Journal of Ultrafine Grained and Nanostructured Materials
https://jufgnsm.ut.ac.ir
Vol. 57, No.2, December 2024, pp. 112-119
Print ISSN: 2423-6845 Online ISSN: 2423-6837
DOI: 10.22059/jufgnsm.2024.02.01

Grain refinement of austenitic stainless steels by cross rolling and annealing treatment: A review

Sara Mohammadzehi, Hamed Mirzadeh*
School of Metallurgy and Materials Engineering, College of Engineering, University of Tehran, Tehran, Iran.

Recieved: 6 July 2024; Accepted: 9 November 2024

**Corresponding author, E-mail:* hmirzadeh@ut.ac.ir

ABSTRACT

The effects of cross rolling and annealing treatment, as an advanced thermomechanical processing route, on the microstructure and grain refinement of metastable austenitic stainless steels were overviewed. It was summarized that cross rolling promotes the formation of intersecting shear bands and leads to higher dislocation density, which are favorable for the formation of strain-induced α′-martensite. Moreover, contrary to unidirectional rolling, cross rolling retains the equiaxed morphology of grains. It was demonstrated and formulated that cross rolling and reversion/recrystallization annealing treatment leads to more intense grain refinement compared to the unidirectional rolling and annealing route, which is quite important for grain refinement of more stable grades. Future prospects include investigating the effects of special alloying elements, initial grain size, and deformation variables on the cross rolled microstructure, analyzing the kinetics parameters of the deformation-induced martensitic transformation during cross rolling, and characterizing the transformation-induced plasticity (TRIP) effect for the grain refined austenitic stainless steels and high-entropy alloys by cross rolling and annealing treatment.

Keywords: *Metastable stainless steels; Unidirectional rolling; Cross rolling; Annealing heat treatment; Grain refinement.*

1. Introduction

Austenitic stainless steels have been used in a wide range of industries due to their excellent corrosion resistance, mechanical behavior, biocompatibility, and processability [1-3]. However, their fundamental disadvantage is relatively low yield stress of these steels [4,5]. In this regard, strengthening by cold rolling is commonly utilized, for which the formation of strain-induced α′-martensite with body-centered cubic (BCC) crystal structure in metastable grades with a low stacking fault energy (SFE) [6,7], work-hardening of the retained austenite [8,9], and grain refinement during subsequent reversion/recrystallization annealing treatment [10-12] are responsible for the remarkable strengthening effect.

Metastable austenitic stainless steels have a martensite start temperature (M_s) of less than 0 °C. However, the temperature below which plastic deformation can trigger the martensitic transformation (M_d) is typically higher than room temperature. As a result, in these alloys, α′-martensite might form during cold deformation at temperatures close to ambient temperature, which is caused by the creation of energetically favorable nucleation sites, such as shear band intersections [13,14]. The formation of the ε-martensite with hexagonal close-packed (HCP) crystal structure might also happen at low strains, which are also among the nucleation sites of α′-martensite. Strain-induced martensitic transformation depends on many factors such as deformation temperature [15,16], chemical composition [17-19], grain size [20], and deformation mode [21,22].

Cold rolling can be implemented in a variety of ways. For instance, besides unidirectional rolling (UR), the multi-step cross rolling (CR) is applied by shifting the rolling direction (RD) by 90° about normal direction (ND) after each pass [23,24]. The cold rolling route has a significant impact on the microstructure, phase transformation, and mechanical properties of metastable austenitic stainless steels. Furthermore, the as-deformed state affects the microstructural evolution during reversion/recrystallization annealing, affecting the mechanical behavior [25,26].

Accordingly, the present review paper is dedicated to summarizing the recent progress in the effects of cross rolling on the microstructure of metastable austenitic stainless steels after both cold rolling and subsequent annealing treatment, which can shed light on the application of cross rolling for the processing of high-performance austenitic stainless steels in future works.

2. Strain-induced martensitic transformation

Cold rolling route has a significant impact on the microstructure and phase transformation of metastable austenitic stainless steels. The effect of UR and CR routes on the formation of α′-martensite in a number of austenitic stainless steels is depicted in Figure 1a [24,27,28]. It can be observed that the CR route promotes the formation of α′-martensite for each class of austenitic stainless steels, where an example of the appearance of α′-martensite peak for the cross rolled AISI 316L stainless steel is depicted in Figure 1b. This is linked to the impact of cross rolling on the deformed microstructure. Sun et al. [23] noticed parallel shear bands within austenite grains at ~20% reduction in thickness for the unidirectionally rolled AISI 304 stainless steel; while for the cross-rolled samples, multiple intersecting shear bands developed after ~20% reduction in thickness due to the activation of additional slip systems. Mohammadzehi et al. [24] validated this effect for AISI 316 stainless steel, as illustrated in the electron backscattered diffraction (EBSD) maps of Figure 2. For the AISI 304 stainless steel, Jiang et al. [29] found that cross rolling promotes multiple twinning, increases nanotwins-intersections, and leads to higher dislocation density in the austenite phase, providing preferred nucleation sites for α′-martensite. The positive effect of cross rolling for increasing the dislocation density has been depicted in Figure 2 [24]. Accordingly, cross rolling promotes α′-martensite formation and increases the dislocation density of retained austenite, which is considered favorable for more stable grades such as 316-type stainless steel.

3. Evolution of grain shape during rolling

Due to the alteration of rolling direction by 90° during multi-pass deformation, the CR route essentially retains the equiaxed status of the microstructure. However, the UR route produced pancaked grains, the degree of which increases by increasing the reduction in thickness. As schematically shown in Figure 3, this effect can be seen on the RD-TD (transverse direction) plane of rolled samples; while on the RD-ND plane, the elongation of grains is similar for both routes. In

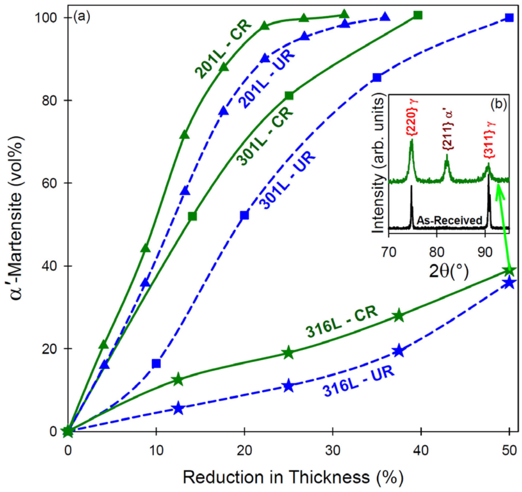

Fig. 1- (a) α′-martensite formation during room-temperature rolling (data taken from [24,27,28]) and (b) XRD patterns of AISI 316L stainless steel for the as-received (0% rolling) and 50% cross rolled conditions [24].

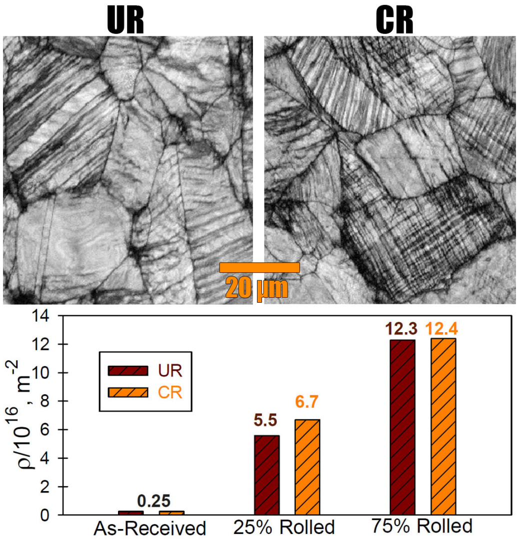

Fig. 2- EBSD contrast maps of 25% rolled AISI 316L stainless steel and summary of dislocation densities [24].

fact, the UR route leads to continuous pancaking of grains; while in the CR route, the pancaked grain in a given step of the multi-pass rolling attains its equiaxed morphology during the subsequent step with rolling direction altered by 90°. It will be emphasized later that the CR route decreases the directionality of properties and anisotropy due to its effects on the microstructure. In this regard, the retention of the equiaxed morphology along both RD and TD is an important effect.

4. Mechanical properties of cold rolled sheets

Cold rolling can improve the yield strength via formation of strain-induced α′-martensite as well as the work-hardening of the retained austenite [30-32]. The achieved hardening during cross rolling is more pronounced compared to the UR route, which is related to the activation of more slip/twinning systems, increasing the dislocation density, and promotion of the formation of strain-induced martensite [24,29]. Recently, the asymmetric cross rolling at room temperature has successfully been used to improve the mechanical properties of AISI 304 stainless steel [33], combining the advantages of asymmetric rolling and cross rolling to boost the stored energy.

Another important issue is the pronounced anisotropy and directionality of mechanical properties for the parts experienced the UR route along the RD (0°), TD (90°), and 45° in the RD-TD plane. This anisotropy can be decreased by cross rolling [24,34]. The Lankford coefficient (plastic anisotropy ratio, R) can be used for the evaluation of anisotropy. It is defined as the ratio of the width to thickness strains and it is measured at 20% elongation in tensile testing for samples prepared with tensile axis along 0° (R_0), 45° (R_{45}), and 90° (R_{90}) to the rolling direction [34-36]. However, more simple methods can also be proposed for the evaluation of anisotropy in the cold rolled sheets. In this regard, Equation 1, as an alternative method based on the obtained values of ultimate tensile strength (UTS), has been suggested, in which, UTS_{avg} can be obtained by Equation 2 [24]:

$$\mathrm{DEV} = \sqrt{\{(\mathrm{UTS}_0-\mathrm{UTS}_{avg})^2+(\mathrm{UTS}_{45}-\mathrm{UTS}_{avg})^2+(\mathrm{UTS}_{90}-\mathrm{UTS}_{avg})^2\}/3} \quad (1)$$

$$\mathrm{UTS}_{avg} = \{\mathrm{UTS}_0+\mathrm{UTS}_{45}+\mathrm{UTS}_{90}\}/3 \quad (2)$$

A lower DEV value indicates a decrease in anisotropy, which has been utilized for the AISI 316L stainless steel to investigate the decrease in the directionality of strength for the CR route compared to the UR route. Simple methods like this should be further evaluated in other cases [37-39].

5. Grain refinement by annealing after CR

If cold deformed austenitic stainless steels with nano/ultrafine martensite induced by micro shear bands are subjected to the annealing treatment at elevated temperatures, the deformed microstructure is replaced by a fine-grained austenitic one via reversion of α′-martensite to austenite (sometimes ultrafine grained austenite) and recrystallization of the retained austenite [40-43]. Then, if the annealing process continues, the reversed and recrystallized austenite will be coarsened, for which the grain growth of the reversed austenite also happens during recrystallization of the retained austenite [44-47].

Accordingly, a higher amount of martensite in the cold rolling step is recommended before applying the annealing treatment. Therefore, cold rolling at lower temperatures and with higher applied strain is usually required. Moreover, adjusting the chemical composition to reduce SFE can be used for this purpose. Furthermore, promoting the

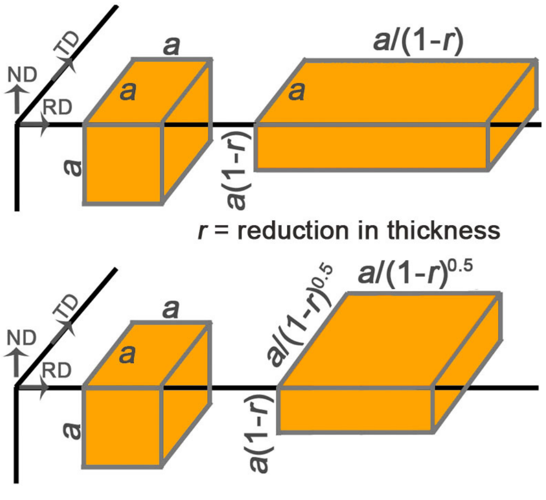

Fig. 3- Schematics for the effects of rolling routes: UR (top) and CR (bottom) [24].

formation of α′-martensite by methods such as the CR route can be implemented, which is quite useful for the more stable grades such as AISI 316L stainless steel, as shown by Nezakat et al. [25] and recently by Mohammadzehi et al. [26].

In this regards, more activated slip systems, higher dislocation density, and greater α′-martensite volume fraction in the deformed state by the CR route lead to the finer grain size after annealing treatment. The grain sizes of AISI 316L stainless steel after cold rolling and annealing based on the report of Nezakat et al. [25] are summarized in Figure 4a, clearly showing this effect. Recently, this subject has systematically been investigated by Mohammadzehi et al. [26], for which the results are summarized in Figure 4b. It can be seen that annealing of cold rolled samples has led to a significant grain refinement. Moreover, finer grain sizes have been obtained by increasing the reduction in thickness in rolling. Furthermore, grain refinement in the CR route is more pronounced compared to the UR route.

After cold rolling and annealing, the grain size can be represented by Equation 3:

$$D = AD_0^l \varepsilon_{\mathrm{def}}^{-m} Z_{\mathrm{def}}^{-n} \tag{3}$$

where Z_{def} is the Zener-Hollomon parameter during deformation, D_0 is the initial grain size, $\varepsilon_{\mathrm{def}}$ is the effective strain, and A, l, m, and n are constants. By consideration of $\varepsilon_{\mathrm{def}}$ as the main variable, this equation is simplified as Equation 4:

$$D = B\varepsilon_{\mathrm{def}}^{-m} \tag{4}$$

where B is a constant. Therefore, m and ln$B$ can be obtained based on the slope and intercept of the straight line fitted to the plot of ln$D$ versus ln$\varepsilon_{\mathrm{def}}$, as shown inlay in Figure 4b [26]. It can be seen that while the m values are close to each other, ln$B$ values are quite different. Accordingly, the regression analysis was applied based on the average m value of 0.56, leading to the B values of 3.22 and 2.48 for the UR and CR routes, respectively. Therefore, the B value for the CR route is smaller, indicating a better grain refining efficiency.

The cold rolling and annealing route is a viable method for grain refinement of austenitic stainless steels [48-52], which can also be applied to other high-performance alloys such as low carbon steels [53,54], dual phase (DP) steels [55-57], and especially high-entropy alloys (HEAs) [18,58,59]. In fact, metastable austenitic stainless steels and HEAs can benefit from the formation of strain-induced martensite during deformation, which can be used for a remarkable grain refinement during subsequent annealing treatment, leading to the improved yield stress (YS), as represented by the

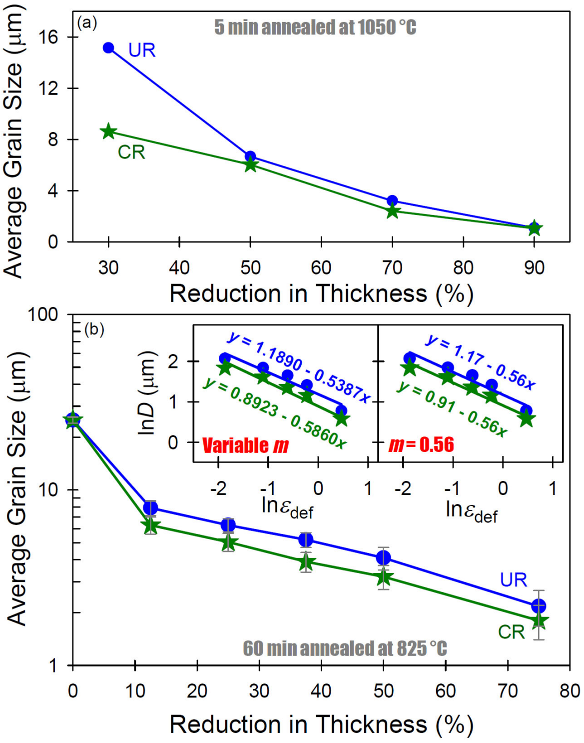

Fig. 4- Grain refinement of AISI 316L stainless steel by cold rolling and annealing (data from [25] and [26]).

Hall-Petch relationship of Equation 5 [60-64]:

$$YS = \sigma_0 + K_Y / \sqrt{D} \tag{5}$$

where σ_0 is the friction stress, describing the overall resistance of the crystal lattice to dislocation slip; while K_Y is the locking parameter or the Hall-Petch slope, denoting the relative hardening contribution of the grain boundaries. Accordingly, more intense grain refinement by the CR route is conducive to improve strength, as depicted in Figure 5 for the reduction in thickness of 75% followed by annealing at 825 °C for the AISI 316L stainless steel. It can be seen that grain refinement by cold rolling and annealing treatment has resulted in the improvement of yield and ultimate tensile strength, where these improvements are more pronounced for the CR route compared to the UR route.

6. Summary and future scope

In summary, grain refinement of austenitic stainless steels by cross rolling and annealing treatment was briefly reviewed in the present monograph. It was revealed that cross rolling promotes the formation of α′-martensite and leads to higher dislocation density in the retained austenite. Cross rolling promotes the formation of intersecting shear bands and also retains the equiaxed morphology of grains. It might improve the mechanical properties and can decrease the directionality of these properties. Cross rolling and annealing treatment leads to more intense grain refinement compared to the unidirectional rolling and annealing route, which is quite important for grain refinement of more stable grades such as AISI 316L stainless steel. This grain refinement depends on the applied strain during rolling, for which simple formulae can be used to predict the grain size.

The mechanical properties of austenite in austenitic stainless steels can be tailored by adding special alloying elements such as carbon and nitrogen. These elements also affect the stability of the austenite phase against the strain-induced martensitic transformation. In these cases, the implementation of the CR route might become advantageous. Moreover, investigating the effects of deformation parameters such as deformation temperature during cross rolling is quite interesting, because near room temperature, the strain-induced martensitic transformation becomes highly dependent on the small change in the temperature. Furthermore, since the initial austenite grain size might determine the nucleation sites of α′-martensite and its effect on the strain-induced martensitic transformation is complicated [19,65], investigating the effect of cross rolling in stainless steels with various initial grain sizes should be conducted in the future works.

The α′-martensite formation kinetics is an important matter. Olson and Cohen proposed a formula expressed as Equation 6 [66]:

$$f_{\alpha'} = 1 - \exp\{-\beta\{1 - \exp(-\alpha\varepsilon_{\text{def}})\}^n\} \tag{6}$$

where α is the rate of the shear band formation, β is the probability of nucleation at shear band intersections, and n is a constant (usually considered as 4.5) [66,67]. Besides the Olson-Cohen formula, other models developed by Guimaraes (based on Johnson-Mehl-Avrami-Kolmogorov analysis) [68], Shin [69], Ahmedabadi [70], and Tavares [71] can be analyzed and critically discussed. Moreover, very recently, a general Hill-based model has been

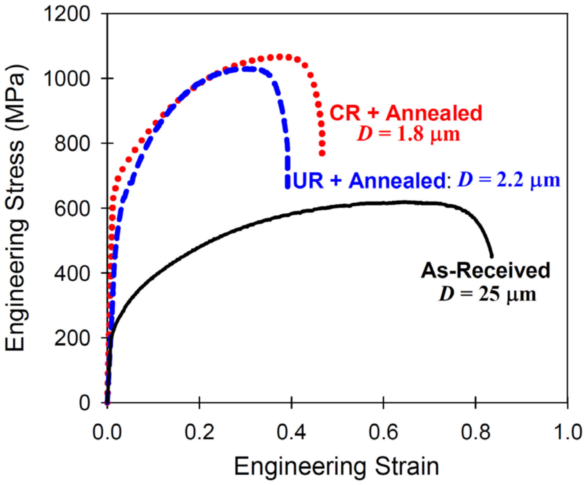

Fig. 5- Tensile stress-strain curves of AISI 316L stainless steel in the as-received condition as well as after 75% cold rolling based on the UR and CR routes and subsequent annealing at 825 °C [26].

formulated for TRIP-assisted steels and high-entropy alloys with promising outcomes [67], which should be applied in this case. Therefore, it is reasonable to expect that cross rolling might have an effect on the parameters of these models, which has been remained to be evaluated to elucidate useful conclusions.

The formation of α′-martensite during deformation can be used to improve the strength-ductility synergy of austenitic stainless steels and HEAs via the transformation-induced plasticity (TRIP) effect [72-74], which depends on the grain size. Accordingly, the more intense grain refinement by the cross rolling and annealing route might have a notable effect on the TRIP effect in the subsequent forming operation, which should be investigated in future works. This subject has recently been explored for austenitic stainless steels [26,75] and HEAs [76-78], resulting in promising outcomes. Moreover, the effect of cross rolling on the anisotropy of deformed sheets needs to be investigated by different approaches for various alloys.

Besides tensile testing, other innovative methods such as shear punch tensing (SPT) can also be applied to investigate the effects of cross rolling and annealing treatment on the mechanical properties of austenitic stainless steels. In fact, the shear mechanical properties of austenitic stainless steels have received less attention. A viable method for investigating the shear properties is SPT [79-82]. Recently, the applicability of SPT has been investigated in this regard, where by relating the ultimate shear stress (USS) to the ultimate tensile strength (UTS) via von Mises criterion, the finalized equation of Hardness = 2.95UTS has been obtained for cross rolled and annealed austenitic stainless steels, confirming the empirical formula of Hardness = 3UTS for steels [75].

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