Microstructure evolution during semi continuous equal channel angular extrusion process of interstitial-free steel

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Abstract. In order to produce ultrafine grained structures, interstitial-free steel sheets have been processed using a novel severe plastic deformation technique semi continuous equal channel angular extrusion (SC-ECAE) which is based on equal channel angular extrusion (ECAE) in an incremental way. The deformation was carried out at room temperature and individual specimen was repeatedly processed to various passes. An overall grain size which is 0.55 µm was achieved after 10 passes (or an equivalent total strain of 4.8). The present paper reports the evolution of microstructures during deformation, which were examined and characterized using high resolution EBSD in a field emission gun SEM. The mechanisms of grain refinement are discussed.

Keywords. Severe plastic deformation, semi continuous equal channel angular extrusion (SC-ECAE), IF steel, ultrafine grained structure, EBSD

1. Introduction
Grain refinement is one of the most important and basic methods for structure control in steel production. The research boom of severe plastic deformation (SPD) has been going for many years for the great potential of the metal grain refinement to the nanometer scale, such as equal channel angular extrusion (ECAE) [1], high pressure torsion (HPT) [2], accumulative roll bonding (ARB) [3-4] and asymmetric rolling (ASR) [5], etc. So far, of all the SPD techniques, equal channel angular extrusion (ECAE) has attracted the most attention, due to its capability of producing UFG structures at scales large enough for structural applications, especially in the field of steel processing [6, 7]. However, the ECAE technique in its original form has disadvantages for commercial application, such as limited scalability, high scrap rate, high load requirement and low production efficiency, etc. Many attempts have been made to transform ECAE into a continuous process, such as continuous constrained strip shearing (C2S2) [8] and ECAP-Conform [9] and continuous frictional angular extrusion (CFAE) [10]. As one of the novel methods based on ECAE, Semi Continuous Equal Channel Angular Extrusion (SC-ECAE) takes advantage of ECAE, which performs the external forces to the dies instead of the specimens. The main benefits that the SC-ECAE technique include the capability of processing large volumes of material into various forms, high productivity, low scrap rate, and reduced extrusion force requirement, etc.

2. Experimental details
The principle of SC-ECAE method is showed in Fig.1, the first extrusion channel is composed by Die A and Plane C, the second channel is made by Die A and Die B. The second channel is shorter than the first one for reducing the resistance of the specimen going through the second channel. The contact surface between Die A and the specimen is smooth with ow friction, on the contrary the contact surface between Plane C and the specimen is rough. In the first step of the procedure, the specimen is fed into the first channel, then it is held by the force N which is applied on Die A. Secondly, Die A is pushed through the extrusion angular Φ
and goes into the second channel for a short step $\Delta s$ by the force $P$ which is affected on Die B. The third step is the two dies return to the initial position when the force $N$ and the force $P$ are released. After that, the above process is repeated until the whole specimen is completely goes through the extrusion angular, which is called one pass. The specimen is processed to various accumulative passes with the orientation maintaining constant throughout. The structure can be refined to submicron level based on the above method. The advantage is the forces of this method acts on the dies instead of the specimen. Then the force can be performed easier, and the shape limits of specimen can be reduced. Especially for the processing of sheets, the workability can be greatly increased. It would be possible to fit the process for practical application.

The SC-ECAE setup was established for the present investigation recently. IF steel sheets, with dimensions of 2 mm×60~70 mm×1000 mm, were used for the SC-ECAE processing. The material was cold rolled and annealed at 820°C for 40 min, giving a fully recrystallized starting microstructure with an average grain size of 24 µm. Deformation was carried out at room temperature with a rate of 0.01 m/s. The sheets were processed to various accumulative passes of up to 10, giving a maximum equivalent true strain of 4.8, with the sheet orientation maintained constant throughout. Plane C was mechanically roughened to enhance friction and MS2 spray was employed to lubricate the interface between the specimen and Die A. The deformation structures of the SC-ECAE processed sheets were examined in a FEG-SEM and using also high resolution EBSD on the TD plane after mechanical and electropolishing. The EBSD data were analyzed by the HKL-Channel5 software.

![Fig.1 Schematic diagram of SC-ECAE](image)

### 3. Results and discussions

In general, as shown in the large-scale micrographs of Fig. 2, the microstructure refinement was intensified with increased strain and deformation passes. The initial grains were elongated and destructed when the deformation began; further elongation occurred in the following passes and the grains evolved into a fiber or lamellar structure after 6 passes. With further deformation, the fiber structure continued to be compressed and became finer.
According to the simple shear deformation model, the structural elongation direction and the simple shear deformation after \( n \) passes should follow the below equations:

\[
\beta_n = \arctan \Gamma^{-1} \quad (1)
\]

\[
\Gamma = 2n \cot \phi \quad (2)
\]

where \( \beta_n \) is the angle between the structural elongation direction and the extrusion direction, and \( \Gamma \) is the total shear strain in the die shear plane after \( n \) passes.

Table. 1 shows that the measured angles fit well with the calculated values based on the simple shear model.

| Passes | 2    | 4    | 6    | 8    | 10   |
|--------|------|------|------|------|------|
| Measurements | 35   | 26   | 20   | 15   | 9    |
| Calculations  | 31.1 | 16.8 | 11.4 | 8.6  | 6.9  |

With the help of high-powered scanning electron microscope, severe plastic deformation, which was essentially caused by simple shear, was found in a narrow region along the intersectional plane of the two extrusion channels (referred to hereafter as the die shear plane) after every pass, and the deformation on the TD plane on the samples processed to all levels of strain was reasonably uniform over the whole cross-section of the specimen. Backscatter electron images in Fig. 3 show the general features of microstructure evolution with increased passes. As shown in Fig. 3(a), the initial equiaxed grains began to be elongated after two passes. In the following passes, the trend of grain elongation was clearer with increasing strain as shown in Fig. 3(b). After 6 passes, the elongated grains formed bands gradually, which were approximately aligned with the die shear plane, at an angle of 45° to the extrusion direction (ED). When the deformation went on, the bands became the main characteristic of the microstructure. It can be observed from Fig. 3(d) that the bands were compressed to be narrower and more uniform leading to a lamellar structure. The spacing of the lamellar structure in the normal direction (ND) can be estimated to be less than 1 µm.
In the present work, high resolution EBSD technique was used to determine HABs. The evolution of the deformation structure has been illustrated by the EBSD maps, as shown in Fig. 4. In the EBSD maps presented, HABs in black lines are defined as having misorientations higher than 15° and low angle boundaries (LABs) in white lines misoriented between 5° to 15°. Boundaries misoriented below 5° are cut off due to the noise effect. The misorientation distributions and the microstructural parameters determined from EBSD measurements are given in Table 2, respectively. It is seen that the substructures formed in the first stage of deformation which only subdivided the initial grain structure by LABs misoriented a few degrees, and the HAB structure began to be elongated after 2 passes. After 4 passes, LABs began to convert to HABs gradually with increasing strain, although their distribution was very heterogeneous, as shown in Fig. 4(b). After six passes, from Fig. 4(c) the elongated HAB structure can be seen obviously and HABs dominated with a fraction over 80%, and the average spacing of HABs was close to a submicron scale, but the spacing of HABs was not uniform. With further passes (total strain of 4.8 in 10 passes), the spacing of most HABs was decreased continuously and the fraction of final HABs kept above the level of 80%, whereas the overall boundary spacing, including both HABs and LABs, was down to about 0.55 µm. However, there were still several regions of HABs with larger size and the LAB structure that made the substructures still can be observed inside the HAB structures, as shown in Fig. 4(d). The existing LABs would be continued to be converted into HABs and the
fraction of LAB area still has potential to increase, and the microstructure would be more uniform, upon further deformation. This substantially refined UFG structure occurred at a lower strain than that expected from conventional ECAE processing of the comparable IF steel. In addition, the application of EBSD technique to ultrafine grained and nanostructured materials processed by SPD methods is still a challenging work, especially in the observation of the specimen with severe residential stress after multi passes deformation \[11, 12\], the pattern quality of EBSD map after 6 passes is hard to reach high level. Nevertheless, the overall trends of microstructural evolution and grain refinement observed in the present work are in agreement with previous ECAE studies.

(a) After 2 passes  
(b) After 4 passes  
(c) After 6 passes  
(d) After 10 passes

Fig. 4 EBSD maps of IF steel samples with different SC-ECAE passes

| Pass | Equivalent True strain | HAB ND- spacing [µm] | Overall ND- spacing [µm] | HAB fraction [%] |
|------|------------------------|-----------------------|--------------------------|-----------------|
| 0    | 0                      | 26.04                 | 24                       | 83.2            |
| 1    | 0.48                   | 16.67                 | 10.69                    | 63.1            |
| 2    | 0.96                   | 11.08                 | 6.6                      | 60.0            |
| 3    | 1.43                   | 8.02                  | 4.03                     | 82.7            |
| 4    | 1.91                   | 8.64                  | 4.31                     | 88.9            |
With increasing deformation, the angle of misorientation increases (Fig. 5) LABs transferred to HABs, meanwhile the material achieved more plastic deformation in macro scale. The variation of misorientation distribution in different passes is in agreement with the results of other SPD methods.

### 4. Conclusions

IF steel sheets were processed to various numbers of passes by the method of SC-ECAE. The microstructure evolution and mechanisms of grain refinement during deformation were studied using high resolution backscatter electron imaging. The angles between the structural elongation direction and the extrusion direction at different passes fit well with the estimation of the simple shear model, suggesting that deformation primarily took place in simple shear along the intersection plane of the two extrusion channels, and the deformation structures were reasonably uniform over the whole cross-section of the specimen.

Misorientation distribution showed that the fraction of LABs increased firstly in early stages. After four passes, the fraction of HABs began to increase gradually with increasing strains. Elongated HAB structures were formed after six passes, and the average spacing of HABs was close to a submicron scale. Processing up to 10 passes (equivalent strain of 4.8), a final HAB fraction of about 90% was obtained and the HAB spacing decreased to less than 1 µm, whereas the overall boundary spacing, including both HABs and LABs, was reduced to about 0.55 µm.
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