Microstructural Evolution and Mechanical Properties of an Interstitial-Free Steel Processed by Equal-Channel Angular Pressing

Preface

Grain refinement in a polycrystalline material to micron level is known for its beneficial effects of enhancing both strength and ductility. Severe plastic deformation (SPD) methods specially equal-channel angular pressing (ECAP) can be adopted for refinement to ultrafine level where grain size of the order of ~200-300 nm can be achieved, which is an order of magnitude higher than that can be obtained by thermomechanical treatment (TMT). Ultra high strength, ductility and high toughness are required along with reduce mass, improved safety and performance in transportation equipments. ECAP of alloys enhances yield strength to grain refinement to ultra high range due to ultrafine level (<1μm) but ductility gets reduced comparison to their coarse grained counterpart due to limited dislocation activity. The solutions to these problems are not yet found because of which, the applications of UFG materials are delayed. Grain refinement of low carbon steel suggests that microstructure of bimodal grain size distribution where majority of grains are in ultrafine range carrying high stress or load and small fraction of grains in 1-10 μm range restore ductility can be achieved in metals and alloys of mixed microstructure. Wang et al. have shown that ductility in electrical grade copper can be regained while retaining strength to about six times that of coarse grained counterpart by effecting bimodal grain size distribution through cryorolling followed by short annealing above secondary recrystallization temperature. Therefore, the solution to the problem is to optimize strength and ductility by producing mixture of ultrafine grains and micron sized grains in the microstructure. Such a microstructure can be achieved by combining optimized ECAP parameters with post ECAP deformation and annealing. Interstitial free (IF) steels are known for their high formability but their low strength acts as a detriment to their wider applications. There is a need to improve the strength of the material with maintaining ductility. Therefore, the present investigation is focused on the refinement of interstitial-free steel to ultrafine-grain level by ECAP to get ultra high strength and post deformation of UFG IF steel by cold rolling and cryorolling to refine the material further. One of the other major objectives is to get bimodal/multimodal grain size distribution in IF steel processed by ECAP and post ECAP deformation followed by flash annealing. Structure-property correlations are to be established.

The present thesis is divided into **EIGHT CHAPTERS**. **Chapter-1** presents a brief introduction along with the review of IF steel, its properties and applications. It also gives the details of the strengthening mechanisms in metals and alloys, ECAP processing to synthesize ultrafine grains in alloys, especially in steel, texture and mechanical properties of ultrafine -grained materials, and suitable methods to get both high strength and high ductility in metals. At the end of the Chapter objectives of the present investigation are elaborated.

Chapter-2 deals with the detailed experimental procedures for the preparation and characterization of UFG IF steel. The hot rolled steel billets are deformed at room temperature by equal-channel angular pressing adopting the route Bc using an ECAP die of an inner intersection angle of 120^o and an outer arc angle of 60^o to a maximum of 40 passes or equivalent strain of 24 to get ultrafine-grained structure. This ECAP die introduces an equivalent strain (ε_{vm}) of ~0.6 during every passage of the billet. About 20 mm lengths from both the ends of the ECAPed billet are removed to eliminate the end effects. X plane is the transverse plane perpendicular to extrusion direction. Y plane is the flow plane vertical to the extruded billet containing the extrusion direction and Z plane is the horizontal plane but parallel to top surface along extrusion direction. The major deformations take place on flow plane, therefore, the deformed samples are sectioned along Y plane for microstructural investigation at the central zone of the billet.

ECAPed (ε_{vm}=12) billets are coldrolled at room temperature to 90% reduction in area and cryorolled at -50°C to the reduction in areas of 70 and 96% along their extrusion direction for studying the effect of temperature on post deformation of ECAPed material.

ECAP, ECAP+coldrolled and ECAP+cryorolled samples are flash annealed from 550-750°C in molten $NaNO_3$ salt for 5 minutes, followed by water quenching to study the stability of ECAPed and post ECAP deformed material. It is also an attempt to optimize conditions to get bimodal grain size distribution. Selective samples are flash annealed directly in air furnace for same duration at temperature 700°C or higher.

IF steel samples are characterized for microstructure by optical microscopy, scanning electron microscopy, electron back scattered diffraction and transmission electron microscopy. Bulk texture is studied by X-ray diffraction. Lattice strain, stored energy and domain size are calculated from X-ray scans. Mechanical properties are evaluated by hardness measurements and tensile testing. Fractured surfaces are investigated by scanning electron microscopy.

Chapter-3 presents the details of microstructure, grain refinement mechanisms operative during SPD from low to high strain level. The microstructural refinement involves elongation of grains, their subdivision to bands having high dislocation density at initial pass of ECAP (ε_{vm}=0.6), splitting of bands to cell blocks and eventually cell blocks to cells (at ε_{vm}=1.8). The degree of reduction in grain size is highest at low strain level, however, most of the boundaries at this stage are low angle boundaries (at ε_{vm}=3). Thereafter, misorientation angle increases by progressive lattice rotation. The coarse bands transform step by step from lamellar structure to ribbon shaped grains (at ε_{vm}=12) and finally get split. With further strain, the transverse boundaries of subgrains interacted with dislocations, resulting in an increase in their misorientation angle. At very large strain ε_{vm}>15, the ribbon shaped grains transform to the near-equiaxed grains structure and grain fragmentation continues till ε_{vm}=18. From ε_{vm}=21 to 24, the grain size almost remains unchanged. Finally average grain size, low angle grain boundary area fraction and average misorientation angle stabilize at respective saturation values. The coarse grained microstructure of IF steel has been refined from 57.6±21 µm in as-received condition to 257±48 nm at equivalent strain ε_{vm}=24 and a low angle grain boundary fraction of 0.34.

Chapter-4 records the development of bulk texture in IF steel processed by ECAP. A strong shear texture develops as a result of ECAP adopting route Bc. At low strain level, ε_{vm}=0.6, components of {110} fiber $J_{\theta},\bar{J}_{\theta}$ and $E_{\theta},\bar{E}_{\theta}$ common components of <111> fibers are existing but their intensities are low. However at intermediate strain ε_{vm}=1.8-6, texture consist mainly of <111> fiber components. At higher strain range, ε_{vm}=9-24 <111> fiber texture develops with high intensity of components $D_{1\theta},D_{2\theta}$ as main components during simple shear deformation from low to high strain level. With the increase in strain the intensity of these components enhances. All components get shifted from their deviated position towards negative shear to exact (ideal) position at large strain level. It is possible to achieve monoclinic symmetry after ECAP process at $\varepsilon_{vm}=9$ by route Bc of ECAP process in IF steel and a texture index that is 3.6 times more than that of as-received IF steel. The grain misorientation in microstructure with increasing equivalent strain is correlated with texture development. Texture initially increases with strain due to the formation of texture components at different strain level.

At $\varepsilon_{vm}=0.6$, coarse grains begin to align in the direction of deformation. Alignment of cells and cell blocks (at $\varepsilon_{vm}=2.4$) in deformation bands lead to increase in texture intensity with concentrated clouds of $\bar{J}_{\theta}$and J_{θ} components. At $\varepsilon_{vm}=6$, high angle grain boundaries, cell blocks and cell structures get aligned to deformation direction to form lamellar structures mainly with $D_{1\Theta}$, $D_{2\Theta}$. At $\varepsilon_{vm}=9$, oriented ribbon grains result in strong $D_{1\Theta}$, $D_{2\Theta}$ components with <111> fiber. At $\varepsilon_{vm}=15$, partial conversion of ribbon grains to near-equiaxed grains produces enhanced intensity of $D_{1\Theta}$ and $D_{2\Theta}$ components. At $\varepsilon_{vm}=15\text{-}21$, grain refinement attains saturation with further increase in intensity of $D_{1\Theta}$, $D_{2\Theta}$. At $\varepsilon_{vm}=24$ results in near-equiaxed grain formation and that attain increased intensity of $D_{1\Theta}$, $D_{2\Theta}$ components.

Chapter-5 gives the influence of microstructural refinement of ECAPed IF steel on mechanical properties. Both the yield strength and ultimate tensile strength increase sharply upto $\varepsilon_{vm}=3$ due to the rapid microstructural refinement with high defect density. Thereafter, the strength increases appreciably upto $\varepsilon_{vm}=9$ as LAGB fraction decreases and average misorientation angle increases. The strengthening continues to occur even upto

ε_{vm}=24, since the increase in high angle grain boundary fraction continues to take place although the grain refinement at ε_{vm}>9 is not significant. The strengthening of IF steel of yield strength of 227 MPa to 895 MPa by ECAP for ε_{vm}=24 at 298K is itself noteworthy. The uniform elongation of IF steel reduces to 0.5% by ECAP due to lack of work hardening ability at a low strain level (ε_{vm}=0.6). with increasing strain, the elongation improves marginally by 1.5-2% upto εvm=9 and thereafter it remains almost constant. The ECAPed sample fails by ductile fracture at lower strain range ε_{vm}=0.6-6 but by mixed mode of ductile-brittle fracture at larger range of ε_{vm}=9-24.

Chapter-6 deals with IF steel samples deformed by ECAP upto equivalent strain, ε_{vm}=12, followed by (i) coldrolling to 90% and (ii) cryorolling at -50°C for 70 and 96% reduction in area. ECAPed material consists of ultrafine-grained ribbon grain structures of about one cell width. When ECAPed samples are deformed further by coldrolling/cryorolling for large percentages of reduction in area, the dynamic recovery results in conversion of cell structures into subgrain structures. ECAP followed by cryorolling to a low strain level produces non-equilibrium grain boundaries and thick cells containing dislocations. ECAP followed by cryorolling can be utilized for further refinement. Deformation subsequent to equal-channel angular processing is found to have improvement in area fraction of the high angle grain boundaries. Enhancement in high angle grain boundary fraction strengthens the material significantly, however, reduction in grain size to ultrafine level lowers work hardening ability of the material that limits ductility. Changing mode of deformation from ECAP to cryorolling leads to the formation of γ fiber, whereas no sharp γ fiber is formed by cold rolling of ECAPed material.

Chapter-7 presents microstructural stability of deformed samples processed by ECAP and post deformation of ECAP by coldrolling/cryorolling followed by flash annealing. All the above mentioned deformed samples are flash annealed in $NaNO_3$ melt maintained at required temperature of 550˚C to 700˚C for 5 minutes and quenched in water. Results show that bimodal grain size distribution in ultrafine grain size range can be achieved in the IF steel processed by equal-channel angular pressing followed by flash annealing. Recrystallization temperature of the steel decreases with increase in amount of equivalent strain. Abnormal grain growth is observed in ECAPed IF steel where temperature of abnormal grain growth in ECAPed IF steel increases with decrease in equivalent strain. Hardness of severely deformed IF steel can be maintained upto 650°C at least for short duration of 300 seconds. When UFG IF steel samples processed by ECAP at ε_{vm}=12 followed by cold rolling/cryorolling to >90% reduction in area and flash annealed at 675°C, the samples get partially recrystallized. The increased subgrain size and reduced residual lattice strain lower the hardness and strength with marginal recovery of ductility. But lack of dislocation activities due to reduced grain size and residual lattice strain fails to recover the ductility to the level of as-received material. Even though at that condition the yield strength is maintained at 2-3 times higher to that of the same.

Chapter-8 presents a summary of the present investigation along with the suggestions for future work. IF steel can be deformed by ECAP upto ε_{vm}=24 adopting route Bc. The microstructural refinement involves elongation of grains at ε_{vm}=0.6, subdivision of grains to bands with high dislocation density at ε_{vm}=1.8, splitting of bands to cell blocks and cell blocks to cells. The degree of reduction in grain size is highest at low strain level, however, most of the boundaries at this stage are low angle boundaries (at ε_{vm}=3). Thereafter misorientation angle increases by progressive lattice rotation. The thickness of bands decreases with strain. The coarse bands transform step by step to lamellar structure at ε_{vm}=6 and to ribbon shaped grains at ε_{vm}= 9 and finally to near-equiaxed grain structure with subgrains of a saturated low angle grain boundary fraction of 0.34 at very large strain ε_{vm}>15. As-received coarse grained (grain size of 57.6±21 µm) microstructure of IF steel has been refined to 257±48 nm at equivalent strain ε_{vm}=24 with low angle grain boundary fraction of 0.34. A strong shear texture forms as a result of ECAP adopting route Bc. The texture density increases with strain and mainly consists of $J_\theta/\bar{J}_\theta$ and $D_{1\theta}/D_{2\theta}$ componentsAt ε_{vm}=24, texture index records a value which is 3.6 times more than that of as-received IF steel. It is possible to achieve monoclinic symmetry after ε_{vm}=9.

Strength of IF steel increases with equivalent strain due to microstructural refinement with high defect density. Strengthening rate with respect to equivalent strain reduces with strain as microstructural refinement rate decreases at higher strain. Strengthening at lower strain is due to refinement and high defect density but at later stage strengthening continues due to increase in average misorientation angle and high angle grain boundary fraction. The yield strength of selected IF steel can be enhanced by ECAP from 227 MPa to 895 MPa at ε_{vm}= 24. Ductility of UFG IF steel is low due to lack of work hardening ability at low strain level but it improves marginally at high strain level. ECAPed IF steel fails by ductile fracture at lower range of εvm=0.6-6 but by the mixed mode of ductile-brittle fracture at large strain range (εvm=9-24).

Microstructure of ECAPed and post ECAP rolled IF steel with suitable thermal treatment can be further investigated by high resolution transmission electron microscopy to explore details of refining mechanisms, the nature of non-equilibrium grain boundaries, migration mechanism of grain boundaries and dislocations. ECAPed IF steel can be deformed further by cold rolling at room temperature/cryorolling at -50°C for >90% reduction in area. The cold rolling/cryorolling of ECAPed IF steel refines the material further along with generation of non-equilibrium grain boundaries that strengthens the material significantly. Decrease in grain size with increased lattice strain lowers work hardening ability of the material and limits its ductility.

Flash annealing can be utilised to get bimodal grain size distribution in ECAPed IF steel ultrafine grain range. The recrystallization temperature decreases at low to intermediate equivalent strain but it increases at high equivalent strain due to lower defect density. Hardness of UFG IF steel can be maintained upto flash annealing temperature of 650°C at least for short duration of 300 S. Flash annealing of cold rolled and cryorolled IF steel after ECAP followed by coldrolling/cryo rolling (At 650°C) recrystallizes the steel partially. The increased subgrain size, grain size and reduced residual strain reduces hardness and strength with marginal recovery of ductility even though the yield strength is maintained 2-3 times when compared to the as-received IF steel.

Correlation of texture with mechanical properties can be further investigated. Formability and impact resistance of UFG IF steel can be studied in detail which is extremely important for processing and application of the steel in automobile manufacturing.