MICROSTRUCTURAL INVESTIGATION AND MECHANICAL TESTING OF AN ULTRAFINE-GRAINED AUSTENITIC STAINLESS STEEL

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Abstract

Special thermomechanical treatment based on high degree deformation followed by reversion annealing was applied to 301LN austenitic stainless steel to achieve ultrafine-grained (UFG) structure with considerably enhanced mechanical properties. Two different conditions of the thermomechanical treatment were adopted and resulting microstructures with different grain sizes were characterised by optical and high resolution scanning electron microscopy (FEG SEM). Hardness measurements and tensile tests were performed to characterize mechanical properties. To reveal structural changes induced during thermomechanical treatment and during tensile tests a magnetic induction method was additionally applied. Experimental study validated the ability of the treatment to produce an austenitic stainless steel with the grain size of about 1.4 μm which exhibits tensile strength of around 1000 MPa while ductility remains close to 60 %. The results obtained for both thermomechanical conditions are compared and the relationship between microstructure refinement, phase content and mechanical properties is discussed.

Keywords: Austenitic steel, ultra-fine grained microstructure, thermomechanical heat treatment, mechanical properties, hardness

1. INTRODUCTION

Ultrafine-grained (UFG) materials are in the focus of growing interest. There is a considerable amount of methods of grain refinement [1, 2] of various materials [3, 4]. Usually, UFG materials exhibit exceptional high strength properties but also drop of ductility. Austenitic stainless steels (ASS) have a great potential in many industrial applications due to their excellent corrosion resistance, weldability and good mechanical properties [5]. Moreover, recently some possibilities of utilization in medical research were indicated when a favourable pre-osteoblast response to UFG structure of austenitic stainless steel was observed [6]. For further improvement of mechanical properties, i.e. increase of the strength of ASS, the effect of grain refinement is considered. The reversion annealing is an efficient method to produce ASS with nano/submicron grain size [7]. This thermo-mechanical treatment is based on the phase transformation of austenite during cold rolling to fine grained strain induced α-martensite, which is during reversion annealing transformed again to austenite. Using this procedure the UFG structure can be reached with the supposed effect of strength enhancement while retaining the excellent ductility of the resulting material. The phase and microstructure evolution of ASS type 301 before and after similar annealing conditions was discussed recently by Rajasekhara et al. [8].

The aim of this paper is to contribute to the knowledge of microstructure and mechanical properties of a conventional stainless steel modified by the special thermo-mechanical treatment to the high performance UFG material using two different conditions.

2. EXPERIMENTAL

2.1 Materials and methods

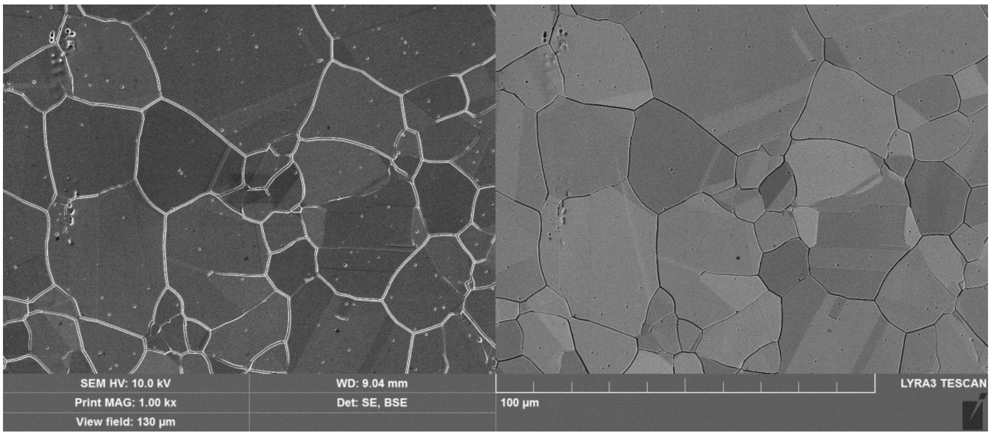
A commercial stainless steel AISI 301 LN having nominal chemical composition (in wt.%) of Fe-0.017C-0.15N-0.52Si-1.29Mn-17.3Cr-6.5Ni-0.15Mo was thermo-mechanically treated at the University of Oulu, Finland. Sheet material with fully austenitic initial microstructure was deformed with cold rolled reduction (CRR) of 63 % and 72 % to obtain the microstructure with deformation induced martensite. After reversion annealing performed at 1000 °C/200 s and 800 °C/1 s the microstructure containing coarse and ultrafine equiaxed austenitic grains was achieved. From the processed sheet material the flat „dog-bone" shape specimens were cut. The shape and dimensions were chosen according to the size of the annealed zone and specimen gage length of 10 mm was placed in the middle of annealed zone, i.e. in the part having the uniform thermo-mechanical treatment. Vickers hardness measurement with 10kg load (HV10) was performed using a testing machine Zwick Z2.5 equipped with a hardness head ZHU0.2 with optics. Tensile test was conducted at the room temperature using a universal test system INSTRON 8862 with electromechanical actuator. Microstructure was characterised using an optical metallographic microscope Neophot and a scanning electron microscope Tescan LYRA 3 XMU equipped with an EBSD analyser. Specimens for the microstructure analysis were mechanically and finally electrolytically polished. Chemical etching of annealed zone was performed to visualize grain boundaries. For EBSD analysis the mechanical polishing was followed by mechanical-chemical polishing using colloidal solution (OPS). The EBSD analysis was performed using AZTEC software. The presence and volume fraction of α'-martensite (MVF), which is magnetic contrary to paramagnetic austenite, was also assessed using Feritscope Fischer FMP 30 with an attached probe EGAB1.3. This device measures changes in voltage induced by the changes of the magnetic field of the specimen. Because of the shape and thickness of measured specimens several correction factors have to be taken into account. X-ray diffraction was performed as well to check the MVF in the microstructure using a diffractometer XPert MPD.

3. RESULTS AND DISCUSSION

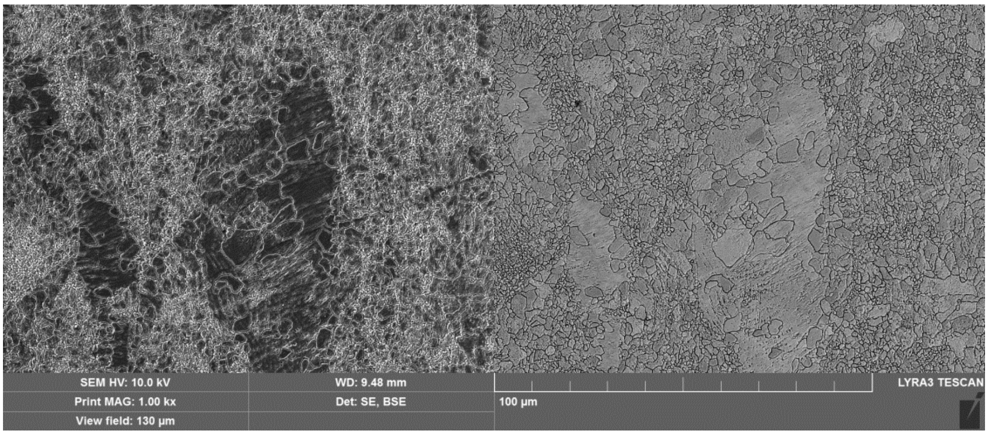
3.1 Microstructure

The results of the microstructure observation after thermo-mechanical treatment are illustrated in ***Fig. 1***. Annealing of the cold-rolled material was performed at two conditions: 1000 °C/200 s and 800 °C/1 s which resulted in two levels of the grain size. Analysis of the grain size and the distribution, texture and orientation were performed using both optical and electron microscopy. Annealing at 1000 °C/200 s results in the coarse grained microstructure (see ***Fig. 1a)***) with equiaxed austenitic grains containing typical annealing twins and with the average grain size of reverted austenite evaluated by the linear-interception method of 14 μm. Annealing at 800 °C/1 s gives more or less uniform fine-grained microstructure. It is approximately 10 times finer and consists of grains with average size of 1.4 μm. Also some individual bigger grains (around 20 μm) can be found in the microstructure (i.e. some of austenitic grains were not transformed to martensite during cold rolling). Presence of two sizes of grains, i.e. bimodality is more pronounced in case of UFG material (see ***Fig. 1b)***) and is caused by the level of rolling reduction. Due to the metastability of 301LN type steel at different conditions of cold rolling (different thickness reduction) different martensite volume fraction is achieved. Results concerning MVF evaluation will be presented, compared and discussed later.

Results concerning the grain size of UFG material are in good agreement with results in the literature for similar treatment, i.e. annealing at 800 °C for 10 s [9]. Nevertheless our grain size is about 2 times higher than in the work of Rajasekhara et al. [10] where UFG austenite produced by cold rolling and annealing at 800 °C for 1 s had a grain size as small as 0.54 μm.



a)



b)

Fig. 1 SEM micrograph in SE (left) and BSE (right) mode of the austenitic stainless steel 301 LN in non-deformed state (a) coarse grained structure after annealing at 1000 °C/200 s, (b) ultrafine grained material annealed at 800 °C/1 s

3.2 Tensile tests

According to the literature the UFG materials usually exhibit higher strength than theirs coarse-grained counterparts; however, the ductility of the most UFG materials is suppressed. It is caused by the low work hardening rate, which contributes to the reduction of the uniform elongation prior necking under uniaxial loading. The favourable and desirable combination of high strength and high ductility of the UFG 301LN steel was reached using special thermo-mechanical treatment. Stress-strain plots obtained at uniaxial tensile tests for two types of used materials are presented in ***Fig. 2***. The main parameters evaluated from the tensile tests for both CG and UFG materials are listed in ***Table 1***.

The UFG sample exhibits the tensile strength higher than 1000 MPa together with extensive plasticity characterized by total elongation almost 60 % at final fracture. The yield stress for the UFG material is two times higher than for the coarse grained steel. The hardening ratio (ratio of the ultimate tensile strength to the yield stress) of the UFG material is lower than for CG material.

3.3 Hardness measurement

The Vickers hardness measurement (HV10) of cold rolled specimens, annealed specimens and specimens after the tensile test for two microstructural states, i.e. annealed at 1000 °C/200 s and 800 °C/1 s was performed. Loading curves obtained for annealed state and state after the tensile test are in ***Fig. 3.*** Final values of the Vickers hardness calculated from the applied maximum load and the size of corresponding indent are listed in ***Table 1.***

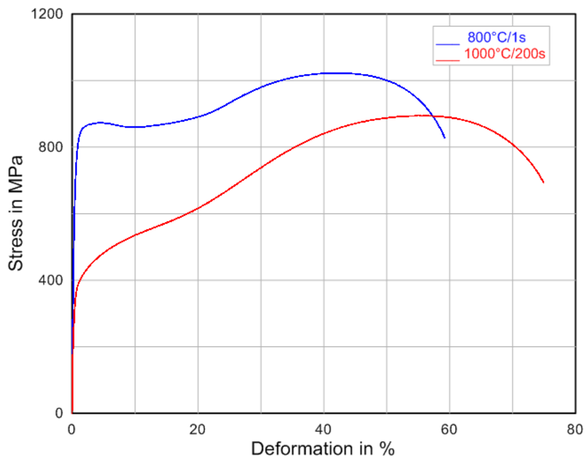


Fig. 2 Stress-strain curves obtained from tensile test for 301LN steel annealed at 800 °C/1 s and 1000 °C/200 s

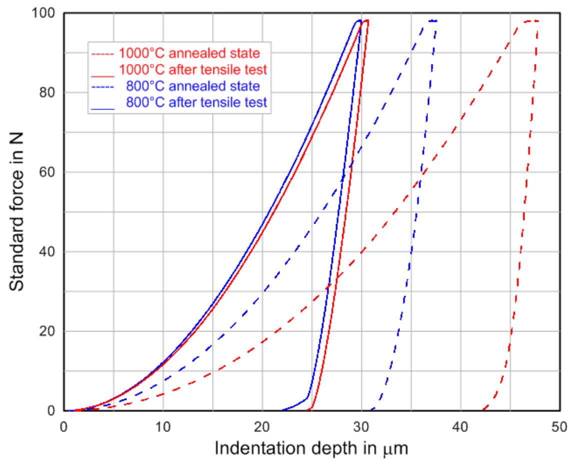


Fig. 3 HV10 loading curves of material before and after tensile tests for 301LN steel annealed at 1000 °C/200 s and 800 °C/1 s

From the plot of loading curves (***Fig. 3***) as well as from the values of the hardness measurement (***Table 1***) we can see that hardness of CG and UFG specimens after tensile test are approximately equal. Nevertheless the hardness of the UFG is slightly higher. Significant difference between hardness of CG and UFG material in the annealed state is due to the refinement of the microstructure and by the reinforcing effect of grain boundaries.

Table 1 Mechanical properties obtained from the tensile tests of specimens of 301LN steel with two types of thermo-mechanical treatment and the hardness measurement in cold rolled and annealed state and after tensile test.

Material	Rp0.2 [MPa]	Rm [MPa]	A_f [%]	HV 10 cold rolled	HV 10 annealed	HV 10 after tension
CG (1000 °C/200 s)	297	889	74.5	522	196	478
UFG (800 °C/1 s)	685	1056	58.9	549	313	495

3.4 Martensite volume fraction

Martensite volume fraction (MVF) in the microstructure is a function of many variables as annealing time and temperature, temperature of cold rolling, thickness reduction, chemical composition of the material etc. In the present work the MVF for different structural states of the material was evaluated by various methods and results are listed in ***Table 2***.

To assess the MVF in the material under investigation the evaluation was performed using three different techniques: magnetic properties measurement by Feritscope, EBSD technique and X-ray diffraction method.

Ferritoscope: Results of the measurement of the ferrite fraction using ferritoscope fall in expected ranges. Material in cold rolled state has 50 to 60 % MVF which is slightly lower than reported by Rajasekhara et al. [10]. In annealed state the values of about 0.2 % correspond to the state when all martensitic phase introduced into the material during cold rolling was successfully reverted to austenite. After tensile test the value of MVF (measured in the vicinity of the fracture surface) in both materials was from 52 to 55 %.

Table 2: Martensite volume fraction of AISI 301 LN stainless steel in two different structural states evaluated by measurement of magnetic properties using Feritscope, from EBSD analysis and X-ray diffraction.

Martensite volume fraction (MVF)	CG material (1000 °C/200 s)			UFG material (800 °C/1 s)		
Material state	Cold rolled	Annealed	After tension	Cold rolled	Annealed	After tension
Method	% of ferromagnetic phase					
Feritscope	50	0.2	52	58	0.2	55
EBSD	*	(39)	57	*	(31)	55
XRD	*	1	94	*	1	92

EBSD: MVF measured by the EBSD analysis (***Table 1***) are in good agreement with the results of the Ferritoscope only for material states containing high percentage of ferromagnetic phase. In annealed samples the EBSD results yield high values of MVF contrary to the measurement by Ferritoscope. This disagreement is due to the effect of the surface preparation using grinding and polishing. In a metastable steel AISI 301 LN the grinding and polishing of the specimen surface causes plastic straining of the surface layer an thus generation of stress induced martensitic transformation.

Moreover the results for both materials (UFG and CG) were strongly dependent on the resolution and place of mapping. When the analysed area was small the level of uncertainty of phase identification, was low (the “zero solution” was up to 10%). Nevertheless, the results were strongly dependent on the analysed place (due to local microstructural properties and heterogeneity of cold rolled and annealed material). When bigger area was analysed to integrate the properties then almost 20 to 30% of all measured point were uncertain in the identification of the crystal microstructure. The difference was higher for UFG material because of higher amount of grain boundaries. At grain boundary it is not possible to avoid the phase overlapping.

X-Ray diffraction: This method evaluates phase composition from higher depth (volume) of the material than EBSD and therefore it is not so much influenced by the surface preparation quality. MVF of the specimens in annealed state are in good accord with the results from Ferritoscope, i.e. after annealing the complete austenitization was reached. MVF of the specimens subjected to a tensile test is considerably higher than measured by the Feritscope. It is not yet clear which value is closer to the actual value of MVF. The results of X-Ray measurement could be influenced by the texture created in the material during the tensile test which decreases the X-Ray measurement accuracy and resulting values can be overestimated.

4. SUMMARY

Reversion annealing performed using two different sets of parameters was used to prepare two types of microstructure of austenitic stainless steel 301 LN. Annealing at 1000 °C/200 s resulted in equiaxed coarse grained microstructure with the grain size of 14 μm while annealing at 800 °C/1 s resulted in the UFG microstructure with average grain size of 1.4 μm. Results of tensile tests of two types of material confirmed higher mechanical properties of UFG material compared to CG material. Yield stress of the UFG material was more than twice of that of CG material and tensile strength of UFG material reached the value above 1000 MPa together with an extensive ductility. Vickers hardness after tensile test are almost the same (HV10 of 480 or 490), they are independent on the coarseness of the microstructure. Martensite volume fraction was evaluated using three methods. Ferritoscope yields MVF for specimens in annealed and in deformed state. The results of the EBSD method must be interpreted with care since data are collected from the surface and are thus influenced by surface preparation, especially in metastable steels. X ray method has tendency to overestimate the MVF content in specimens with texture.

ACKNOWLEDGEMENT

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LITERATURE

[1] Valiev RZ, Islamgaliev RK, Alexandrov IV. Progress in Materials Science 2000;45:103.

[2] Todd I, Chlup Z, O'Dwyer JG, Lieblich M, Garcia-Escorial A. Materials Science and Engineering a-Structural Materials Properties Microstructure and Processing 2004;375:1235.

[3] Lukas P, Kunz L, Svoboda M. Materials Science and Engineering a-Structural Materials Properties Microstructure and Processing 2005;391:337.

[4] Chlupova A, Chlup Z, Kruml T, Kubena I, Roupcova P, Ltd T. Nanocon 2011 2011:259.

[5] Talonen J, Nenonen P, Pape G, Hanninen H. Metallurgical and Materials Transactions a-Physical Metallurgy and Materials Science 2005;36A:421.

[6] Misra RDK, Thein-Han WW, Pesacreta TC, Hasenstein KH, Somani MC, Karjalainen LP. Advanced Materials 2009;21:1280.

[7] Hamada AS, Karjalainen LP, Surya PKCV, Misra RDK. Materials Science and Engineering: A 2011;528:3890.

[8] Rajasekhara S, Karjalainen LP, Kyröläinen A, Ferreira PJ. Materials Science and Engineering: A 2010;527:1986.

[9] Hamada AS, Karjalainen LP. Materials Science and Engineering a-Structural Materials Properties Microstructure and Processing 2010;527:5715.

[10] Rajasekhara S, Ferreira PJ, Karjalainen LP, Kyrolkainen A. Metallurgical and Materials Transactions a-Physical Metallurgy and Materials Science 2007;38A:1202.

[11] Huang JX, Ye XN, Gu JQ, Chen X, Xu Z. Materials Science and Engineering a-Structural Materials Properties Microstructure and Processing 2012;532:190.