Deformation and Annealing Behavior of Nitrogen Alloyed Duplex Stainless Steels.  Part I: Rolling

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The deformation behavior, i.e. the microstructure development and the deformation mechanisms in nitrogen alloyed ferritic-austenitic duplex stainless steels were studied by means of microstructure investigations and texture measurements in both phases. By comparison with corresponding single phase bcc and fcc materials and Taylor type deformation texture simulations it was possible to reveal the influence of the complementary phase on deformation of each phase during hot and cold rolling. To determine the effect of nitrogen on the deformation mechanisms the nitrogen content in the investigated steels was varied between 0.15 to 0.62 %. The investigations prove that during rolling both phases deform similar to the respective single phase material and that there is a minor influence of the second phase on the deformation mechanisms due to the morphological constraints of the typical microstructure in rolled duplex materials.

KEY WORDS: duplex stainless steel; deformation mechanism; texture; microstructure; nitrogen.

1. Introduction

Duplex stainless steels (DSS) have been known since the 1930’s, but due to an insufficiently accurate chemical analysis and too high residual stress levels, both of which are important prerequisites for reproducible properties, their commercial use in corrosive environments under high mechanical duty occurred only recently. In this class of materials the high nitrogen alloyed grades seem to have the most promising properties and economic qualifications. The development of high nitrogen duplex steels appears, therefore, very attractive, because in addition to the specific improvements resulting from the coupling between ferrite and austenite and from nitrogen-enriched austenite, the interphase boundaries strongly confine grain growth. In order to improve the DSS and to optimize specific properties the physical mechanisms and thus, the differences with single phase materials have to be understood. The aim of this work is to elucidate how the second phase affects deformation and recrystallization in duplex stainless steels and to determine how nitrogen modifies the involved micromechanisms. In this part the hot rolling of DSS is investigated by microstructure investigations and texture measurement. By means of Taylor simulations the influence of different processing routes of the DSS and the role of the second phase on texture development can be probed.

While the texture development of cold rolled and annealed single phase steels (ferritic and austenitic steels) is well known, the texture evolution in duplex stainless steels has not been systematically investigated. It has been shown that the activity of crystallographic slip during deformation leads to pronounced textures in bcc and fcc metals. Changes in the strain distribution and even recrystallisation (see Part II: annealing) mechanisms will alter these textures. An analysis and comparison of the textures in DSS and single phase materials provides valuable information on the influence of the second phase on hardening and softening mechanisms of DSS. Figure 1 shows the most important texture components in the ODF for ferritic and austenitic single phase steels and allow to identify the mentioned orientations of this paper.

2. Experimental

The chemical composition of the investigated steel grades is given in Table 1. These duplex and super duplex stainless steel grades—the terms refer to their pitting resistance equivalent number (PREN)—are commercial hot deformed alloys with different thermo-mechanical histories with the exception of DSS 5 which came from a test alloy and was available as a slab of an ingot. DSS 1 was an extruded and round forged rod material $\Phi 30$ mm. DSS 2 and DSS 3 were hot rolled rod materials. The rectangular rod profiles $45 \times 12$ mm$^2$ were produced by alternately changing the rolling plane (horizontal and vertical). DSS 4 was a hot rolled plate with 6 mm thickness.

Subsequent to the different industrial thermo-mechanical treatments all the samples were homogeneously unidirectionally cold rolled with the same rolling schedule under oil.
lubrication at room temperature on a laboratory rolling mill to thickness reductions up to 90%. The microstructure development was investigated by optical microscopy. All textures were determined from the normal direction (ND) (i.e. in the sheet plane) with Co-Kα1 radiation (λ = 1.79Å) by means of incomplete pole Figs. (5°/H11349 b/75°), three \{200\}g, \{113\}g for the austenite phase \( g \) and two \{200\}a, \{112\}a for the ferrite phase \( a \) in back reflection mode on a fully automatic texture goniometer. By use of a position sensitive detector (PSD) it was possible to record and deconvolute the \{111\}g and \{110\}a pole figures. Despite of the close interplanar spacing \( d_{a}^{111} - d_{g}^{110} \). Taylor type deformation texture simulations were performed for DSS 2 and DSS3 by discretizing the textures before the last hot rolling sequences of these steels. Simulations were performed for different deformation modes changing from full to relaxed constraints \(^{4,5}\) and for various numbers of active slip systems. For DSS 2 additionally the change of width during hot rolling of the rod profile was accounted for in the used displacement gradient tensors. The displacement gradient tensors employed were estimated from the change of the sample dimensions and the rolling schedules which were known.

The austenite/ferrite ratio alters with increasing nitrogen content due to its strong austenite stabilizing properties from 45/44 % for DSS 1, 50/50 % for DSS 2 and 3, 55/45 % for DSS 4 to 60/40 % for DSS 5. The austenite stabilizing effect of nitrogen is well known and thus will not be further discussed.

3. Results
3.1. Hot Rolling
Hot rolling was conducted only for DSS 2, DSS 3 and DSS 4 on an industrial scale. The extruded and subsequently round forged DSS 1 and the cast material DSS 5 were excluded from the hot rolling examinations because of too different thermo-mechanical treatments for the latter materials.

The microstructure development during hot rolling is given in Fig. 2 for DSS 4 but is also representative for DSS 2 and DSS 3. In the presented longitudinal sections the microstructures of these steels do not differ remarkably after hot rolling (Fig. 2(b)). There are some obvious differences in the microstructures due to different production schemes of rod (DDS 2, 3) and sheet (DSS 4) material. The macroscopic product shapes are reflected by the initial phase morphology with regard to the extension in transverse direction. So the phase morphology of both phases receives a flatter morphology (DSS 4) in the sheet material than in the rod material (DSS 2 and DSS 3).

With increasing deformation during hot rolling austenite and ferrite align in such a way that both phases arrange in alternate layers and elongated flat shapes parallel to the rolling direction as shown in the longitudinal section (Fig.

| Steel | % N | % Cr | % Ni | % Mo | % Mn | % Cu | % C | % Fe | austenite / ferrite |
|-------|-----|------|------|------|------|------|-----|-----|-------------------|
| DSS 1 | 0.153 | 24.16 | 5.48 | 3.27 | 1.20 | - | 0.022 | balance | 45/55 |
| DSS 2 | 0.25 | 25.00 | 6.50 | 3.50 | - | 1.5 | -0.03 | balance | 50/50 |
| DSS 3 | 0.25 | 25.30 | 7.20 | 3.50 | 0.50 | 0.62 | 0.014 | balance | 50/50 |
| DSS 4 | 0.4 | 25.20 | 7.10 | 4.10 | 2.90 | 0.10 | 0.023 | balance | 55/45 |
| DSS 5 | 0.62 | 22.50 | 2.80 | 5.52 | 0.46 | 0.025 | 0.025 | balance | 60/40 |

Fig. 1. (a) Typical orientation distribution function (ODF) of ferrite (ϕ2 = 45° section) and (b) austenite texture (fibers and components: W – Cube, G – Goss, MS – brass, BR – brass recrystallization, D – Taylor, C – Copper, S – S orientation); (c) definition of the relaxed shear components.
2(b)). After hot rolling the “flat” morphology of the ferritic phase areas appears slightly more continuous than that of the austenitic phase in concordance with the matrix role played by ferrite.

The textures of the austenitic phases of DSS 2, DSS 4 and of a single phase austenitic stainless steel after hot rolling are presented in Fig. 3. The austenite texture of the rod material (DSS 2) consisted mainly of cube orientation \{001\}/H20855 \(100\)/H20856, Goss orientation \{011\}/H20855 \(100\)/H20856 and brass orientation \{011\}/H20855 \(211\)/H20856 with some spread (Fig. 3(a)). The textures of the sheet material were composed of cube and brass orientation. All three ODFs exhibit a weak \(\beta\)-fiber. Clearly the austenite textures of sheet products—two phase DSS 4 and single phase austenite—are very similar.

The hot rolling textures of the ferrite phases and of the single phase ferritic steel compare well among the sheet material (Figs. 4(b), 4(c)) but are very different from the rod material DSS 2 (Fig. 4(a)). The ferrite ODF of this rod shaped sample (DSS 2) exhibits strong intensities of the cube orientation with spread toward the 45° rotated (about ND) cube orientation \{001\}/(110) and from there along the \(\alpha\)-fiber up to \{112\}/(110) (Fig. 4(a)). DSS 4 reveals an \(\alpha\)-fiber with decreasing intensity toward \{111\}<110> (Fig. 4(b)). In contrast to duplex samples the ferritic single phase steel comprises a complete \(\alpha\)-fiber of constant intensity up to \{111\}/(110) and extending beyond with lower intensities.

Figure 5 shows the results of respective Taylor simulations. The simulated hot rolling textures of DSS 2 and DSS 4 were obtained by using 48 slip systems \{110\}-, \{112\}- and \{123\}-slip planes. For DSS 2 the \(\varepsilon_{23}\) strain rate tensor component was relaxed and the widening observed for this geometry during rolling due to the small width/thickness ratio of the rod profile, was taken into account for the rolling deformation. There was no need to consider a change of width for the sheet format of DSS 4, but both the \(\varepsilon_{23}\) and the \(\varepsilon_{13}\) strain tensor components were relaxed as typical for a texture simulation of rolled microstructure morphologies. Qualitatively, the simulated textures compared well to experimental results but generally the predicted intensities were too high. Also, the computed \(\alpha\)-fiber intensities close to \{112\}/(110) for the DSS 2 and the intensities between \{112\}/(110) and \{111\}/(110) for DSS 4 were far too high compared to the other texture components.

3.2. Cold Rolling

The anisotropy of microstructure generated during hot rolling was intensified by cold rolling. Figure 6 illustrates the strong microstructure refinement up to an extremely fine thickness of the constituent phases of 1–5 \(\mu\)m in sheet normal direction. Between 60 and 90% thickness reduction some lenticular bulges formed in the austenitic (light) phase areas. However, it is stressed that no well-defined shear band development was apparent in the microstructure.
in contrast to fcc single phase materials. There were some shear bands in both phases but their extension was obstructed by the phase boundaries and did not reach dimensions comparable to observations in single phase materials.

The texture development of the austenite phases was qualitatively very similar among the investigated duplex stainless steels (here only shown for DSS 1 and DSS 4) and resembled single phase brass (Fig. 7). The textures mainly consisted of an incomplete $\alpha$-fiber extending to the Brass $\{011\}\langle211\rangle$ orientation. A sensible difference between the duplex and single phase textures is the comparatively lower Goss orientation density of the single phase material.

The ferrite cold rolling textures of DSS 1 and of single phase ferritic stainless steel were very similar, while the textures of the other DSS represented by DSS 4 showed some differences. The $\alpha$-fiber in the latter terminated between $\{112\}\langle110\rangle$ and $\{111\}\langle110\rangle$ and especially the $\alpha$-fiber is more pronounced from $\{001\}\langle110\rangle$ to $\{112\}\langle110\rangle$. While DSS 1 and the single phase steel also exhibited $\{111\}\langle112\rangle$ besides a weak $\gamma$-fiber, this orientation is nearly completely missing in the other DSSs.

4. Discussion

4.1. Strain Partitioning

Hot rolling of DSSs was performed in the temperature range between 1250°C and 900°C. Due to the high starting temperatures the austenite phase fraction was reduced remarkably. With falling temperature during the hot rolling process the austenite phase content increased. From hot
rolling of single phase steels it is well known that the rolling forces are reduced upon a phase change from austenite to ferrite. The cause of this material softening is the temperature dependence of the Peierls-stress in the ferrite phase, which is the dominant stress component for plastic deformation of bcc metals. The flow stress in fcc metals is determined by the passing stress of parallel dislocations and the intersection of forest dislocations. The long range passing stress is virtually independent of temperature. With regard to the DSS this means that the austenite phase, the volume fraction of which increases with decreasing rolling temperature seems to be slightly harder than the ferritic phase, so that more strain is partitioned to the ferrite. Thus, in DSS decreasing rolling temperatures have a double negative effect, i.e. generally increasing rolling forces and reduced formability of the materials as well as the additionally developing austenitic phase with a higher strength at these temperatures. Below a critical temperature (approx. 950°C) precipitation will degrade formability and the material is liable to fail easily.

4.2. Hot Rolling Textures

The textures of the ferrite phase appear more strongly developed and reveal notably higher intensities. The texture of the austenite phase of DSS and of the austenitic single phase material are very similar especially for the sheet material of DSS 4 (Fig. 3). The texture similarity of these two and single phase materials suggests the activation of the same deformation mechanisms, i.e. the deformation mechanisms and their relative contribution to the total deformation must be comparable. The different texture, in particular the generated Goss orientation in the materials (DSS 2) was apparently due to the different strain path to produce rod material, i.e. the rolling plane normal altered between normal and transverse direction (Fig. 3(a)).

The rod rolling of DSS 2 led to a strongly different ferrite orientation distribution (Fig. 4(a)) compared to the sheet material DSS 4 and the single phase ferritic sheet material, with the latter two showing very similar textures (Figs. 4(b), 4(c)).

We surmise that the higher texture intensities and the qualitatively stronger influence of the different rolling modes in ferrite can be attributed to the lower tendency of ferrite to undergo dynamic recrystallization. The high stacking fault energy (SFE) of the bcc ferrite promotes dynamic recovery, which in turn conserves the rolling orientations. In contrast the austenite will undergo dynamic recrystallization especially during the early stages of hot rolling where the temperatures are high. Dynamic recrystallization continuously annihilates rolling orientations and thus prevents the development of a rolling texture. A retention of the deformation texture in the austenite phase is only possible for the final rolling passes or even the last pass, which was parallel to the “sheet” plane for the rod material. This explains the minor influence of the different rolling modes for DSS 2 and DSS 4 in the austenitic phase.

4.3. Taylor Simulations

The results of the Taylor simulation of the ferrite phase substantiate that the different textures of sheet (DSS 4) and rod material (DSS 2) are due to the different deformation modes (rod, sheet). Because the previous steps of hot rolling already had generated a pancake grain microstructure, the Taylor simulation was carried out with relaxed constraints for \( e_{13} \) and \( e_{23} \). In the sheet material a relaxation of the shear \( e_{13} \) and \( e_{23} \) produced the best agreement with experimental results. For DSS 2 (rod material) a relaxation of \( e_{23} \) was not sensible due to the alternating rolling plane (Fig. 1(c)), which prevents shear in this direction. A relaxation of the \( e_{12} \) shear component, which is conceivable for a thickness reduction in transverse direction did not lead to an improved texture prediction owing to the consecutive rolling from sheet normal direction which reversed the \( e_{12} \) shear. Since the used simulation algorithm did not take into account influences of a second phase the good agreement between prediction and experimental single phase texture revealed a negligible influence of the second phase on deformation mechanisms during hot rolling.

The shown hot rolling textures of the individual phases in the DSS were not identical to the respective single phase ones, particularly the textures of ferritic stainless steel showed fluctuations especially in the formation of \( \alpha \)-fiber intensities. It should also not be forgotten that all materials compared were not rolled under exactly the same conditions.
4.4. Microstructure Development During Cold Rolling

In the course of cold rolling both phases further developed the typical rolling morphology and both phases showed only slight differences in thickness reduction. With higher degrees of deformation the ferrite layers got thinner slightly faster than the austenite layers due to the higher hardening of the austenitic phase, which is related to its low stacking fault energy and thus, depends on the nitrogen content. The hardening of the austenitic phase rises with increasing nitrogen content of the DSS. Nitrogen is likely to increase the stacking fault energy of austenitic stainless steel\(^\text{10}\) up to nitrogen concentrations of about 0.4\% and to lower it at even higher concentrations.\(^\text{7}\) Nitrogen dissolved to 90\% in the austenite phase and thus, the concentration in the austenite of the investigated DSS was higher than 0.4\%, and one can expect a decreasing stacking fault energy in the steels from DSS 1 to DSS 5. In addition to solid solution hardening the lower stacking fault energy due to nitrogen also increased work hardening. Nevertheless, the cold rolling strain was almost equally partitioned among the two phases.

Between 60 and 90\% thickness reduction some lenticular shaped areas developed (DSS 1, 2 and 4), which were associated with deformation induced martensite\(^\text{11}\) or may have originated from an interaction of shear bands and deformation twins,\(^\text{9}\) meanwhile for the latter no evidence was found in this study. The texture development, specifically the high Goss intensities, also indicated that deformation induced martensite was responsible for this microstructure effect. Lense shaped areas were also found for deformation induced martensite in single phase austenitic steels. This was explained by a preferential martensite transformation of the Brass component during deformation\(^\text{10}\) while grains with a (100) axis parallel to the direction of principal stress were found particularly resistant to martensitic transformation.\(^\text{11}\) Obviously, the Goss orientation \{011\}{(100)} complies with this criterion while the Brass orientation \{011\}{(211)} does not. The DSS with higher nitrogen concentration (DSS 3 and 5) did not show these lenticular areas, which is consistent with the austenite stabilizing role of nitrogen similar to that of carbon.

4.5. Cold Rolling Textures

As in single phase ferritic stainless steel the rolling textures of the ferrite phase in DSS and in general were not very homogeneous and often exhibited a variance of intensity along the \(\alpha\)-fiber. The found differences in the cold rolling textures of the ferrite phase in the investigated DSS originated, of course, in the different starting textures, which in turn were due to the hot rolling textures because of different forming modes, \textit{i.e.} rolling (DSS 2, 3 and 4), forging (DSS 1) and casting (DSS 5). Apparently the development of a rolling type texture during the whole rolling process was not as continuous as for single phase materials, especially in the ferritic phase. Furthermore, the formation of the typical fibres and stable end orientations was delayed to higher rolling degrees. This slow and discontinuous texture development was likely affected by changing strain partitioning between austenite and ferrite during the rolling process. However, the finally evolving textures were qualitatively comparable to the textures of single phase material. Therefore, the activated major deformation mechanisms did not change from single phase ferrite or austenite steels to DSS.

4.6. Conclusions on Ferrite and Austenite Interactions during Rolling

The negligible influence of the second phase on deformation mechanisms in both the ferritic and austenitic volumes can be understood from the specific microstructure development during rolling (Fig. 9). The major strain accommodation between ferrite and austenite occurs in those areas (\(\lambda\)) where phase boundaries separate ferrite and austenite in rolling directions. These areas represent only a small fraction of the total interface. The largest part of the total phase boundary area (\(\Lambda\) in Fig. 9) lies parallel to the sheet plane and as long as the strains in both phases are comparable no strong interaction of both phases is expected at the phase boundaries if not the mutual influence could be compared to a friction effect. The Taylor simulation gave good agreement with experimental results, when \(\varepsilon_{11}\) and \(\varepsilon_{23}\) were relaxed. This indicates only negligible phase interaction across the interfaces denoted as \(\lambda\) in Fig. 9. Most of the
volume of each phase (A in Fig. 9) can, therefore, deform almost independently like a “single phase” and thus, develops a typical rolling texture as known from single phase material. In fact, the textures give evidence that the two connected phases deform like independent single phase materials.

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