Cold Rolled Texture and Microstructure in Types 304 and 316L Austenitic Stainless Steels

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Two grades of austenitic stainless steel (ASS), types 304 (UNS S 30400) and 316L (UNS S 31603), were cold rolled to different reductions by unidirectional and by cross-rolling. The steels had reasonable difference in stacking fault energy (estimated respectively as 15 and 61 mJ/m² in types 304 and 316L) and also in starting (or pre-deformation) crystallographic texture—being relatively weak and reasonably strong in types 304 and 316L respectively. The cold rolling increased texturing in type 304, but not in type 316L ASS. The more significant effect of cold rolled texture development was in the relative increase of Brass ([011]<211>/H20856) against Copper ([112]<111>/H20856) and S ([231]<346>/H20856) orientations. In type 304 the increase in Brass was significant, while in type 316L the increase in Copper and S was stronger. This effect could be captured by Taylor type deformation texture simulations considering stronger twinning contributions in type 304—for example the respective ‘best-fits’ (in terms of matching the changes in the volume fractions of Brass against Copper and S) were obtained by full constraint Taylor model with 1 : 100 and 1 : 10 slip : twin activities in types 304 and 316L ASS respectively.

Microstructural developments during cold rolling were generalized as strain induced martensite formation and developments of dislocation substructure. The former, as estimated by vibrating sample magnetometer (VSM), increased with cold reduction, being significantly more in type 304 and was also noticeably stronger in both grades under cross-rolling. The most significant aspect of substructural developments was the formation of strain localizations. These were observed as dense dislocation walls (DDWs), micro-bands (MBs) and twin lamellar structures (TLS). The TLS contribution gained significance at higher reductions and during cross-rolling, especially in type 304. Large misorientation development and the accompanying grain splittings were always associated with such strain localizations. Efforts to relate Taylor factor (M) and textural softening (dM/de) values (of ideal texture components) with relative misorientation developments was, however, unsuccessful. No consistent trend could be established for any unique combination(s) of slip-twin in the respective alloys.

KEY WORDS: deformation; texture; microstructure; microtexture; strain localizations; strain induced martensite; austenitic stainless steel; twin; Taylor Models.

1. Background

Austenitic stainless steels (ASS) are used for their excellent corrosion resistance. The localized corrosion resistance of such steels can be substantially improved through control of chemistry, grain size and grain boundary nature. In a recent study, it was shown that extreme randomization of grain boundaries is an effective means of improving localized corrosion resistance in ASS—in particular types 304 (UNS S 30400) and 316L (UNS S31603). The extreme randomization of grain boundaries were obtained through large cold rolling followed by conventional solution annealing. Presence of strain localizations was suggested to be responsible for the effective randomization of grain boundaries during annealing. The present study is a subset of the larger study and makes an attempt at summarizing the observations on texture and microstructure developments in types 304 and 316L ASS during cold rolling.

In ASS, cold rolling is expected to have three effects—development in deformation texture, developments in dislocation substructure and strain induced martensite formation. Such developments, as in other fcc alloys, are expected to depend on stacking fault energy (SFE). The respective SFEs of types 304 and 316L ASS, as calculated empirically from their chemical compositions, are 15 mJ/m² and 61 mJ/m². This, on the other hand, is expected to induce different slip-twin activities and critical resolved shear stresses (CRSS) ultimately accounting for differences in deformation texture developments. The dislocation substructure development also depends significantly on the relative stacking fault energies, especially in terms of relative mobility and recoverability of the dislocations and...
on the twin activities.\textsuperscript{11,17–19,25,29,32–34} The formation of strain induced martensite formation is also expected to depend on strain, chemistry and dislocation activity.\textsuperscript{35–42} In a word, the difference in SFE between types 304 and 316L ASS are expected to bring substantial differences in structural developments during cold working. The understanding such developments is the objective of the present study.

2. Experimental Methods

2.1. Material

The present study used commercial ASS types 304 (UNS S 30400) and 316L (UNS S 31603), chemical composition of the respective types are given in Table 1. The former was received as a hot band bar with a relatively weak initial crystallographic texture, while type 316L was received as a finished plate with a strong crystallographic texture.\textsuperscript{12} Both the materials were solution annealed and then cold rolled to different reduction percentages (20–80\%) by unidirectional and by cross rolling\textsuperscript{*1} in a laboratory rolling mill and were then investigated through various characterization techniques.

2.2. Texture Measurement, Analysis and Simulation

Bulk texture measurements were obtained by inversion of 4 incomplete pole figures. The X-ray ODFs (Orientation Distribution Functions) were calculated by standard series expansion method\textsuperscript{43} using the program MTM-FHM.\textsuperscript{44} The starting bulk textures (generalized as as-received texture) were discretized\textsuperscript{44} into 3,000 representative orientations and were then subjected to Taylor type deformation texture simulations.\textsuperscript{12–16,47,48} The results of texture measurements and simulations were analyzed and compared from the respective volume fractions of ideal texture components.\textsuperscript{55,48} Volume fractions were estimated by convoluting the ODFs with suitable model functions, with an integrated ODF value of 1 and 16.5° Gaussian spread.\textsuperscript{45}

2.3. Microstructural Studies

Optical samples were prepared through conventional metallography followed by electrolytic etching, conducted at room temperature with a platinum cathode and at 6 volts dc in an electrolyte of 10 g oxalic acid and 90 mL water. The samples were studied under a crossed polarizer. The TEM thin foils were prepared in a Struers Tenupol 3 twin-jet electropolisher at −30°C, 11 volts dc and an electrolyte of 80:20 methanol: perchloric acid. A Philips CM200 TEM was used at 200 keV operating voltage. Local orientation measurement techniques were obtained using online interface of CM200 series, which uses deflector coil movements to measure distances in the microstructures. All microscopic observations were made on the rolling plane (containing rolling direction (RD) and transverse direction (TD)).

2.4. Strain Induced Martensite Formation

An effort was made to estimate strain induced martensite formation through following characterization techniques: (i) micro-hardness measurements, (ii) ferritoscope measurements, (iii) conventional X-ray diffraction (XRD) and (iv) vibrating sample magnetometer (VSM). The micro-hardness measurements may provide an index, that is somewhat corrupted by simultaneous substructural developments. The ferritoscope measurements are the quickest, but their accuracy may be questionable. The XRD technique is sensitive to the volume fraction of the detectable phase. For large reductions (20\% and above) in type 304, XRD was used to quantify percentage martensite using the standard formulation.\textsuperscript{29} At lower martensite percentages, however, XRD was not effective. The VSM, undoubtedly the most accurate and sensitive of all the methods in quantifying presence of magnetic phase(s) (strain induced martensite, in case of the present study), was used to estimate magnetic saturation per unit mass, using EGG PAR model 400 machine. The VSM results were then converted to percentage strain induced martensite, assuming a direct proportionality of percentage martensite (as estimated by XRD) and saturation magnetization.

3. Results

The observed structural changes during cold rolling were sub-divided in three categories:

3.1. Developments in Deformation Texture

Figures 1 and 2 show the ODFs outlining broadly the texture developments in types 304 and 316L ASS. Type 304 as-received material was with weak crystallographic texture (maximum ODF intensity was 2.07 times random). Cold reduction increased the texturing (maximum ODF at 80\% reduction was about 4), see Fig. 1. Type 316L as-received material, on the other hand, was a relatively textured material to start with (maximum ODF intensity was 6.12). Cold reduction did not alter the texturing substantially, though changes in texture was apparent, see Fig. 2.

The results of the ODFs, as in Figs. 1 and 2, were further elaborated in Figs. 3 and 4, in terms of volume fractions of major texture components. The major texture components were identified from usual\textsuperscript{11} ideal fcc texture components with, on average, more than 5\% volume. The changes in the texture components, with increasing percentage reduction, was assumed to be linear and the trends of such changes were estimated from the slope. As given in Table

| Material | C  | S  | P  | Si | Mn | Cr | Ni | Mo | Cu | N  |
|----------|----|----|----|----|----|----|----|----|----|----|
| 304      | 0.059 | 0.010 | 0.035 | 0.41 | 1.43 | 18.10 | 8.05 | 0.09 | 0.23 | 0.052 |
| 316L     | 0.025 | 0.07 | 0.025 | 0.46 | 1.58 | 17.11 | 11.69 | 2.57 | 0.026 | 0.027 |

\*1 Cross rolling had equal reductions in alternating direction.

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the important difference between types 304 and 316L were differences in changes of Brass (\{011\}/H20855 \{211\}/H20856) and Copper (\{112\}/H20855 \{111\}/H20856) and S (\{231\}/H20855 \{346\}/H20856). In type 304 the increase in Brass was significant, while in type 316L Copper and S had more significant increase, see Table 2(b), irrespective of unidirectional or cross-rolling.

An effort was made to capture such an effect, as in Table 2, with Taylor type deformation texture models and different CRSS (critical resolved shear stress) values for slip and twin.12–16,47–49) Table 3 summarizes the results of texture simulations. As shown in the table for unidirectional rolling,*2 increased twin activity (by adjusting the respective CRSS45,49) enhanced the increase in Brass along with a drop in Cu and S, an expected trend explaining classical Brass and Cu type textures.10) The ‘best-fit’, in terms of trend predictability of all texture components (as given in Table 2(a)), was obtained by full constraint (FC) Taylor model, closely matched by relaxed constraint pancake (RCP). The only difference, for FC respective ‘best-fit’ slip : twin ratios were 1 : 10 and 1 : 100 for 316L and 304 respectively (also indicated in Table 3), while the respective ratios for RCP were 1 : 1 and 1 : 10. In other words, it was possible to capture the basic difference of cold rolled texture developments in types 316L and 304 (as given in Table 2) by increasing the relative contribution of twin. It is to be noted that both FC and RCP gave reasonable fits for other (as in Table 2) major texture components with the ‘best-fit’ (as in Table 3) slip-twin ratios, the match for relaxed control lath (RCL) was, however, less impressive.

3.2. Microstructural Developments

Figure 5 shows optical structures (few selective ones) of cold rolled samples. The first interesting feature of Fig. 5(a), was the apparent subdivision or splitting of the grains.17–22,25–29) Often the boundaries creating such splitting (as viewed under crossed polarizer) did exist as ‘packs’, see Fig. 5. Such packs, however, always did not follow the classical angles of fcc strain localizations (generalized as Cu type and Brass type shear bands20,22)). At the higher reduction or in case of type 304, the grain splitting and the accompanying strain localizations were clearly more extensive, see Figs. 8(b) and 8(c). At the same reduction and for the same grade of stainless steel more strain localizations were observed in the cross rolled samples. TEM

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*2 Similar trends were observed for cross-rolling as well.
based local orientation measurements were used for more in-depth characterization of such strain localizations.

In both grades of stainless steel, the substructure evolved from the so-called random dislocation structure or Taylor lattice to the strain localizations. As shown in Fig. 6, the first visible strain localizations were in the form of first-generation or non-crystallographic micro-bands (MBs) and dense dislocation walls (DDWs). These appeared at reductions as low as 10% at approximately 45° with RD, and as their traces did not fall within 5° of the closed packed planes, they were generalized as first generation or non-crystallographic. Relatively large misorientation developments were always associated with such strain localizations, see Fig. 6. Increased reduction increased the relative presence and also the misorientation developments across the strain localizations (see Fig. 7) — they remained mostly first generation in type 316L (second generation MBs appeared at and above 60% reduction, their relative presence being less than the first generation MBs), while in type 304 the so-called second generation or non-crystallographic MBs appeared at 40% reduction and were more extensive (than first generation MBs) at and above 60% reduction in thickness. As shown in Fig. 7, the MBs were the major source of misorientation development and accompanying grain splitting.

The other type of strain localization, as in Fig. 8, was the twin lamellar structure (TLS). At times, but not always, the presence of martensitic phase was associated with the TLS. The TLS was also associated with large misorientation (naturally, as a result of twin boundaries) and significant grain splitting. TLS was more in type 304 and in respective cross-rolled samples.
The other interesting, though negative, result of microtexture studies was an apparent inability to link substructural developments with Taylor factor ($M^{26}$) or textural softening ($dM/da^{48}$). As shown in Table 4, large differences in $M$ and especially in $dM/da$ was observed between the ideal texture components (as given in the table for only ideal $\beta$-fibre components, under unidirectional and cross rolling). The differences in $dM/da$ also varied significantly with different twin contributions. Our microtexture measurements, however, did not bring out any consistent trend of misorientation development with $M$ or $dM/da$, considering any unique slip-twin contribution. For example, as given in Table 4, without any twin contribution the $S$ component is expected$^{26,48}$ to have more misorientation development under unidirectional rolling than under cross rolling. Any such trend was not observed in misorientation development.$^3$ The different ideal texture components did not have clear differences in their respective dislocation substructures—especially in terms of misorientation developments at higher reductions.

### 3.3. Strain Induced Martensite Formation

The other interesting feature in microstructure development is the strain induced martensite formation.$^{35-42}$ The VSM proved to be an effective tool in quantifying the relative amounts, even in type 316L, see Fig. 9 and Table 5. Using standard formulation$^{22}$ and assuming a direct proportionality (irrespective of the strain path and the type of stainless steel) of martensite percentage and saturation magnetization, the respective martensite percentages were estimated and are presented in Table 6. As shown in Tables 5 and 6, the strain induced martensite formation was clearly more in type 304 and more in both grades under cross-rolling.

Table 7 summarizes the results of hardness developments in both types of stainless steels. As shown in the table, type 316L had relatively sluggish hardness development as compared to type 304—respective slopes (as estimated for that matter, any consistent trend for any unique combination(s) of slip and twin.)
Fig. 6. (a) TEM micrograph of 20% unidirectionally rolled type 316L austenitic stainless steel. Local orientation measurements were taken at the marked points. Visible micro-bands (MBs) and dense dislocation walls (DDWs) were first generation or non-crystalligraphic\textsuperscript{17–19,26} as their traces did not fall within 5° of the closed packed plane. (b) (111) pole figure representing the local orientation measurements, as in figure (a). (c) Point to point misorientation (estimated from the points shown in figure (a)). In absence of strain localizations (MBs or DDWs) the misorientation development did not exceed 2°, while across the strain localizations misorientation was exceeding 5°.

Fig. 7. (a) TEM micrograph of 60% unidirectionally rolled type 316L austenitic stainless steel. Local orientation measurements were taken at the marked points. (b) (111) pole figure representing the local orientation measurements, as in figure (a). (c) Point to point misorientation (estimated from the points shown in figure (a)). Though measurements were taken across a suspected pre-deformation grain boundary (marked by arrowheads, see figure (a)), MBs (often first-generation, but second generation MBs were also observed at this stage) created large misorientation changes and accompanying grain splittings.
mated by best-fit straight lines) were 3.24 and 3.88, while the relative fits were 0.94 and 0.99. In case of type 316L the relative fit was less as the hardness reached a relative saturation at and above 60% reduction, while in type 304 a monotonic hardness increase (with increasing reduction) was noted.

4. Discussion

The developments of texture, microstructure, strain induced martensite and micro hardness has been studied during cold rolling (unidirectional and cross rolling) of types 304 and 316L stainless steels. As given in Table 2, the important difference in development of texture between types 304 and 316L were differences in changes of Brass (\(\{011\}\)/\(\{211\}\)) and Copper (\(\{112\}\)/\(\{111\}\)) and S (\(\{231\}\)/\(\{346\}\)). In type 304 the increase in Brass was significant, while in type 316L Copper and S had more significant increase, see Table 2(b), irrespective of unidirectional or cross-rolling. Texture simulations by Taylor type deformation texture models and different CRSS values for slip and twin\(^{12-16,47-49}\) showed that for unidirectional rolling, increase in twin activity leads to increase in Brass component along with a drop in Cu and S components. Relative contributions of twins were able to explain the basic difference of

Table 4. Estimated values of Taylor factor (\(M^2\)) and textural softening (\(dM/d\epsilon\)).\(^{48}\) The values were calculated for respective ideal texture components using only shear and full constraint (FC) Taylor model. For \(dM/d\epsilon\), a small strain of 0.05 was considered. It is to be noted that very large variations in respective \(dM/d\epsilon\) were observed with different twin contributions, effect of relative twin contribution often exceeding effects of ideal orientation.

| Strain Path                  | Copper \(\{112\}\)/\(\{211\}\) | S \(\{231\}\)/\(\{346\}\) | Brass \(\{011\}\)/\(\{211\}\) |
|-----------------------------|---------------------------------|--------------------------|-----------------------------|
|                             | \(M\) \(dM/d\epsilon\)        | \(M\) \(dM/d\epsilon\)  | \(M\) \(dM/d\epsilon\)    |
| Unidirectional Rolling      | 3.674 0.083                    | 3.369 -0.628             | 3.265 0.001                |
| Cross Rolling               | 3.0655 -2.443                  | 3.138 0.814              | 3.674 0                   |

Table 5. Saturation magnetization values at different reductions, as measured from the respective hysteresis loops (as in Fig. 9) of VSM measurements. The values are given for both types 316L and 304 under unidirectional and under cross-rolling.

| Percentage of reduction | Magnetic saturation \(4\)IM, (emu/gm) in Type 316L | Magnetic saturation \(4\)IM, (emu/gm) in Type 304 |
|-------------------------|---------------------------------------------------|--------------------------------------------------|
|                         | Unidirectional | Cross-Rolled | Unidirectional | Cross-Rolled |
| 0%                      | 0            | 0            | 1.25           | 1.25         |
| 20%                     | 0.25         | 0.25         | 2.5            | 2.5          |
| 40%                     | 0.26         | 0.34         | 5.5            | 9.0          |
| 60%                     | 0.27         | 0.6          | 9.5            | 12.5         |
| 80%                     | 0.95         | 1.1          | 12.5           | 20.0         |

Table 6. Percentage martensite was estimated using standard formulation and direct XRD measurements\(^{42}\) in 80% deformed type 304 stainless steel. The respective saturation magnetization values (for lower martensite percentages, and hence not amenable to XRD estimations), as in Table 5, were then converted to martensite percentages, assuming direct proportionality (irrespective of the strain path and the type of stainless steel) of martensite percentage and saturation magnetization.

| Percentage of reduction | Percentage Martensite in Type 316L | Percentage Martensite in Type 304 |
|-------------------------|-----------------------------------|-----------------------------------|
|                         | Unidirectional | Cross-Rolled | Unidirectional | Cross-Rolled |
| 0%                      | 0            | 0            | 2.14           | 2.14         |
| 20%                     | 0.42         | 0.42         | 4.28           | 4.8          |
| 40%                     | 0.44         | 0.58         | 9.43           | 15.43        |
| 60%                     | 0.46         | 1.02         | 16.29          | 21.43        |
| 80%                     | 1.62         | 1.88         | 21.43          | 34.3         |

Fig. 8. 60% unidirectionally rolled type 316L austenitic stainless steel. Twin lamellar structure is marked with arrowheads.

Fig. 9. VSM plots for type 316L cross-rolled samples—magnetization (\(M\) in emu/gm) vs. applied magnetic field (\(H\) in Oe). The respective reduction percentages (40 to 80%) are marked. Saturation magnetization values for all sample (both types 316L and 304, under unidirectional and under cross-rolling) are listed in Table 5.

Fig. 10. VSM plots for type 316L cross-rolled samples—magnetization (\(M\) in emu/gm) vs. applied magnetic field (\(H\) in Oe). The respective reduction percentages (40 to 80%) are marked. Saturation magnetization values for all sample (both types 316L and 304, under unidirectional and under cross-rolling) are listed in Table 5.
cold rolled texture developments in types 316L and 304 (as given in Table 2).

Microstructural studies showed (Fig. 5) the apparent subdivision or splitting of the grains during cold rolling. The boundaries creating such splitting existed as ‘packs’. At the higher reduction and specifically in case of type 304, the grain splitting and the accompanying strain localizations were clearly more extensive (as shown in Figs. 5(b) and 5(c)). At the same reduction and for the same grade of stainless steel more strain localizations were observed in the cross rolled samples.

Detailed studies in TEM showed that a reduced dislocation recoverability is expected to create,25) based on the so-called Dillamore’s22) plastic instability criteria, more strain localizations. This was observed even in the optical images, see Figs. 5(b) and 5(c). The initial strain localizations were identified (in both types 316L and in 304) as the so-called first generation MBs and DDWs, see Fig. 6, responsible for noticeable misorientation changes in the deformed grains. Second generation MBs and TLS were observed at the subsequent stages of deformation, see Figs. 7 and 8. These, especially the TLS, were more in type 304 and were also more in cross-rolled samples of both grades. This statement is largely quantitative*4 based on observations, both optical and TEM, on the respective samples. A similar pattern, as that of TLS, was followed for strain induced martensite formation.

The total number of strain localizations, as estimated based on optical images, was more in type 304, though no noticeable difference was observed between unidirectional and cross-rolled samples of either of the grades. This observation, coupled with observations of strain induced martensite formation (as in Tables 5 and 6), clearly indicated that differences in relative hardening (as in Table 7) was created by the relative presence of strain localizations, irrespective of their possible nature. The distinct differences in relative hardening exist in the frame-work of stacking fault energy differences and associated effects on dislocation recoverability.17–19) The differences in micro hardness during cold rolling (as shown in Table 7) cannot be explained from strain induced martensite formation. Though the strain induced martensite formation was more in type 304, it was also significantly more in cross-rolled samples (see Tables 5 and 6)—however hardness of both unidirectionally rolled and cross rolled material were almost identical (see Table 7).

Perhaps the most significant, albeit negative, observation was the apparent inability to link $M$ or $dM/d\varepsilon$ (at any unique slip-twin combination(s) in either of the grades) with misorientation development, as discussed in Sec. 3.2. In typical fcc50–52) and bcc26–48) material without any twinning component, such ‘linking’ is common and too distinct to miss in ideal texture components with extreme differences in $M$ or $dM/d\varepsilon$. This was not the case in the present study. Substructure development, especially the development of misorientation, appeared independent of the orientation (or $M$ or $dM/d\varepsilon$) of the deformed grain85). One possible explanation is that the presence of twinning and the TLS (also responsible for large orientation changes) is not fully accounted for by the relative trends in $M$ or $dM/d\varepsilon$. Such an explanation, though far from being definitive, appears reasonable in view of the present experimental observations. It is interesting to note that respective recrystallization textures17–19) were nearly identical as that of the parent deformation textures. This is possible only in case of an apparent absence of preferred nucleation and/or selective growth/micro-growth selection,25) a possibility which may arise from an apparent ‘orientation independent’ substructural development. Such a hypothesis is far-fetched and definitively speculative at this stage, but the ‘negative’ observation of apparently ‘orientation independent’ substructural development in materials with strong twinning contribution remains a clear possibility.

5. Summary

• The significant difference in cold rolling texture development between types 304 and 316L austenitic stainless steels was in the relative increase of Brass \((\{111\}\{211\})\) against Copper \((\{112\}\{111\})\) and S \((\{231\}\{346\})\) components. In type 316L increases in Copper and S were significant, while in type 304 increase in Brass was dominant. Such differences could be accounted in Taylor type deformation texture simulations considering increasing contributions of twinning—for example the respective ‘best-fits’ (in terms of matching the relative increases in Brass against Copper and S) were obtained by full constraint Taylor model respectively with 1 : 10 and 1 : 100 slip : twin activities in types 316L and 304.

• The contribution from strain induced martensite increased monotonically with increasing reduction, as estimated by VSM. It was significantly more in type 304 and was also more in cross-rolled samples (for the respective grades).

• The most interesting feature of the substructural developments was the strain localizations. These were first generation MBs and DDWs at the early deformation stages, while second generation MBs and TLS (twin lamellar structure) appeared at the latter stages. The TLS was more

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*4) Quantifying such an observation is difficult in optical structure, while a pure TEM based statistics is inherently weak.

*5) This cannot be explained from limited TEM statistics, as similar studies in bcc26–48) and in fcc50–52) systems without twinning could bring out major differences in substructures linked to the orientation aspect.
in type 304 and also more in cross-rolled samples of both grades. Large misorientation developments (inside the deformed grains) were always across such strain localizations.

- We failed to observe any links between respective Taylor factor ($M$) and/or textural softening ($dM/de$) and the substructural development, especially in terms of misorientation development. This is in clear contradiction to the existing knowledge in common fcc and bcc alloys and/or textural softening ($dM/de$) substructural development in materials with strong twinning contributions.

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