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# Microstructure evolution in a 316L stainless steel subjected to multidirectional forging and unidirectional bar rolling

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**Abstract.** The formation of ultrafine-grained structures was studied in a 316L stainless steel during severe plastic deformation. The steel samples were processed up to a total amount of strain of 4 at ambient temperature using two different methods, i.e., multidirectional forging and unidirectional bar rolling. The large strain developed upon cold working resulted mechanical twinning and partial martensitic transformation. The latter was readily developed during multidirectional forging. After straining to the total amount of strain of 4, the austenite fractions comprised approximately- 0.45 as well as 0.15 in the rolled and forged samples, respectively. Both the multidirectional forging and bar rolling led to extensive grain refinement. The uniform microstructures consisting of austenite and ferrite crystallites with the transverse size of 60 nm and 30 nm were evolved at a total amount of strain of 4 in the rolled and forged samples, respectively. The grain refinement by severe plastic deformation was accompanied by an increase for the microhardness to 5380 and 4970 MPa for the forged and rolled samples, respectively.

**Keywords:** austenitic stainless steels, cold rolling, multidirectional forging, nanocrystalline structure.

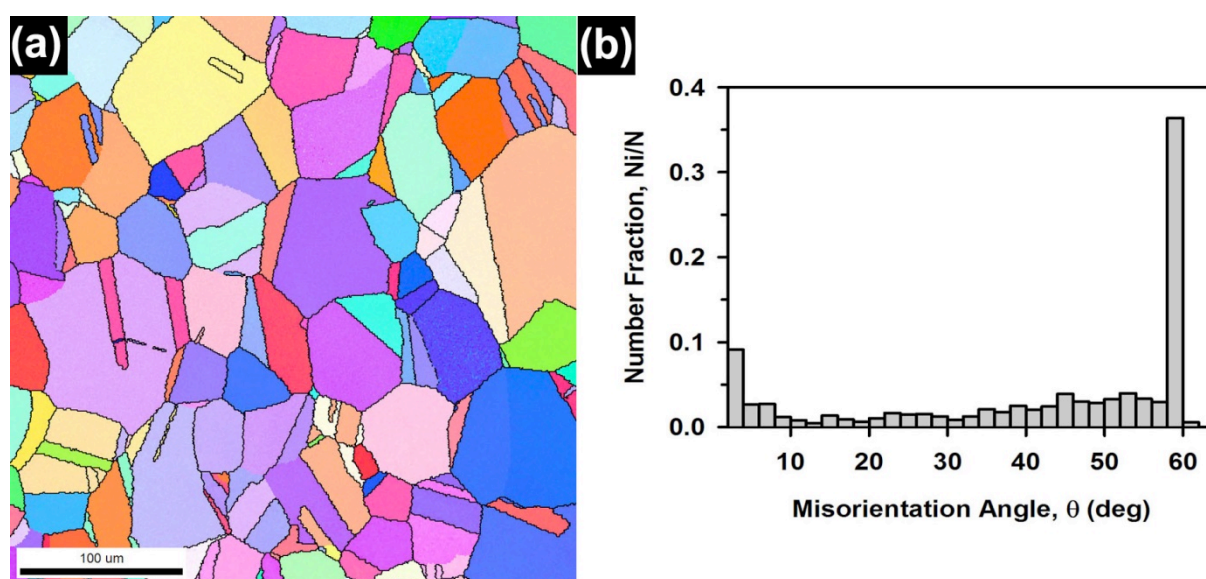
## 1. Introduction

Austenitic stainless steels are one of the most popular classes of structural materials because they possess a unique combination of mechanical, technological and functional properties [1, 2]. However, applications of austenitic stainless steels as structural materials are limited due to their relatively low yield strength [1, 3]. The yield strength of structural steels can be significantly improved by the formation of an ultrafine-grained structure upon severe plastic deformation. Up to date, a number of different methods using severe plastic deformation have been developed [4]. Among those, multiple forging and rolling are the simplest techniques, which do not require any specific equipment and can be utilized for processing of diverse metallic materials with sufficient ductility [5]. These techniques are particularly attractive for processing materials exhibiting grain refinement with rapid kinetics during cold working as for instance austenitic stainless steels. It has been shown that the deformation by cold working of austenitic stainless steels is accompanied by the development of mechanical twinning and martensitic transformation, which lead to significant grain refinement up to the formation of ultrafine-grained structures at relatively small true strains [6]. However, the effect of the processing method on the kinetics of microstructure evolution in such materials has not been studied in sufficient detail. The aim of the present work is to examine the effect of deformation technique on the mechanisms of microstructure evolution in a 316L stainless steel. The grain refinement in this steel was studied by the comparison of multidirectional forging with unidirectional bar rolling at ambient temperature. Particular attention is paid to examination of the kinetics of grain refinement to nanocrystalline level during severe plastic deformation.



## 2. Experimental Procedure

A 316-type austenitic stainless steel (0.04C, 17.3Cr, 10.7Ni, 1.7Mn, 0.4Si, 0.04P, 0.05S, 2Mo, 0.09V, 0.04Ti, 0.05Nb, 0.4 Cu, 0.19Co, all in mass%, and the balance Fe) was investigated. The steel was hot rolled at 700°C and then annealed at 1100°C for 10 min followed by air cooling to produce a uniform microstructure having a mean grain size of about 20  $\mu\text{m}$  and a large fraction of annealing twins of  $\sim 0.4$  (figure 1). The multidirectional forging (MDF) was carried out by multi-pass compressions introducing a strain of approximately 0.4 by each pass while the compression axis was varied by 90° from pass to pass (i.e., x to y to z, etc.) [7]. Samples for multidirectional forging were machined in rectangular shape with initial dimension of 10  $\times$  12.2  $\times$  15 mm<sup>3</sup>. The unidirectional cold rolling (CR) was carried out on square bars with initial cross section of 9.2  $\times$  9.2 mm<sup>2</sup>. The samples were forged/rolled to total true strains ( $\epsilon$ ) of about 0.4, 1.2, 2 and 4 at ambient temperature.

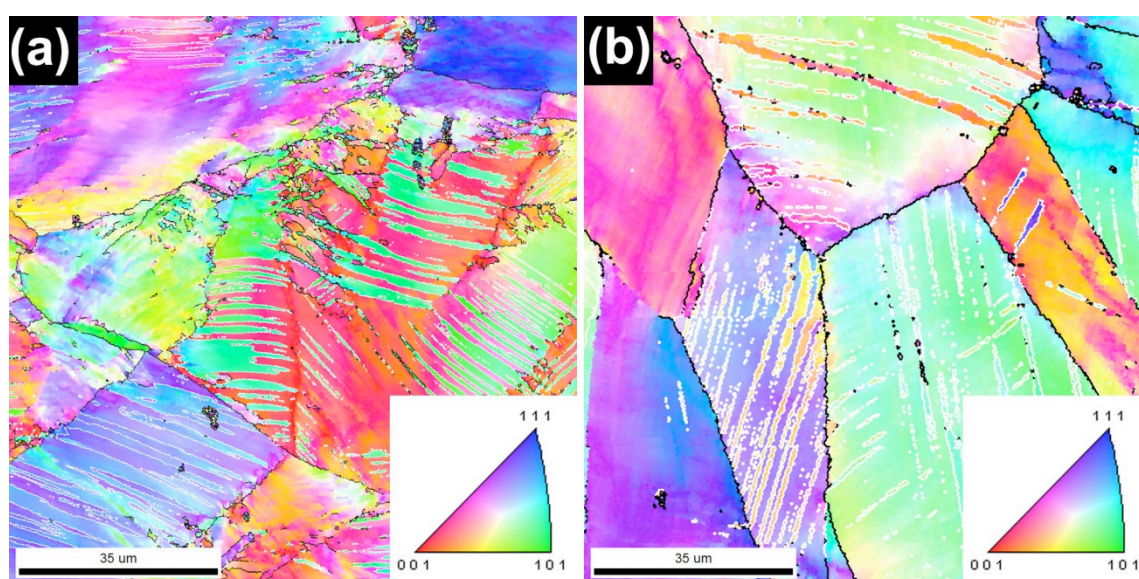


**Figure 1.** Representative OIM micrographs of the starting microstructure (a) and grain boundary misorientation distribution (b) of the 316L-type austenitic stainless steel.

The structural characterization was performed using a JEM-2100 transmission electron microscope (TEM) and a Nova Nanosem 450 scanning electron microscope incorporating an orientation imaging microscopy (OIM) system. Microstructure after MDF and CR was studied on the sample sections parallel to the final compression axis and the rolling axis, respectively. The grain/subgrain sizes were measured by a linear intercept method along the axis of the final compression pass for the forged samples, and crosswise with the rolling axis for the rolled samples. The dislocation density was estimated by counting individual dislocations revealed by conventional TEM in the grain/subgrain interiors. The hardness of forged/rolled samples was studied using Vickers hardness tests using a load of 3N. Misorientations between ultrafine-grains were analyzed by Kikuchi-lines in conventional TEM using convergent-beam electron diffraction (CBED) [8]. The volume fractions of the austenite were estimated by X-ray analysis, magnetic induction method and electron back scattered diffraction (EBSD) technique. The equilibrium volume fraction of the austenite was calculated with the software program ThermoCalc using a TCFE7 database at 20°C.

### 3. Results and Discussion

Microstructures developed in the 316L austenitic stainless steel at ambient temperature during MDF and CR by a strain of 0.4 are shown in figure 2. Relatively small amounts of strain (0.4) lead to an elongation of the original grains towards the flow direction of the deformation and the development of deformation twins. Deformation twinning starts to operate at an early stage of straining as a significant deformation mechanism. The number density of deformation twins in the forged samples is remarkably higher than that in the cold rolled samples (cf. figures 2a and 2b). The twin areas per unit volume are  $0.87 \mu\text{m}^{-3}$  and  $0.45 \mu\text{m}^{-3}$  in the forged and rolled samples, respectively. Therefore, the mechanical twinning results rapidly in the formation of subgrains at small strain. Applying further strain the lamellar microstructures are retained in the case of CR, while the MDF leads to evolution of granular structure (figure 3).

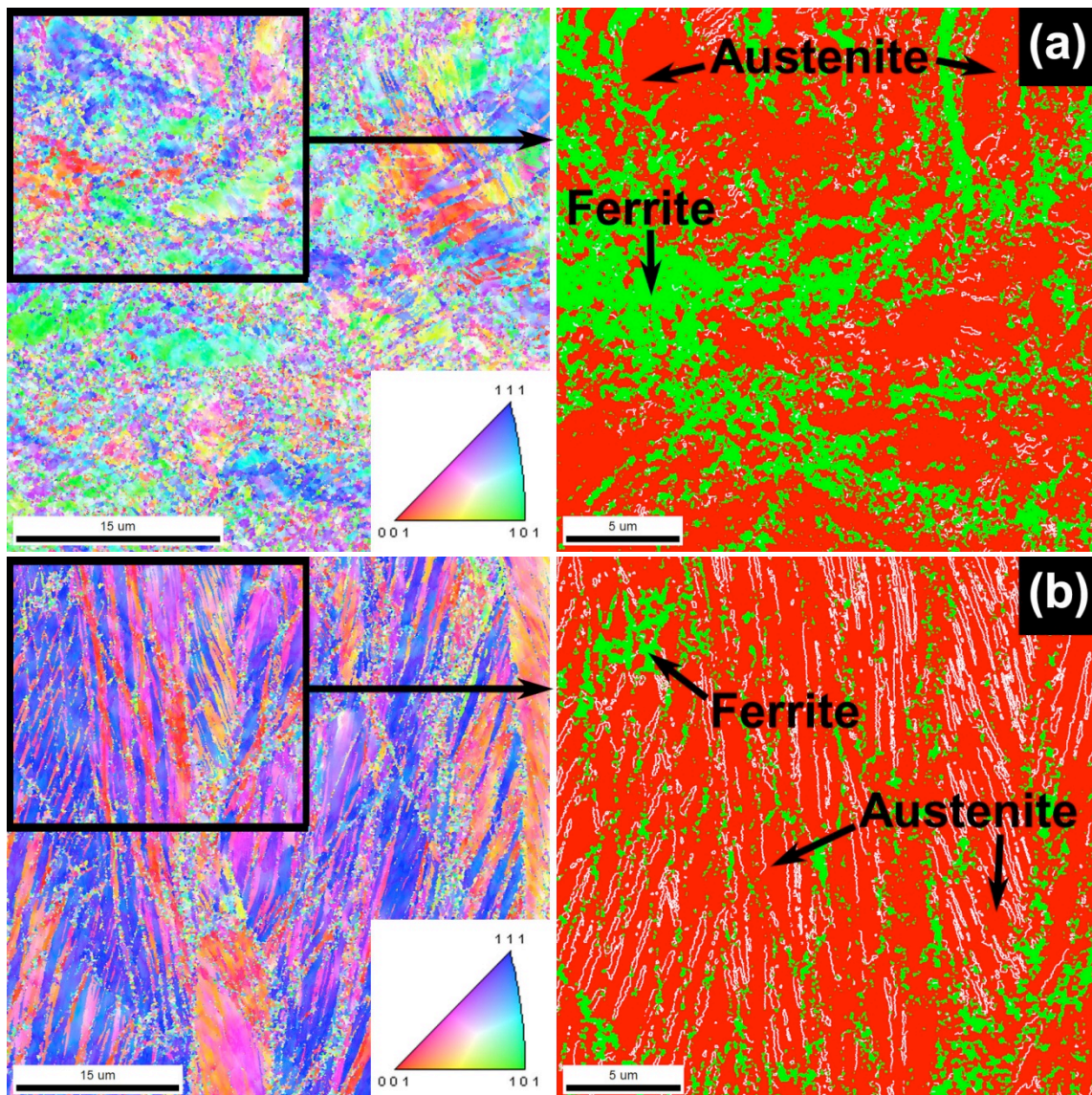


**Figure 2.** Representative OIM micrographs of the microstructure in the 316L austenitic stainless steel after forging (a) and rolling (b) to a strain of 0.4. The inverse pole figures in (a) and (b) are shown to indicate the forging axis and the rolling direction, respectively.  $\Sigma 3$  and high-angle grain boundaries are indicated by white and black lines, respectively.

At  $\varepsilon \sim 1.2$ , the appearance of strain-induced ferrite results from partial martensitic transformation (figure 3). Deformation martensite originates at the deformation twins and within regions of large strain gradients such as vicinities of grain boundaries and shear bands. In contrast to other studies on metastable austenitic stainless steels, in which the mechanical twins were considered as primary nucleation sites for deformation martensite, the present results suggest that the martensite readily develops at the shear bands. It should be noted that the forged samples are characterized by a higher number density and a larger width of the micro shear bands as compared with the rolled samples. Hence, the fraction of strain-induced ferrite increases faster during MDF than during CR.

Representative microstructures of a micro shear bands are shown in figure 4. Due to the martensitic transformation the microstructure of micro shear bands consists of alternating elongated ferrite and austenite grains with a crosswise size of about 60 nm. The orientation relationships between the strain-induced ferrite and the austenite matrix are close to those predicted by Kurdjumov-Sachs ( $42.85^\circ < 0.178, 0.968, 0.178 >$ ) and Nishiyama-Wasserman ( $45.98^\circ < 0.083, 0.201, 0.976 >$ ) orientation relationships [9]. For example, the

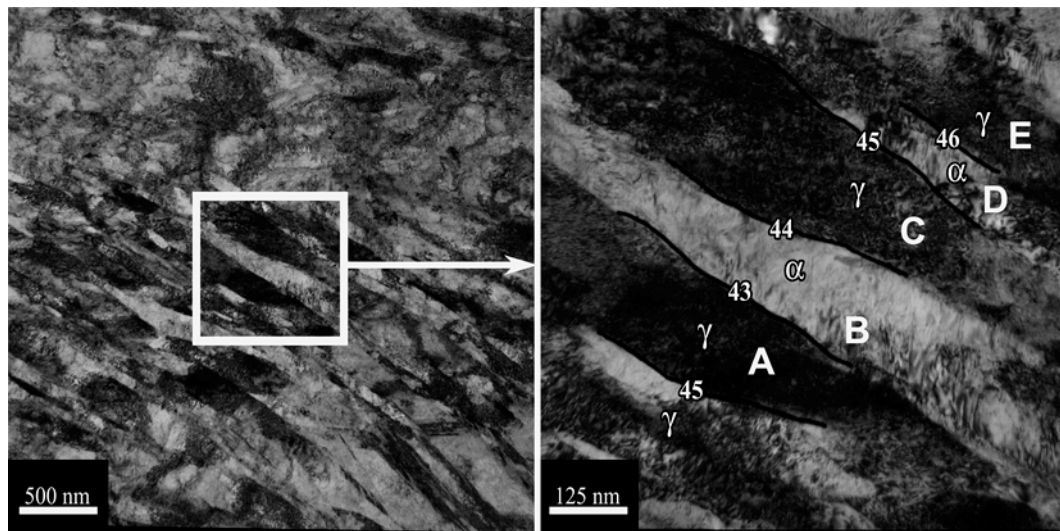
minimum misorientation angle-axis pairs are  $42.5^\circ \langle 0.138, -0.983, -0.122 \rangle$  between crystallites A and B and  $45.6^\circ \langle -0.064, 0.154, -0.986 \rangle$  between crystallites D and E in figure 4. The former is close to Kurdjumov-Sachs orientation relationship, whereas the latter is almost identical with Nishiyama-Wasserman.



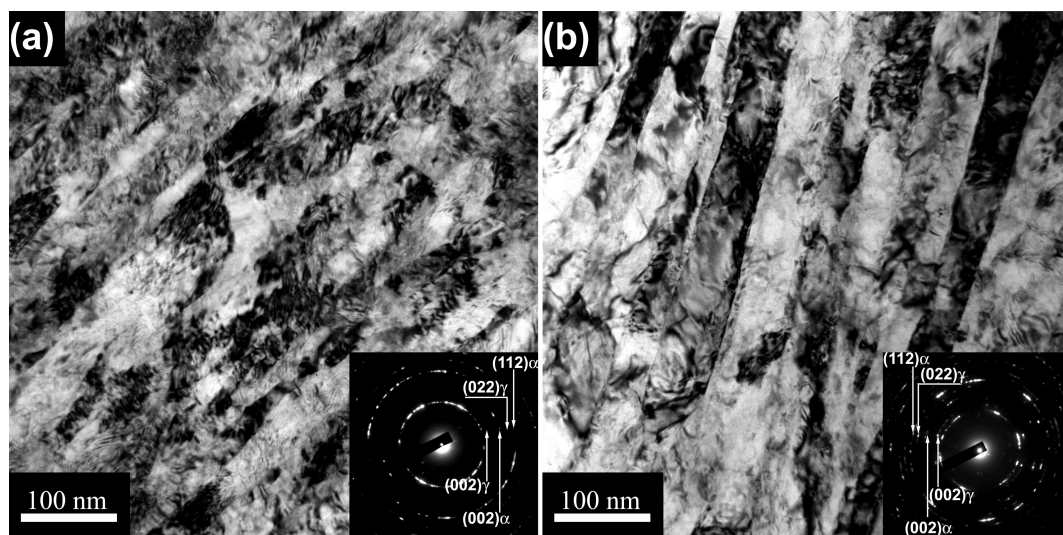
**Figure 3.** Representative OIM micrographs of the microstructure in 316L austenitic stainless steel after multidirectional forging (a) and unidirectional bar rolling (b) to a strain of 1.2. The inverse pole figures in (a) and (b) are shown to indicate the axis of the last compression pass and the rolling direction, respectively.  $\Sigma_3$  twin boundaries are indicated by white lines in the enlarged portions.

An increase in the total strain is accompanied by the progressive development of the martensitic transformation leading to the evolution of ultrafine-grained structures with high dislocation densities. Representative deformation microstructures developed at a total strain of 4 are shown in figure 5. MDF results in the formation of nanocrystallites with a crosswise size of about 30 nm (figure 5a). This microstructure is almost fully composed of ferrite

grains. On the other hand, the lamellar type microstructure with the crosswise grain size of about 60 nm was formed in the cold rolled samples (figure 5b). The kinetics of martensitic transformation during the CR is slower than the MDF. Therefore, the severely cold rolled microstructures consist of a mixture of highly elongated austenitic and ferritic grains.



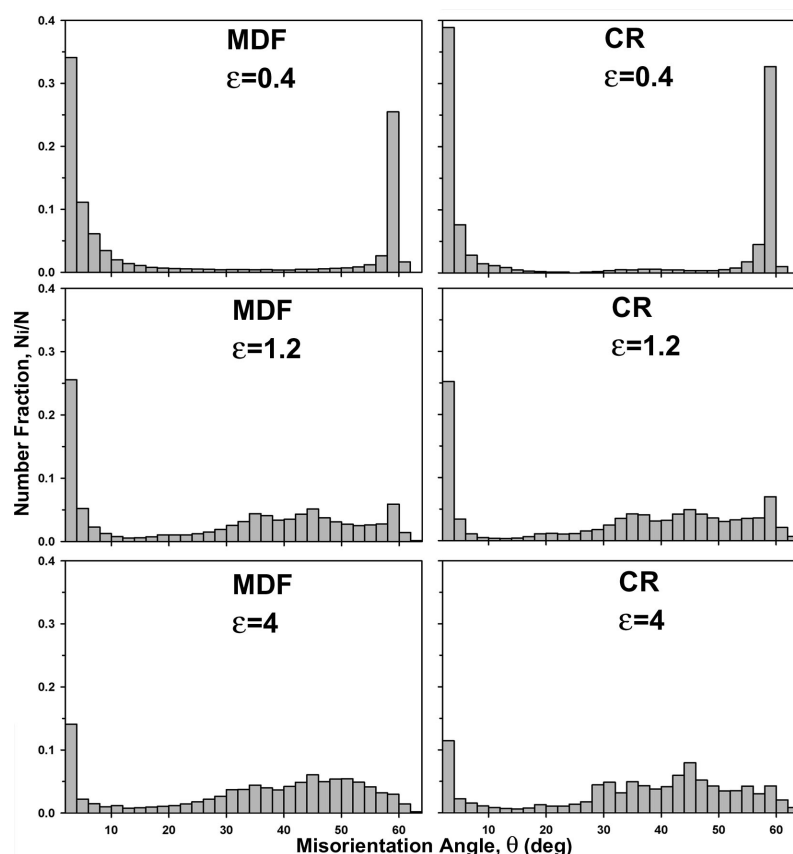
**Figure 4.** Representative deformation microstructure developed after bar rolling to a strain of 1.2. The numbers indicate the nominal misorientations angles between neighbouring cubic crystallites in degrees.



**Figure 5.** Representative deformation microstructures developed after multidirectional forging (a) and unidirectional bar rolling (b) to a strain of 4.

The changes of the deformation microstructures and the hardness increase with strain suggest a strong effect of the deformation method on the grain refinement (figures 6 and 7). Plastic deformation to  $\epsilon \sim 0.4$  provides a double increase in microhardness, which is the same for the two different techniques (figure 7) investigated. However, the microstructural evolution during MDF and CR is different. The grain boundary misorientation distributions that developed at relatively small strains look like a superimposition of two characteristic

spectrums, namely, the sharp peak corresponding to  $\theta \sim 60^\circ$ , which is attributed to  $\Sigma 3$  twin boundaries, and a peak at low-angle misorientations. In contrast to CR, MDF facilitates the formation of sub-boundaries by dislocation networks with low-to-moderate misorientations (figure 6) and an increase in dislocation density (figure 7). Therefore, the MDF samples that experienced significant work hardening are characterized by higher hardness compared with the CR samples.

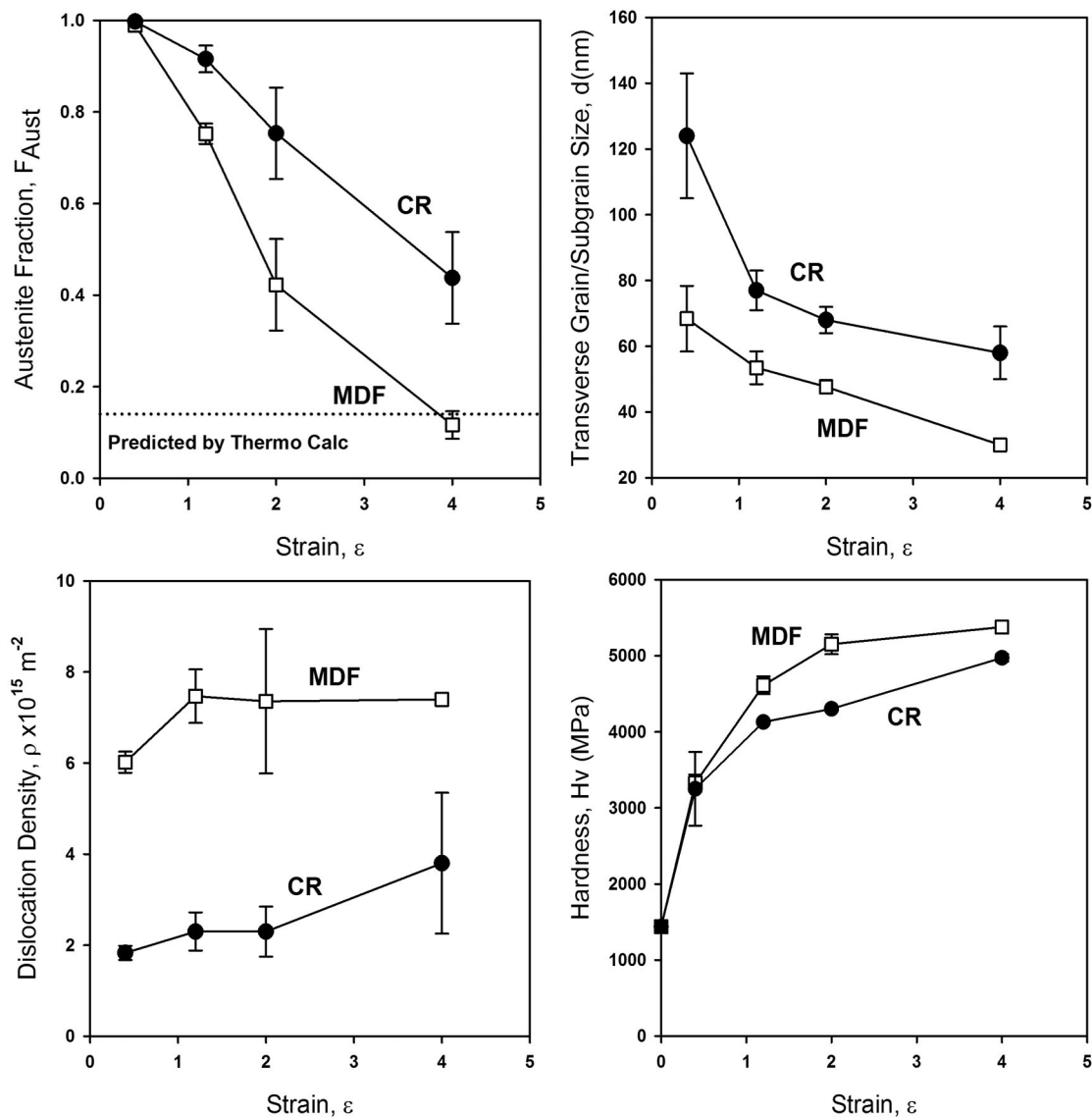


**Figure 6.** Distributions of (sub)-grain boundary misorientations developed in 316L austenitic stainless steel during multidirectional forging (MDF) and unidirectional cold rolling (CR) during different strain levels.

Upon further straining, the numbers of  $\Sigma 3$  twin boundaries and low-angle sub-boundaries tend to decrease (figure 6). The numbers of low-angle grain boundaries decrease from 0.34 to 0.14 and from 0.38 to 0.11 with increase in strain from 0.4 to 4 in the forged and rolled samples, respectively. However, remarkable peaks against small misorientations,  $\theta < 4^\circ$ , are observed at all strains investigated. Therefore, the fraction of high-angle grain boundaries increases with straining. On the other hand, the dislocation densities hardly changed for large strains. Following a rapid increase to  $2 \times 10^{15} \text{ m}^{-2}$  and  $6 \times 10^{15} \text{ m}^{-2}$  after the rolling and forging to a strain of 0.4, the dislocation density gradually approaches  $4 \times 10^{15} \text{ m}^{-2}$  and  $7.5 \times 10^{15} \text{ m}^{-2}$  during subsequent CR and MDF to a total strain of  $\sim 4$  (figure 7). Therefore, a dynamic equilibrium between the number of dislocations emitted by sources and the number of dislocations trapped by grain boundaries is established which provides an increase in grain boundary misorientations during straining.

MDF has a more pronounced effect on the martensitic transformation. This effect may be associated with the formation of 3D arrays of geometrically necessary boundaries

delimitating the micro shear bands upon MDF. Therefore, the formation of granular-type structure highly promotes martensitic transformation. As consequence, the deformation method, which facilitates the formation of 3D arrays of high-angle grain boundaries instead of lamellar structure, is favorable for inducing martensitic transformation. The increased hardness of the MDF samples is attributed to the larger fraction of strain-induced ferrite (figure 7). Processing to  $\epsilon \sim 4$  leads to an increase in the hardness to about 5380 and 4970 MPa in the forged and rolled samples, respectively (figure 7).



**Figure 7.** Effect of multidirectional forging (MDF) and unidirectional cold rolling (CR) on the austenite fraction, the crosswise grain/subgrain size, the dislocation density and the hardness in 316L austenitic stainless steel.

#### 4. Summary

Both the multidirectional forging and the unidirectional rolling of a 316L stainless steel at ambient temperature result in the development of ultrafine-grained structures with a high dislocation density exceeding  $10^{15} \text{ m}^{-2}$ . The formation of deformation sub-grain boundaries



followed by their transformation to high-angle grain boundaries and deformation twinning in austenite promote the martensitic transformation. Superposition of these mechanisms provides grain refinement during cold working. The martensitic transformation during multidirectional forging develops faster than during unidirectional bar rolling. The crosswise grain/subgrain sizes evolved after multidirectional forging and unidirectional rolling to a total strain of 4 are about 30 nm and 60 nm, respectively. The grain refinement during cold working is accompanied with an increase in the hardness, which yields to 5380 MPa and 4970 MPa in the forged and rolled samples, respectively.

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