On the microstructural-textural characterization and deformation analysis of a nano/ultrafine grained Fe-20Cr-8Mn-0.3N duplex alloy with superior mechanical properties

Moallemi, Mohammad; Zarei-Hanzaki, Abbas; Kim, Sung Joon; Alimadadi, Hossein

Published in:
Materials Characterization

Link to article, DOI:
[10.1016/j.matchar.2019.109878](https://doi.org/10.1016/j.matchar.2019.109878)

Publication date:
2019

Document Version
Peer reviewed version

[Link back to DTU Orbit](#)

Citation (APA):
Moallemi, M., Zarei-Hanzaki, A., Kim, S. J., & Alimadadi, H. (2019). On the microstructural-textural characterization and deformation analysis of a nano/ultrafine grained Fe-20Cr-8Mn-0.3N duplex alloy with superior mechanical properties. *Materials Characterization*, 156, [109878].
<https://doi.org/10.1016/j.matchar.2019.109878>

General rights

Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

- Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
- You may not further distribute the material or use it for any profit-making activity or commercial gain
- You may freely distribute the URL identifying the publication in the public portal

If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.

On the microstructural-textural characterization and deformation analysis of a nano/ultrafine grained Fe-20Cr-8Mn-0.3N duplex alloy with superior mechanical properties

Mohammad Moallemi, Abbas Zarei-Hanzaki, Sung Joon Kim, Hossein Alimadadi

PII: S1044-5803(18)33560-5

DOI: <https://doi.org/10.1016/j.matchar.2019.109878>

Reference: MTL 109878

To appear in: *Materials Characterization*

Received date: 23 December 2018

Revised date: 16 August 2019

Accepted date: 18 August 2019

Please cite this article as: M. Moallemi, A. Zarei-Hanzaki, S.J. Kim, et al., On the microstructural-textural characterization and deformation analysis of a nano/ultrafine grained Fe-20Cr-8Mn-0.3N duplex alloy with superior mechanical properties, *Materials Characterization* (2019), <https://doi.org/10.1016/j.matchar.2019.109878>

This is a PDF file of an article that has undergone enhancements after acceptance, such as the addition of a cover page and metadata, and formatting for readability, but it is not yet the definitive version of record. This version will undergo additional copyediting, typesetting and review before it is published in its final form, but we are providing this version to give early visibility of the article. Please note that, during the production process, errors may be discovered which could affect the content, and all legal disclaimers that apply to the journal pertain.

On the microstructural-textural characterization and deformation analysis of a nano/ultrafine grained Fe-20Cr-8Mn-0.3N duplex alloy with superior mechanical properties

Mohammad Moallemi^{1,2*}, Abbas Zarei-Hanzaki^{1†}, Sung Joon Kim², Hossein Alimadadi^{3,4}

¹ The Complex Laboratory of Hot Deformation & Thermo-Mechanical Processing of High Performance Engineering Materials, School of Metallurgy and Materials Engineering, College of Engineering, University of Tehran, Tehran, Iran.

² Graduate Institute of Ferrous Technology, Pohang University of Science and Technology, Pohang, South Korea.

³ DTU Danchip/Cen, Technical University of Denmark, Fysikvej, Building 307, 2800 Kgs. Lyngby, Denmark.

⁴ Danish Technological Institute, Kongsvang Alle' 29, 8000 Aarhus C, Denmark.

Abstract

The microstructure of a Fe-20Cr-8Mn-0.3N duplex stainless steel was tailored by combination of cold deformation and subsequent martensite reversion annealing. It was shown that by reversion treatment at proper temperature and time nano/ultrafine grained (NG/UFG) with superior mechanical properties (1GPa yield strength and 40% elongation to fracture) can be achieved. Supplementary dilatometry analysis and thermodynamic studies proved that the shear reversion mechanism of α -martensite to austenite was responsible for the formation of NG/UFG microstructure. Detailed transmission Kikuchi diffraction (TKD) characterization revealed that austenite grain size distribution was broad and narrowed by increasing the reversion annealing time. The observed inhomogeneity in grain size distribution was ascribed to the large variation in morphology and deformation level of martensite. Additionally, it was proved that certain stable original orientations of austenite grains i.e. $\langle 110 \rangle //$ ND are highly resistant to martensitic transformation, causing stability of originally large austenite grains. The austenite texture after reversion annealing in the present duplex alloy was found to be similar to that of single phase austenitic steels. It was shown that Brass and Goss textures, which were formed during cold rolling, persist after reversion annealing irrespective of austenite metastability degree. Transmission electron microscopy (TEM) revealed that superior mechanical strength observed in the NG/UFG material, stems from both grain refinement and chromium nitride precipitates which form during revision annealing. The extraordinary plasticity of the UFG microstructure was recognized to be due to the complex deformation mechanisms of NG/UFG austenite, specifically nano size twin/twin-like bands, as well as deformation accommodation by ferrite phase.

Keywords: Microstructure; Phase transformation; duplex stainless steels; Strain-induced martensite; Nano/ultrafine grain structure; Texture

* Corresponding author. Tel: +98 9370490237; Fax: +98 21 88006076
E-mail address: m.moallemi@ut.ac.ir (Mohammad Moallemi).

† Co-corresponding author. Tel: +98216114116; Fax: +98 21 88006076
E-mail: areih@ut.ac.ir (Abbas Zarei-Hanzaki)

1. Introduction

Ultrafine grained (UFG) ferrous materials with an average grain size of less than 1 μm are of high technological interest due to their superior mechanical properties [1–6]. The most common methods for achieving a UFG/nano-grained (NG) material are [7]: (i) severe plastic deformation (SPD) techniques, and (ii) advanced thermo-mechanical processing (ATP) methods. SPD is solely based on mechanical deformation. In this method a large plastic strain is exerted often by several stage of deformation. This and general low ductility of final material are the main shortcomings of SPD processes. In ATP methods, thermal and mechanical processes are combined to achieve the intended grain refinement. However, it is very challenging task to obtain average grain size below 1 μm by ATP [3,4]. Recently, the combination of severe cold deformation and phase reversion annealing is employed to obtain UFG/NG in metastable austenitic stainless steels (ASSs) [8–12]. In this approach, severe deformation of austenite leads to a strain-induced transformation of face centered cubic (FCC) γ -austenite to body centered cubic (BCC) α -martensite, hence, achieving a notable grain refinement. Subsequent short-duration annealing leads to reversion of the strain-induced martensite (SIM) to NG/UFG austenite [8,13]. This approach requires phase transformation, as observed in most metastable austenitic alloys [8–10]. By optimization of deformation and annealing, UFG material with an ultrahigh yield strength (YS) and excellent ductility is obtained [14].

1.1 Achieving NG/UFG structures by $\gamma \rightarrow \alpha$ transformation

The process of cold deformation to activate $\gamma \rightarrow \alpha$ transformation and subsequent annealing to revert austenite ($\alpha \rightarrow \gamma$ transformation) is identified as an effective method to produce NG/UFG material [8–10,14]. This method has been successfully applied to different commercial ASSs such as 301[14,15], 301LN [16], 316L [17], 201 [18] , 201L [19], and 204Cu [20]. To

successfully produce a NG/UFG material: (a) γ -austenite should be easily transformed to α -martensite by cold deformation to achieve a large fraction of α -martensite. (b) The α -martensite should be further deformed by cold rolling to maximize the dislocation density of α -martensite. (c) The deformed α -martensite should be reverted to austenite by minimal thermal treatment to suppress the growth of the newly forming austenite grains. (d) The martensite-start temperature (M_s) of the reversed austenite should be below ambient temperature to achieve a fully austenitic material. Considering all these points, it is of high importance to deliberately tailor the cold deformation stage and also reversion annealing.

Cold deformation: saturation strain (ϵ_s) is the strain above which the fraction of α -martensite remains constant i.e. by applying further strain α -martensite volume does not increase. By cold deformation beyond ϵ_s , martensite fragmentation takes place, i.e. dislocation density increases. From this point of view, a low ϵ_s is advantageous for an effective grain refinement. ϵ_s is affected by many parameters such as chemical composition, cold rolling temperature, route of imposing strain, and strain rate [21–23] .

The stability of austenite dictates the progress of phase transformation by deformation and often studied in terms of stacking fault energy (SFE) and the chemical driving force of austenite transformation to α -martensite ($\Delta G^{\gamma \rightarrow \alpha}$) [24,25]. In general, the stability of austenite decreases by decreasing the SFE and increasing $\Delta G^{\gamma \rightarrow \alpha}$. The driving force of austenite to α -martensite transformation is often expressed by M_{d30} temperature. M_{d30} is a temperature at which 50% martensite is formed at 0.3 true strain in uniaxial tension. According to Nohara et al. [26], M_{d30} temperature is calculated using the following equation.

$$M_{d30}(^{\circ}\text{C}) = 551 - 13.7(\% \text{Cr}) - 29(\% [\text{Ni} + \text{Cu}]) - 8.1(\% \text{Mn}) - 18.5(\% \text{Mo}) - 9.2(\% \text{Si}) - 68(\% \text{Nb}) - 462(\% [\text{C} + \text{N}]) - 1.42(\text{GS}-8) \quad (1)$$

where elemental composition is in weight percent. Eq. 1 is widely used to estimate the stability of the austenitic alloys. An alloy with high M_{d30} transforms more readily to martensite during deformation and therefore is more suitable for the previously mentioned grain refinement.

As pointed out above, the other parameter that should be considered is SFE as it governs the deformation mechanism of austenitic alloys [27,28]. The dominant deformation mechanisms for high to low SFE is as follows: (i) partial and perfect dislocations glide, (ii) mechanical twinning and (iii) ϵ -martensitic and subsequent α' -martensite transformations. The transformation of ϵ -martensite to α' -martensite is explained based on the Olson and Cohen mechanism [29]. Recently, Behjati et al. [25] reported a critical ratio of $SFE/\Delta G^{\gamma \rightarrow \alpha'}$ for successful grain refinement by martensite reversion. They found that a logical relation between ϵ_s and $SFE/\Delta G^{\gamma \rightarrow \alpha'}$ can be used as criterion for production of UFG alloys.

Reversion annealing: The reversion annealing parameters are important factors in producing NG/UFG microstructure. In one hand, the annealing time and temperature should be sufficiently high that the α' -martensite completely transforms into γ -austenite. On the other hand, the annealing time and temperature should be sufficiently low that austenite grain growth is suppressed during reversion annealing. It is noted that microalloying has been also employed to inhibit grain growth of reversed austenite [30].

The α' -martensite $\rightarrow$ γ -austenite transformation takes place both by diffusional and shear mechanisms. The shear reversed austenite grains inherit the martensite morphology and are of high dislocation density immediately after the reversion [31,32]. The shear reversion is more probable to take place at relatively high temperatures where the newly reversed austenite phase tends to be refined by recovery and recrystallization processes [31]. In contrast, diffusional reversed austenite is recognized by the nucleation of equiaxed austenite grains within the

martensitic matrix, in which the austenite grains gradually grow during annealing. Accordingly, it is suggested that [33] the annealing conditions should be selected for reversion transformation by diffusional mechanism instead of shear reversion; this promotes the formation of UFG microstructure. Misra et al. [14,34] and Somani et al. [35] also reported the formation of NG/UFG structure in 301LN ASS by diffusional mechanisms. In contrast, it is also reported that shear reversion was responsible for the formation of NG/UFG structures in 301 ASS (nominal chemical composition: Fe-0.10 C-1.06 Si-1.18Mn-16.7Cr-6.3 Ni-0.65 Mo-0.047N) [14]. Several studies are carried out on the mechanism of α -martensite $\rightarrow$ γ -austenite transformation during annealing, however, the effect of reversion mechanism on the formation of UFG/NC structure is not fully understood. In addition, grain growth after complete reversion and recrystallization is not thoroughly investigated in previous studies.

1.2. Mechanical behavior of NG/UFG material

NG/UFG materials produced by ATP, in contrast to other NG/UFG materials (e.g. SPD), exhibit notably higher ductility. Somani et al. [35] suggested that dislocation glide and enhanced strain-induced α -martensitic transformation are the main governing deformation mechanisms in the NG/UFG austenitic 301LN steels and are responsible for the high ductility. Kisko et al [36] showed that, beside the dislocation glide as main deformation mechanism, formation of SIM in UFG austenitic alloy (of grain size 0.5 μ m) is an effective deformation mechanism, however, the kinetic of SIM formation with strain was faster in fine grains than coarse grains.

There is a general agreement that SIM transformation has a significant role in obtaining a desirable ductility of UFG microstructures, however, many aspects of deformation mechanisms of high-ductility NG/UFG austenitic structures are not fully explained, yet.

1.3 Motivations of present study

In the present study, it is tried to elucidate some less explored aspects of cold deformation and reversion annealing to obtain a NG/UFG duplex stainless steel (DSS). The following points are the motivations behind the study.

1- Many studies are carried out on the formation of nano/ultrafine grain structure in commercial ASSs [14,17,18,37] and non-commercial ASSs including Fe-Cr-Ni-Mn and Fe-Cr-Mn alloying systems [38–40] by reversion of cold worked structures. However, there are also few works on improvement of mechanical properties of DSSs. Chen et al. [41] produced a micro-duplex microstructure consisting of austenite and ferrite from a UNS S32304 using equal channel angular pressing (ECAP) and subsequent annealing. The average yield stress and uniform elongation were increased to 540 MPa and 30% as from 403 MPa and 26% of original coarse-grained material. In another study, Mandal et al. [42] produced a grain refined microstructure of both ferrite and austenite in a lean duplex stainless steel (LDSS) with low-N content using cold rolling and annealing process. Their developed material exhibits YS and tensile strength as high as 798 and 1088 MPa, respectively. They reported uniform and total elongation of 32% and 41%, respectively. The present study is motivated by the question that how cold rolling and annealing can help producing NG/UFG structure in DSS. Accordingly, one of the key points explored in this study is NG/UFG formation in high nitrogen DSS using thermo-mechanical treatment in order to develop a new high strength-high ductility material.

2- As the formation of martensite in each austenite grain during deformation is affected by austenite grain characteristics such as size and orientation (texture), consequently, the reversed austenite grain size is indirectly influenced by these parameters. Thus, special attention is paid to study the homogeneity of grain size obtained by thermo-mechanical process and also the texture

of the material. Although some microstructural features of reversed austenite grains have been investigated by some researchers [11,43,44], many aspects have remained unclear so far. In addition, most of the available studies in the literature are conducted on ASSs, especially 301LN ASS [11,16,37]. However, the DSSs were not extensively explored. The present study aimed to establish a relation between grain size homogeneity and martensite transformation features in a high nitrogen DSS.

3- Many aspects of the mechanical properties of NG/UFG material, specifically high elongation to fracture is not fully understood, yet. It has been reported that [35,36] transformation induced plasticity (TRIP) can affect the plasticity of NG/UFG steels. However, from a general viewpoint, the TRIP effect can be hindered by grain refinement as SFE of austenite increase with a decreasing grain size. For the first time, in a cold rolled and reversed 301LN stainless steel, the inverse dependence of grain size on TRIP effect for the UFG material was recorded by Somani et al. [35]. Kisko et al. [36] also reported that in an austenitic steel (15Cr-9Mn-Ni-Cu), the highest austenite stability was in the austenite of grain size 1–2 μm . Apposing to these reports, Misra et al. [45,46] did not find martensite formation but twinning in a UFG 301LN steel, deformed by nano-indentation or tensile straining. So, there is no general agreement on the influence of grain size on the deformation characteristics of austenite. Hence, in the present study, we focused on the deformation mechanism in order to find out the dependency of mechanical properties to austenite stability.

2. Materials and methods

An experimental metastable DSS was received in as electroslog remelted condition. The chemical composition of the alloy is presented in Table 1. The material was hot rolled at

temperatures in the range of 1100-1200 °C. The hot rolled material was then solution annealed at 1100 °C for 60 min.

The applied thermo-mechanical processing was consisted of an 80% cold rolling followed by reversion annealing at temperatures in the range of 750-1100 °C for 30, 60 and 180 seconds. The schematic representation of the applied thermo-mechanical processes is shown in Fig. 1.

Dilatometry analysis was carried out in a wide range of heating rates from 0.5 °C/s to 100 °C/s between 300°C and 1000 °C to determine the critical phase transformation temperatures. Phase analysis was performed by Ferritescope (Fischer MP30) and X-ray diffractometer (XRD Philip's X'Pert using Cu K α). The microstructural characterizations were carried out using scanning electron microscopes (SEM Philips XL30 and SEM LEO 1455 VP) after electro-etching in 65% nitric acid solution applying a potential of 1.5 V. The grain size measurements were performed using Image Tool software. Tensile tests were performed according to the ASTM E8 standard [47] at room temperature using a Gotech tensile machine. It is worth mentioning that the tensile direction was parallel to the rolling direction, and all the tests were repeated 3 times.

In order to investigate the deformation mechanism(s), tensile deformed microstructures were characterized by a transmission electron microscopy (TEM), JEOL JEM 2100. TEM specimens were prepared by grinding and polishing a thin foil down to 70 μ m thick, followed by jet polishing at room temperature with a solution of 10% perchloric acid in acetic acid.

For phase differentiation and orientation mapping, electron backscatter diffraction (EBSD) and transmission Kikuchi diffraction (TKD) were performed in an FEI Nova NANOSEM 600TM equipped with a field-emission gun and an EBSD/TKD system from Bruker, e⁻ Flash HR camera (GmbH, Berlin, Germany). The samples for EBSD analysis were first mechanically polished using SiC paper (from 220 to 2000 mesh size) and then electrochemically polished in an electrolyte of 15 vol.% perchloric acid and 85 vol.% acetic acid using LectroPol-5 electrolytic polishing machine from Struers. EBSD measurement was performed in a square grid with a step size of 300 nm. An electron probe current of 3.1 nA at an acceleration voltage of 12 kV and camera exposure time of 7 ms was utilized. TKD measurement was performed on an electron

transparent lamella in a square grid with a step size of 20 nm. An electron probe current of 2.3 nA at an acceleration voltage of 30 kV and camera exposure time of 10 ms was utilized. It is noted that the TKD measurements were carried out using an on-axis set up [48]. Data analysis of EBSD and TKD was carried out using Esprit 2.0 from Bruker.

3. Results and discussion

3.1. Phase characterization and microstructural aspects

Cold deformation stage: X-ray diffraction patterns at different cold reduction levels are shown in Fig. 2a. As can be seen, the solution-treated material is composed of austenite and ferrite. The fractions of ferrite and austenite were measured to be 26% and 76%, respectively. Following Fig. 2a, after 10% and 20% reductions by cold rolling, austenite is partially transformed to the ϵ -martensite (HCP) and α -martensite. By further deformation up to 60% reduction, X-ray diffraction pattern is mainly composed of $(110)_{\alpha/\alpha}$, $(200)_{\alpha/\alpha}$, $(211)_{\alpha/\alpha}$, $(220)_{\alpha/\alpha}$ peaks. Austenite reflections intensity are notably decreased and $(101)_{\epsilon}$ peak is absent. This suggests that austenite transforms to α -martensite in a two-step process, γ -austenite $\rightarrow$ ϵ -martensite $\rightarrow$ α -martensite. This transformation succession was originally discussed by Olson and Cohen [29]. Based on their model, the superimposed shear created by two intersecting ϵ -martensite bands leads to shifting of the original austenite lattice close to the α -martensite structure. It is noted that, formation of SIM is expected as the SFE of the austenite phase in present alloy is calculated to be as low as 20 mJ/m² (based on Curtze et al. model [49]).

Fig. 2b. shows the volume fraction of α -martensite as a function of effective strain. The volume fraction of martensite increases by deformation and reaches ϵ_s of approximately 1.1 at 65% reduction. Comparing ϵ_s of the steel in this study and other austenitic steels provides a better understanding of the relative stability of the alloy. Järvenpää et al. [50] reported $\epsilon_s=0.4$ for a 301LN ASS which is notably lower than the present alloy. The SFE value of their material

calculated to be 27mJ/m^2 . This means that despite higher SFE value in their ASS, the austenite phase more readily transforms to SIM than the present DSS alloy. This may stem from the presence of ferrite in the microstructure. As a part of imposed strain is accommodated by ferrite, the stored energy in the austenite phase reduces and consequently the driving force of martensite formation decreases. Similar behavior has been also presented by Breda et al. [51]. Despite higher SFE of AISI 304L than that of 2205 DSS, a retarded SIM transformation kinetic and lower martensite fraction were observed for 2205 DSS. However, the stability of DSS in present study is lower than the 2205 DSS, due to lower concentration of alloying elements.

Reversion annealing stage: Fig. 3 shows the variation of Ferritescope number (A number read by Ferritescope instrument) as a function of annealing conditions. The minimum Ferritescope numbers are obtained at $900\text{ }^{\circ}\text{C}$. It means that the complete reversion of α -martensite to austenite occurs at approximately $900\text{ }^{\circ}\text{C}$. The dilatometry result in Fig. 4 shows that the complete reversion of α -martensite to austenite is at $880\text{ }^{\circ}\text{C}$ which is in good agreement with the obtained result by Ferritescope analysis. By increasing the annealing temperature above $950\text{ }^{\circ}\text{C}$, the Ferritescope numbers gradually raises. This stems from the increase in ferrite fraction. Since the complete reverse transformation of martensite to austenite occurs at 900°C , the microstructural examinations were mainly carried out on the materials annealed at this temperature.

Fig. 5a is a bright field TEM micrograph of fully reversed specimen upon annealing at $900\text{ }^{\circ}\text{C}$ for 30 s. The ultrafine austenite grains (average grain size of about 400 nm) can be clearly seen in microstructure neighboring coarse ferrite grains. Another noteworthy feature observed in the micrograph is the precipitates (marked by white arrows) which are present in the austenite grains (NG and UFG) as well as ferrite grains adjacent to austenite phase. For better visibility, a selected part of Fig. 5a is magnified and presented in Fig. 5b. The inset images show selected

area diffraction (SAD) pattern from precipitates which are indexed as Cr_2N phase. It has been reported that [52–55] in high nitrogen steels ($>0.1\text{wt}\% \text{ N}$) the formation of stable Cr_2N with HCP lattice structure takes place at temperatures of 800°C and above. Previous investigations carried out by Machado and Padilha [54] and Padilha and Rios [55], have shown that Cr_2N precipitates are more stable than the other nitrides such as CrN at high temperatures (800°C and above). This finding suggests that the precipitation hardening in addition to the grain refinement may synergistically enhance the mechanical properties of UFG alloy.

In order to evaluate the homogeneity of grain size distribution in the NG/UFG structure, TKD analysis was conducted on the sample annealed at 900°C for 30 s. The corresponding phase map is shown in Fig. 6a (face centered cubic (FCC) is shown in green phase and body centered cubic (BCC) in red). Austenite grain size distribution is not homogenous, which is visually reflected as areas with relatively large and fine grains. UFG and FG areas are enclosed by white and black circles on the map, respectively. Fig. 6b, c depict the EBSD results of samples annealed at 900°C for 60 s and 180 s, respectively (note the difference in scale bar in Fig. 6a and 6b-c). Calculating the austenite grain size of sample annealed for 60 s reveals that the average grain size corresponding to FG area and CG area are approximately $1.5\ \mu\text{m}$ and $10\ \mu\text{m}$, respectively. By extending the annealing time from 60 s to 180 s the inhomogeneity in the austenite grain size diminishes. The observed inhomogeneity in austenite grain size is in agreement with previous studies on austenite grain refinement via reversion annealing [11,12,44]. It is noted that in the reverse annealed samples, there is a significant difference between austenite and ferrite grain size where the CG ferrite are neighboring UFG austenite (Fig. 6a, 6b). Fig. 7 shows that in the initial stage of annealing treatment ($750^\circ\text{C}/30\ \text{s}$), the fine recrystallized ferrite grains are observed adjacent to the none-reversed SIM. This indicates the ferrite recrystallization takes place prior to

the austenite reversion and recrystallization. This is in agreement with previous studies indicating that the recrystallization of ferrite in nitrogen containing DSSs occurs rapidly and prior to austenite recrystallization [56,57]. Hence, a significant difference between ferrite and austenite grain size is anticipated.

Regarding the inhomogeneity of austenite grain size in reversion annealed samples, there are two major points to consider: (i) The morphology of α -martensite and (ii) lack of martensite formation in austenite grains.

(i) *α -martensite morphology*: it has been reported [33,34] that two types of α -martensite, namely lath and dislocation cell type, can form during plastic straining of metastable austenitic alloys. Misra et al [34] suggested that the formation of dislocation cell type martensite is a prerequisite for obtaining the NG/UFG structure on reversion annealing stage. In other words, the success of the reversion approach in obtaining (NG/UFG) structure depends on the predominance of dislocation cell type structure in the severely deformed martensite. The TEM micrographs of deformed samples at 40% and 80% reduction are presented in Fig. 8a, b, respectively. As can be seen, at 40% reduction, lath α -martensite is dominant in the microstructure. The inset is the corresponding SAD pattern of α -martensite from [111] zone axis. At 80% reduction, the microstructure is a mixture of two types of martensitic structures which are differentiated by SAD patterns. The elongated lath martensite is identified by spot like SAD, while the dislocation cell type martensite has a ring like diffraction pattern. The dislocation cell type martensite is formed due to deformation of lath type martensite. Therefore, there are regions where lath and dislocation cell type martensite coexist. It is noted that with the increase in applied strain, the width of martensite lath and packet size decreases due to the break up and destruction of lath structure, before formation of dislocation cell type martensite. With further deformation the

dislocation cell type martensite forms from the broken laths. Several authors have pointed out that the number of nucleation sites for austenite reversion for low-deformed martensite (lath-type) is considerably low, hence such microstructure cannot effectively be used for grain refinement [46,58,59]. In contrast, the number of nucleation sites for dislocation cell type martensite is considerably large. Thus, the variation in number of nucleation sites of different constituents of microstructure causes variation in the grain size upon reverse treatment. It is noteworthy that the inhomogeneity is inherent to deformation process since the accommodated strain can differ from one austenite grain to another. Thus, there is a variation in the types of martensite in austenite grains for a fixed macro scale reduction level. The variation may include dislocation cell type martensite, dislocation cell + lath type martensite and or ϵ -martensite + α' -martensite. Even assuming SIM fraction is the same inside all grains, the order of formation of martensite causes variation in nucleation sites. For example, the last-transformed SIM are inevitably less deformed than those forming earlier, and it is suggested that this causes variation in grain size upon reversion treatment.

(ii) *lack of martensite formation*: Fig. 9a shows the phase map of the material after 30% reduction. Most of the austenite grains in the microstructure contain ϵ -martensite bands (yellow color), however there are several austenite grains which are almost free of any ϵ -martensite phase. The normal direction orientation map of the austenite phase is shown in Fig. 9b. The austenite grains which are less ready to transform to ϵ -martensite are marked with numbers 1–5. As can be seen, the orientation of these grains is near $\langle 101 \rangle // ND$ (hence, color coded in greenish colors). This orientation is the preferred orientation during rolling. Although there has not been any consistent study about the dependence of ϵ -martensite formation on grain orientation under rolling deformation mode, some researchers reported dependency of twin formation to

orientation in compression mode. Meg et al. [60] reported that the grains with orientation near the $\langle 100 \rangle$ parallel to compression direction are more suitable for twin formation and the grains near the $\langle 101 \rangle$ orientation are mainly free of twins. This was attributed to the higher Schmid factor of mechanical twins shearing system near the $\langle 100 \rangle$ pole. As the shearing system of mechanical twins and ε -martensite is similar, hence, the ε -martensite formation (which is prerequisite for α -martensite formation) is anticipated to follow such trend. In other words, during rolling the matrix grains rotate towards the $\langle 101 \rangle$ directions, which suppresses further ε -martensite formation. Therefore, some of the grains may contain the martensite phase and some may be martensite free depending on their orientation before deformation. During the reversion treatment, grains with a lower amount of SIM, mainly undergo recrystallization (if the imposed deformation is insufficient then even recrystallization may not occur). In such condition, the inhomogeneity in austenite grain size is anticipated. Kisko et al. [44] have also observed the non-uniform distribution of the grain size distribution in a cold rolled and annealed 204Cu ASS with different level of Nb alloying element. They suggested that the non-uniform distribution of the grain size is due to the fact that ultrafine grains are formed from the reverted α' -martensite and larger grains from recrystallization of cold rolled austenite. However, they didn't focus on the orientation dependency of martensite formation. Poulon et al. [61] pointed out that the brass texture (i.e. $\langle 110 \rangle // \text{ND}$) is highly stable against SIM formation. Järvenpää et al. [11] reported that in the FG, UFG 310LN ASS, Goss component disappeared but the Brass $\{110\} \langle 112 \rangle$ component remained (or $\{110\} \langle 111 \rangle$ appeared in FG) during tensile straining. Even though they were more concentrated on the stability of austenite during tensile tests, they observed that the Brass oriented grains (i.e. $\langle 110 \rangle // \text{ND}$) are most stable during cold rolling as well as in tensile deformation. These findings are in agreement with the results in present work where the

$\langle 110 \rangle$ //ND are more stable against SIM formation. So, some retained stable austenite grains during cold work stage, experience recrystallization and grain growth during reversion annealing and consequently have a different size from nano-sized reversed austenite grains.

3.2. α -martensite $\rightarrow$ γ -austenite reversion mechanism

In general, the reversion of $\alpha \rightarrow \gamma$ transformation may occur either by diffusionless (shear) mode or diffusional transformation, which have been well explained for various alloys such as Cr-Ni and ASSs [31,62]. However, the effect of $\alpha \rightarrow \gamma$ reversion mechanism on the formation of UFG material is not completely clear. It has been well established that if shear reversion is the dominant mechanism, the reversion temperatures (i.e. austenite start (A_s) and finish (A_f) temperatures) does not significantly change with heating rate [31,62]. The variation of A_s and A_f temperatures over a wide range of heating rates were measured by dilatometry analysis. Such measurement is carried out and the results are graphed in Fig. 10. The A_s and A_f temperatures are increased by raising the heating rate up to 10 °C/s and then reach a steady state ($A_s=720$ °C and $A_f=880$ °C). This indicates that the diffusional reversion is the dominant mechanism below 10 °C/s heating rate, while the shearing mechanism is dominant above 10 °C/s. Accordingly, due to the small thickness of the specimens (1mm) and the heating procedure in the salt bath, the heating rate in the present study is higher than 10 °C/s. Therefore, the shearing is most probably the dominant reversion mechanism.

Other than dilatometry analysis, the Tomimura et al. [32] approach can be used for the determination of governing reversion mechanism in Fe-Cr-Ni alloys. This approach also successfully applied for Fe-Cr-Ni and Fe-Cr-Mn-N-C alloy systems by other researchers [35]. Somani et al. [35] used a model to evaluate the reversion mechanism of several ASSs. They

calculated the driving force of reversion ($\Delta G^{\alpha' \rightarrow \gamma}$) in terms of temperature and chemical composition by applying Kaufman and et al. [63] equation for ternary Fe-Cr-Ni alloys, as follows:

$$\begin{aligned} \Delta G^{\alpha' \rightarrow \gamma} (J/ m) = & 10^{-2} \Delta G_{Fe}^{\alpha' \rightarrow \gamma} (100 - Cr - Ni) - 97.5 Cr \\ & + 2.02 Cr^2 - 108.8 Ni + 0.52 Ni^2 - 0.05 NiCr \\ & + 10^{-3} T (73.3 Cr - 0.67 Cr^2 + 50.2 Ni - 0.84 Ni^2 - 1.51 CrNi) \end{aligned} \quad (2)$$

where $\Delta G_{Fe}^{\alpha' \rightarrow \gamma}$ is the free energy difference in pure iron, T is the temperature in Kelvin, and the symbols of Ni and Cr represent the chemical composition of each element in wt%. In order to take other alloying elements (i.e. C, N, Mn, Si) into account in Eq. (2), Ni_{eq} (Ni equivalent) or Cr_{eq} (Cr equivalent) are used instead of sole Ni and Cr. Ni_{eq} and Cr_{eq} are determined using Eqs. (3) and (4).

$$Ni_{eq} = Ni + 0.31 Mn + 22 C + 14.4 N + Cu \quad (3)$$

$$Cr_{eq} = Cr + 1.4 Mo + 1.5 Si + 3 Ti + 2 Nb \quad (4)$$

Tomimura et al. [32] reported that the critical driving force for complete shear reversion was approximately -500 J/mol. Regarding the austenite chemical composition of the present alloy and employing Eq. (2)-(4), the driving force of the reversion at 900 °C amounts to -542 J/mol, suggesting that the predominance of shear-controlled mechanism for experimental alloy at this temperature.

3.3. Textural study

Fig. 11 shows the macrotexture analysis results in the form of orientation distribution function (ODF) maps. Fig. 11a-d represent ODF maps of cold rolled and annealed samples at 900 °C for 30 s, 60 s, and 180 s. The cold rolled specimen shows a sharp α -fiber i.e. $\langle 110 \rangle // ND$, Goss $\{110\} \langle 001 \rangle$ and Brass $\{110\} \langle 112 \rangle$ orientations with some spread around the main components.

According to the work of Chowdhury et al. [64] on the texture development of a single phase austenitic steel, the austenite phase after 90% cold roll reduction exhibited Goss and Brass texture along with α -fiber. Regarding the specimens annealed at 900 °C/30 s, the main orientations resemble the cold rolled state, though of a lower intensity. In addition, Copper{112}<111> and S {123}<634> orientations can be detected in ODF map. With prolonged annealing, the texture changes during the grain growth in the austenite phase in a way that the existing texture components become weaker (Fig. 11b). In addition, the formation of an entirely new orientation, {001}<100> Cube, is detected as shown in Fig. 11c, d. Considering the texture evolution in cold rolled and reversed austenite phase, Somani et al. [35] conducted textural study on several metastable ASSs. They found that the texture of reversed austenite phase austenite is dominated by strong Brass and Goss orientations and minor Copper and S {123}<634> orientations. This finding is very similar to the findings in this study. Somani et al. [35] also reported the same main orientations (Brass, Goss, Copper, and S) with equal intensities after the reversion annealing irrespective of reversion mechanism (shear or diffusional). They stated that the reversion mechanism does not affect the texture of the reverted austenite phase. Chowdhury et al. [64], investigated the texture evolution of 316L stainless steel with a stable austenite phase after cold rolling and annealing. They reported that the Brass and Goss texture components persist after annealing.

Based on the above-mentioned findings it is concluded that, first, as in single phase ASS, the rolling textures of the austenite phase in experimental DSS is homogeneous and exhibits a strong α -fiber. This stems from the fact that there is a negligible interaction across FCC/BCC interface, hence, austenite in duplex steel behaves similar to single phase austenite. Second, the texture inheritance of reversed austenite phase is irrespective of austenite metastability so that Goss and

Brass textures remain after reversion annealing. There are several explanations for this observation in duplex steel. In the case of stable austenite phase, retention of the deformation texture or texture components can be attributed to a suppressed growth selection due to low grain boundary mobility caused by solute drag, etc. The high nitrogen content of the experimental duplex alloy may reduce the grain boundary mobility during recrystallization of the austenitic portion of DSS. The other possible reason for texture retention is oriented nucleation or strain-induced boundary migration (SIBM). These two are the determining mechanisms of texture evolution in austenite which may cause recrystallization textures stemming from (and similar to) the deformation textures. These explanations can be applied for that part of initial austenite grains which do not envelope SIM phase. Regarding the reversed austenite phase from SIM, it is shown that [65] the main components in α -martensite giving rise to special texture in austenite, are $\{332\}\langle 113 \rangle$ and $\{113\}\langle 110 \rangle$, which promote austenite Brass and Copper, respectively. Grains with orientations belonging to α -fiber ($\langle 110 \rangle$ direction parallel to rolling direction) of α -martensite also bring about the Copper texture in austenite. The transformation component S, also tends to form from α -martensite orientations mentioned above, near the $\{112\}\langle 113 \rangle$ and also close to α -fiber components. The Goss and Brass component can originate from γ -fiber ($\langle 111 \rangle$ parallel to rolling plane) of α -martensite based on the broadly accepted Kurdjumov–Sachs orientation relationship between α -martensite and austenite [66–68]. It is noted that the typical recrystallization component of FCC alloys is cube which has been observed with further annealing. All the above-mentioned explanations manifest that the remaining texture components form cold deformation after annealing is possible for both metastable and stable austenite phase. In other words, both UFG and FG parts in austenite phase can develop the texture retention after annealing.

3.4. Mechanical behavior of NG/UFG material

To study mechanical properties, tensile testing of reversion annealed samples (those annealed at 900 °C for 30, 60 or 180 s) and also solution annealed sample (as reference) are carried out. The results are shown in Fig. 12a,b. As can be seen in Fig. 12a, the solution annealed specimen has low YS (350 MPa) and a large elongation (75%). Surprisingly, the reversion annealed sample at 900 °C for 30 s with UFG austenite (grain size of 400 nm) shows superior mechanical properties, i.e. 1 GPa YS and 40% elongation. On the other hand, by increasing the reversion annealing duration, the YS decreases moderately but the work hardening potential increases notably, e.g. sample reversion annealed at 900 °C for 180 s shows ~85% elongation at the expense of moderately lower YS of ~630 MPa. This is obviously due to grain growth. It is worth to mention that the decreasing trend of YS with increasing annealing duration at 900 °C is not very pronounced so that YS for highest annealing time i.e. 180 s of the current study, is notably higher than that of solution annealed sample. This may be due to precipitation hardening effect via chromium nitride formation with increasing annealing duration, in addition to the grain refined structure of reversion annealed samples.

Fig. 12c shows the strain hardening rate (SHR) curves of the same samples shown in Fig. 12b. All samples, especially the ones reversion annealed at 900 °C for 60 and 180 s, show a three-stage behavior: (i) decreasing to a minimum level, (ii) increasing to a maximum level and (iii) decreasing down to the fracture strain. This behavior is often referred to as abnormal work hardening since it differs from the behavior of the most metals and alloys showing continuously decreasing work hardening rate [69]. The maximum level of work hardening is attributed to the formation of the α -martensite phase during straining [70] while minimum work hardening is attributed to the formation of ϵ -martensite and partial dislocation glide [71]. Dynamic softening

effect due to initiation of α -martensite formation is another mechanism proposed to be responsible for decreasing SHR in initial stage. In this regard, α -martensite transformation acts as an additional deformation mechanism along with the slip deformation [72]. Regarding the reversion annealed samples, the SHR slope in region ii (where the role of SIM formation on work hardening is the largest) increases with the increase of annealing duration. However, the slope of the SHR curve of 30 s is not notably large. These observations indicate that the SIM is more likely to form by increasing reversion treatment duration. In order to understand the deformation mechanisms of samples produced by different thermo-mechanical processes and their work hardening behavior, the microstructure of the samples after tensile testing are studied. Bright field TEM micrograph, underneath the fracture surfaces of samples annealed at 900 °C/30 is shown in Fig. 13. Fig. 13a shows the deformed Non-UFG area. As can be seen the Non-UFG austenite (Fig. 13a) consists of a small area of deformation induced α -martensite. However, the fraction of α -martensite is not significantly high. Fig. 13b, c show the deformed microstructure of UFG austenite and ferrite, respectively. Dislocations are clearly visible in ferrite, indicating that this phase accommodates part of plastic strain. However, the NG/UFG austenite phase exhibits a complex behavior presenting two characters: (1) the grains possessing dense planar defect features and (2) grains with wavy glide dislocation structures (both characters are shown with white arrows). In the former case, dislocations with straight configuration and consequently planar defects are arranged on some slip planes; whereas, in the latter, there is no regular arrangement of dislocations and a mixture of straight and curved dislocation is recognized. It is noted that the grains with the dense planar defect are more populous in the microstructure. The more populous grains with planar features may be associated with nitrogen element in present alloy and its effect on the planar glide through the short range

ordering (SRO) phenomenon [73]. As is reported, increasing the nitrogen content in nitrogen bearing steels would promote the planarity through SRO effects. SRO in these alloys assists planar slip because each successive dislocation needs a lower stress to pass over the slip plane as compared to the first one which passes through the ordered crystal and consumes an additional energy to shift the atoms from the low energy sites [74]. On the other hand, the variation in grain orientation can cause the observed twofold deformation characteristic (i.e. planar and wavy) in UFG austenite. It has been reported that [75] the planar dislocations structure preferentially form when the tensile axis is close to [111]. In contrast, in the grains with orientation close to [001], the dislocation structure changes to tangled dislocations. Therefore, the dual nature of deformation in the NG/UFG austenite phase is in agreement with previous studies.

For clarification of the characteristics of dense planar defect inside UFG austenite grains, a bright field TEM micrograph with higher magnification is presented in Fig. 13d. Corresponding SAD patterns of two defective areas (A and B) are provided as inset images. The SAD pattern of area “A” shows sole austenite diffraction spots and elongated line between them which are due to the high strain inside the austenite grain. The SAD pattern of area “B” shows spots which corresponds to twinning. It suggests that some of the planar defects inside NG/UFG austenite grains are nano-twins and the others are dense stacking fault (SF) bundles. It means that the deformation accommodation of austenite phase may proceed with twin and/or twin-like nano bands. It is well established that by decreasing grain size, SFE increases [76]. This brings about a change in deformation mode [27], i.e. twinning takes place instead of α -martensite formation, in a manner similar to twinning induced plasticity effect in TWIP steels. The reason for the activation of twin/twin-like features can be also related to the constraining slip mechanism in UFG structures. In general, the slip or shear bands induced plastic deformation is based on the

continuous nucleation of dislocations from Frank Reed sources and their glide through the well-defined slip system. Since the source of dislocation cannot exceed the grain size, smaller grain size dictates a high magnitude of stress for generation and migration of the dislocations. Thus, the dislocation slip is considered to be the main operating deformation mechanism in materials with a grain size of greater than 1 μm [77]. However, our observations show there are some dislocation planar features inside austenite grains which have been formed on the several primary and conjugate slip systems. The proceeding partial dislocations slip in such planar way can lead to the formation of local SF areas inside austenite grains. Finally, the successful overlapping nano SF bands results in nano twin/twin like structure in UFG austenite structure. Thus, the excellent observed plasticity is attributed to the formation of twin/twin-like features in UFG austenite phase of DSS and their effect on the work hardening of the alloy. Misra et al. [77] suggested that the high strength and high ductility of NG/UFG 301 ASS stems from SIM transformation during deformation. However, no SIM phase after plastic straining was detected in the UFG grains in the present work. This is attributed to the strain partitioning between ferrite and austenite. Since a part of strain is accommodated by ferrite (Fig. 13c), the mechanical driving force for SIM transformation decreases and SIM formation is suppressed. Thus, it can be suggested that the extraordinary plasticity of the UFG alloy results firstly from twin/twin-like induced plasticity effect and secondly from strain accommodation of the ferrite phase.

Fig. 14a, b show the deformed microstructures under tension of the samples reversion annealed at 900 °C/ 60 and 180 s, respectively. As can be seen, the microstructure is composed of heavily deformed austenite of high dislocation densities and α -martensite (marked with white arrows). Therefore, the extraordinary plasticity can be associated with the TRIP effect in these conditions. However, since the amount of α -martensite is not very high, SIM accounts as a synergetic

mechanism besides the other active deformation mechanism. In other words, high dislocation density in austenite accounts for a larger amount of strain, and therefore, the most important deformation mechanism is dislocation glide and to a lesser extent TRIP deformation mechanism. If local necking occurs at certain points in the gage length, SIM will be formed preferentially there. The α -martensite/ austenite interfaces act as strong barriers for dislocation glide, and significant strain hardening takes place in the vicinity of local necks. No further deformation in these strain-hardened regions will occur, instead, local necking takes place in areas of lower strain. This repeating process of local necking brings about an extended macroscopic uniform elongation. Therefore, a pronounced work hardening occurs, as illustrated by the plotted SHR curves in Fig. 12c.

All the above microstructure features (Fig. 13, Fig. 14), in connection with presented work hardening behavior (Fig. 12) can explain the mechanical behavior of the processed alloy. Four factors should be considered for interpretation of mechanical behavior. First, the effect of the grain refinement on the SIM formation since the ultrafine grains limit the SIM formation by their size constraints; second, the ferrite phase which accommodate a part of plastic strain; third, the grain orientations which affect the SIM formation and forth, chromium nitride which can lead to pinning effect as well as reducing austenite matrix stability due to depletion of chrome and nitrogen. Accordingly, the moderate SHR slope of 30 s annealed sample in comparison with two others annealed for 60 s and 180 s, can be attributed to the lack of SIM formation in UFG structures and also ferrite role on strain accommodation so that the strain accumulation inside austenite grains is not sufficient for SIM transformation. However, the formation of nano-size twin/twin like bands seems be an effective route to extend work hardening and plasticity of the UFG alloy. In comparison, the formation of SIM in two other samples (i.e. 60 s and 180 s) led to

higher work hardening and plasticity of those ones. Regarding the ferrite phase, however, it seemingly does not exert a significant effect on work hardening as its fraction is not very high and is approximately similar in three reversed samples. For samples annealed for 60 s and 180 s, SIM formation during straining can strongly affect the work hardening and mask the ferrite role. Ferrite phase in present DSS is a discontinuous phase and can just act as a lubricant between austenite grains and be easily deformed during straining. At higher strain level, where the most of austenite phase transforms (fully or partially) to SIM, however, the strain accommodation by ferrite can help enhancing the elongation of the alloy. Mandal et al. [42] applied a thermo-mechanical process including cold rolling and annealing for 3h of a low nitrogen DSS and achieved 798 YS and 41% total elongation. They showed that through the reverse transformation of SIM to austenite an appropriate refinement occurs in the austenite phase. Ferrite grains were also mostly refined by the pinning effect from several tiny austenite islands. Due to the grain refinement in both austenite and ferrite and homogeneous distribution of γ -islands, an optimum combination of strength and ductility has been observed. In their DSS, austenite was discontinuous and its deformation was restricted to an area by the surrounding ferrite. On the contrary, owing to ferrite continuous character, the ferritic matrix suffers less of such restrictions and the imposed strain can find easier deformation paths as deformation proceeds, allowing the activation of a considerable number of slip systems at high deformation levels. However, the austenite phase could participate in work hardening through the formation of SIM. In our DSS, the austenite phase is a continuous phase and can readily deform during straining and SIM forms with more ease which has a significant effect on the work hardening than ferrite.

Regarding two other factors, i.e. precipitation and grain orientation, it is considered that the precipitation cannot strongly affect the deformation mechanisms in the processed alloy. The

microstructural observation of the deformed UFG samples revealed that precipitations present both in grains with planar-glide and grains wavy-glide defects. So, in the present DSS, the precipitation does not seem to change the stability to such an extent that SIM would form in UFGs. However, the precipitations may influence the strengthening and grain growth during reversion annealing. It is supposed that the grain orientation to be more effective on deformation mechanisms of the austenite phase. As it was explained earlier, the grain orientation can influence the stability of austenite in both UFG and CG materials. Accordingly, with controlling the texture of material, the stability of austenite and corresponding mechanical behavior of alloy can be controlled.

A comparison between mechanical properties of NG/UFG sample produced in this study with the results of some recently published works [12,13,17,36,39,41,42,78–80] on the grain refined ASSs and DSSs is presented in Table 2. As can be seen, the developed alloy has an attractive combination of ultrahigh YS and UTS accompanied with good ductility. The combination of strength and ductility can be evaluated using the concept of formability factor (product of UTS and total elongation). The formability index values are also presented in Table 2 and clearly show that the processed alloy in the present study possesses a higher corresponding value than most of its counterparts. Focusing on DSSs, the formability index of the material processed in current study, is higher than that what is reported in [41] but lower than [42]. However, the YS of material processed in current study is higher than that of presented DSSs in Table 2. In addition, the present alloy utilizes alloying elements of lower cost compared to conventional ASSs and DSSs, hence is economically a superior option. Thus, it is appropriate for use in lightweight structures where superior mechanical properties are required.

4. Conclusions

In the present study, the microstructural features and deformation mechanisms of a UFG Ni-free DSS, produced by cold rolling and reversion annealing, were studied. The following conclusions are drawn:

- 1- The volume fraction of α -martensite was increased through the $\gamma \rightarrow \epsilon \rightarrow \alpha$ transformation sequence by increasing the imposed strain at ambient temperature. The measured saturation strain of the material amounts to 1.1 (cold roll reduction about 65%). Despite the lower SFE of austenite phase in the duplex alloy compared with single phase ASSs, a higher saturation strain was observed. This is attributed to the role of ferrite in strain accommodation and subsequently reducing the mechanical driving force of SIM formation.
- 2- The finest austenite grain size of 400 nm was achieved with complete $\alpha \rightarrow \gamma$ reversion by annealing at 900°C/30s, however, an inhomogeneity in the austenite grain size was observed. This was attributed in one hand to the difference in the degree of deformation and morphology of SIM and on the other hand to the lack of martensite formation in some austenite grains due to certain stable original orientations of austenite grains i.e. $\langle 110 \rangle // \text{ND}$.
- 3- The dilatometry analysis accompanied with thermodynamic calculations revealed that the $\alpha \rightarrow \gamma$ reversion transformation was shear controlled at 900 °C. The shear mechanism was responsible for producing UFG austenitic structure.
- 4- The produced NG/UFG structure exhibited a combination of high YS around 1 GPa and 40% elongation. This superior strength is attributed to the grain refinement of austenite phase in present alloy. However, the precipitation hardening effect of nano size

chromium nitride which formed during the annealing stage may be another factor to increase the strength level of the processed alloy.

- 5- The textural study of cold deformed and reversed austenite phase comparing with literature findings indicates that austenite texture is similar to that of single-phase austenitic steels. In addition, the texture inheritance of reversed austenite phase is irrespective of austenite metastability degree. Hence, Brass and Goss texture components remain in the austenite phase after reversion annealing.
- 6- The microstructural study of deformed NG/UFG austenite phase proved that the austenite grains possess both planar and wavy defects which was ascribed to the special grain orientation; however, the grains with planar features are more populous. The superior plasticity of the NG/UFG structure was attributed to the extended work hardening due to nano size twin/twin like bands in NG/UFG austenite as well as deformation accommodation of ferrite phase in processed DSS.
- 7- The TEM studies of the tensile deformed CG and FG samples with grains size of 1.5 μm and 10 μm showed that the deformation induced α -martensite transformation is responsible for increasing SHR as well as high strength-high ductility prosperities of the processed alloy.

Table Captions

Table 1 The chemical composition (wt. %) of the experimental DSS alloy.

Table 2 Comparison between mechanical properties of some nano/ultrafine grained stainless steels with the ultrafine grained DSS produced in the current study.

Figure Captions

Fig. 1 Schematic representation of the applied thermo-mechanical processes.

Fig. 2 (a) X-ray diffraction patterns of cold rolled samples at different reduction level, (b) Normalized α -martensite fraction to the initial austenite amount of experimental DSS as a function of rolling effective strain.

Fig. 3 Variation of Ferritescope numbers with annealing time and temperature.

Fig. 4 Dilatometry test result of 80% cold roll specimen with a 25 °C/s heating rate.

Fig. 5 (a) Bright field TEM micrograph of sample 80% cold rolled and annealed at 900 °C for 30 s. (b) the magnified square area shown in (a). (c-e) selected area diffraction patterns of the austenite, ferrite and chromium nitride precipitations, respectively.

Fig. 6 (a) TKD phase map superimposed on the pattern quality map of a reversion annealed sample at 900 °C/30 s after 80% cold rolling (b,c) EBSD phase map superimposed on the pattern quality map for reversion annealed samples at 900 °C/60 s and 900 °C/180 s, respectively. In all figures green and red are representative of the FCC and BCC, respectively.

Fig. 7 Topographical secondary electron image of electro-etched 80% cold rolled and annealed sample at 750°C/30 s. The fine recrystallized ferrite grains are observed neighboring non-reversed SIM.

Fig. 8 Bright field TEM micrograph showing (a) lath martensite in 40% cold deformed sample (b) combination of lath and dislocation cell type martensite in 80% cold deformed sample. The insets are corresponding selected area diffraction pattern from lath-type showing spots from $[-111]$ zone axis and dislocation cell type martensite showing a ring pattern.

Fig. 9 EBSD results of sample 30% cold rolled (a) phase map. Green, red and yellow colors are representative of FCC, BCC and HCP phases, respectively. (b) inverse pole figure color coded orientation map parallel to the normal direction (ND) of austenite.

Fig. 10 Variation of reverse transformation start and finish temperatures (A_s and A_f) with various heating rates.

Fig. 11 The $\varphi_2=0^\circ$, 45° , 65° sections of ODF maps of austenite phase; (a) 80% cold rolled at room temperature, annealed at 900 °C for (b) 30 s (c) 60 s (d) 180 s.

Fig. 12 (a) Engineering stress-strain curves at different thermo-mechanical processing conditions. (b) True stress-strain curves of samples in (a). (c) Corresponding SHR curves of samples shown in (b).

Fig. 13 Bright field TEM micrograph of underneath the fracture surface of the 80% cold rolled and annealed sample at 900 °C/30 s (a) Non-UFG austenite phase (b) UFG austenite phase (c) ferrite phase. (d) The dense planar defects area inside the UFG austenite phase under magnified observation with corresponding selected area diffraction patterns.

Fig. 14 Bright field TEM micrograph of austenite phase, underneath the fracture surface of 80% cold rolled and annealed samples at (a) 900°C/60 s (b) 900°C/180 s. The insets are selected area diffraction patterns representing α -martensite phase from [100] and [111] zone axis, respectively.

Reference

- [1] Y.Z. Tian, Y. Bai, L.J. Zhao, S. Gao, H.K. Yang, A. Shibata, Z.F. Zhang, N. Tsuji, A novel ultrafine-grained Fe–22Mn–0.6C TWIP steel with superior strength and ductility, *Mater. Charact.* 126 (2017) 74–80.
- [2] A. Karmakar, R.D.K. Misra, S. Neogy, D. Chakrabarti, Development of ultrafine-grained dual-phase steels: mechanism of grain refinement during intercritical deformation, *Metall. Mater. Trans. A.* 44 (2013) 4106–4118.
- [3] S.G. Hashemi, B. Eghbali, Evolution of high strength and ductile ultrafine grained dual phase superferrite low carbon V-Nb-Mo steel, *Mater. Sci. Eng. A.* 705 (2017) 32–41.
- [4] C.P. Scott, F. Fazeli, B.S. Amirkhiz, I. Pushkareva, S.Y.P. Allain, Structure-properties relationship of ultra-fine grained V-microalloyed dual phase steels, *Mater. Sci. Eng. A.* 703 (2017) 293–303.
- [5] B. Poorganji, G. Miyamoto, T. Maki, T. Furuhashi, Formation of ultrafine grained ferrite by warm deformation of lath martensite in low-alloy steels with different carbon content, *Scr. Mater.* 59 (2008) 279–281.
- [6] C. Lei, X. Deng, X. Li, Z. Wang, G. Wang, R.D.K. Misra, Mechanical properties and strain hardening behavior of phase reversion-induced nano/ultrafine Fe-17Cr-6Ni austenitic structure steel, *J. Alloys Compd.* 689 (2016) 718–725.
- [7] R. Song, D. Ponge, D. Raabe, J.G. Speer, D.K. Matlock, Overview of processing, microstructure and mechanical properties of ultrafine grained bcc steels, *Mater. Sci. Eng. A.* 441 (2006) 1–17.
- [8] H.S. Baghbadorani, A. Kermanpur, A. Najafizadeh, P. Behjati, M. Moallemi, A. Rezaee, Influence of Nb-microalloying on the formation of nano/ultrafine-grained microstructure and mechanical properties during martensite reversion process in a 201-type austenitic stainless steel, *Metall. Mater. Trans. A.* 46 (2015) 3406–3413.
- [9] H. Samaei Baghbadorani, A. Kermanpur, A. Najafizadeh, P. Behjati, A. Rezaee, M. Moallemi, An investigation on microstructure and mechanical properties of a Nb-microalloyed nano/ultrafine grained 201 austenitic stainless steel, *Mater. Sci. Eng. A.* 636 (2015) 593–599.
- [10] A.S. Hamada, A.P. Kisko, P. Sahu, L.P. Karjalainen, Enhancement of mechanical properties of a TRIP-aided austenitic stainless steel by controlled reversion annealing, *Mater. Sci. Eng. A.* 628 (2015) 154–159.
- [11] A. Järvenpää, M. Jaskari, J. Man, L.P. Karjalainen, Austenite stability in reversion-treated structures of a 301LN steel under tensile loading, *Mater. Charact.* 127 (2017) 12–26.
- [12] A. Järvenpää, M. Jaskari, L. Karjalainen, Reversed microstructures and tensile properties after various cold rolling reductions in AISI 301LN steel, *Metals (Basel)*. 8 (2018) 109.

- [13] K. Tomimura, S. Takaki, S. Tanimoto, Y. Tokunaga, Optimal chemical composition in Fe-Cr-Ni alloys for ultra grain refining by reversion from deformation induced martensite, *ISIJ Int.* 31 (1991) 721–727.
- [14] R.D.K. Misra, S. Nayak, P.K.C. Venkatasurya, V. Ramuni, M.C. Somani, L.P. Karjalainen, Nano grained/ultrafine-grained structure and tensile deformation behavior of shear phase reversion-induced 301 austenitic stainless steel, *Metall. Mater. Trans. A.* 41 (2010) 2162–2174.
- [15] M. Eskandari, A. Kermanpur, A. Najafizadeh, Formation of nanocrystalline structure in 301 stainless steel produced by martensite treatment, 40 (2009) 2241–2249.
- [16] S. Rajasekhara, L.P. Karjalainen, a. Kyröläinen, P.J. Ferreira, Microstructure evolution in nano/submicron grained AISI 301LN stainless steel, *Mater. Sci. Eng. A.* 527 (2010) 1986–1996.
- [17] M. Eskandari, a. Najafizadeh, a. Kermanpur, Effect of strain-induced martensite on the formation of nanocrystalline 316L stainless steel after cold rolling and annealing, *Mater. Sci. Eng. A.* 519 (2009) 46–50.
- [18] M. Moallemi, A. Najafizadeh, A. Kermanpur, A. Rezaee, Effect of reversion annealing on the formation of nano/ultrafine grained structure in 201 austenitic stainless steel, *Mater. Sci. Eng. A.* (2011) 4–7.
- [19] A. Rezaee, A. Kermanpur, A. Najafizadeh, M. Moallemi, Production of nano / ultrafine grained AISI 201L stainless steel through advanced thermo-mechanical treatment, *Mater. Sci. Eng. A.* 528 (2011) 5025–5029.
- [20] A. Kisko, A. Hamada, L.P. Karjalainen, J. Talonen, Microstructure and Mechanical Properties of Reversion Treated High Mn Austenitic 204Cu and 201 Stainless Steels, *HMnS2011*, Gd. Hilt. Hotel. Seoul, Pap. B-19, May15À18. (2011).
- [21] B. Ravi Kumar, B. Mahato, N.R. Bandyopadhyay, D.K. Bhattacharya, Comparison of rolling texture in low and medium stacking fault energy austenitic stainless steels, *Mater. Sci. Eng. A.* 394 (2005) 296–301.
- [22] A. Rezaee, A. Kermanpur, A. Najafizadeh, M. Moallemi, H.S. Baghbadorani, Investigation of cold rolling variables on the formation of strain-induced martensite in 201L stainless steel, *Mater. Des.* 46 (2013) 49–53.
- [23] J. Chiang, B. Lawrence, J.D. Boyd, A.K. Pilkey, Effect of microstructure on retained austenite stability and work hardening of TRIP steels, *Mater. Sci. Eng. A.* 528 (2011) 4516–4521.
- [24] M. Moallemi, A. Kermanpur, A. Najafizadeh, A. Rezaee, H.S. Baghbadorani, P.D. Nezhadfar, Deformation-induced martensitic transformation in a 201 austenitic steel: the synergy of stacking fault energy and chemical driving force, *Mater. Sci. Eng. A.* 653 (2016) 147–152.

- [25] P. Behjati, A. Najafizadeh, Role of chemical driving force in martensitic transformations of high-purity Fe-Cr-Ni alloys, *Metall. Mater. Trans. A.* 42 (2011) 3752–3760.
- [26] K. Nohara, Y. Ono, N. Ohashi, Composition and grain size dependencies of strain-induced martensitic transformation in metastable austenitic stainless steels, *Tetsu-to-Hagané*. 63 (1977) 772–782.
- [27] A. Das, Revisiting stacking fault energy of steels, *Metall. Mater. Trans. A.* 47 (2016) 748–768.
- [28] M. Moallemi, A. Zarei-Hanzaki, A. Mirzaei, On the stacking fault energy evaluation and deformation mechanism of Sanicro-28 super-austenitic stainless steel, *J. Mater. Eng. Perform.* 24 (2015) 2335–2340.
- [29] G.B. Olson, M. Cohen, A mechanism for the strain-induced nucleation of martensitic transformations, *J. Less Common Met.* 28 (1972) 107–118.
- [30] A. Kisko, J. Talonen, D.A. Porter, L.P. Karjalainen, effect of Nb microalloying on reversion and grain growth in a high-Mn 204Cu austenitic stainless steel, 55 (2015) 2217–2224.
- [31] S.-J. Lee, Y.-M. Park, Y.-K. Lee, Reverse transformation mechanism of martensite to austenite in a metastable austenitic alloy, *Mater. Sci. Eng. A.* 515 (2009) 32–37.
- [32] K. Tomimura, S. Takaki, Y. Tokunaga, Reversion mechanism from deformation induced martensite to austenite in metastable austenitic stainless steels, *ISIJ Int.* 31 (1991) 1431–1437.
- [33] S. Takaki, K. Tomimura, S. Ueda, Effect of pre-cold-working on diffusional reversion of deformation induced martensite in metastable austenitic stainless steel, *ISIJ Int.* 34 (1994) 522–527.
- [34] R.D.K. Misra, S. Nayak, S.A. Mali, J.S. Shah, M.C. Somani, L.P. Karjalainen, On the significance of nature of strain-induced martensite on phase-reversion-induced nanograined/ultrafine-grained austenitic stainless steel, *Metall. Mater. Trans. A.* 41 (2010) 3–12.
- [35] M.C. Somani, P. Juntunen, L.P. Karjalainen, R.D.K. Misra, A. Kyröläinen, Enhanced mechanical properties through reversion in metastable austenitic stainless steels, *Metall. Mater. Trans.*
- [36] A. Kisko, R.D.K. Misra, J. Talonen, L.P. Karjalainen, The influence of grain size on the strain-induced martensite formation in tensile straining of an austenitic 15Cr-9Mn-Ni-Cu stainless steel, *Mater. Sci. Eng. A.* 578 (2013) 408–416.
- [37] S. Rajasekhara, P.J. Ferreira, L.P. Karjalainen, A. Kyröläinen, Hall-Petch behavior in ultra-fine-grained AISI 301LN stainless steel, *Metall. Mater. Trans. A.* 38 (2007) 1202–1210.

- [38] P. Behjati, A. Kermanpur, A. Najafizadeh, H.S. Baghbadorani, L.P. Karjalainen, J.-G. Jung, Y.-K. Lee, Effect of nitrogen content on grain refinement and mechanical properties of a reversion-treated Ni-free 18Cr-12Mn austenitic stainless steel, *Metall. Mater. Trans. A*. 45 (2014) 6317–6328.
- [39] Y. Ma, J. Jin, Y. Lee, A repetitive thermomechanical process to produce nano-crystalline in a metastable austenitic steel, *Scr. Mater.* 52 (2005) 1311–1315.
- [40] P. Behjati, A. Kermanpur, A. Najafizadeh, H. Samaei Baghbadorani, J.G. Jung, Y.K. Lee, Enhanced mechanical properties in a high-manganese austenitic steel through formation of nano grains, nanotwinned austenite grains, nano carbides and TRIP, *Mater. Sci. Eng. A*. 610 (2014) 273–278.
- [41] L. Chen, F.P. Yuan, P. Jiang, J.J. Xie, X.L. Wu, Simultaneous improvement of tensile strength and ductility in micro-duplex structure consisting of austenite and ferrite, *Mater. Sci. Eng. A*. 618 (2014) 563–571.
- [42] A. Mandal, S. Patra, D. Chakrabarti, S.B. Singh, Effect of Rolling and Subsequent Annealing on Microstructure, Microtexture, and Properties of an Experimental Duplex Stainless Steel, *Metall. Mater. Trans. A*. 48 (2017) 5960–5977.
- [43] M. Naghizadeh, H. Mirzadeh, Microstructural Evolutions During Annealing of Plastically Deformed AISI 304 Austenitic Stainless Steel: Martensite Reversion, Grain Refinement, Recrystallization, and Grain Growth, *Metall. Mater. Trans. A*. (2016). 4210–4216.
- [44] A. Kisko, A.S. Hamada, J. Talonen, D. Porter, L.P. Karjalainen, Effects of reversion and recrystallization on microstructure and mechanical properties of Nb-alloyed low-Ni high-Mn austenitic stainless steels, *Mater. Sci. Eng. A*. 657 (2016) 359–370.
- [45] R.D.K. Misra, X.L. Wan, V.S.A. Challa, M.C. Somani, L.E. Murr, Relationship of grain size and deformation mechanism to the fracture behavior in high strength–high ductility nanostructured austenitic stainless steel, *Mater. Sci. Eng. A*. 626 (2015) 41–50.
- [46] R.D.K. Misra, Z. Zhang, Z. Jia, P.K.C.V. Surya, M.C. Somani, L.P. Karjalainen, Nanomechanical insights into the deformation behavior of austenitic alloys with different stacking fault energies and austenitic stability, *Mater. Sci. Eng. A*. 528 (2011) 6958–6963.
- [47] A.A. of S.H. and T. Officials, A.S. for T. and Materials, E8M-04 Standard Test Methods for Tension Testing of Metallic Materials (Metric) 1, ASTM International, 2004.
- [48] A.B. Fanta, M. Todeschini, A. Burrows, H. Jansen, C.D. Damsgaard, H. Alimadadi, J.B. Wagner, Elevated temperature transmission Kikuchi diffraction in the SEM, *Mater. Charact.* 139 (2018) 452–462.
- [49] S. Curtze, V. Kuokkala, A. Oikari, J. Talonen, H. Ha, Thermodynamic modeling of the stacking fault energy of austenitic steels, 59 (2011) 1068–1076.
- [50] A. Järvenpää, M. Jaskari, L.P. Karjalainen, Properties of Induction Reversion-Refined

- Microstructures of AISI 301LN under Monotonic, Cyclic and Rolling Deformation, in: Mater. Sci. Forum, Trans Tech Publ, 2018: pp. 601–607.
- [51] M. Breda, K. Brunelli, F. Grazzi, A. Scherillo, I. Calliari, Effects of Cold Rolling and Strain-Induced Martensite Formation in a SAF 2205 Duplex Stainless Steel, Metall. Mater. Trans. A. 46 (2014) 577–586.
 - [52] A.F. Padilha, R.L. Plaut, P.R. Rios, Annealing of Cold-worked Austenitic Stainless Steels., ISIJ Int. 43 (2003) 135–143.
 - [53] A.J. Ramirez, J.C. Lippold, S.D. Brandi, The relationship between chromium nitride and secondary austenite precipitation in duplex stainless steels, Metall. Mater. Trans. A. 34 (2003) 1575–1597.
 - [54] I.F. Machado, A.F. Padilha, Aging behaviour of 25Cr–17Mn high nitrogen duplex stainless steel, ISIJ Int. 40 (2000) 719–724.
 - [55] A.F. Padilha, P.R. Rios, Decomposition of Austenite in Austenitic Stainless Steels, ISIJ Int. 42 (2002) 325–327.
 - [56] J. Yong, J. Hoon, S. Woo, K. Park, Strain induced martensitic transformation of Fe – 20Cr – 5Mn – 0 . 2Ni duplex stainless steel during cold rolling : Effects of nitrogen addition, Mater. Sci. Eng. A. 528 (2011) 6012–6019.
 - [57] Q. Ran, Y. Xu, J. Li, J. Wan, X. Xiao, H. Yu, L. Jiang, Effect of heat treatment on transformation-induced plasticity of economical Cr19 duplex stainless steel, J. Mater. 56 (2014) 959–965.
 - [58] R.D.K. Misra, Z. Zhang, Z. Jia, M.C. Somani, L.P. Karjalainen, Probing deformation processes in near-defect free volume in high strength–high ductility nanograined/ultrafine-grained (NG/UFG) metastable austenitic stainless steels, Scr. Mater. 63 (2010) 1057–1060.
 - [59] C. Celada-Casero, B.M. Huang, M.M. Aranda, J.-R. Yang, D. San Martin, Mechanisms of ultrafine-grained austenite formation under different isochronal conditions in a cold-rolled metastable stainless steel, Mater. Charact. 118 (2016) 129–141.
 - [60] L. Meng, P. Yang, Q. Xie, H. Ding, Z. Tang, Dependence of deformation twinning on grain orientation in compressed high manganese steels, 56 (2007) 931–934.
 - [61] A. Poulon, S. Brochet, J. Glez, J. Mithieux, J. Vogt, Influence of Texture and Grain Size on Martensitic Transformations Occurring During Low-Cycle Fatigue of a Fine-Grained Austenitic Stainless Steel, Adv. Eng. Mater. 12 (2010) 1041–1046.
 - [62] Y.-K. Lee, H.-C. Shin, D.-S. Leem, J.-Y. Choi, W. Jin, C.-S. Choi, Reverse transformation mechanism of martensite to austenite and amount of retained austenite after reverse transformation in Fe-3Si-13Cr-7Ni (wt-%) martensitic stainless steel, Mater. Sci. Technol. 19 (2003) 393–398.

- [63] L. Kaufman, E. Clougherty, R. Weiss, The lattice stability of metals—III. Iron, *Acta Metall.* 11 (1963) 323–335.
- [64] S.G. Chowdhury, S. Das, B. Ravikumar, S. Kumar, G. Gottstein, Textural development in AISI 316 stainless steel during cold rolling and annealing, *Mater. Sci. Forum*, (2002) 1371–1376.
- [65] D.P. Escobar, S.S.F. de Dafé, D.B. Santos, Martensite reversion and texture formation in 17Mn-0.06 C TRIP/TWIP steel after hot cold rolling and annealing, *J. Mater. Res. Technol.* 4 (2015) 162–170.
- [66] J.J. Jonas, Transformation textures associated with steel processing, *Microstructure and Texture in Steels*, Springer, 2009: pp. 3–17.
- [67] J.J. Jonas, Effect of austenite recrystallization on toughness of pipeline steels, *Mater. Sci. Forum*, (2013) 546–553.
- [68] R.K. Ray, J.J. Jonas, Transformation textures in steels, *Int. Mater. Rev.* 35 (1990) 1–36.
- [69] J. Talonen, Effect of Strain-induced α' -Martensite Transformation on Mechanical Properties of Metastable Austenitic Stainless Steels Doctoral Dissertation, Helsinki University of Technology, Espoo, Finland, 2007.
- [70] J. Talonen, H. Hänninen, P. Nenonen, G. Pape, Effect of strain rate on the strain-induced $\gamma \rightarrow \alpha'$ -martensite transformation and mechanical properties of austenitic stainless steels, *Metall. Mater. Trans. A.* 36 (2005) 421–432.
- [71] A.K. De, J.G. Speer, D.K. Matlock, D.C. Murdock, M.C. Mataya, R.J. Comstock, Deformation-induced phase transformation and strain hardening in type 304 austenitic stainless steel, *Metall. Mater. Trans. A.* 37 (2006) 1875–1886.
- [72] G.L. Huang, D.K. Matlock, G. Krauss, Martensite formation, strain rate sensitivity, and deformation behavior of type 304 stainless steel sheet, *Metall. Trans. A.* 20 (1989) 1239–1246.
- [73] V.G. Gavriljuk, B.D. Shanina, H. Berns, On the correlation between electron structure and short range atomic order in iron-based alloys, *Acta Mater.* 48 (2000) 3879–3893.
- [74] V.G. Gavriljuk, H. Berns, High nitrogen steels: structure, properties, manufacture, applications, Springer Science & Business Media, 2013.
- [75] G. Dini, R. Uejii, Effect of grain size and grain orientation on dislocations structure in tensile strained TWIP steel during initial stages of deformation, *Steel Res. Int.* 83 (2012) 374–378.
- [76] A. Saeed-Akbari, J. Imlau, U. Prahl, W. Bleck, Derivation and variation in composition-dependent stacking fault energy maps based on subregular solution model in high-manganese steels, *Metall. Mater. Trans. A.* 40 (2009) 3076–3090.

- [77] R.D.K. Misra, B.R. Kumar, M. Somani, P. Karjalainen, Deformation processes during tensile straining of ultrafine/nanograined structures formed by reversion in metastable austenitic steels, *Scr. Mater.* 59 (2008) 79–82.
- [78] J. Huang, X. Ye, J. Gu, X. Chen, Z. Xu, Enhanced mechanical properties of type AISI301LN austenitic stainless steel through advanced thermo mechanical process, *Mater. Sci. Eng. A.* 532 (2012) 190–195.
- [79] F. Forouzan, A. Najafizadeh, A. Kermanpur, A. Hedayati, R. Surkialiabad, Production of nano/submicron grained AISI 304L stainless steel through the martensite reversion process, *Mater. Sci. Eng. A.* 527 (2010) 7334–7339.
- [80] P. Behjati, A. Kermanpur, A. Najafizadeh, H.S. Baghbadorani, Effect of annealing temperature on nano/ultrafine grain of Ni-free austenitic stainless steel, *Mater. Sci. Eng. A.* 592 (2014) 77–82.

Table and table captions

Table 1 Chemical composition (wt.%) of the experimental alloy.

Grade	C	Cr	Mn	N	Ni	Si
DSS	0.02	20.02	8.21	0.32	0.05	0.36

Table 2 Comparison between mechanical properties of some nano/ultrafine grained stainless steels with the ultrafine grained duplex stainless steel produced in the current study.

Alloy(Initial microstructure)	Mean grain size of austenite after processing (μm)	Yield strength (MPa)	Ultimate Tensile strength (UTS) (MPa)	Elongation (%)	UTS $\times$ Elongation (MPa%)	Ref
301LN (100% γ)	0.6	900	1150	49	56350	[12]
204Cu	0.5	899	1345	38	51110	[36]
301LN (100% γ)	<3	830	953	36	34308	[78]
316L (100% γ)	0.035	-	1385	5.5	7917	[17]
304L (100% γ)	0.33	1000	1010	40	40400	[79]
Fe-0.1%C-10%Cr-	0.1	779	1102	32	35267	[39]

5%Ni-8%Mn (100% γ)						
Fe-17.5%Cr-11.5%Mn- 0.25%N (100% γ)	0.24	1150	1293	21	27153	[80]
Low N S32101(50% γ +50% α) (austenite island size)	19.6	798	1088	41	44608	[42]
UNS S32304 (γ + α)	1.69	540	800	48	38400	[41]
Fe-20.5%Cr-8.2%Mn- 0.32%N (74% γ +26% α)	0.4	993	1028	40	41120	Present work

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8

Figure 9

Figure 10

Figure 11

(a)

(b)

(c)

Figure 12

Figure 13

Figure 14