of magnetic phase content along the specimen length was measured for specimens cycled with various strain amplitudes. Cycling was terminated either when the crack criterion was reached or when the specimen was fractured.



(a)



(b)

Fig. 4: A profile of magnetic phase content along the specimen length. (a) Comparison of CG and UFG materials cycled with the same strain amplitude. (b) Magnetic phase in UFG material cycled with various strain amplitudes.

301LN steel is very sensitive to the strain-induced phase transformation. Measured data revealed certain difference between CG and UFG material. During cyclic loading with the same strain amplitude the higher fraction of martensite is produced in CG material (see **Fig. 4a**) (the difference in the area of gage length is approximately 10%). The fraction of martensite (i.e. magnetic phase, denoted in **Fig. 4** as % of ferrite) increases with the applied strain amplitude (**Fig. 4b**). Martensite volume fraction in gauge length is about 60 % for specimen cycled with strain amplitude 0.8 %. Peak visible in some plots (e. g. in **Fig. 4a**) is related to the presence of a principal crack in specimen. Local increase of martensite volume fraction is connected with the cyclic plastic zone formation in the vicinity of fracture surface. The local strain amplitude in the plastic zone of the growing crack is higher than the applied strain amplitude and martensite volume content has increased accordingly.

4. CONCLUSIONS

Influence of grain refinement on low cycle fatigue behavior of 301LN stainless steel was investigated. The initial austenitic structure of metastable austenitic AISI 301LN steel undergoes deformation-induced martensitic transformation under monotonic and cyclic loading. This phase transformation strongly influences its microstructure and mechanical properties.

Low cycle fatigue properties were studied and cyclic hardening/softening curves and fatigue life curves were determined. UFG material shows initial cyclic softening followed by hardening in contrast to CG where mostly strong cyclic hardening was observed. UFG material exhibits longer low cycle fatigue life than CG counterpart.

Considerable difference can be seen in the stress-strain behavior of both types of material. UFG material exhibits constriction effect on the hysteresis loop. Constriction of hysteresis loop is the most pronounced in the first closed cycle. It was ascribed to the reversed magneto-elastic effect (Villari effect) of the residual magnetic phase in the UFG material. The constriction diminishes within the first tens of cycles which is connected with the difficult reorientation of magnetic domain due to production of martensite needles and accompanying dislocations.

Measurement of the magnetic phase content along the specimen length revealed that both CG and UFG material cycled with various strain amplitudes revealed increasing martensite volume fraction with increasing strain amplitude. Nevertheless grain refinement influences the amount of martensite. UFG material exhibits lower martensite volume fraction along the specimen length in comparison with CG material cyclically loaded with the same strain amplitude.

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LITERATURE

[1] RAJASEKHARA, S., FERREIRA, P. J.; KARJALAINEN, L. P.; et al.: Hall-Petch behavior in ultra-fine-grained AISI 301LN stainless steel, *Metall. Mater. Trans. A*, 38A (2007) 1202–1210.

[2] FOROUZAN, F., NAJAFIZADEH, A., KERMANPUR, A., et al.: Production of nano/submicron grained AISI 304L stainless steel through the martensite reversion process, *Mater. Sci. Eng. A*, 527 (2010) 7334-7339.

[3] MISRA, R. D. K., NAYAK, S., VENKATASURYA, P. K. C., et al.: Nanograined/Ultrafine-Grained Structure and Tensile Deformation Behavior of Shear Phase Reversion-Induced 301 Austenitic Stainless Steel, *Metall. Mater. Trans. A,* 41A (2010) 2162–2174.

[4] MISRA, R.D.K., ZHANG, Z., VENKATASURYA, P.K.C., SOMANI, M.C., KARJALAINEN, L.P.: The effect of nitrogen on the formation of phase reversion-induced nanograined/ultrafine-grained structure and mechanical behavior of a Cr–Ni–N steel, *Mater. Sci. Eng. A*, 528 (2011) 1889–1896.

[5] REZAEE, A., NAJAFIZADEH, A., KERMANPUR, A., MOALLEMI, M.: The influence of reversion annealing behavior on the formation of nanograined structure in AISI 201L austenitic stainless steel through martensite treatment, *Mater. Design*, 32 (2011) 4437–4442.

[6] HAMADA, A.S., KARJALAINEN, L.P., SURYA, P.K.C.V., MISRA, R.D.K.: Fatigue behavior of ultrafine-grained and coarse-grained Cr–Ni austenitic stainless steels, *Mater. Sci. Eng. A*, 528 (2011) 3890–3896.

[7] MATEO, A., FARGAS, G., ZAPATA, A.: Martensitic Transformation during Fatigue Testing of an AISI 301LN Stainless Steel, *IOP Conf. Series: Mater. Sci. Eng.*, 31 (2012) 012010.

[8] HAMADA, A.S.: Deformation and damage mechanisms in ultrafine-grained austenitic stainless steel during cyclic straining, *Metall. Mater. Trans. A*, 44A (2013) 1626–1630.

[9] JÄRVENPÄÄ, 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.

[10] SOMANI, M. C., KARJALAINEN, L. P., KYROLAINEN, A., TAULAVUORI, T.: Processing of submicron grained microstructures and enhanced mechanical properties by cold-rolling and reversion annealing of metastable austenitic stainless steels, *Mater. Sci. Forum*, 539-543 (2007) 4875–4880.

[11] CHLUPOVÁ, A., MAN, J., POLÁK, J., KARJALAINEN, L.P.: Microstructural investigation and mechanical testing of an ultrafine-grained austenitic stainless steel, Conference Proceedings NANOCON 2013, 5th International Conference on NANOCON, Brno, Czech Republic (2013).

[12] POLÁK, J., PETRENEC, M., DEGALLAIX, S.: On the analysis of the hysteresis loop of ferritic steel in cyclic straining, *Scripta mater.*, 47 (2002) 731–736.

[13] MORRISON, D.J., JIA, Y., MOOSBRUGGER, J.C.: Cyclic plasticity of polycrystalline nickel at low plastic strain amplitude: constricted hysteresis loops, *Scripta mater.*, 44 (2001) 449–453.

[14] JIA, Y., MORRISON, D.J., MOOSBRUGGER, J.C.: The influence of magnetostriction on the shape of the hysteresis loop of cyclically deformed single crystal nickel, *Scripta mater.*, 53 (2005) 1025–1029.