On strengthening of ultrafine grained austenitic steels subjected to large strain deformation

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Abstract. Our current results on the structure-property relationship in ultrafine grained (UFG) austenitic steels, subjected to large strain deformation under conditions of cold to warm working are critically reviewed. Commonly, the grain size and dislocation density that evolve in UFG steels and alloys during large strain deformation depends sensitively on the processing conditions, i.e., temperature and strain rate. The grain size decreases, and the dislocation density increases with a decrease in deformation temperature. Therefore, a unique power-law relationship with a grain size exponent of \(-0.6\) holds between the deformation grain size and the dislocation density. A power-law function between the grain size and dislocation density is obtained for UFG steels even after annealing. Thus, the yield strength of UFG steels can be expressed by a modified Hall-Petch-type relationship, which includes the dislocation strengthening, by using the grain size or dislocation density as a unique variable.

1. Introduction
Structural steels and alloys are commonly processed by various thermo-mechanical treatments [1]. Depending on the processing conditions, the thermo-mechanical treatment can involve dynamic and post-dynamic recovery and/or recrystallization. Both recovery and recrystallization are intended for controlled microstructure evolution during deformation and heat treatment. The most important microstructural parameters affecting mechanical behavior and properties of structural metallic materials are the grain size and dislocation density. Effect of hot working conditions on dynamic recrystallization (DRX) and the dynamic grain size evolved after sufficiently large strains is fairly clarified [2-4]. The power law relationships between the grain size and flow stress or temperature-compensated strain rate have been revealed for various metals and alloys. Studying the dislocation density in DRX grains evolved during hot working is complicated by heterogeneous dislocation distribution in DRX microstructures [5], although an inverse proportional stress dependence of the substructural parameters has been suggested [6].

The revealed dependencies for the microstructural parameters and the deformation conditions imply a power law function between the grain size and the dislocation density evolved in DRX microstructures. The aim of the present work, therefore, is to clarify the structural relationships in DRX microstructures evolved during large strain warm deformation, and to elucidate the strengthening mechanisms aroused in DRX microstructures. In contrast to DRX microstructures, the structural/substructural relationships in ultrafine grained (UFG) microstructures developed by large strain cold deformation followed by an annealing have not been elaborated. Thus, another aim of this
study is to reveal a possible relationship between the grain size and dislocation density and their effect on the yield strength of UFG materials processed by severe plastic deformation and annealing.

2. Experimental
Two widely used austenitic stainless steels, 304L (0.05%C – 18.2%Cr – 8.8%Ni – 1.65%Mn – 0.43%Si – 0.05%P – 0.04%S) and 316L (0.04%C – 17.3%Cr – 10.7%Ni – 2%Mo – 1.7%Mn – 0.4%Si – 0.04%P – 0.05%S) were used in the present study as typical representatives of the classical DRX materials during hot working and susceptible to rapid grain refinement during severe plastic deformation. The steel samples were subjected to plate rolling at room temperature and a temperature of 573 K to a total strain of 3 and bar rolling at temperatures of 773-1173 K to a total true strain of 2. Then, the samples subjected to plate rolling were annealed at 873-1073 K. Structural investigations were carried out by using a JEM – 2100 transmission electron microscope (TEM) and a Nova Nanosem 450 scanning electron microscope, equipped with an orientation image microscopy (OIM) system based on an electron back-scattered diffraction pattern analyzer. The dislocation density was evaluated as the number of dislocations crossing an arbitrary selected area on at least six representative TEM images. Tensile tests were carried out with dog-bone specimens having a gauge length of 16 mm and a cross section of 3×1.5 mm² using an Instron 5882 universal testing machine.

Figure 1. Typical microstructures evolved in 304L stainless steel after warm rolling at 773 K (a) and 973 K (b) and those after cold rolling followed by annealing at 973 K (c) and 1073 K (d). Colours in (a, b) and (c, d) correspond to the rolling axis (RA) and the normal direction (ND), respectively.

3. Results and discussion
Typical microstructures that evolve in austenitic stainless steels during warm rolling or cold rolling followed by annealing are shown in figure 1. The elongated grains containing numerous dislocation subboundaries testify to continuous DRX as the mechanism of microstructure evolution during warm
working. The transverse grain size, measured along the normal direction, increases with an increase in rolling temperature. Correspondingly, the dislocation density in the grain interiors increases with a decrease in the rolling temperature (figure 2a). Both the grain size and the dislocation density are characterized by weakening temperature dependencies as the rolling temperature decreases. Such behavior leads to a power law relationship between the grain size and the dislocation density with a grain size exponent of -0.6 in the warm rolled steel samples, namely, $\rho = 18D^{-0.6}$ in figure 2b.

![Figure 2](image1.png)

**Figure 2.** Effect of the rolling temperature on the grain size and the dislocation density (a) and the relationship between the grain size and the dislocation density evolved by warm rolling (b).

![Figure 3](image2.png)

**Figure 3.** Effect of annealing for 30 min and 2 h on the grain size and the dislocation density (a) and the relationship between the grain size and the dislocation density (b) in steel samples subjected to rolling at room temperature (CR) and 573 K (WR).

Annealing of cold rolled steel samples results in the development of an almost equiaxed grain microstructure (figures 1c and 1d). An increase in the annealing temperature leads to a growth of the mean grain size, while the dislocation density decreases (figure 3a). Both the annealed grain size and the dislocation density within the grains can be expressed by power law functions of inverse temperature. Similar to the warm worked microstructures, the dislocation density in the cold rolled and annealed steel samples can be expressed by a unique power law function of the annealed grain size with an exponent of 2, i.e., $\rho = 0.61D^2$ in figure 3b. It can be concluded, therefore, that deformation microstructures that evolved at sufficiently large strains under conditions of warm working and
annealed microstructures developed in largely strained samples are characterized by unique relationships, which link the grain size and the dislocation density through power law functions.

The fine grained samples processed by warm rolling and cold rolling, followed by annealing, exhibit enhanced strength properties. The yield strength \( \sigma_{0.2} \) of structural steels depends on their mean grain size \( D \) and is commonly expressed by the Hall-Petch relationship \([7, 8]\). In the case of high dislocation density \( \rho \) in grain interiors, the modified Hall-Petch relationship is used for yield strength calculation taking into account substructural strengthening as follows \([9, 10]\).

\[
\sigma_{0.2} = \sigma_0 + kD^{0.5} + \alpha Gb\rho^{0.5}
\]  

Here \( \sigma_0 \) is the strength of the same material with an infinite grain size, \( k \) and \( \alpha \) are strengthening factors (\( \alpha \) involves Taylor factor), \( G \) is the shear modulus, and \( b \) is the Burgers vector. It is interesting to note that the yield strength can be expressed by using one microstructural/substructural parameter, i.e., either grain size or dislocation density, using equation (1), if there is a unique relationship between these parameters. Thus, the yield strength was calculated using equation (1), substituting the corresponding power law functions, \( \rho \sim D^\alpha \) indicated above, for the grain size and the dislocation density. Figure 4 illustrates a good correspondence between the experimentally measured values of the yield strength and those calculated using the grain size, \( \sigma(D) \), or the dislocation density \( \sigma(\rho) \) taking \( k = 300 \text{ MPa } \mu\text{m}^{-0.5} \) and \( \alpha = 0.5 \).

**Figure 4.** Relationship between the experimental and calculated yield strength for UFG stainless steels processed by warm rolling and annealing (a) or cold rolling and annealing (b).

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