Cao-Son Nguyen, Hoang Le, Anh-Hoa Bui*

School of Materials Science and Engineering, Hanoi University of Science and Technology
No. 1, Dai Co Viet Road, Hai Ba Trung District, Hanoi, Vietnam
*hoa.buiyanh@hust.edu.vn

MICROSTRUCTURAL CHARACTERIZATION OF ULC STEEL

ABSTRACT
In the present study, microstructure of the ULC steel was investigated by using the X-ray diffraction (XRD), optical microscopy (OM) and electron back scattering diffraction (EBSD) analysis. The pure ferrite phase consisting of various crystalline orientations, e.g. (110) and (200) etc., existed in the ULC steel. Ultra-fine grains of ferrite were observed in the ND-TD cross-section (⊥RD), meanwhile, typical lamina were seen in the ND-RD cross-section (//RD) of the steel sheet. Grain size of the annealed steel was observed to be coarser and equiaxed in all direction. According the EBSD results, intensities of the beneficial texture {111}<001> increased in the annealed steel, but weakened in the cross-section that was parallel to rolling direction. Ratio of low-angle grain boundaries (1°<LAGBs<15°) in the annealed steel was estimated as the higher value (93.1%) than that in the cold-rolled steel (69.1%).

Keywords: ULC steel; microstructure; EBSD analysis; crystalline orientation; texture

INTRODUCTION

Nanomaterials and steels play an important role in food processing and packaging applications [1,2]. Since reduction of greenhouses emissions and volatile organic carbon is necessary in order to reduce their effects on the environment, ultra-low carbon (ULC) steel coated with an inorganic or organic resin such as polypropylene or polyethylene terephthalate (PET) have been strongly attracted [3,4]. The C content in ULC steel is typically less than 50 ppm (the same as 0.005 wt.%), thus most of the interstices are not occupied, making an interstitial-free (IF) steel so that excellent ductility can be obtained. The ultra-low C of the steel is beneficial for ductility, toughness, formability and weldability but the loss in solid-solution strengthening accompanied by the decrease in C level needs to be compensated by the other strengthening mechanisms [5,6]. For example, the addition of silicon (Si) strengthens the ferrite matrix; manganese (Mn) improves the hardenability and promotes the formation of intermediate transformation products such as, bainitic and acicular ferrite. However, high Si and Mn addition has an adverse effect on the ductility of the ULC steel. For a practical application, these steels are known as good formability together with non-aging
properties, which it has given opportunity to the automotive industry to produce complex parts more easily and with high resistance [7,8].

The microstructure of steel is influenced by chemical compositions and thermo-mechanical processing parameters, so many metallurgical processes of rolling or/and heat treatment have been done [9-12]. According to Pan et al., the superior mechanical properties of steel were attributed to the influence of microstructure refinement on the work-hardening and subsequent inter-critical annealing, for example, ultra-grained dual-phase steel with an average ferrite grain size of about 2.7 μm and an martensite island of 2.9 μm was achieved [9]. Chen et al. experimentally studied on the microstructure and texture evolution, as well as the morphology, size and distribution of second phase precipitates during hot rolling, cold rolling and continuous annealing processes of Ti bearing and Ti+Nb stabilized ULC steels [13]. Meanwhile, Liu et al. investigated the influence of continuous annealing soaking temperature on microstructure, precipitation behaviour and fish-scaling resistance of ULC steel for porcelain enameling; the ferrite grain size grew from 13.4 up to 17.3 μm with the continuous annealing soaking temperature increasing from 750 to 840°C [4]. In the study conducted by Shukla et al., a thermo-mechanical controlled processing followed by water quenching was used to produced ULC micro-alloyed steel with consisting of polygonal ferrite, acicular ferrite as well as granular bainite with the average grain sizes less than 5 μm [14].

Being different from the low carbon steel which contains inter- and intra-granular carbides, ULC steel includes single ferrite phase with a very low content of alloying elements [15]. Guo et al. found that the α-fiber at the surface as well as at the midsection intensified during cold rolling and weakened during annealing, {001}<110> dominated at the surface and {111}<112> was the main component at the midsection in the hot band [10]. The study of crystallographic texture is important because the manufacturing process of the steel induces certain preferred crystallographic orientations. In ferritic steel, the slip direction is always <111>, but the slip planes can be {110}, {112} and, perhaps {123}. The typical texture of rolled ULC metals is composed of a partial α-fiber (running from {001}<110> to {111}<110>) [16]. It has long been recognized that the presence of favourable texture component in ULC steels is responsible for their excellent deep drawability, and it is well known that strong {111} and weak {001} components parallel to the sheet plane produce good formability [14]. Therefore, investigation of microstructure and crystallization orientation is very essential for research of ULC steels; and grain boundary characterization is necessary to clearly identify the mechanism which governs the mechanical properties of such steel. There are several methods, but currently, electron backscatter diffraction (EBSD) can be extensively applied to crystal orientation mapping, defect studies, phase identification, grain boundary and morphology studies, regional heterogeneity investigations, microstrain mapping, density of geometrically necessary dislocation [17-20]. By using the EBSD technique, the microstructural maps have shown that the cold work stored energy is distributed heterogeneously among different texture components in the steel [21]. Precise control of microstructure is important for developing high-performance and high-functionality steels, and therefore analysis technology for precision observation of microstructure is very important. Furthermore, the microstructure of severe cold-rolled ULC steel was rarely studied before. In this paper, microstructure of ULC steel was characterized using not only optical microscopy and X-ray diffraction (XRD) but also EBSD - a modern technique to examine the crystallographic orientation of steel by recording diffraction patterns along the sample surface.
EXPERIMENTAL

ULC steel was melted in a high frequency induction furnace (ALD) under argon vacuum pressure of 0.2 atm. An amount of the initial steel (500 g) was contained in a magnesia crucible (ID $\phi 40 \times 70$ mm), then melted and remained at 1600°C for 10 minutes. Thereafter, a given amount of high purity Si and Mn were added into the liquid steel to adjust the compositions as obtained in Table 1. The molten steel was casted into plate ($40 \times 25 \times 20$ m$\times$) which was hot-forged to 10 mm thickness, then directly cold-rolled to sheets with several passes at room temperature. The final cold reduction was controlled as 90 % with 1 mm thickness of the steel sheets. Finally, cold-rolled sheets were annealed at 800°C for 15 minutes.

Table 1. Compositions of the ULC steel (wt%)

|   | C   | Mn  | Si  | S   | P   | Fe   |
|---|-----|-----|-----|-----|-----|------|
|   | 0.004 | 0.415 | 0.175 | 0.003 | 0.001 | Balance |

Specimens of the cold-rolled and annealed steel were cut longitudinally to the rolling direction (see Fig. 1, RD – rolling direction, TD – transverse direction, ND – normal direction). The cross-section was observed using an optical microscope subjected to microstructural characterization by optical microscope (Axiovert 25A), X-ray diffraction (XRD, D8 Advance, Bruker) and electron back scattering diffraction (EBSD) analysis (EDAX). The specimens were grinded, polished and 2% nital-etched for optical and SEM-EBSD analysis. For EBSD, the step size was fixed to 0.1 μm in a field of 100×200 μm. The grain size of the annealed steels was defined as the average diameter which was determined by the mean linear intercept method from micrographs, meanwhile the one of the cold-rolled steels was calculated from EBSD histograms for grains statistic. Standard specimens (ASTM A370) of the cold-rolled and annealed steels were cut longitudinally the rolling direction and subjected to tensile testing using a MTS 809.10A machine.

RESULTS AND DISCUSSION

The optical micrographs of cold-rolled and annealed steel were shown in Fig. 2. After cold rolling, ultra-fine grains appeared in the RD–ND section as seen in Fig. 2a, but deformation bands were the main microstructure features in the TD–ND section as observed in Fig. 2b. It was seen that the grains could not be determined after cold rolling to a large
reduction. The grains were elongated along rolling direction after cold rolling. The obvious characteristic of the cold-rolled sample’s microstructure was formation of the in-grain bands for the present research. Meanwhile, the annealed steel demonstrated the image in Fig. 2c,d with morphology change and grain size was large and approached an polygonal one. The average grain size was calculated as 30 µm, and small grains were found to embed in large ones for the annealed steel. The phenomenon that there were more equiaxed grains present in annealed steel than in cold-rolled one was the same with other findings [22,23]. During annealing of the deformed ferrite, the fiber grains having high stored energy were likely to recrystallize so that polygonal grains were formed. Guo et al. remarked that with the temperature increasing to 700°C, more and larger equiaxed grains formed but some small grains still stayed beside large ones [10]. Thus, controlling annealing temperature is very helpful for the deformed ULC steel.

![Fig. 2. Optical micrographs of the cold-rolled (a,b) and anneal (c,d) ULC steel](image)

The XRD pattern of the ULC steel was shown in Fig. 3 where the ferrite peaks were presence of the (110), (200) and (211) orientations corresponding to BCC α-ferrite phase. Clearly, pure ferrite microstructure was present in the steels for both cold-rolled and annealed condition. There was no presence of FCC γ-austenite phase in all sample although the transformation of $\alpha \rightarrow \gamma \rightarrow \alpha$ might have occurred during annealing and cooling process. This obtained result was consistent with the other research [24]. In addition, width of the peaks was seen to broaden due to grain refinement for the cold-rolled sample. Similarly, the broadening of the peaks was concluded to be occurred mainly due to the refinement of crystalline size and increase in the dislocation lattice strain [25]. With regard to texture formation during cold rolling, it was observed in ULC steel that orientations had a trend to evolve to a stationary texture; but results were dependent on the deformation condition and the chemical composition of the steel [16]. Figure 4 shows the orientation of EBSD imaging microscopy for the ULC steel. The mapping was carried out on the both sections (RD–ND and TD–ND) to clarify variation of the morphology. The obtained result indicated that the
cold-rolled bands were not single crystals but polycrystalline. The same characteristic was noted by Gautam et al., who found a single layer of grain with specific texture (predominantly <110>) and morphology (elongated along rolling direction) presenting in the rolled steel [26].

Comparison of EBSD micrographs in Fig. 4 pointed out a difference microstructure between the two sections for the cold-rolled sample. A large number of ultrafine grains could be observed in the magnifying EBSD image of the perpendicular section due to the severe deformation, as in Fig. 4a; however, only in-band grains distributed along the rolling direction, as in Fig. 4b. The same result was achieved by Lim et al., who explained that the grain refinement occurred on the basis of intersection of micro-bands formed due to strain localization leading to formation of subgrains which subsequently rotated to form ultrafine crystallites [27]. Recrystallized microstructure including coarsen and equiaxial grains was observed in the annealed steel, see Fig. 4c, because of nucleation of stressed grains and the joining of several grains to form larger ones. Azushima et al. have reported that the severe plastic deformation (SPD) could be applied to make ultra-fine grains and high strength metal in order to produce lightweight parts for safety and reliability; by equal channel angular pressing (ECAP) at ambient temperature, ultra-fine grains with 0.2 µm wide and 0.5 µm long developed in the metal which was severely deformed up [28]. In this case, grain subdivision during SPD was a method of bulk metal grain refinement in the nano-size scale. Also, ULC steel was severely strained by accumulative roll-bonding (ARB) process and subsequently annealed for 0.5 h at various temperatures ranging from 400 to 800°C for strengthening by grain refinement; the tensile strength of the as-ARB processed IF steel increased with strain, reached a maximum of 813 MPa which was about 3 times higher than the initial value [29]. It was concluded that severe deformation caused the grain refinement, which was an effective way to increase strength without addition of alloying elements [30,31]. In this research, histogram for grains statistics of the ULC steels were shown in Fig. 5, where average grain size of the cold-rolled ferrite was 3 and 0.5 µm in parallel (∥ RD) and perpendicular (⊥ RD) section, respectively. Combination of Fig. 4 and Fig. 5 referred that columnar grains of the cold-rolled steel were formed with variation in the average lengths of the major and minor grain axes. The change of this ratio could be reflexed by the grain evolution from columnar to equiaxed which was observed in EBSD map. For the annealed sample, the average grain size was estimated as 30 µm. This result was consistent with calculation from the optical
micrographs. The similar grain evolution from columnar to equiaxed was also reported in several researches [22,26,32].

![EBSD micrographs of the cold-rolled (a,b) and anneal (c) ULC steel](image)

**Fig. 4.** EBSD micrographs of the cold-rolled (a,b) and anneal (c) ULC steel

The EBSD analysis of misorientation angle distribution in the samples was given in Table 2, where the statistical results shown that almost grain boundaries misorientation angles were distributed in the cold-rolled steel. The ratio of boundaries with misorientation changed significantly in the two samples. In terms of grain boundary misorientation, the boundaries are usually divided into two categories: low angle boundaries are known to contribute to the sub-grain strengthening, while the high angle boundaries constitute the Hall-Petch contribution; the large misorientation indicates the presence of stress in the material [14]. The low-angle grain boundaries (1°<LAGBs<15°) and the high-angle grain boundaries (HAGBs>15°) were 93.1 and 6.9 % in the annealed sample, respectively. This is attributed to the recrystallization occurred in the annealing process, in which the stored energy is released and larger grains are formed by the nucleation of stressed grains and the joining of several grains. The grain boundaries misorientation angles was more disordered in the cold-rolled one, in which the ratio of HAGBs increased to the value of 30.9 % in the cold-rolled steel. During severe cold-rolling, the grains in the present ULC steel fragmented into smaller ones of different orientation. With ultrafine grains in the cold-rolled steel, the higher angle boundaries indicated a higher degree of misorientation, and thus a high stored energy in the grains. This was in accordance with the tested yield strengths (YS) which were 461 and 285 MPa for the cold-rolled and the annealed steel, respectively. It was also concluded by Cizek et al., who studied the strengthening behaviour of IF steel after the hot pressure torsion at room temperature and demonstrated that the improvement of the YS occurred mainly due to the dislocation strengthening and grain size refinement [33].
For EBSD analysis, individual orientation measurements from the whole sample were plotted together and shown as points in the pole figure which revealed the texture of the sample [34-36]. The orientation distribution close to a \{001\} texture component would appear as a cluster of points on the pole figure map as seen in Fig. 6. The result was similar to a typical steel rolling texture with strong concentration at \(\alpha\)-fiber component in the investigation of Wenk et al. [34]. In addition, textures can also be represented on an inverse pole figure which shows the distribution of crystallographic directions parallel to certain sample directions, namely as \(<001>\//RD\) in Fig. 7. According to Guo et al., although the major characteristics of the BCC rolling texture were almost unchanged during deformation, decrease the \{001\} components in the annealed steel indicated that ferrite has undergone extensive recovery and recrystallization during annealing [10]. In this study, the \{111\} intensity in the annealed steel was higher than one in the cold-rolled steels, so it could be concluded that development of the \{111\} texture caused an increase in drawability of a ULC steel sheet.

Fig. 5. Histograms for grains statistics of the cold-rolled steel

Fig. 6. EBSD pole figure maps of the cold-rolled (left) and the annealed (right) steel
It is known that the texture can also be viewed as a continuous orientation distribution with contour levels which show the strength of the texture as the number of times random occurrence. From Fig. 6, the $\alpha$-fiber {111}<001> intensities were seen as 2.7 and 5.8× random intensity (RI) in the cold-rolled and annealed steel, respectively. In Fig. 7, the values were 4.9 and 8.0× RI in the cold-rolled and annealed steel, respectively. The fact that intensities of the beneficial texture components such as {111}<001> were lower in the cold-rolled steel referred that annealing was capable of strengthening the {111}<001> recrystallization texture; resulting the formability of the steel could be improved. As described in the study of Guo et al., the intensity of the {111} texture components, the favourable texture for ULC steel sheet, seemed to correlate with the degree of recrystallization of ferrite [10]. Meanwhile, it is well established that plastic strain ratio r value increases with ratio of the {111}/(001) intensity; when the annealing temperature increased to 750°C, the strongest component {111}<112> reached higher than {111}<110>, resulting in an improvement of r value [14]. The present study has achieved the same result with Wenk et al. who studied texture changes during the $\alpha$ (BCC) $\rightarrow$ $\gamma$ (FCC) phase transformation in ULC steel during annealing and found that upon cooling from 900 to 400°C, the steel reverted completely to BCC though there was a reduction of the {111} intensity [34]. Wakita et al. studied the microstructural transformation of low-carbon steel during heating, and then concluded that the $\gamma$ phase was confirmed to have formed at 800°C but disappeared during cooling to 300°C [35].

CONCLUSIONS

In this study, microstructure of the ULC steel after cold rolling and annealing were characterized using optical microscopy, XRD and EBSD analysis. It was acknowledged that severe cold reduction of 90% caused a formation of the ultrafine ferrite grains which were found in the cold-rolled ULC steel and estimated as 0.5 and 3 μm in accordance with the two main axes. Grain evolution from columnar to equiaxed occurred during annealing process. The EBSD results referred that intensity of the beneficial texture {111}<001> increased in the annealed steel, but weakened in the cross-section that was perpendicular to rolling direction. The ratio of low-angle grain boundaries ($1°$<LAGBs $<15°$) in the cold-rolled steel (69.1%)
was lower than the annealed steel (93.1%), meaning that cold deformation caused the higher misorientation. The ultrafine ferrite grains in the cold-rolled ULC steel were attributed to the high yield strength, while the equiaxed ones in the annealed ULC steel contributed for the high ductility.

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