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## Summary and Suggestions for Future Work

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### 8.1 Summary of the Present Work

Ti+Nb stabilized interstitial-free steel (IF) can be deformed by the equal-channel angular pressing (ECAP) upto very large equivalent strain of 24 by adopting route Bc. The degree of reduction in the grain size is highest at low strain level and thereafter it decreases. However, most of the boundaries at this stage are low angle boundaries created by the rearrangement of the dislocations (at  $\epsilon_{vm}=3$ ). The microstructural evolution which strongly depends on amount of equivalent strain, involves the elongation of grains at  $\epsilon_{vm}=0.6$ , the subdivision of grains to the bands with high dislocation density. With increasing strain the bands split to cell blocks and finally cell blocks to cells at  $\epsilon_{vm}=1.8$ . The width of bands and the size of cells decrease with increase in the equivalent strain. Major high angle boundaries (original high angle boundary, boundaries of bands and cell block boundaries) align to deformation direction at  $\epsilon_{vm}=3$  and form lamellar structure at  $\epsilon_{vm}=6$ . The inter lamellar spacing decreases with strain and finally becomes one subgrain wide at  $\epsilon_{vm}=9$  and the grains take ribbon shape. With further strain the ribbon grains get sheared partially into near-equiaxed grains. The transverse boundaries of subgrains interacted with dislocations and increase its misorientation angle. At large strain  $\epsilon_{vm}=15$ , the ribbon grains split into the near-equiaxed grains. At very large strain  $>15$ , the ribbon shaped grains transform to the near-equiaxed grain structure and fragmentation continues till  $\epsilon_{vm}=18$ . At  $\epsilon_{vm}=21-24$ , the grain size almost remain unchanged. It is interesting to be

noted that even though the size the size remains unchanged, the microstructure is metastable at large equivalent strain of 24 containing low angle grain boundary fraction of 0.34. Finally at  $\varepsilon_{vm}=24$ , average grain size, low angle grain boundary area fraction and average misorientation angle stabilize to respective saturation values.

ECAP processing of IF steel develops a strong shear texture. The texture intensity continuously increases with strain even though saturation in grain refinement reaches saturation at higher strain. Texture is correlated to properties of materials such as strength, work hardening, grain refinement and fracture. At low strain level,  $\varepsilon_{vm}=0.6$ , components of  $\{110\}$  fiber,  $J_\theta$ ,  $\bar{J}_\theta$  and common components of both  $\{110\}$  and  $\langle 111 \rangle$  fibers,  $E_\theta$ ,  $\bar{E}_\theta$  are existing but their intensity is low. At intermediate strain  $\varepsilon_{vm}=1.8-6$ ,  $J_\theta$ ,  $\bar{J}_\theta$  and  $D_{1\theta}$ ,  $D_{2\theta}$  components are intensifying. At higher strain range,  $\varepsilon_{vm}=9-24$ ,  $\langle 111 \rangle$  fiber texture forms with high intensity of main components,  $D_{1\theta}$ ,  $D_{2\theta}$ . At low strain level, major components get deviated from their exact position level and come to their ideal position at large strain level. Monoclinic symmetry is noticed after  $\varepsilon_{vm}=9$ . At  $\varepsilon_{vm}=24$ , texture index reaches a very high value of 3.6 times the value of as-received IF steel. At  $\varepsilon_{vm}=0.6$ , coarse grains begin to align in the direction of deformation along with splitting into deformation bands. Alignment of cells and cell blocks (at  $\varepsilon_{vm}=2.4$ ) in deformation bands lead to increase in texture intensity with concentrated clouds of  $\bar{J}_\theta$ ,  $J_\theta$  components. Fragmentation of bands (at  $\varepsilon_{vm}=3$ ) randomise orientations that suppresses  $\bar{J}_\theta$ ,  $J_\theta$  components. At  $\varepsilon_{vm}=6$ , high angle grain boundaries, cell blocks and cell structures get aligned to deformation direction to form lamellar structures with mainly  $D_{1\theta}$ ,  $D_{2\theta}$  as dominating components. At  $\varepsilon_{vm}=9$ , oriented ribbon grains result in strong  $D_{1\theta}$ ,  $D_{2\theta}$  components with  $\langle 111 \rangle$  fiber. At  $\varepsilon_{vm}=15$ , partial conversion of ribbon grains to near-

equiaxed shaped grains produces enhanced intensity of  $D_{1\theta}$  and  $D_{2\theta}$  components. At  $\epsilon_{vm}=15-21$ , grain refinement attains saturation with further increase in intensity of  $D_{1\theta}$ ,  $D_{2\theta}$ . At  $\epsilon_{vm}=24$  results near-equiaxed grain formation complete and leads to increased intensity of  $D_{1\theta}$ ,  $D_{2\theta}$  components.

The yield strength and the ultimate tensile strength increase sharply upto  $\epsilon_{vm}=3$  due to the rapid microstructural refinement with high defect density. Thereafter, the strength increases appreciably upto  $\epsilon_{vm}=9$  as LAGB fraction decreases and average misorientation angle increases. The strengthening continues to occur even upto  $\epsilon_{vm}=24$ , as the increase in high angle grain boundary fraction and average misorientation angle take place although the grain refinement at  $\epsilon_{vm}>9$  is not significant. The strengthening of selected IF steel which increases from 227 MPa to 895 MPa by ECAP for  $\epsilon_{vm}=24$  at 298K (25° Celsius) is itself noteworthy. The uniform elongation of the IF steel reduces to 0.5% by ECAP due to a lack of work hardening ability at a low strain level ( $\epsilon_{vm}=0.6$ ). With increasing strain the elongation improves marginally by 1.5-2% upto  $\epsilon_{vm}=9$ , and thereafter it remains almost constant. The ECAPed IF steel fails by ductile fracture at lower range of  $\epsilon_{vm}=0.6-6$  but by the mixed mode of ductile-brittle fracture at larger equivalent strain (9-24). The strategy to enhance the ductility further proceeds to be a successful method as described below. From the ECAP processing alone, one could achieve enhanced strength by grain refinement which saturates from strain levels of 21. Therefore additional methods of pumping in more dislocations to further reduce grain size were adopted and found to yield expected results.

The ECAPed IF steel can be deformed further by coldrolling/cryorolling at 223K (-50°C) to >90% reduction in area. The post ECAP deformation of IF steel by cold

rolling/cryorolling to 90% reduction in area decreases the grain size and improves the area fraction of the high angle grain boundaries. When ECAPed IF steel samples are cryorolled heavily stressed non-equilibrium grain boundaries are formed. No sharp  $\gamma$  fiber is formed by coldrolling of ECAPed material but cryorolling of ECAPed IF steel results in formation of sharp  $\gamma$  fiber. The enhancement in high angle grain boundary fraction, the non-equilibrium boundaries and the reduction in grain size strengthen the material significantly. Decrease in grain size to ultrafine level with increased lattice strain that lowers work hardening ability of the material and that consequently limits its ductility.

Though cryorolling is effective in further refining the microstructure and enhance desirable texture component, it was in effective in producing bimodal distribution of grain sizes, a key to gaining ductility. The second strategy for this was to subject the above processed steel to flash annealing treatment. The bimodal grain size distribution in ultrafine-grains range has been successfully achieved by flash annealing the IF steel processed by equal-channel angular pressing. The recrystallization temperature of ultrafine-grained IF steel decreases with increase in equivalent strain. The hardness of severely deformed IF steel can be maintained upto 923K (650°C) at least for short duration of 300 seconds. The temperature of abnormal grain growth in IF steel increases with decrease in equivalent strain.

When UFG IF steel samples processed by ECAP at  $\epsilon_{vm}=12$  followed by coldrolling/cryorolling to >90% reduction in area are flash annealed at 948K (675°C), the materials are partially recrystallized. The increased subgrain size, the grain size and reduced residual lattice strain lower the hardness and the strength with marginal recovery of ductility. But lack of dislocation activities due to reduced grain size and residual lattice

strain fail to recover the ductility to the level of coarse-grained as-received steel even though at that condition the yield strength is maintained at 2-3 times to that of the same.

## **8.2 Suggestions for Future Work**

Future prospects and plans include high resolution microscopy of UFG materials to explore details of refining and migration mechanisms of grain boundaries and dislocations. Recovery, recrystalliation and grain growth takes place by grain boundary migration mechanism during annealing also boundary migration is activated during equiaxed grains formation by dynamic recrystallization process in which new grains of high angle misorientation are formed via progressive rotation of subgrains. It is also mandatory to explore equilibrium state of grain boundaries as there is supposed be high diffusion activity in non-equilibrium state of grain boundaries (introduced by absorption of lattice dislocations) which changes behavior with heat treatment. There is substantial effect of non-equilibrium grain boundaries on uniaxial tensile properties. There is also a need to understand the nature and establish a correlation between the level of structural order of grain boundary (or non-equilibrium state), triple junctions and mechanical properties of materials. In the present thesis correlation of microstructure with texture is already established but there is still need to establish correlation of texture with selected mechanical properties of the material.

There are reports which proof that grain refinement as strengthening mechanism also increases the toughness to reduce weight and improve safety performance in transportation and heavy machinery. But IF steel containing BCC crystal structures are very sensitive to a decrease in impact toughness during service under impact loading. In the present thesis, IF steel is refined to saturation grain size to find saturation strength but

toughness and ductile to brittle transition temperatures are not determined with refinement.

IF steels contain very low amount of interstitials (C, N, and O) in their ferritic matrix which make them promising candidate for applications in the automotive industries for the fabrication of complicated body parts due to their high formability and planar anisotropy. In the present thesis, studies on mechanical properties (tensile strength, hardness, ductility) of ultrafine-grained IF steel are conducted however the formability or workability of materials after ECAP process can be worth to evaluate in future.