Substructure Formation, Texture, and Mechanical Behavior of Warm Rolled 2205-Type Duplex Stainless Steel

Francislayne Lages Dias¹ · Dagoberto Brandão Santos¹

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Abstract

The microstructural evolution and mechanical behavior of 2205 duplex stainless steel were studied after plate warm rolling at 600 °C with 60 and 80% thickness reduction, using different microscopy techniques, X-ray diffraction, and tensile and hardness testing. The microstructures of the warm-rolled specimens consisted of flattened wavy grains. The texture evolution during rolling in the ferrite domain presented α -fiber and rotated cube components, while the austenite showed brass, copper, and cube components to a lesser extent. Intense formation of entanglement and dislocation forests characterized the microstructure. In ferrite, there was an intense formation of dislocation substructures and cell blocks. In austenite, the substructure was characterized by planar gliding and the formation of dislocation entanglements. After warm rolling, the tensile strength reached 1185 and 1328 MPa at 60 and 80% thickness reduction, respectively. Compared with the as-received steel, the warm work raised the mechanical strength level by between 65 and 72%. These results highlight the prospects for innovative routes to the industrial production of this class of duplex stainless steel, such as cold rolling suppression, considering even ductility reduction.

Keywords Warm rolling · Texture · Duplex stainless steel · TEM · Mechanical properties

Introduction

Duplex stainless steels (DSSs) may be defined as two-phase ferritic-austenitic steels in approximately equal volume fractions [1]. The amount, morphology, and distribution of ferrite (α) and austenite (γ) phases in a DSS determine the required properties for application in severe conditions, such as in oil and gas, pulp and paper, desalination, transport, and other general industries [2–4]. The γ -phase contains a face-centered cubic structure, which has non-magnetic characteristics and promotes superior toughness and strength. The α -phase, a body-centered cubic crystal structure, is responsible for the excellent crevice and chloride-pitting corrosion resistance properties [5, 6]. 2205, a standard of current DSS grades, is the workhorse of this generation, responsible for nearly 60% of DSS use because of its high strength, which allows a meaningful wall thickness reduction [2, 6].

The thermomechanical process industrially applied to these steels is hot rolling after continuous casting, performed in the range of 1000 to 1200 °C, to obtain a large thickness reduction. Then, DSS is annealed to recover the ductility for the following cold rolling to attain the final thickness and fine microstructure. A final annealing is required to guarantee desirable characteristics for future applications, such as plasticity [7, 8]. This process favors a microstructure with alternating lamellae of α - and γ -phases because of the lower interfacial energy of these phases related to the other interfaces between them [7, 9, 10]. However, the two phases present in the DSS do not exhibit the same behavior during deformation [10, 11]. The ferritic phase has numerous slipping systems, including dislocation cells and high stacking fault energy (SFE). The austenitic phase presents slip bands, a lower amount of slipping systems, and significantly lower SFE [10–12].

Because hot working requires great amounts of thermal energy, there has been an initiative to work at lower temperatures, which may produce steel close to its final shape and reduce or even eliminate cold rolling. Cold work leads to drastic degradation of plasticity, commonly experiencing an austenitic phase to strain-induced martensitic transformation

✉ Dagoberto Brandão Santos
dsantos@demet.ufmg.br; dagoberto.santos2000@gmail.com

¹ Metallurgical and Materials Engineering Department,
Universidade Federal de Minas Gerais, Av. Antonio Carlos,
6627, Belo Horizonte, Minas Gerais 31270901, Brazil

(SIMT) and requires higher forces in rolling mills [8]. In some applications, final annealing treatment may also be avoided when warm rolling is applied [13–16]. Warm rolling can be used to produce sheet steel products at low cost at temperatures within the ferrite region (600–800 °C), while hot rolling occurs in the austenite region (900–1200 °C). It is applicable in materials with weak {001} and strong {111} texture components, leading to good formability [17].

In a 0.017% C carbon steel, warm-rolling natural strains of up to 1.6 at 650 °C produce strength increases of up to 60%, but only with ductility reduction, which is associated with the formation of a recovered substructure. Conversely, the transition temperature for the highest rolling strain was better than that of the as-hot-rolled steel and was associated with the formation of a {100} texture [13]. In DSS, ultrahigh strength (> 1 GPa) was achieved, SIMT was avoided, and an ultrafine microstructure was obtained after warm-rolling, producing successful ultrahigh-strength steel [18–20].

In the present work, a detailed study was performed of the substructure, texture, and mechanical behavior, of a 2205-type DSS after warm rolling at 600 °C. Some studies [18–20] have described the microstructure of DSSs after warm deformation, but none of them have described the substructure of warm-rolled DSS with this specific chemical composition.

Experimental Procedure

Materials and Heat Treatments

A 2205-type DSS (0.026 C–22.43 Cr–5.44 Ni–3.04 Mo–0.15 N) was received in hot-rolled sheets of 5.15-mm thickness and annealed condition.

The phase volume fraction as a function of temperature was calculated using the mentioned chemical composition on Thermo-Calc® software and the data were plotted on Origin™ software, as shown in Fig. 1. It is important to choose a rolling temperature that can avoid the nucleation of unwanted phases. At 600 °C, the presence of 4 phases can be seen: α , γ , σ (sigma), and χ (chi) phases, the last two being deleterious phases. However, the initial formation of the σ and χ phases only occurs after 10,800 s of soaking at 600 °C [10]. In this way, the thermomechanical processing of DSS at 600 °C to modify its performance, without precipitation of deleterious phases, is possible.

Subsequently, the samples were submitted to warm rolling, performed in a Fröhling laboratory rolling mill with roll diameters of 0.20 m, upon reaching strains (ε) of $\varepsilon = 0.60$ (thickness reduction from 5.15 to 2.20 mm in nine passes) and $\varepsilon = 0.80$ (thickness reduction from 5.15 to 1.15 mm in fifteen passes) with a line speed of 0.104 m/s. Before warm rolling, the samples were soaked in a

Fig. 1 Volume phase fraction as a function of temperature, calculated using Thermo-Calc® software for the 2205 DSS composition used in the present work

Table 1 Rolling pass and respective thickness obtained

Pass	Per pass reduction	
	WR 60%	WR 80%
0	5.15	5.15
1	4.75	4.7
2	4.5	4.5
3	4.0	4.0
4	3.7	3.65
5	3.3	3.25
6	3.1	3.05
7	2.8	2.75
8	2.5	2.45
9	2.2	2.15
10		1.8
11		1.5
12		1.4
13		1.3
14		1.2
15		1.15

conventional muffle furnace for 1800 s at 600 °C. After each pass, a necessary 900 s of reheating was employed to ensure a 600 °C strip temperature before the next pass. Table 1 shows the thickness obtained in each pass for both conditions. Following the last pass, the strip was cooled to approximately 25 °C through air cooling without subsequent annealing. The process is schematically illustrated in Fig. 2. In summary, three conditions were analyzed: as-received (AR), warm-rolled at 60% reduction (WR 60%), and warm-rolled at 80% reduction (WR 80%). For microhardness and tensile tests, three other conditions were produced. The specimens, AR, WR 60%, and WR 80%, were annealed at 1050 °C for 300 s in a muffle furnace.

Fig. 2 Schematic warm rolling processing applied to DSS in as-received condition, that is, industrial hot rolling and homogenizing annealing

The orientations corresponding to the warm-rolled product are ND, RD, TD, which denote normal, rolling, and transverse direction, respectively.

Microscopy

Microstructural characterization was carried out for as-received and warm-rolled samples. The microstructure was examined by scanning (SEM) and transmission electron microscopy (TEM).

The samples were prepared for SEM via the conventional procedure of cutting, grinding, and ultimately polishing with diamond paste. Behara etchant was used to reveal the microstructure. All micrographs are of the ND–RD plane. The images were obtained with secondary electron emission in a FEI Inspect™ S50 SEM with an accelerating voltage of 15 kV, spot size of 5.5 nm, and a working distance of 10 mm.

The sample preparation for TEM was conducted on the RD–TD plane, consisting of cutting, then mechanical grinding to reach 200 μm thickness, with a final reduction to 50 to 70 μm thickness. Then, samples were polished with a 1 μm diamond paste. After polishing, disks with diameters of 3 mm were drilled using a puncher device. Thin foils were prepared using twin-jet-polishing in a 5% perchloric acid plus 95% methanol solution between -30 and -20 °C at an etching potential of 20 V using a Struers Tenupol Electro polishing device. Micrographs were recorded via transmission electron microscopy (Tecnai G2-20 SuperTwin FEI, 200 kV) and energy-dispersive X-ray spectroscopy (EDS) was also conducted using the same equipment.

Texture

The microstructure was also characterized by electron backscattered diffraction (EBSD). The EBSD samples were also prepared via the conventional SEM procedure on the ND–RD plane of the center layer of the thickness and were

automatically polished in a 0.04- μm particle-size colloidal silica for 5400 s using a Buehler Minimet 1000® automatic machine. Scan data were obtained using a FEI Inspect™ S50 SEM with an accelerating voltage of 20 kV and a working distance close to 20.0 mm. A step size of 0.2 μm was used to scan an area of approximately 45×120 μm with a confidence index of 0.1. The raw data were clean-up post-processed by EDAX OIM Analysis™ software, using grain dilation, and neighbor orientation correlation with a grain tolerance angle of 5. The microtexture analysis and grain size of phases determined by EBSD were quantitatively calculated using TSL™ software. The ferrite and austenite microtextures were investigated using specific conditions of the inverse pole figure (IPF) and a constant section of the orientation distribution function (ODF) in the $\varphi_2 = 45^\circ$ of Euler space.

X-ray Diffraction

To determine the phase constituents and their proportions, X-ray diffraction (XRD) experiments were conducted on the mechanically polished RD–TD plane using a Pan Analytical Empiryan instrument under Cu-K α radiation. The Cu source was excited at an acceleration voltage of 45 kV and current of 45 mA. The records were acquired at room temperature, with 2θ ranging from 35° to 110° in a 0.02° step size and a counting time of 1 s per step. Origin™ software was used for quantitative analysis from the database by the integration of all diffraction peaks (for ferrite and austenite) using the Lorentzian peak function. This method of quantitative estimation of phase fractions relies on the fundamental proportional equivalence between the phase volume fraction and the sum of the integrated intensities of the diffraction peaks for each phase in the mixture [21].

Tensile Testing

Triplicate tensile tests per condition were performed at room temperature, with a constant cross head speed and average strain rate of 10^{-3} s^{-1} , in a 5582 model Instron machine, using the Blue Hill software for data acquisition. The specimens were taken from the RD–TD plane in the RD direction using the dimensions of the ASTM A-370-15 subsize, as shown in Fig. 3.

Microhardness Testing

Microhardness samples were turned from the ND–RD plane, mechanically ground, and polished. The test was conducted using a Future Tech FM700 Vickers microhardness instrument loaded to 4.9 N for 10 s. The average value was calculated from 20 indentations.

Fig. 3 Dimensions of the tensile test sample

Fig. 4 Diffraction patterns: (a) as-received; (b) 60% and (c) 80% warm-rolled samples

Table 2 Phase volume fraction calculated using X-ray diffraction data

Condition	% Ferrite	% Austenite
As-received	35	65
Warm-rolled 60%	32	68
Warm-rolled 80%	40	60

Results and Discussion

X-ray Diffraction

The diffraction patterns of AR, WR 60%, and WR 80% are shown in Fig. 4, and the quantitative results of the fraction volume phases are summarized in Table 2. The results confirm the presence of austenite and ferrite in all the samples. There is a slight difference between the warm-rolled conditions when compared with the as-received. The volume fraction of α -ferrite in the WR 60% decreased by a small amount compared with that in the as-received condition. In WR 80%, the fraction of α -ferrite increased. It should be noted that the precision of the method does not allow for major conclusions regarding the phase variation. As expected, no deleterious phase was observed.

Scanning Electron Microscopy

Figure 5 shows SEM micrographs of the AR, WR 60%, and WR 80% samples. The gray color difference between these phases is a result of their different corrosion resistances during etching, where ferrite is the darkest and most corroded phase. An elongated morphology of alternative bands is noticeable for ferrite and austenite along the RD in the deformed structure. These lamellae appear as approximately parallel-lined grains from the early stages of deformation.

Fig. 5 Micrographs of the samples obtained using SEM: (a) AR; (b) and (c) WR 60% and WR 80%, respectively. Behara etch

In Fig. 5b and c, it is also possible to see that the bands or lamellae of the phases ($\alpha + \gamma$) became wavy because of the intense warm deformation imposed on the steel. The refinement of the microstructure because of the thickness reduction of ferrite and austenite lamellae is also noteworthy.

The microstructure in Fig. 5 observed after industrial hot rolling is slightly different from that after warm-rolling processing [7]. During plastic deformation, the behavior of ferrite and austenite phases is quite different. Austenite presents slip bands, while ferrite presents dislocation cells. Strain hardening also occurs in different ways in both phases, as the causes of shear bands, characterized by the wavy microstructure observed in Fig. 5b and c. Shear banding is a common phenomenon in metals deformed to high strains and is favored in low-SFE metals, as austenite, which develops layered microstructures, is often partially twinned, which inhibits dislocation glide and thereby promotes shear banding [22]. This is caused by local plastic instabilities when work hardening becomes exhausted [22]. The lower SFE of austenite also controls the deformation and recrystallization texture proportions and the stored energy during deformation before annealing [23–25].

Microtexture

Figures 6 and 7 illustrate the orientation distribution function (ODF) for $\varphi_2 = 45^\circ$ for ferrite and $\varphi_2 = 0^\circ$, $\varphi_2 = 45^\circ$, and $\varphi_2 = 65^\circ$ for austenite, respectively. Some important fibers and orientations in the Euler space ($\varphi_2 = 0^\circ$, $\varphi_2 = 45^\circ$, and $\varphi_2 = 65^\circ$ section) are also plotted. The microtextures represented in the ODF sections were measured from the half-width of the ND–RD plane.

The ferrite texture is characterized as a typical rolling texture, primarily centered on the rotated cube orientation

($\{001\} \langle 110 \rangle$) and α -fiber orientations under warm-rolled conditions, with the strongest component being $\{112\} \langle 110 \rangle$. The austenite phase shows a cube component $\{001\} \langle 100 \rangle$ transforming to typical copper rolling texture features with a pronounced fraction of copper orientation $\{112\} \langle 111 \rangle$ as the deformation increases, along with S (123) $\langle 364 \rangle$ and brass $\{110\} \langle 112 \rangle$.

The average grain diameter of the phases measured using EBSD are shown in Fig. 8. It is possible to see the effect of the warm-rolling strain on the ferrite and austenite grain size. As opposed to results reported by Ma and co-authors [26], where the austenite morphology remains almost undeformed after straining in a super DSS, in this study, austenite was the first phase to accommodate deformation, visible by its pronounced thickness reduction, Fig. 5b and c. This observation was reported in other study realized by Ahmed and Bhattacharjee of a warm rolled super DSS type with high Ni content (10.5%) [18].

The presence of brass components after deformation in austenitic stainless steels and fcc metals has been studied by several authors [23, 25, 27–32], and a possible mechanism is due to the low SFE, such as deformation twinning and shear banding. Some authors [33] have attributed the presence of an additional Goss ($\{110\}$) component in DSSs after hot rolling with an elevated deformation temperature, which tends to produce extra shear strain, especially near the sheet surface [34, 35]. A decrease in brass ($\{112\}$) and an increase in Goss and copper ($\{112\}$) components after annealing in cold- and warm-rolled samples has also been reported [8]. However, an increase in the S ($\{123\}$) component was only reported in cold working [8].

When the stored energy during cold deformation was not steep, a partial retention of these deformation texture components was observed throughout annealing. For the α -phase,

Fig. 6 Illustration of $\varphi_2 = 45^\circ$ ferrite sections of the ODF determined in (a) AR; (b) WR 60% and (c) WR 80% samples. (d) Shows the location of the ideal components in the ODF

Fig. 7 Illustration of $\varphi_2 = 0^\circ$, $\varphi_2 = 45^\circ$, and $\varphi_2 = 65^\circ$ austenite sections of the ODF determined in (a) as-received; (b) WR 60% and (c) WR 80% samples. (d) Shows the location of the ideal components in the ODF

Fig. 8 Average grain diameter in the as-received and warm-rolled samples

the texture is typical of hot rolling, in which a strong rotated cube component is usually created. Additionally, the cube component incorporates a strong tendency to recover rather than recrystallize, and consequently, to be conserved and still reinforced during the annealing of various steels (pure iron, low carbon, or stainless steels) [23, 31, 34–36].

The microstructure after cross-warm rolling of a high Ni (10 wt.%) super DSS revealed a lamellar structure with alternating bands of the two phases (α , γ) [35]. Strong brass and rotated brass texture components were observed in austenite in this steel. Ferrite in the warm-rolled steel showed a remarkably strong α -fiber and $\{001\}$ component. An increase in ND-fiber in warm-rolled 10.5 wt% Ni DSS has also been reported [19]. This trend was seen at this steel only at warm rolling with 80%, where strong α -fiber and the first components of γ -fiber could be seen. As reported by Ahmed et al. [20], ferrite in DSS warm-rolled showed much stronger α -fiber (RD// $\langle 110 \rangle$) than γ -fiber (ND// $\langle 111 \rangle$). This can be explained by the phenomenon of interaction of carbon atoms with dislocations [19]. The warm-rolling temperature (425 and 615 °C) is within the dynamic strain aging regime of DSS alloys resulting in the preferential retardation of slip on the $\{110\} \langle 111 \rangle$ system and in the strengthening of the α -fiber components at the expense of the γ fiber components as evidenced in the texture of the ferrite in Fig. 6b and c. Ahmed [20] noticed that the strengthening of the α -fiber happens beyond 70% deformation during warm-rolling being affected by carbon locking of dislocations. Therefore, while the strength of the α and γ texture fibers in carbon steels is known to be greatly affected by deformation [36, 37], in the present case the texture development is also greatly affected by the temperature of warm-rolling. The advancement of texture in the austenite and ferrite after cross-warm rolling can be explained by the stability of the texture components.

Along with isothermal annealing of the 90% cross-warm-rolling DSS, the lamellar microstructure was retained before the breakdown of such morphology to the mutual interpenetration of the phase ensembles [35]. Ferrite showed recovery leading to an annealing texture quite like the deformation texture. Due to the low-temperature (600 °C) deformation applied here, there is no recrystallization, only recovery. Development of γ fiber from strong $\{111\} \langle 112 \rangle$ orientation is additionally evident during warm rolling in carbon steel [14, 36]. Verbeken et al. [36] observed a strong γ -fiber in an ultra-low carbon steel after cold rolling to 70 and 90% of reduction. In this direction, Samajdar et al. [37] found also the same behavior in an IF steel cold rolled to 90%. The point to enhance in the work of Samajdar et al. [37] is the initial texture of the steel, a hot band completely recrystallized, i.e., an extraordinarily strong γ -fiber. In the present work, the initial texture of the as-received material was a rotated cube, Fig. 6a. This component was maintained along the warm rolling processing, Fig. 6b and c.

In this study, it can be concluded that the warm-rolling textures of austenite in DSS undergo the cube and brass to copper texture transition during warm rolling, indicating some possible restoration phenomena [8]. Goss is also noticeable in the WR 80%. In ferrite, the texture can be adequately described by a strong cube component and RD fiber (RD//) and slight development of ND fiber (ND//). The evolution of deformation and annealing texture in two phases of the DSS developed, in principle, independently of one another, corroborating the results reported above [25, 38].

Transmission Electron Microscopy

TEM micrographs corresponding to observations made in the RD-TD plane are displayed in Figs. 9, 10, 11, 12, 13, 14 and 15. Figures 9 and 10 show the substructure formed in the DSS in the AR condition. Figure 9a shows the presence of deformation slip bands [23, 24, 27, 38], characteristic of the austenite phase (region in the upper right corner of the micrograph, indicated by an arrow). Figure 9b shows subgrains in ferrite, and Fig. 9c illustrates the planar arrangements of dislocation slip occurring in the austenite grain. Figure 9d illustrates discrete arrangements and dislocation loops in ferrite with defined high-angle grain boundaries. In Fig. 10a, it is possible to see the substructure of ferrite containing some subgrains, and Fig. 10b reveals the intra-granular dislocation arrays, indicated by an arrow. It is also noticeable that the grain size, according to the EBSD results, is 3 μm for ferrite and 2 μm for austenite.

The 60% warm-rolled sample substructure in Fig. 11a–d contains a cellular structure (Fig. 11a and b) and dense dislocation walls (DDWs). Dislocation tangles can also be seen in Fig. 11c. Figure 11d displays an electron diffraction pattern with respect to austenite in the region indicated by an

Fig. 9 Bright-field mode TEM images of the hot-rolled and annealed as-received sample, showing: (a) and (b) austenite and ferrite high-angle grain boundaries (HAGB), and BC-bending contours. Disloca-

tions in ferrite and microbands in austenite (arrows); (c) planar slip bands in austenite; (d) discrete dislocation loops in ferrite and high-angle grain boundaries

arrow in the micrograph of Fig. 11c. Figure 12 shows TEM bright-field images of the 60% warm-rolled specimen, where Fig. 12a and b illustrates parallel sub-boundaries formed by dislocation walls [39]. Austenite shows a substructure with tangles and dislocation walls in the austenite, and the mottled contrast in ferrite is associated with substructure formation. The grain size, according to the EBSD results, is 1.7 μm for ferrite and 1.5 μm for austenite.

Representative TEM micrographs after 80% reduction by warm rolling are shown in Figs. 13, 14 and 15. Long DDWs bounded by free dislocations in austenite grain interior result from the relatively low SFE of austenite, as seen in Fig. 13a and b. Conversely, in ferrite, randomly distributed tangled dislocations were dominant. A large portion of the phase boundaries were apparently random, as shown in Fig. 14a. The EBSD results in Fig. 8 show a grain size of 0.9 μm for ferrite and 0.8 μm for austenite. This indicates that the microstructure developed by the warm work was not fully

balanced. The EDS chemical analysis (Fig. 15b and c) helps to identify the ferrite phase (point 1) with less Ni and more Mo contents. In contrast, the austenite phase had more Ni and less Mo contents (point 2). This means that an intense dislocation was formed at the α/γ interface that was concentrated in the γ phase (see Fig. 15a, upper corner).

During the warm deformation of steel at 600 °C, dislocations begin to cluster inside individual grains, and these clusters eventually join to form a cell structure as the amount of deformation is increased along with the warm rolling passes. After more than approximately 10% strain, most grains are subdivided into light-defined cells whose walls consist of a tangled network of dislocations. Within each cell, the dislocation density is low. The low temperature at which the specimen is deformed increases the number of long, straight screw dislocations by inhibiting cross-slip. The substructure of steel after large deformation, approximately 20% reduction or more, cannot be described solely based on

Fig. 10 Bright-field mode TEM micrographs of the (a) as-received sample. Substructure showing a smooth structure and some incipient dislocation walls in austenite, dislocation arrays with subgrains in fer-

rite (arrow). (b) Subgrain boundary (SGB), and discrete dislocation loops in ferrite (arrow)

dislocation glide, dislocation cell formation, and changes in cell dimensions. It has become apparent that in heavily deformed metals, deformation occurs by a series of different processes, each of which operates within a particular range of deformation. With increasing deformation of DSS, the sequence is: (1) slip by dislocation glide, (2) formation of microbands, and (3) macroscopic shearing.

Figures 9, 10, 11, 12, 13, 14 and 15 show TEM images of samples in the as-received and warm-rolled conditions. It is clear that warm deformation introduces a large amount of dislocations in the microstructure, resulting in a characteristic arrangement, depending on the phase, ferrite or austenite. The original grains of ferrite and austenite are subdivided into small arrangements of cell blocks, DDW subgrains, and irregular assembly of dislocations, called forests. The micrographs (Figs. 9, 10, 11, 12, 13, 14, and 15) confirm that grain sizes are reduced to the micrometer scale, or even to lower dimensions, that is, the nanometer scale. The dislocation structure in austenite changed from a planar slip and discrete loops to subgrains. In ferrite, there were cellular block structures. Formation of DDW occurs in both austenite and ferrite phases. This intense formation of DDW in both phases was observed after warm deformation at 600 °C. Hong and Lee [40] working on a low-cycle fatigue (LCF) 316L stainless steel emphasized that for temperatures below 250 °C, a cellular substructure changes to planar arrangements when the temperature reaches 250–600 °C in a dynamic strain aging regime (DSA). At a high strain rate, it changed back to equiaxial cells. In addition, there was a subgrain structure formation at a low strain rate above 600 °C, for instance, in Figs. 10b and 11a. This indicates that the plastic deformation mechanism goes from the wavy slip mode in the

non-DSS regime to the planar slip mode in the DSA regime, thus restricting the cross-slip of screw dislocations [40–42]. This appears to be consistent with the gradual development of larger-angle planar dislocation walls on the background of misoriented tangled cells, observed by TEM in hot deformed austenite at low strains, as described by Cizek [32].

Dini and Ueji [30] have reported that the planar glide mode is the dominant mechanism before mechanical twinning commencement in TWIP steels (austenitic matrix). According to the authors, at low strains, the activation of primary slip systems from dislocation sources during plastic deformation leads to the formation of dislocation pile-ups against grain boundaries. With increasing strain, the number of dislocation sources increases, and with the activation of two or three slip systems and the lack of mobility, dislocations rearranged themselves in a complex geometric configuration, as can be seen in Fig. 9c, which is a Taylor lattice. In addition, the deformed ferrite microstructure consisted of undefined or undeveloped dislocation cells, as shown in Figs. 11 and 12. In previous cold-rolled DSSs, the formation of equiaxed and elongated cells was reported for a ferritic structure when it was deformed at room temperature and low deformation, $\epsilon = 0.18$ [43].

Experiments were carried out in austenitic stainless specimens to elucidate the dislocation substructure under LCF loading conditions at 20 and 350 °C. Typical dislocation substructures were found to be cell block structures at 20 °C, as shown in Fig. 11a and b. Still, slip planarity seemed to prevail at 350 °C [42]. In agreement with a previous report [42], for the current research, this condition was found for the as-received sample. Figure 9c represents the typical well-developed planar slip band that characterizes

Fig. 11 TEM micrographs obtained in bright-field mode corresponding to substructure of the warm-rolled sample with 60% reduction at 600 °C: **(a)** Formation of cell block along with low-angle tangled dislocation boundaries and dislocation walls. The slip band in **(c)** shows

dislocation entanglements with high dense dislocation walls (DDWs) in ferrite and austenite. **(d)** Electron diffraction pattern with respect to austenite in the central region of the micrograph indicated by the arrow in **(c)**. SB: shear band

Fig. 12 TEM micrographs in bright-field mode of the 60% warm-rolled: **(a)** Substructure showing dislocation walls (DDW) in the austenite (left side), and the mottled contrast in ferrite associated with

incipient subgrain formation (upper center of the micrograph). **(b)** Original high-angle grain boundaries between ferrite and austenite (arrows)

Fig. 13 TEM micrographs in bright-field mode of the 80% warm-rolled sample, (a) and (b). (a) Formation of elongated cells and dense dislocation walls in austenite. (b) Details of the region delineated with a white rectangle in (a). (b) Cell block in ferrite (arrow) and DDW in austenite

Fig. 14 (a) Micrograph obtained in TEM bright-field mode of the warm-rolled sample with 80% reduction; (b) respective electron diffraction pattern. (a) Dense dislocation walls and poorly defined channels in the ferrite, fragmented structure near a grain boundary (red

arrow). (b) Electron diffraction pattern with respect to ferrite in the central region of the micrograph in (a), indicated by the blue arrow inside

the Taylor lattice. Ferrite, on the other hand, has a crystalline bcc system with high SFE. Because the probability of generating stacking faults is very small, the screw dislocations can cross-slip, allowing for the accommodation of large deformation with a small change in the orientation of their crystallographic substructure. Thus, grain refinement is achieved by successive subdivision of the original grains in the dislocation walls [23, 40, 43–45].

TEM images of the fine substructures that evolved at the final stage of warm deformation clearly show the development of very intense gliding of dislocations in the austenite,

primarily at the interphase boundary (Fig. 15a and b). Conversely, the ferrite phase is characterized by a conventional cold-worked substructure containing a high amount of DDWs and dislocation cell blocks. In the austenite phase, deformation twin boundaries are usually distinguished by their characteristics, such as straight shapes, close parallel locations, and misorientations that quickly change with straining. However, no evidence was found in warm-rolled DSS specimens because of the high temperature employed for the latter. The increase in deformation temperature increases the SFE, reducing the probability of mechanical

(a)

(b)

Fig. 15 (a) TEM bright-field micrograph of the 80% warm-rolled specimen, showing DDW and dislocation tangles, with incipient cell formation in the ferrite substructure. (b) Results of chemical analysis by EDS in regions identified by points EDS1 and EDS2 in (a)

twinning formation [46]. The dislocation substructures in the austenite appear as very dense irregular parts with rather thick diffuse sub-boundaries, presenting a wide spectrum of low- to high-angle misorientations (Figs. 12b and 13a). The presence of the cell structure of dislocations is attributed to cross slipping in screw dislocations, observed in ferrite grains [47]. The dislocation density in ferrite is lower than that in austenite because of the recovery process through annihilation of dislocations; only this mechanism operates at a low temperature of 600 °C [29, 45, 47, 48]. This condition can be confirmed by examination of Figs. 9 and 10. The austenite phase has more non-resolved Kikuchi bands during the EBSD examination, that is, the austenite grain

(a)

(b)

Fig. 16 Engineering stress–strain curves for: (a) AR, WR 60%, and WR 80%. (b) AR and WR samples after annealing at 1050 °C for 300 s

structure is not resolved as ferrite grains, as shown in Fig. 5. This means that a high deformation state is translated by intense dislocation gliding. This condition implies that it is more difficult for an EBSD system to identify and classify the Kikuchi bands [31, 35].

Tensile and Hardness Tests

Figure 16a corresponds to the engineering stress–strain curve for the as-received and warm-rolled samples, as well as the curves for all the samples after annealing at 1050 °C for 300 s in Fig. 16b. Figure 16a shows that warm rolling led to a significant increase in ultimate tensile strength and a reduction in ductility. In addition, the Vickers hardness

(Table 3) of the warm-rolled samples converged to an average value of 408 HV, well above the steel in the as-received condition. Therefore, it is possible to conclude that the warm-rolling route of the DSS used here is suitable to produce a high-strength microstructure, as the warm-rolled condition can double the yield and tensile strength (Fig. 16a).

The Vickers hardness of the annealed samples (Table 3) converged to an average value of 255 HV, slightly inferior to that of the as-received steel. In addition, when the warm-rolled and as-received samples were annealed at 1050 °C for 300 s, there was an increase in ductility and strength reduction, as shown in Fig. 16b. Comparing the as-received sample in Fig. 16a with the three conditions in Fig. 16b, there is a possible advantage in applying warm rolling and annealing to the steel, to reach industrial conditions, without the need for cold rolling and subsequent annealing. However, this strength level has already been reached in hot rolling conditions. However, to reach a thickness of 1–1.5 mm for commercial applications, warm rolling would be necessary.

As shown in Figs. 5 and 8 the grain size results, the austenite phase undergoes a more intense deformation, resulting in a fine substructure as consequence of strain hardening. In contrast, the ferrite was not able to undergo strain hardening at the same intensity, so it was prone to deformation by a cleavage mechanism [7].

There is a pronounced loss of ductility associated with warm rolling at 600 °C, as shown in Fig. 16a. However, it is worth observing that the 80% warm-rolled sample did not have its ductility reduced compared with the 60% sample. In fact, it showed a slightly larger ductility than the sample warm-rolled with 60% reduction (Fig. 16a).

After warm rolling, both the austenite and ferrite phases are self-distinguishable because of differences in the structure and intense heterogeneity (Fig. 5b and c). Conversely, the as-received sample has a more uniform microstructure, with a lamellar structure oriented according to the rolling direction (Fig. 5a). However, the dominant dislocation arrangements in both conditions (60 and 80% reduction) are still the same: intense formation of DDWs in austenite and subdivision in ferrite grains by dislocation, forming extended dislocation walls. In the specimen with the highest total strain, the appearance of deformation was very swift during the tensile tests. Necking occurred above the plastic

deformation over the yield strength. The total elongation of the severely strained steel in a small volume was significantly reduced. Such behavior is typical for nanostructured materials [48, 49]. According to the literature [27], microcracks or shear bands (Fig. 5b) should terminate at the grain boundaries. However, it is difficult to determine the mechanisms responsible for the fracture because of the small volumes (reduced grain size, Fig. 8) in which the deformation takes place, for instance, grain diameter or α/γ interface [27].

The engineering stress–strain curves of the tensile specimens, corresponding to the microstructures shown in Fig. 5, are presented in Fig. 16a. For comparison, the tensile curves of the specimens annealed at 1050 °C for 300 s, after warm rolling, are also plotted in Fig. 16b. A distinct loss in strength when compared with its warm-rolled counterpart process can be easily observed. The difference stems from the grain size in both phases, which is greatly reduced to approximately the nanometer scale, for the warm work applied here. The structure after deformation at 600 °C, containing an ultrafine substructure, showed a stress peak soon after the yield point and failed at a percentage near the total elongation (Fig. 16a). These warm-worked samples displayed a small uniform strain-hardening characteristic, but showed a reasonable post-necking deformation, that is, total elongation. The lack of strain hardenability of the microstructure with nanostructured configuration has been anticipated by some authors [40, 47] and is considered to be the primary cause of low uniform tensile elongation. In addition, the ultrafine grained (UFG) size microstructure consisted of an intense deformed substructure with cellular regions, tangles of dislocation arrays, and high-density dislocation walls (HDDW), which strongly contribute to high strength and a lesser strain work hardenability [50, 51], in agreement with a previous report [51, 52].

A similar level of mechanical strength to that depicted here was reached in an experiment that involved a hydrostatic extrusion process with a true deformation of 1.4 in a DSS with 0.3%C, 22%Cr, 5%Ni, and 3.0%Mo. In this case, a tensile strength of 1370 MPa was reached, but with only 5% of the total elongation [27].

The high level of mechanical strength found in the present work is due to the substructure formation: dislocation tangles, cell block dislocations, incipient formation of subgrains, and DDWs. An additional increase in disorientation by dislocation accumulation; development of deformation heterogeneities, such as deformation and shear bands, which intensify to a large gradient of orientation in a small volume of the microstructure; and coalescence of the contours for large deformations to form cell blocks also contribute to the strength. The texture formation mechanisms involve relative rotation between different parts of a grain for different final orientations because of differences in the slip systems selection [28–30].

Table 3 Results of Vickers microhardness tests (4.9 N–10 s)

Condition	HV	HV (annealed at 1050 °C for 300 s)
As-received	281 ± 12	265 ± 7
Warm-rolled 60%	410 ± 9	244 ± 5
Warm-rolled 80%	405 ± 14	256 ± 6

Ratuszek and Witkowska [53] have reported that the process of texture formation proceeds in different way depending on the initial orientation distribution and, also, that the formation and character of the final deformation textures is strongly influenced by the previous morphology of DSS phases. They show that ferrite–austenite band-like structure exerts considerable effect on slip behavior. Since Burgers vectors of slip dislocations in each DSS phase are different thus only slip transfer may occur across the α/γ interfaces. It seems that the interphase boundaries are strong barriers even for the co-ordinate dislocation motion throughout the band-like structure and the extension of shear banding is apparently strongly restrained by the α/γ interfaces, affecting the rolling texture.

Notwithstanding of processing conditions, the residual dislocation density in the all samples subjected to cold or warm rolling followed by annealing at different temperatures varies locally in a rather wide range [28]. The density is not the same in different recovered grains. As should be expected, the values of local dislocation density decrease with an increase in the warm rolled pass due to the interval between them, when the steel recovery the temperature for the next pass. As a result, the variation range of the residual dislocation density tightens with a progress in recovery because of reduction of local high dislocation densities first. Nevertheless, the dislocation density varies locally in a wide range even in apparently well recrystallized microstructures of an austenitic stainless steel after annealing, as suggest by Odnobokova et al. [28].

The grain refinement mechanisms are different between austenite and ferrite, for instance, during the ECAP process was described by Chen et al. At the early stage of ECAP, the ferrite should be refined by successive subdivision of dislocation walls due to the operation of multi-slip systems. Further deformation will increase the portion of high-angle grain boundaries owing to rotation of subgrains, leading to the formation of nanostructure in ferrite [29]. However, the deformation mechanisms of austenite are associated with dislocation slip and deformation twinning. The intersection of deformation twins may result in rapid subdivision of coarse grains into submicron-sized blocks, which could be further refined by the formation of micro twins and dislocation boundaries inside the interiors [29, 30]. During the warm rolling, as function of temperature, the deformation twin is not formed.

Deformation microstructures of polycrystalline single phase materials with medium to high SFE show a continuous evolution with strain in which stable glide dominates. An important characteristic of this evolution is the subdivision of the grains into cell blocks, which deform by scarcer slip systems than specified by the Taylor criterion for strain accommodation. However, the groups of neighboring cell block fulfill the Taylor criterion collectively, with increasing

strain the size of the cell block shrinks faster than usual dislocation cells. Subgrains are formed as a last step in this evolution. New cells blocks can be formed principally through the formation of microbands [50].

Hence, the grain refinement mechanisms are different between austenite and ferrite in DSS, leading to heterogeneous microstructure after thermomechanical pressing [28]. Therefore, the work hardening can be a function of both the dislocation structure and the specific microstructural features such as cell blocks and DDW.

It is generally accepted that austenite with low SFE results in limited cross-slips, and hence, a low recovery rate. Consequently, the main softening mechanism in austenite for hot working would be static or discontinuous recrystallization. Ferrite, in turn, has high SFE, so its recovery rate is high, alongside a higher diffusion rate. Consequently, the recrystallization driving force remains far too low, making it unobserved, mostly for low temperatures employed in warm work at 600 °C.

Conclusions

In the present study, a 2205-type duplex stainless steel was warm rolled at 600 °C with 60 and 80% thickness reduction, followed by air cooling. The main results can be summarized as follows:

1. The warm rolling increased the tensile strength, reaching 1185 MPa for 60% reduction and 1328 MPa for 80% reduction. The as-received steel tensile strength was 774 MPa, and the hardness was 281 HV in the hot-rolled and solution-annealed condition. The hardness of the steel was approximately 408 HV for both warm-rolled conditions.
2. The ferrite microtexture presented α -fibers and a rotated cube component, while the austenite showed brass and S components. The cube and brass components undergo a copper transformation as long as the deformation increases. The α -fiber for ferrite and the copper and brass components for austenite are representative of the deformation texture of their respective phases. Goss in austenite is present at the highest deformation.
3. The substructure is characterized by intense formation tangles or forests of dislocations and dense dislocation walls, as revealed by transmission electron microscopy. For the ferritic phase, intense formation of dislocation substructures and cell blocks with discrete subgrains formation was observed. In the austenitic phase, the substructure was categorized by planar glide of dislocations and formation of dislocation tangles and dense dislocation walls. Tangled dislocation formation, along with a great amount of dense dislocation walls and cell blocks,

represent strong obstacles for dislocation movement, which led to a high strength in the fine substructured DSS.

4. Mechanical behavior comparison with the steel in the as-received condition revealed that the warm work raised the strength level of the DSS by between 65 and 72%. These results draw attention to innovative routes for the industrial production of this duplex stainless-steel class, even considering the ductility reduction.

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