Preface

The low-carbon low-alloy steels lack in strength. As a result, their application in structural parts is overdesigned. Grain refinement in micron level is considered as a major strengthening mechanism for this group of steels as it increases both strength and ductility. In industry thermomechanical treatments are utilized to refine low carbon steel where grain size is in micron level. Severe plastic deformation (SPD) techniques are used to produce bulk ultrafine-grained structure in low carbon steel with improved strength and hardness. Among all SPD techniques equal-channel angular pressing (ECAP) is a most promising one due to many advantages. Various low-carbon steels have been deformed by ECAP up to a maximum equivalent strain of 10 to attain a microstructural refinement up to ultrafine-grain (UFG) size of 200 to 300 nm. Strength continues to increase while grain size decreases in the range of micron to ultrafine level but ductility increases as grain size decreases in micron size range only. Tensile ductility decreases as grain size decreases in ultrafine range. The tensile strength of UFG alloys goes up 2 to 5 times compared to that of the coarse-grained or annealed material, but the tensile ductility decreases as a consequence of low strain-hardening ability. Therefore, there is a possibility of obtaining high strength with high ductility by producing a microstructure of bimodal grain size distribution of ultrafine grains with micron-sized grains in the material.

This thesis comprises eight chapters. **Chapter-1** gives a brief overview about low carbon steels, its properties and applications. The Chapter comprises details of severe plastic deformation for synthesis of UFG materials, keeping steels in focus. Importance of ECAP and the effect of its processing parameters on grain refinement are elaborated. The Chapter also covers the details of grain refinement mechanisms and strengthening mechanisms from low to high strain levels. Properties and texture development in UFG steels are reviewed. Effect of short time annealing on UFG material is also explained.

The objectives of the present investigation are to develop UFG low carbon steel by ECAP to get high strength and post deformation of ECAPed low carbon steel by cold rolling and cryorolling to further grain refinement of the material. One of the other major objectives is to regain the ductility in UFG low carbon steel with maintaining high strength. By optimized microstructure consisting of bimodal grain size distribution of ultrafine grains and micron sized grains in low carbon steel with suitable thermal treatment. Finally, correlations among microstructure, texture and mechanical properties will be established.

Chapter-2 includes the details of experimental procedures. In the present study, low carbon steel is deformed using ECAP process upto an equivalent strain of 16.8 adopting route Bc at room temperature. ECAPed material is deformed by coldrolling upto 80% reduction in area and cryorolling at -50°C upto 75% reduction in area followed by flash annealing at various temperatures (475-675°C). The microstructures are examined by optical microscopy, scanning electron microscopy, transmission electron microscopy (TEM) and Electron back scattered diffraction (EBSD) from bulk specimens. EBSD Data is analysed for misorientation, grain boundary fraction and microtexture. Bulk texture, dislocation density and elastic stored energy are measured from X-ray diffraction (XRD) study. Mechanical properties are evaluated by microhardness measurement, tensile testing, and fractography.

Chapter-3 comprises the details of microstructural development. At low strain (ε_{vm} = 0.6), the coarse-grained equiaxed microstructure of as-received low carbon steel gets modified to deformation bands with a high density of dislocations and reduced grain size. The microstructural refinement involves elongation of grains, subdivision of grains to bands with high dislocation density, as the imposed strain increases, the bands are elongated, and their thickness decreases. At intermediate strain (ε_{vm} = 6), the boundaries of bands, few cell block boundaries, and grain boundaries all get aligned to the deformation direction which results in the lamellar structures. With the increase in strain, at ε_{vm} = 9, the lamellar width decreases to a few subgrain wide and their length increases. The elongated bands take the shape of ribbons. At ε_{vm} = 16.8, the ribbon grains are partially broken into near-equiaxed grained structures with increased misorientation angle and dislocation density. Rate of grain refinement is high at low strain level and it decreases with increasing strain. As-received coarse grained (grain size of 67±7 μm) of low carbon steel has been refined to 0.2 μm of grain size at equivalent strain ε_{vm}=16.8 with average misorientation angle of 40° and dislocation density of 17.1 $x10^{14}/m^2$. Defect density is low in as-received material. It increases with imposed strain. The dislocation density reaches maximum value at intermediate strain.

Chapter-4 presents the development of bulk texture in low carbon steel processed through ECAP. The texture strength of the ECAPed low carbon steel increases, continuously with imposed strain in the range (ε_{vm} = 0.6 - 1.2) due to grain subdivision to bands. With further increase in strain the index decreases due to randomization. At intermediate strain (ε_{vm}=3-6) the material is strongly textured due to alignment of bands though it fluctuates due to randomisation. At high strain level (ε_{vm}=9-16.8), though texture strength increases but due to (randomisation) breakage of ribbon grains followed by randomisation the rate of increase in texture intensities decreases. ECAP of low carbon steel at low strain level (ε_{vm}=0.6 - 1.2) forms F_θ (110)[001], J_θ (110)[$1\bar{1}2$], $\bar{J}_\theta$ ($\bar{1}\bar{1}0$)[$\bar{1}1\bar{2}$] and $D_{1\theta}$ ($\bar{1}\bar{1}2$)[111] components. At intermediate strain level (ε_{vm}= 3-6), $F_{\theta,}$ $\bar{J}_\theta$ and $D_{1\theta}$ components get strengthened and new component $D_{2\theta}$ component is formed. At high strain level (ε_{vm}=9-16.8), intensities of $D_{1\theta,}$ and $D_{2\theta}$ components are maintained, but new component E_θ (110)[$1\bar{1}1$] is formed and becomes intense.

The presence of pearlite influence the deviation of texture components from their ideal position at $\varepsilon_{vm}<9$ and deviation gets reduced at high strain level as cementite get dissolved partially. The pearlitic carbides allows the formation of E_θ at $\varepsilon_{vm}>3$ whose intensity reaches maximum 17 at ε_{vm}=12.

Chapter-5 gives the strengthening mechanism in UFG low carbon steel produced through ECAP. At low strain (0.6 to 1.8), strengthening takes place with rapid rate due to rapid grain refinement and increase in dislocation density. In intermediate strain (ε_{vm} = 3 - 6) pumping of dislocations and their annihilation by dynamic recovery occur simultaneously. Reduced grain refinement rate, annihilation of dislocation and increase in average misorientaion angle of grain boundaries, leads to enhancement in strength at lower rate. At higher strain level (ε_{vm} = 9 - 16.8), strength increases due to increase in average misorientaion angle, significant dissolution of cementite in ferrite matrix and reduction in size of carbides. Ultimate tensile strength of the low carbon steel increases from 367 MPa (as-received) to 1009 MPa after ε_{vm} = 16.8. Low carbon steel fails by ductile fracture at early stage of deformation by ECAP but brittle or shear fracture area increases with increasing imposed strain. As a result at ε_{vm} = 16.8, the material fails mainly by cleavage or brittle fracture.

Chapter-6 deals with cold-rolling and cryo-rolling of ECAP-12 and ECAP-16,8 respectively. ECAP followed by cold rolling of low carbon steel for 80% reduction in area refines the material to ultrafine level (band width = 0.11 µm) with high dislocation density. Bulk nanostructured low carbon steel of grain size of 87 nm is produced by ECAP at ε_{vm} = 16.8 followed by cryorolling at -50ºC for 75% reduction in area. Flash annealing of ECAPed+cold-rolled low carbon steel above the secondary recrystallisation temperature for a short duration results in bimodal grain size distribution of ultrafine (0.8µm size, 73% volume) and micron-sized (9µm size, 27 % volume) ferrite grains and a small amount of cementite precipitates. UFGs and precipitates endow the material with high strength, and micron-sized grains allow plastic deformation. ECAP followed by cryo-rolling and flash annealing of low carbon steel at 600°C for 5 minutes produces novel microstructure of bimodal grain size distribution of UFG and micron sized grains.

Chapter-7 deal with the mechanical properties of post ECAP processed steels. Grain refinement in ultrafine level after cold rolling of ECAPed at ε_{vm} =12 and high dislocation density provide ultrahigh strength (YS> 900 MPa) but reduces ductility, and the material fails by cleavage fracture due to development of a $(110)[1\bar{1}1]$ texture component at the expense of a γ fiber component of $(111)[1\bar{2}3]$. Ultimate tensile strength of bulk nanostructured steel increases to 1238 MPa after cryo rolling of ECAP-16.8. The ductility of the material is drastically dropped due to high defect density and non-equilibrium nature of boundaries of nano-grains.

Flash annealing at 873 K (600°C) of UFG low carbon steel develops required bimodal grain size distribution in ferrite (fine grain size 0.8µm with 73% volume and coarse grain size 9µm with 27% volume) with a small amount of fine cementite precipitates, where ductility is recovered due to the optimum quantity of micron-sized ferrite grains and precipitates with development of a good amount of γ fiber texture components of (111)[110] and (111)[$\bar{1}\bar{4}$5], where a major contribution toward ductility comes from bimodal grain size distribution UFG and micron sized grains in ferrite rather than precipitation of cementite. Cryo-rolling and flash annealing of ECAPed low carbon steel also produces a novel microstructure of bimodal grain size distribution of ultrafine-grains and micron-sized-grains due to the combination of recrystallisation and secondary recrystallisation respectively.

Chapter-8 deals with the conclusions of the thesis. Microstructural refinement upto a grain size of 0.2µm with average misorientation of ~40°, has been achieved in a coarse-grained low carbon steel by deformation upto ε_{vm}=16.8 at room temperature The rate of refinement is high at initial stages but it decreases with the amount of imposed strain. At initial stages of deformation most of the grain boundaries are of a low angle of misorientation. Lowest level of low angle grain boundary fraction is < 20% at ε_{vm}=16.8. Defect density increases with imposed strain and reaches a maximum value at intermediate strain but decreases to lower level at higher strain.

On ECAP the grains get elongated at the initial stage of deformation (ε_{vm}=0.6) and subdivided into bands. At intermediate strain (ε_{vm}=1.8) a cellular structure develops within the bands due to the rearrangement of dislocations. At high strain (ε_{vm}=9-12), bands are further elongated and form ribbon grains. These ribbon grains are broken down partially to near-equiaxed grains due to the intersection of bands. At large strain (ε_{vm}=16.8) cellular structure gets converted into granular one. In the low carbon steel pearlitic cementite starts dissolving at equivalent strain low as 6 and the lamellae are partially broken. The dissolution of cementite continues with strain but complete dissolution could not be achieved within the equivalent strain of 16.8.

The material is strongly textured upon ECAP with imposed strain. New components are developed at different strain levels. At low to intermediate strain level F_θ, J_θ, $\bar{J}_\theta$, $D_{1\theta}$, and F_θ texture components typical of pure shear are formed. At high strain level new component E_θ is formed. The positions of components are nevertheless deviated from their ideal position. The deviation decreases with the dissolution of cementite in ferrite matrix.

Strength and hardness increase rapidly at early stages of deformation due to rapid rate of grain refinement and increase in defect density but the rate of strengthening and hardening decreases with strain as refinement rate and dislocation density decrease. An ultrahigh level of ultimate tensile strength greater than 1000 MPa has been achieved at $\varepsilon_{vm} = 16.8$ but failure takes place by brittle fracture at high strain level $\varepsilon_{vm}=16.8$. The ECAPed low carbon steel of ultrafine-grain structure is further deformed to greater than 75% reduction in area by cold rolling or cryorolling and the material has been refined to ~100 nm.

Bimodal grain size distribution consisting of ultrafine grains and micron-size grains of ferrite and development of γ-fiber texture component are obtained by post ECAP deformation and followed by optimized thermal treatment where ultrafine grains offers high resistance to deformation, i.e. high strength but micron sized grains adds ductility for the material by changing the nature of failure from brittle to the ductile fracture.

The chapter also provides suggestions for future work. At the end of the thesis list of references is given.