
Effect of Flash Annealing on UFG IF Steel Processed by ECAP and ECAP+Coldrolling/ Cryorolling

7.1 Microstructure

The microstructure of ECAP-3 (ECAP-3-FA550) IF steel, consists of bands of width 298 ± 30 nm containing cells. The bands are aligned towards deformation direction. The microstructure of ECAP-3-FA550 consists of recovered banded structure of average band width of 444 ± 54 nm (Figure 7.1a). Annealing of ECAP-3 at 873K (600°C), i.e. the ECAP-3-FA600 sample shows elongated grains with the recovered structure but with subgrain size of 448 ± 35 nm (Figure 7.1b). When ECAP-3 sample is flash annealed at 923K (650°C), the morphology of grains is maintained (Figure 7.1c) but subgrain growth could be observed. When ECAP-3 samples are annealed at 948K (675°C), the microstructure contains fine subgrains of 409 ± 11 nm along with coarse subgrains of 1.4 ± 0.2 μm (Figure 7.1d, 7.2a) i.e. subgrain growth occurs. Further increase in annealing temperature to 973K (700°C), the material gets recrystallized and the microstructure show bimodal grain size distribution where equiaxed grains coexist with abnormally grown grains (Figure 7.1e, 7.2b). The microstructure consists 77% fine grains of average size of 620 ± 28 nm and 23% of coarse grains of average size of 1.5 ± 0.24 μm (Figure 7.1e, 7.2b, Table 7.1). On annealing of ECAP-3 at 998K (725°C), grains are uniformly coarsened to 1.7 ± 0.38 μm (Figure 7.1f, 7.2c). Flash annealing at 1023K (750°C) results in coarsening of maximum fraction of grains in the microstructure to size 2.6 ± 0.36 μm (Figure 7.1g, 7.2d).

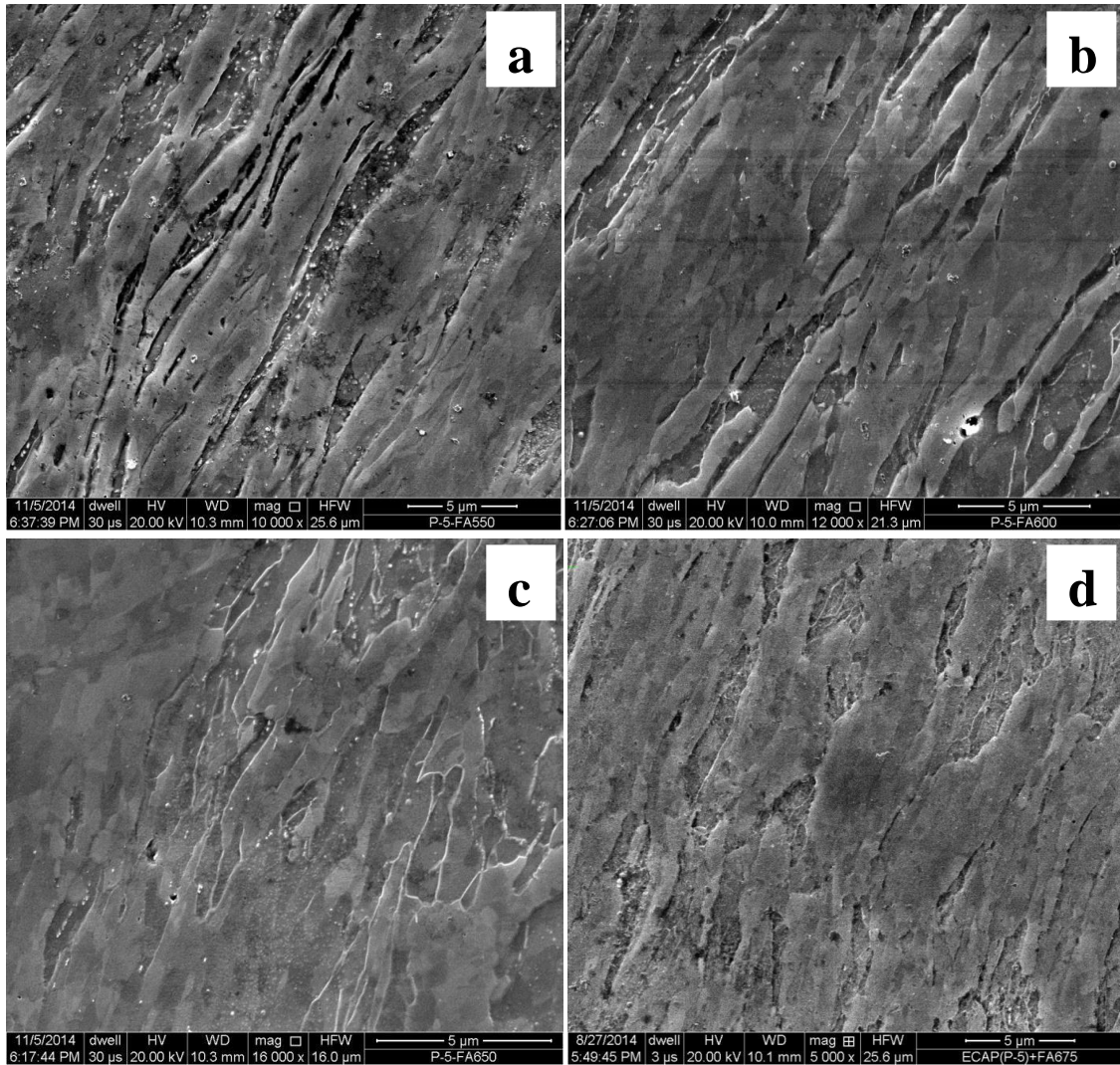


Figure 7.1: SEM images of IF steel deformed upto $\epsilon_{vm}=3$ and flash annealed (a) ECAP-3 FA550, (b) ECAP-3-FA600, (c) ECAP-3-FA650, (d) ECAP-3-FA675.

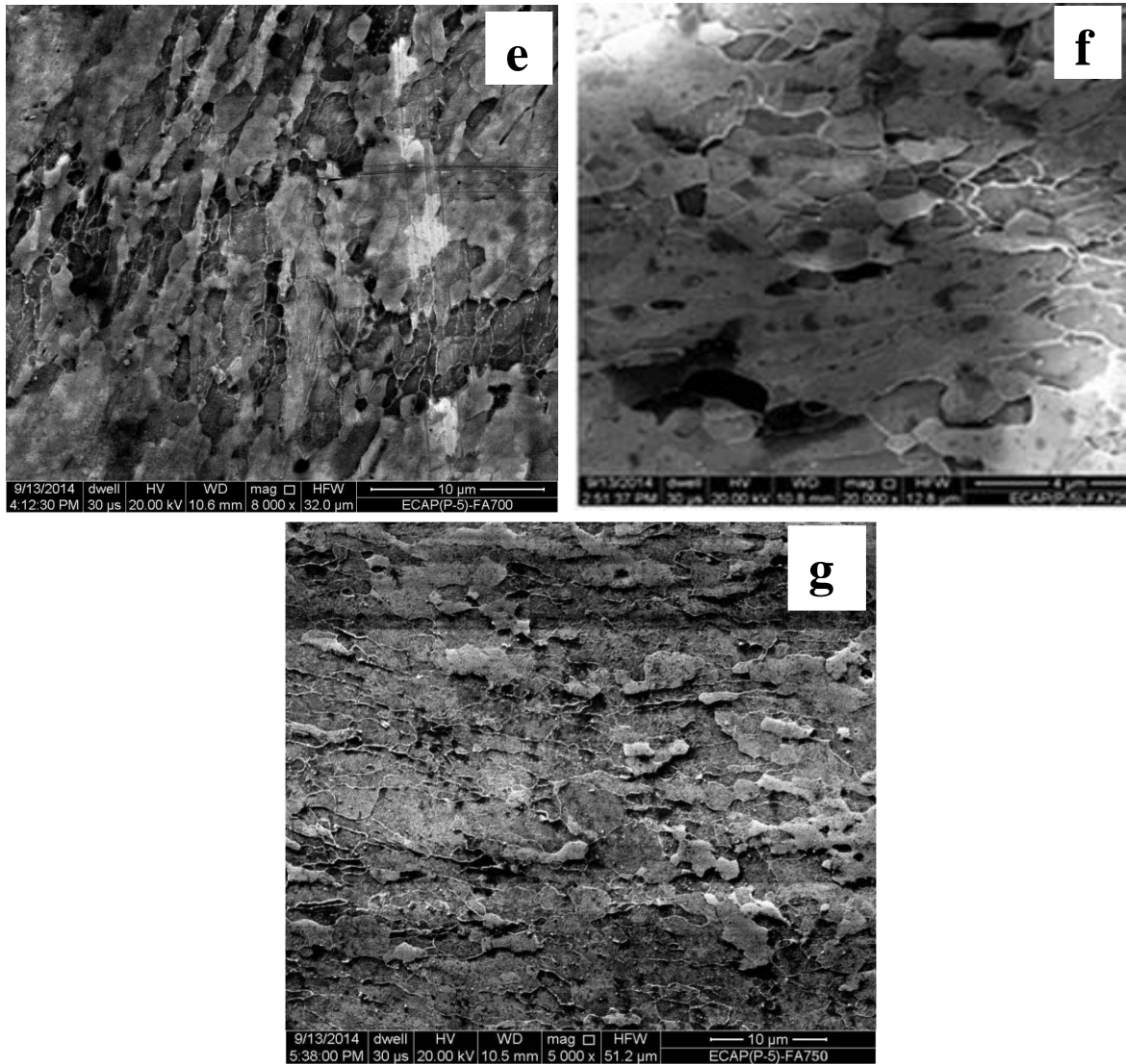


Figure 7.1: SEM images of IF steel deformed upto $\epsilon_{vm}=3$ and flash annealed (e) ECAP-3-FA700, (f) ECAP-3-FA725 and (g) ECAP-3-FA750.

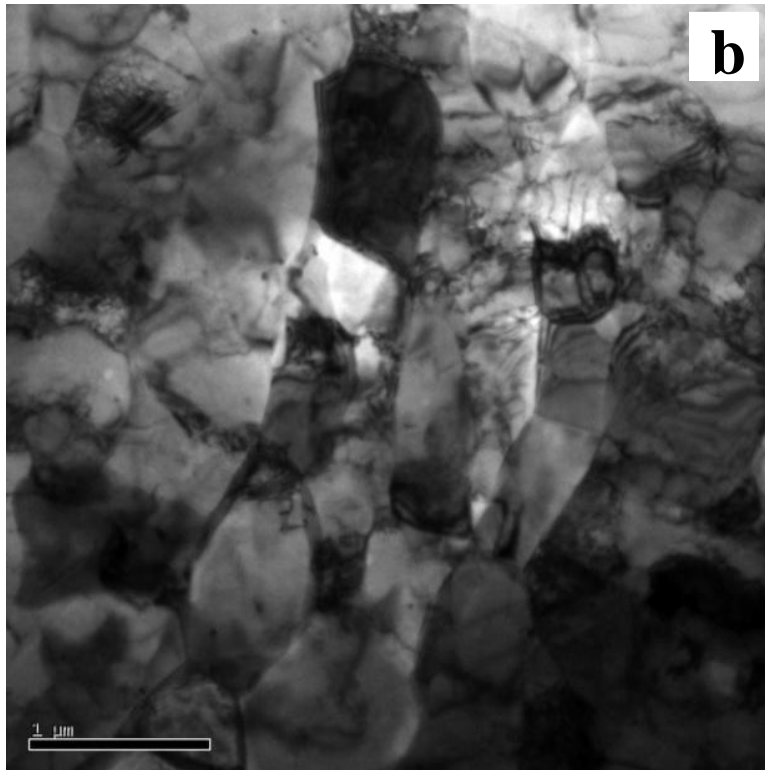
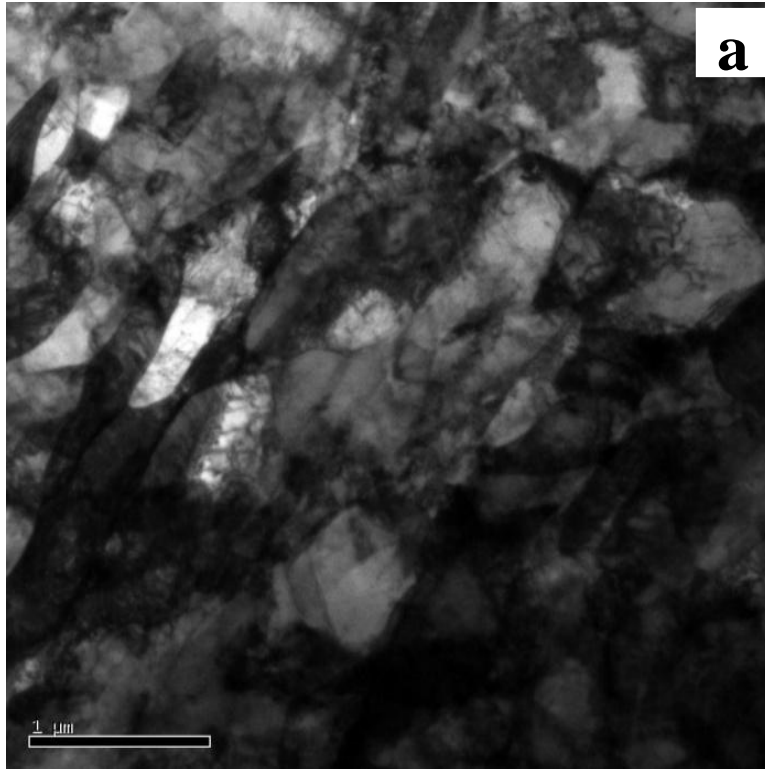


Figure 7.2: TEM bright field images of IF steel deformed upto $\epsilon_{vm}=3$ and flash annealed (a) ECAP-3-FA675 and (b) ECAP-3-FA700.

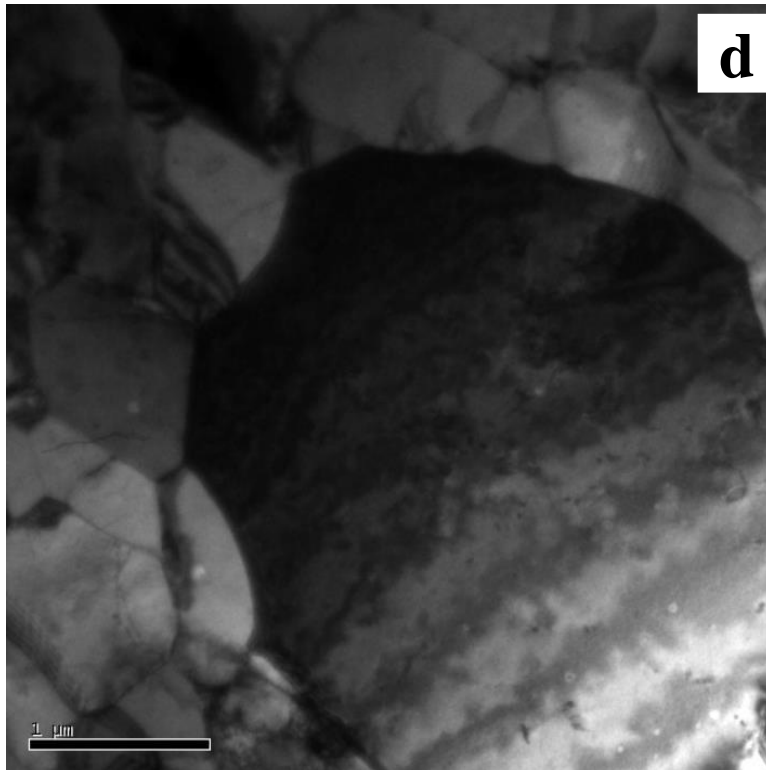
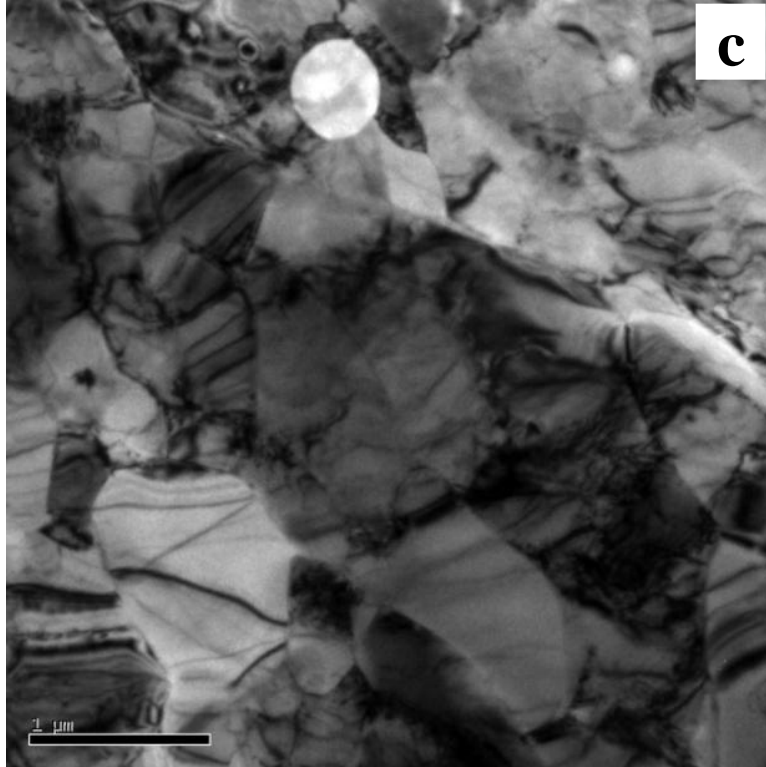


Figure 7.2: TEM bright field images of IF steel deformed upto $\epsilon_{vm}=3$ and flash annealed (c) ECAP-3-FA725 and (d) ECAP-3-FA750.

The microstructure of ECAP-12 IF steel consists of ribbon shaped grains of single subgrain wide and width becomes 196 nm (Figure 3.3i,3.3i). On flash annealing at 873K (600°C), some of the ribbon grains partially convert to near equiaxed grains of size 452 nm (Figure 7.3a). On flash annealing of ECAP-12 IF steel at 898K (625°C), most of the ribbon grains get recrystallized to equiaxed shape and produces bimodal grain size distribution (Figure 7.3b). The finer grains occupying 78 volume fractions are of size 417 nm whereas a few coarse grains are also observed in the microstructure. While ECAP-12 IF steel is flash annealed at 923K (650°C), the bimodality of grain size widens, containing 43% of fine grains i.e. the volume fraction of fine grains decreases. The fine grain size reaches 521 nm but coarse grains grow to 1270 nm size (Figure 7.4a). On flash annealing at 948K (675°C), volume fraction of fine grains further decreases to 29% with an increase in volume fraction (Table 7.1). The size of fine grains is about 367 nm and that of coarse grains is about 1440 nm (Figure 7.4c).

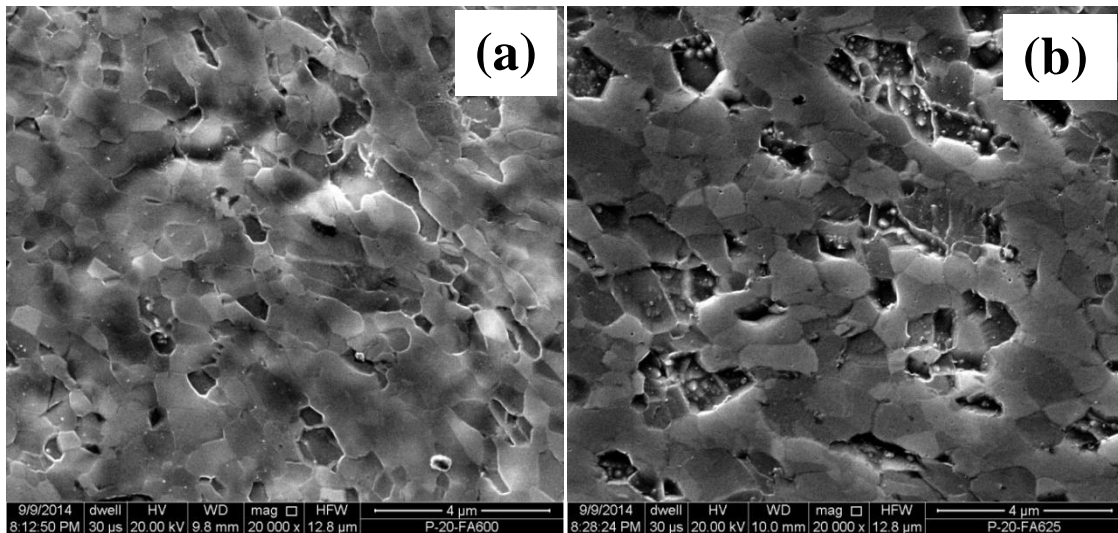


Figure 7.3: SEM images of IF steel deformed upto $\epsilon_{vm}=12$ and flash annealed (a) ECAP-12-FA600 and (b) ECAP-12-FA625.

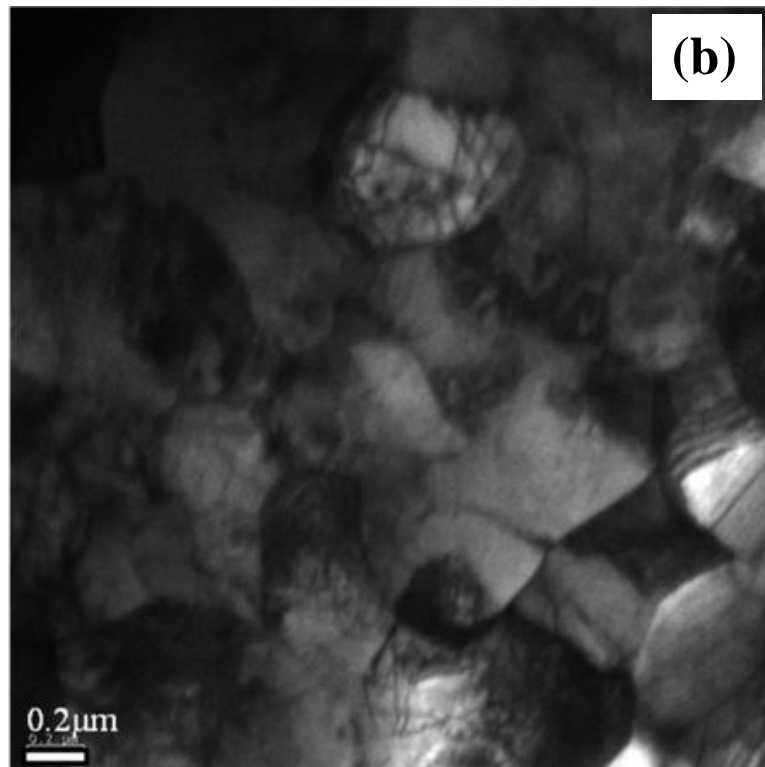
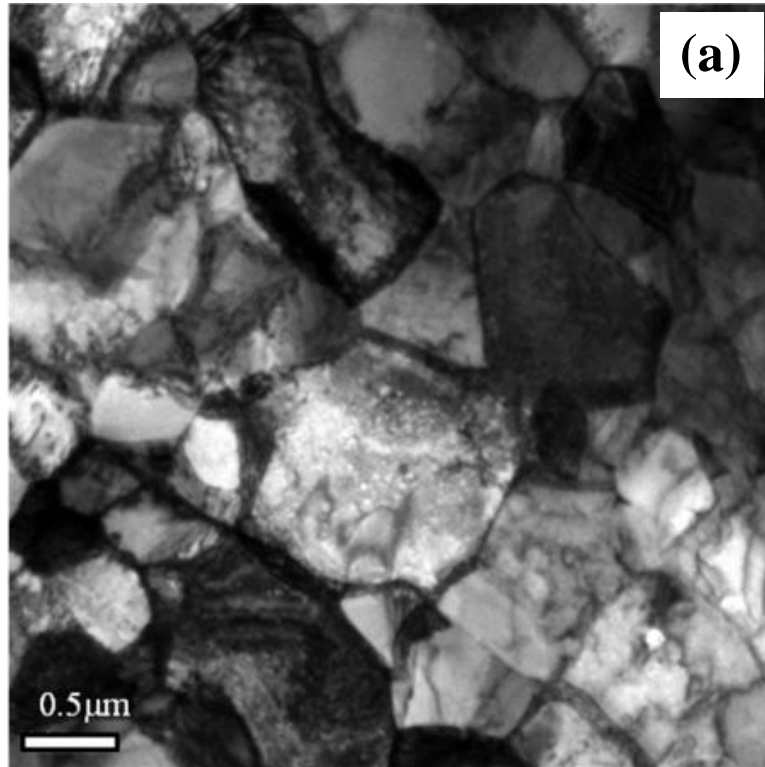


Figure 7.4: TEM bright field images of IF steel after (a) ECAP-12-FA650 and (b) ECAP-12-FA675.

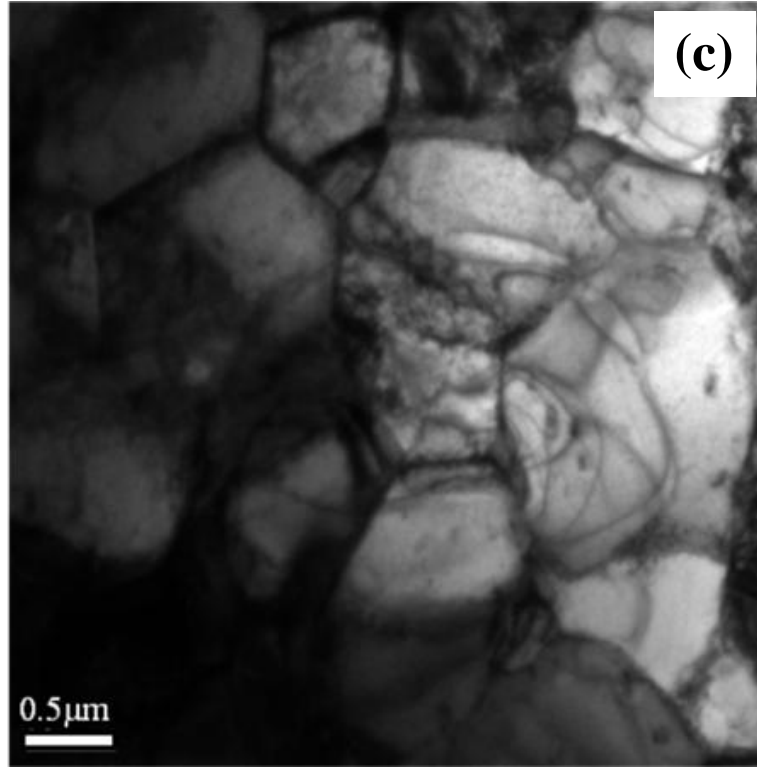


Figure 7.4: TEM bright field images of IF steel after (c) ECAP-12-FA675.

At $\varepsilon_{vm}=21$, the microstructure consist of near-equiaxed ultrafine-grained structure which is developed progressively by shearing of ribbon grains and average grain size reaches 256 ± 61 nm (Figure 3.3k). On flash annealing of ECAP-21 sample at 873K (600°C), the average size of near-equiaxed fine grains increases to 377 ± 94 nm (Figure 7.5a, 7.6a) but the elongated shape is maintained. Due to recovery, grain boundaries existing in deformed state begin to migrate and width of few grains increases to 665 ± 12 μm . When ECAP-21 sample is annealed at 898K (625°C), recrystallization begins and microstructure contains 93% fine grains of average size 388 ± 83 nm size with 7% coarse grains of average size of 1.2 ± 0.26 μm (Figure 7.5b, 7.6b, Table 7.2). On flash annealing at 948K (675°C), the material is recrystallized that results in bimodal grain size distribution. The microstructure contains 81.5% recrystallized grains of average size of 455 ± 60 nm and 18.5% of abnormally grown grains of 1.3 ± 0.21 μm (Figure 7.3c, 7.4c).

Further flash annealing at 973K (700°C) leads to uniform coarsening of grains to $3.8 \pm 0.39 \mu\text{m}$ (Figure 7.5d, 7.6d).

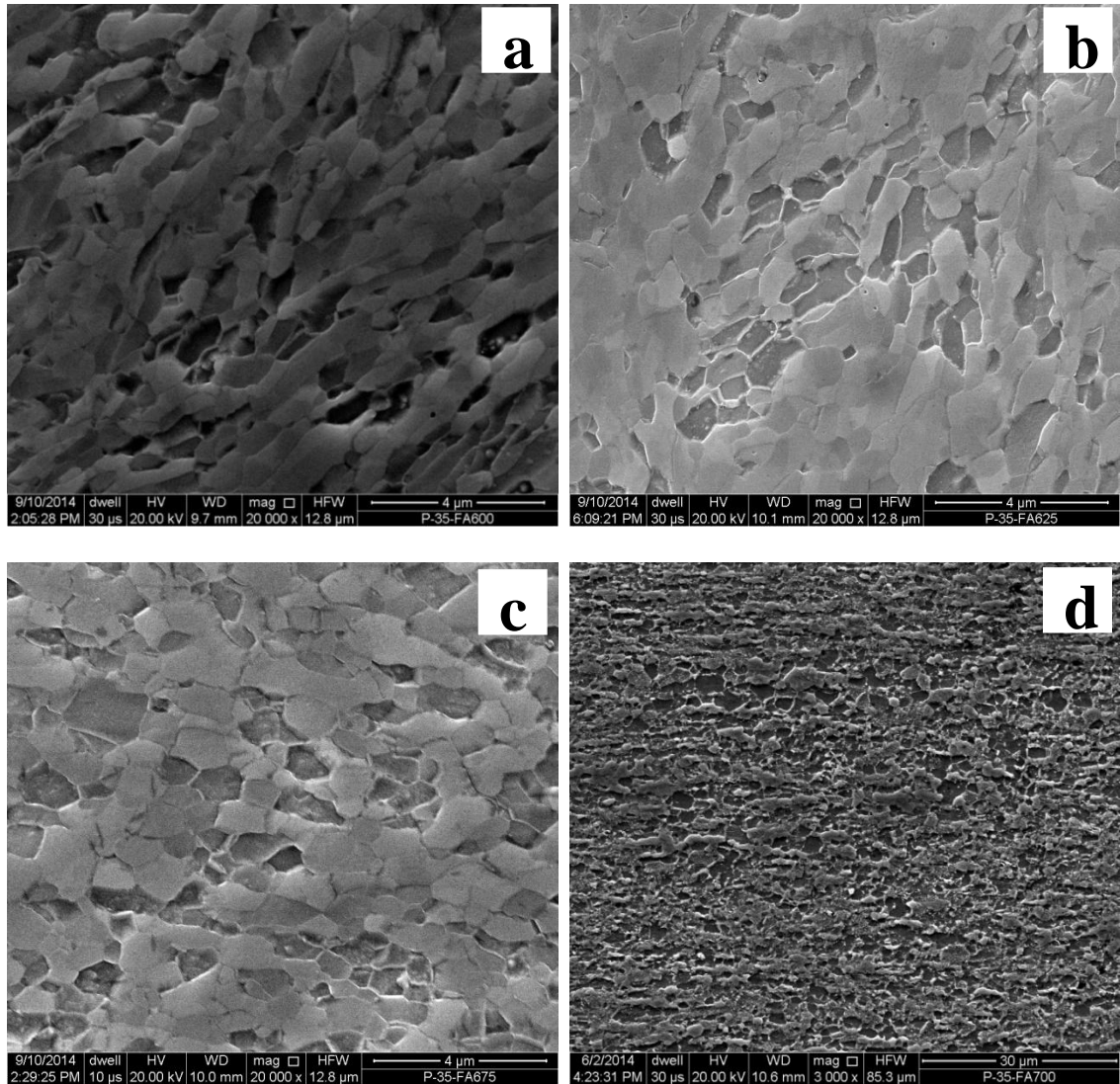


Figure 7.5: SEM images of IF steel deformed up to $\epsilon_{vm}=21$ and flash annealed (a) ECAP-21-FA600, (b) ECAP-21-FA625, (c) ECAP-21-FA675 and (d) ECAP-21-FA700.

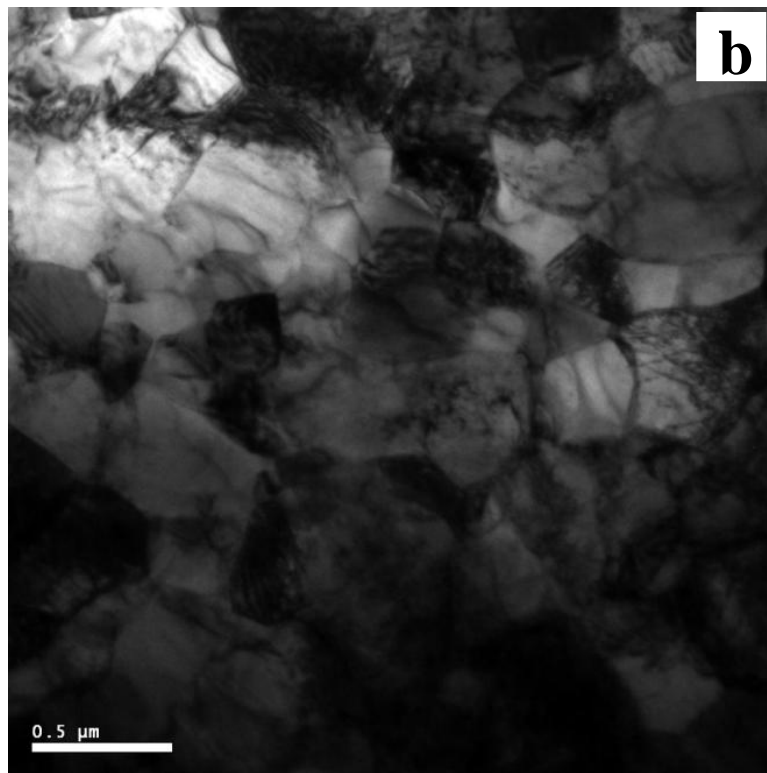
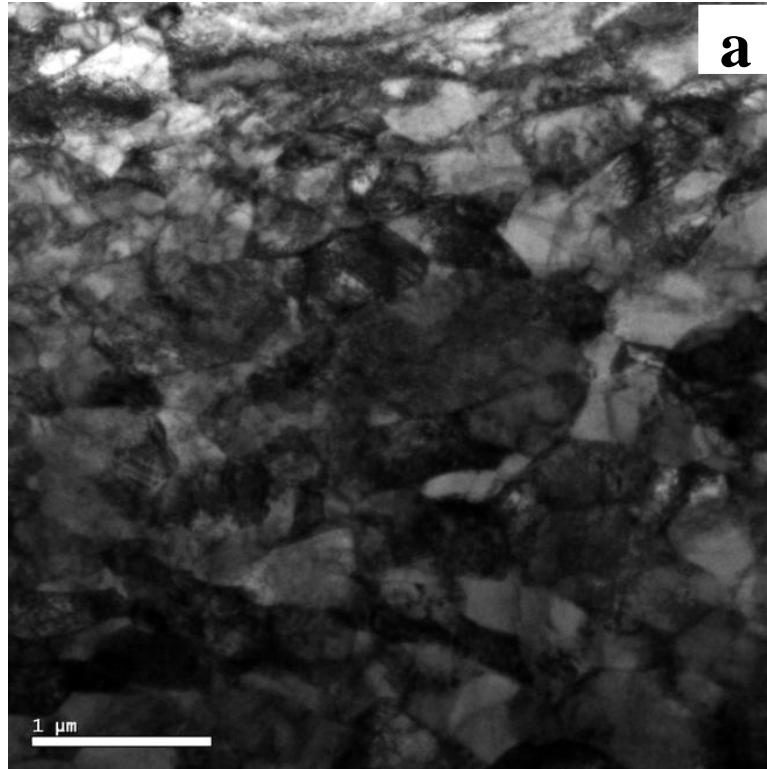


Figure 7.6: TEM images of IF steel deformed upto $\epsilon_{vm}=21$ and flash annealed (a) ECAP-21-FA60 and (b) ECAP-21-FA625.

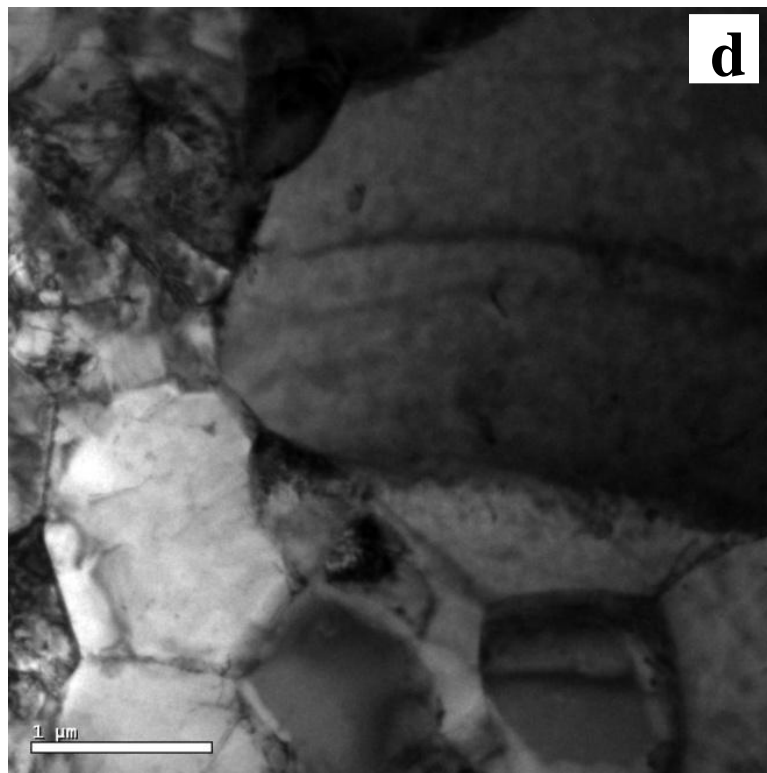
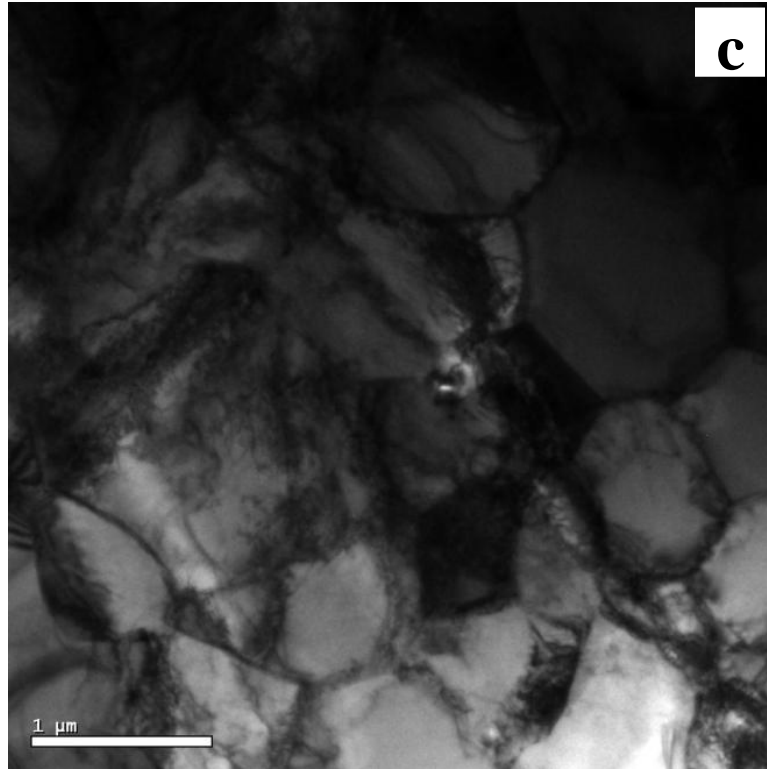


Figure 7.6: TEM images of IF steel deformed upto $\epsilon_{vm}=21$ and flash annealed (c) ECAP-21-FA675 and (d) ECAP-21-FA700.

Table 7.1: Details of microstructure of ECAPed and flash annealed samples.

Sample	Volume Frac. of fine grains	Size of coarse grains	Size of fine grains
As- received	-	57±21 µm	-
ECAP-3	-	-	298±30
ECAP-3-FA550	-	-	444±54
ECAP-3-FA600	-	-	448±35
ECAP-3-FA650	-	-	459±87
ECAP-3-FA675	-	-	409±11
ECAP-3-FA700	77	-	620±28
ECAP-3-FA725	-	1.7±0.4 µm	-
ECAP-3-FA750	-	2.6±0.36 µm	-
ECAP-12	-	-	196±38
ECAP-12-FA 600	-	-	452±42
ECAP-12-FA 625	78	-	417±65
ECAP-12-FA-650	43	1.3±0.3 µm	521±54
ECAP-12-FA-675	29	1.4±0.1 µm	367±32
ECAP-12-FA-700	-	-	-
ECAP-21	-	-	256±61
ECAP-21-FA600	-	-	377±94
ECAP-21-FA625	-	-	388±83
ECAP-21-FA675	81.5	1.3±0.2 µm	455±60
ECAP-21-FA700	-	3.8±0.39 µm	-
ECAP-12-CR-90	-	-	204±24s
ECAP-12-CR-90-FA650	53.9	1.6±0.5 µm	590±61
ECAP-12-CR-90-FA675	41	1.7±0.8 µm	620±23
ECAP-12-CR-90-FA700	-	1.9 ±0.9 µm	407±44
ECAP-12-CRR-96	-	-	127±17
ECAP-12-CRR-96-FA550	-	-	611±86
ECAP-12-CRR-96-FA600	-	630±56 nm	412±59
ECAP-12-CRR-96-FA650	62	1213±75 nm	504±56
ECAP-12-CRR-96-FA675	-	-	564±23

When ECAPed ($\epsilon_{vm}=12$) IF steel is cold rolled to 90% reduction in area, the material is dynamically recovered and dislocations are arranged to cell walls and dislocation tangles are formed but most of the cell interiors are dislocation free (Figure

6.3b). The domain size reduces to 34 nm and lattice strain increases to 0.23%. Flash annealing of ECAP-12-CR90 at 650°C produces inhomogeneous microstructure with dislocations distributed in the interior of grains (Figure 7.7a). Grain size of coarse grains is 1674 nm and of fine grains becomes 590 nm. While ECAPed ($\epsilon_{vm}=12$) and cold rolled samples are flash annealed at 948K (675°C) (Figure 7.7b) uneven coarsening of grains occurs with reduced dislocation density. Few grains have got coarsened to very large size of 1715 nm whereas size of fine grains remains at 620 nm. The domain size is increased to 133 nm but elastic stored energy is reduced from cold rolled condition to 45.6 Joule/mole (Table 7.2). Flash annealing at 700°C leads to coarsening of most of the grains to ~1900 nm (Figure 7.7d).

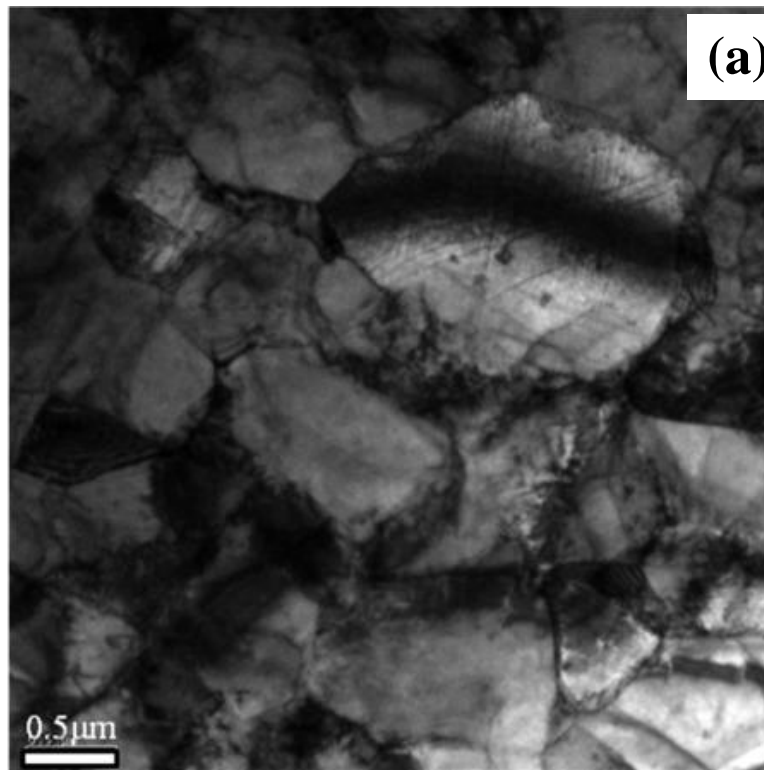


Figure 7.7: TEM bright field images of IF steel after (a) ECAP-12-CR-90-FA650.

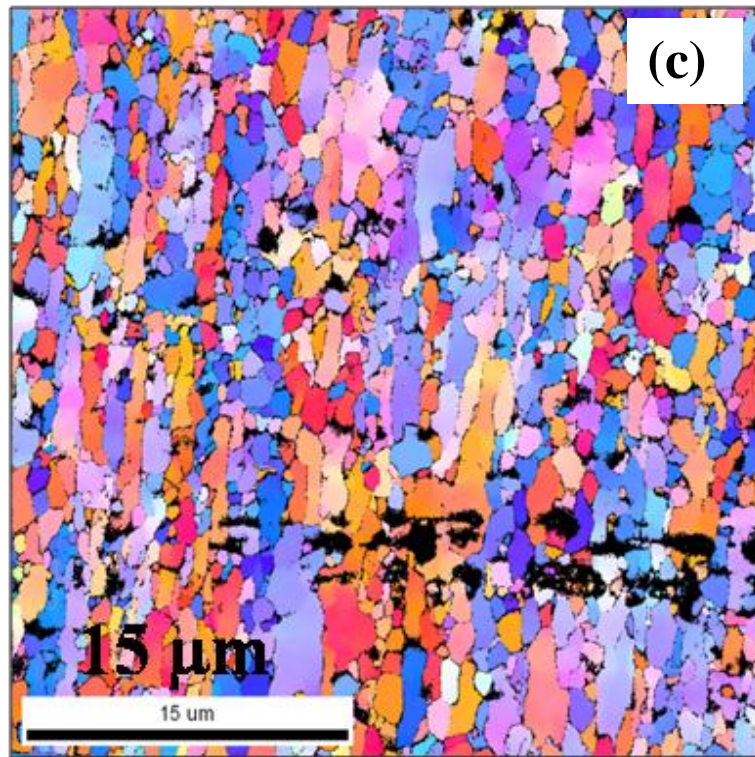
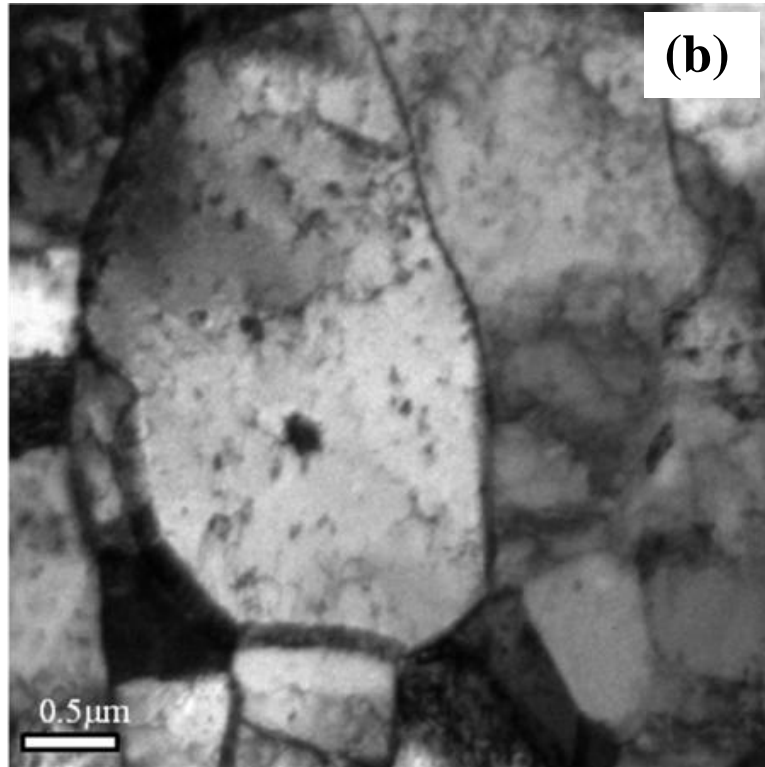


Figure 7.7: (b) and (c) TEM bright field images and [001] IPF maps of IF steel after ECAP-12-CR90-FA675.

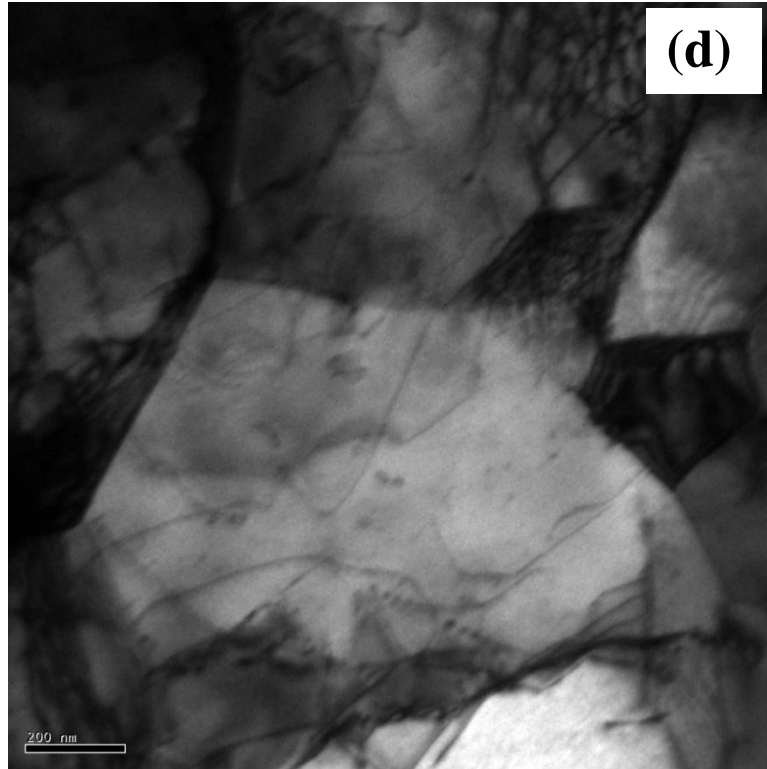


Figure 7.7: (d) TEM bright field image of ECAP-12-CR90-FA700.

When ECAPed billet is cryorolled to 96% reduction in area (ECAP-12-CRR-96), the microstructure contains equiaxed and elongated grain structure of average size of 127 nm with diffuse grain boundaries and scattered dislocation network in the interior of grains. The domain size decreases from 41 nm to 36 nm and elastic stored energy is increased (Figure 6.3d). Appreciable dislocation density is observed in the samples after ECAP followed by cryorolling and flash annealing at 773K (550°C). Further increase in flash annealing temperature at 873K (600°C) the microstructure contains comparatively less amount of dislocations and cell structures having dislocation free interior. Grain boundaries are not well defined but wavy in nature which means that dislocations are not annihilated and there is no sign of recrystallization and grain size is maintained at 412 nm (Figure 7.8a). Grain size remains almost unchanged even when the cryorolled samples (ECAP-CRR-96) are flash annealed at 873K (600°C) (Figure 7.8b) whereas small

fraction of recrystallized grains are also observed at few locations. Flash annealing of ECAP-12-CRR-96 at 923K (650°C) leads to bimodal grain size distribution having fine grains of 504 nm (Figure 7.8c) of ~64% volume fraction and the remaining coarse grains of 1213 nm (Figure 7.8d). ECAP-12-CRR-96-FA675 (Figure 7.8e), contain the elongated as well as equiaxed recrystallized grains of 564 nm. When ECAP-12-CRR-96 samples are flash annealed at 948K (675°C), the domain size increases to 44.8 nm, elastic stored energy reduces to 45 Joule/mole and lattice strain decreases to 0.19% (Table 7.2) compared to its deformed state. Average misorientation angle increases to 26.4° and the HAGB fraction increases to 57.6% (Table 7.2).

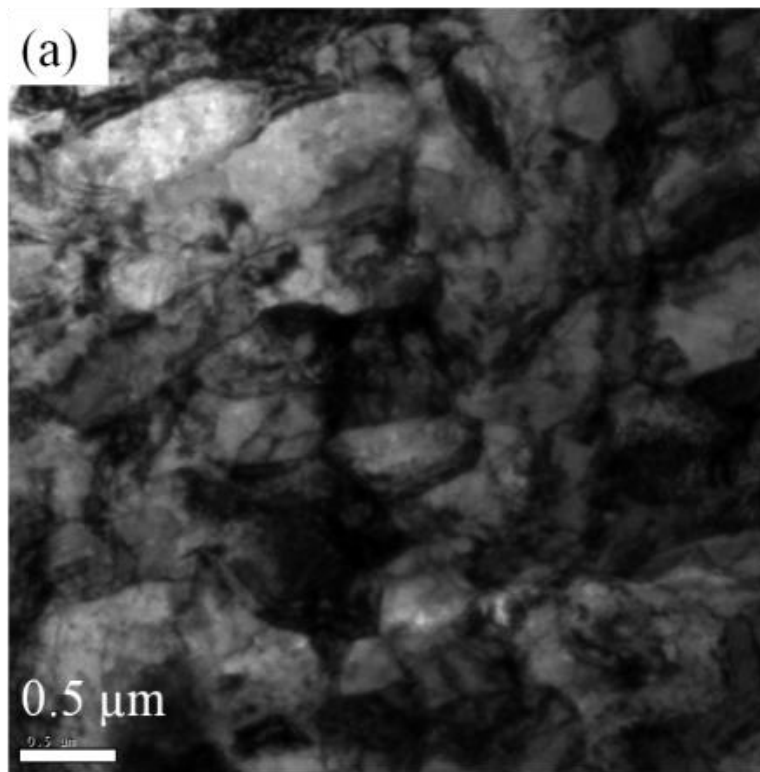


Figure 7.8: TEM bright field images of IF steel after (a) ECAP-12-CRR-96-FA550.

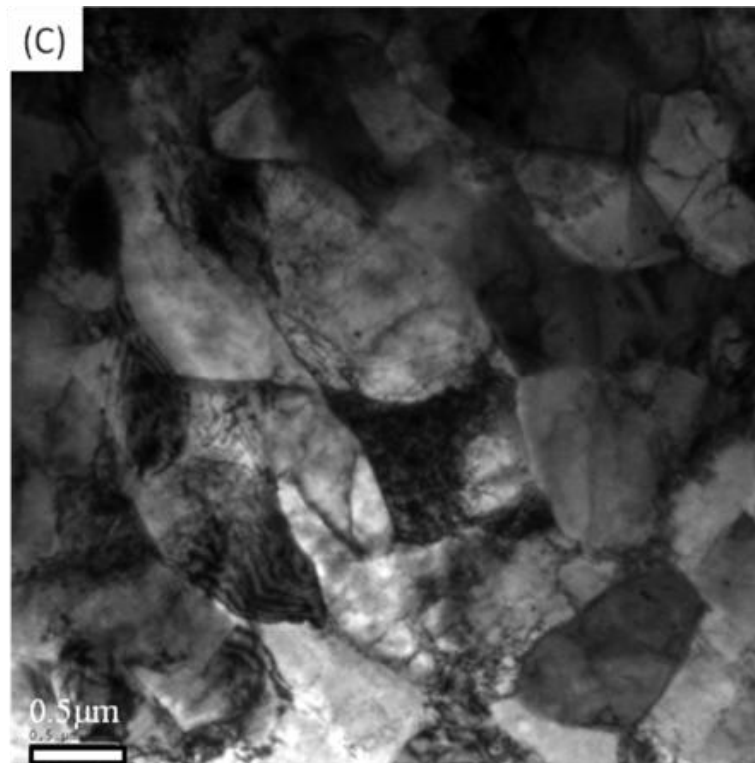
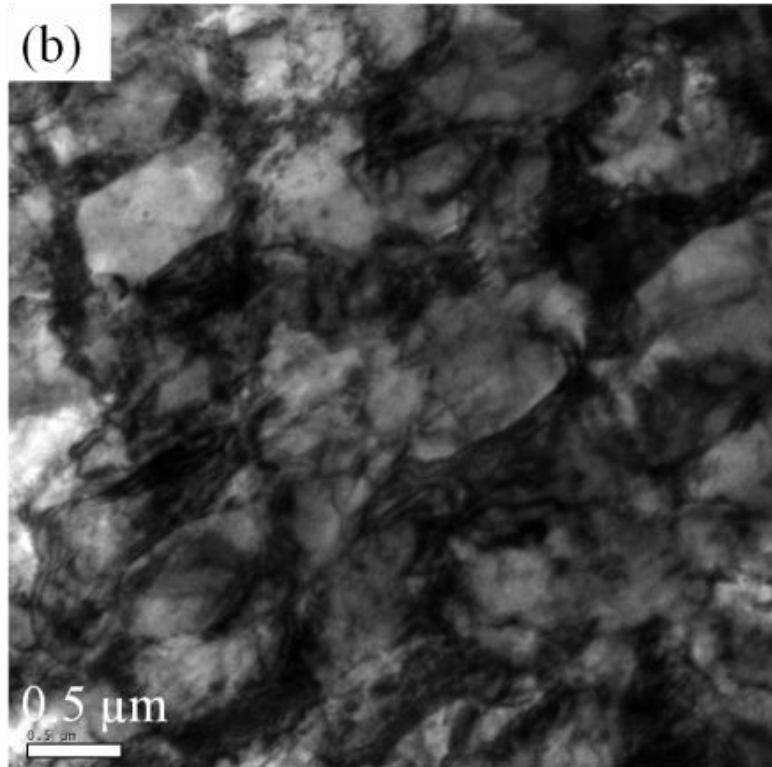


Figure 7.8: TEM bright field images of IF steel after (b) ECAP-12-CRR-96-FA600 and (c) ECAP-12-CRR-96-FA650.

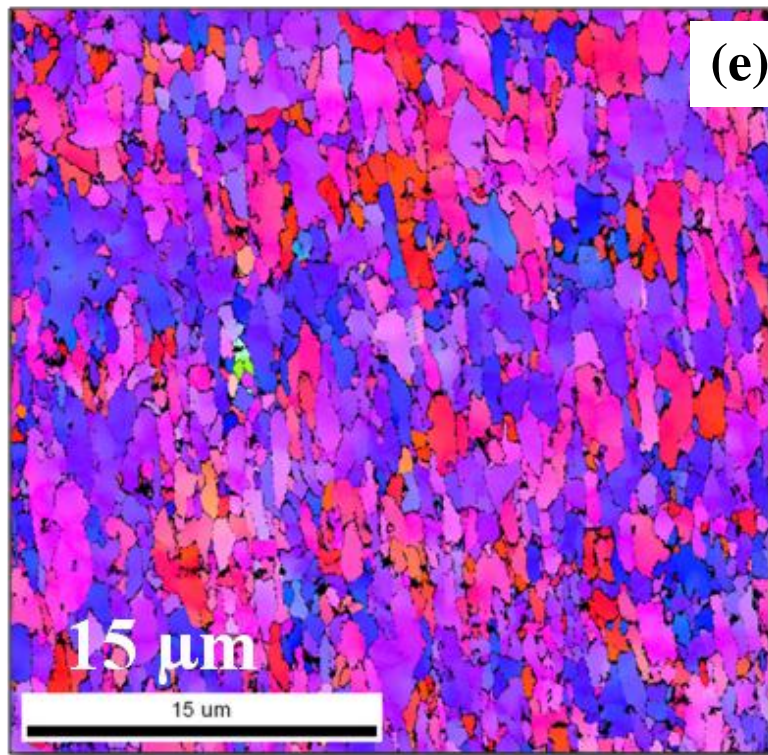
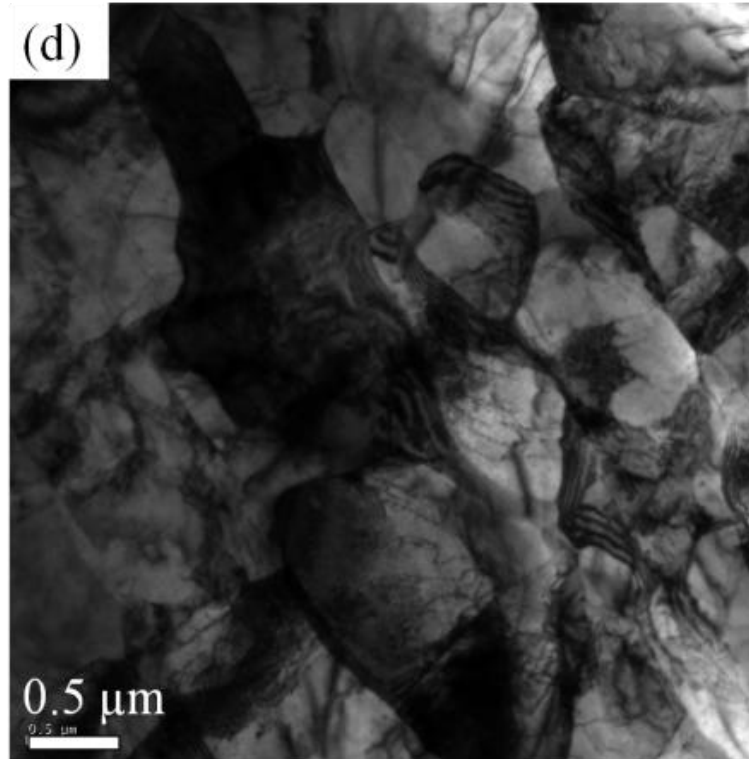


Figure 7.8: TEM bright field images of IF steel after (d) ECAP-12-CRR-96-FA650 and [001] IPF maps of IF steel after (e) ECAP-12-CRR-96-FA675.

Table 7.2: Microstructural parameters of ECAP followed by cold rolled and cryorolled IF steel samples.

	Domain size (nm)	Avg. Grain size (nm)	Lattice Strain (%)	Elastic stored energy (J/mole)	Avg. HAG B (%)	Avg. LAG B (%)	Avg. Mis. angle (°)	Hardness (VHN)
As-received	164	57.6 μm	0.12	26.9	88.4	11.6	37	174 \pm 7.5
ECAP-12	41	196	0.21	50.9	34.5	65.5	15.8	300 \pm 3
ECAP-CR-90	34	204	0.23	57	58.3	41.7	25.9	524 \pm 25
ECAP-CRR-96	36	127	0.23	60	60.8	39.2	25.7	530 \pm 0.5
ECAP-CR-90-FA	133	639	0.14	45.6	55.9	44.1	25.2	450 \pm 22
ECAP-CRR-96-FA	44.8	564	0.19	45	57.6	42.4	26.4	462 \pm 41

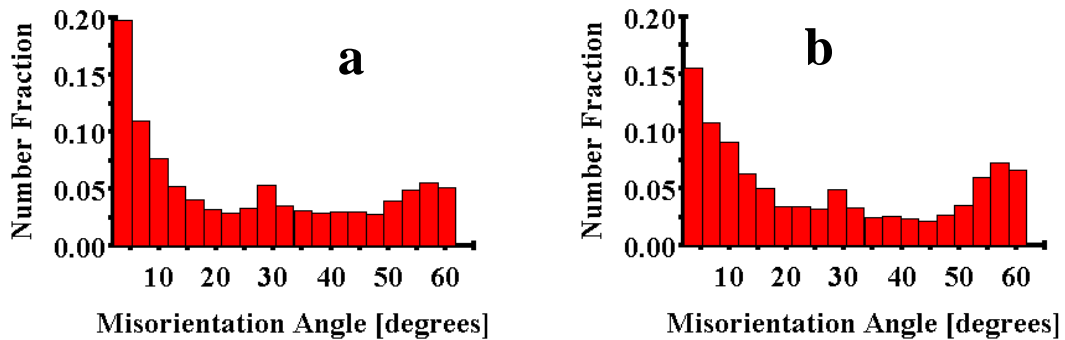


Figure 7.9: The misorientation angle distribution of the IF steel for (a) ECAP-12-CR-90-FA, (b) ECAP-12-CRR-96-FA.

7.2 Mechanical Properties

As-received IF steel shows hardness of 175 VHN (Table 7.3). Figure 7.10 represents the variation of Vickers microhardness with the annealing temperature of ECAP-3. On ECAP for $\epsilon_{vm}=3$, of IF steel hardness increases to 323 VHN due to grain refinement and increase in defect density. As this material is annealed, hardness is maintained at 923K (650°C) due to recovery but it starts decreasing significantly at 948K (675°C) and it continues the decreasing trend due to the onset of recrystallization, which

continues till 973K (700°C) and consequently the hardness value drops down to 243 VHN (Figure 7.10a). At 998K (725°C) the hardness value decreases to 227 VHN due to grain growth.

Table-7.3: Mechanical properties of flash annealed IF steel samples.

Sample	Y.S. (MPa)	UTS (MPa)	U.E. (%)	T.E. (%)	Hardness (VHN)
As- received	227	336.7	26.14	41.8	175±8
ECAP-3	-	-	-	-	323
ECAP-3-FA550	-	-	-	-	360
ECAP-3-FA600	-	-	-	-	355
ECAP-3-FA650	-	-	-	-	342
ECAP-3-FA675	-	-	-	-	302
ECAP-3-FA700	-	-	-	--	243
ECAP-3-FA725	-	-	-	-	227
ECAP-3-FA750	-	-	-	-	220
ECAP-12	795	858	2.2	16.2	300±1.6
ECAP-12-FA 600	764	820	1.7	16.7	-
ECAP-12-FA 625	694	784	3	20.6	-
ECAP-12-FA-650	-	-	-	-	311±18
ECAP-12-FA-675	-	-	-	-	291±13
ECAP-12-FA-700	-	-	-	-	305.3±5.9
ECAP-21	-	-	-	-	366
ECAP-21-FA600	-	-	-	-	368
ECAP-21-FA625	-	-	-	-	375
ECAP-21-FA675	-	-	-	-	378
ECAP-21-FA700	-	-	-	-	368
ECAP-12-CR-90	1104	1657	2.5	2.77	524.3±25.4
ECAP-12-CR-90-FA675	653	693	2.4	4.9	450±21
ECAP-12-CR-90-FA700	730	843	1.3	3.9	347.66±24
ECAP-12-CRR-96	1195	1409	1.5	1.7	530±17
ECAP-12-CRR-96-FA550	-	-	-	-	409.3±7.9
ECAP-12-CRR-96- FA600	-	-	-	-	378.6±21
ECAP-12-CRR-96-FA675	677	711	0.4	2.7	428±14

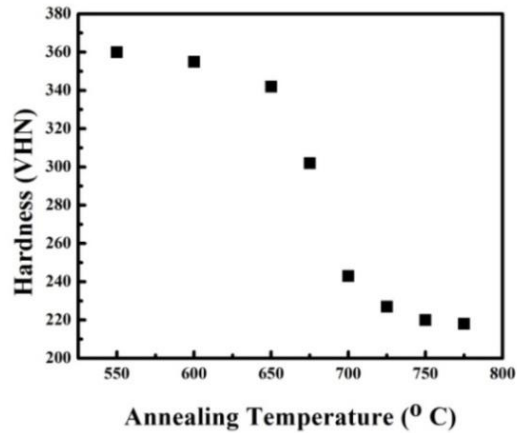


Figure 7.10: Variation of hardness of ECAPed ($\epsilon_{vm}=3$) IF steel with flash annealing temperature.

Figure 7.11 represents engineering stress strain curves of as-received, ECAP-12 and ECAP followed by flash annealed material (Table 7.3). As-received IF steel displays lowest yield strength of 227 MPa and tensile strength of 336 MPa. On ECAP at $\epsilon_{vm}=12$, yield strength and tensile strength increase to 795 MPa and 858 MPa respectively. On flash annealing at 873K (600°C), yield strength and tensile strength decrease to 694 and 820 MPa respectively. On flash annealing of IF steel at 898K (625°C) both yield strength and tensile strength decrease further to 694 MPa and 784 MPa respectively.

As-received IF steel shows high uniform elongation of 26% and total elongation of 41.8%. On ECAP at $\epsilon_{vm}=12$, uniform elongation and total elongation decrease to 2.2% and 16.2% respectively. When ECAPed IF steel ($\epsilon_{vm}=12$) samples are flash annealed at 600°C, both uniform elongation and total elongation are recovered to 1.7% and 16.7% respectively. However, on flash annealing at 898K (625°C), the uniform elongation improves to 3% and total elongation recovers to 20.6% (Table 7.3).

Hardness of flash annealed ECAP-21 IF steel sample remains almost constant in annealing temperature range of 873-973K (600-700°C) (Figure 7.13, Table 7.3).

Figure 7.12a represents engineering stress strain curves of ECAP-12-CR-90-FA675 and ECAP-12-CR-90-FA700. Upon cold rolling to 90% reduction in area/cryorolling of ECAPed IF steel to ~90% reduction in area, the yield strength improves by 1.4 times and the tensile strength significantly, but ductility decreases. The yield strength of cryorolled sample is higher than that of the cold rolled sample. The flash annealing of the cold rolled IF steel (ECAP-CR-90-FA) reduces both the yield strength as well as the ultimate tensile strength to 653 MPa and 693 MPa respectively and the total elongation is partially recovered, however the strength of the flash annealed steel is still 2-3 times the strength of as-received coarse grained material. Annealing at 973K (700°C) leads to decrease in strength from 1657 MPa to 843 MPa and elongation increases to 3.9%. Hardness of flash annealed samples also follow decreasing trend with flash annealing temperature (Table 7.3). The flash annealed cryorolled IF steel is slightly stronger than the flash annealed cold rolled material but ductility is lower (Figure 7.12b, Table 7.3). Hardness of flash annealed ECAPed and cryorolled IF steel samples decreases with flash annealing temperature (Figure 7.13, Table 7.3).

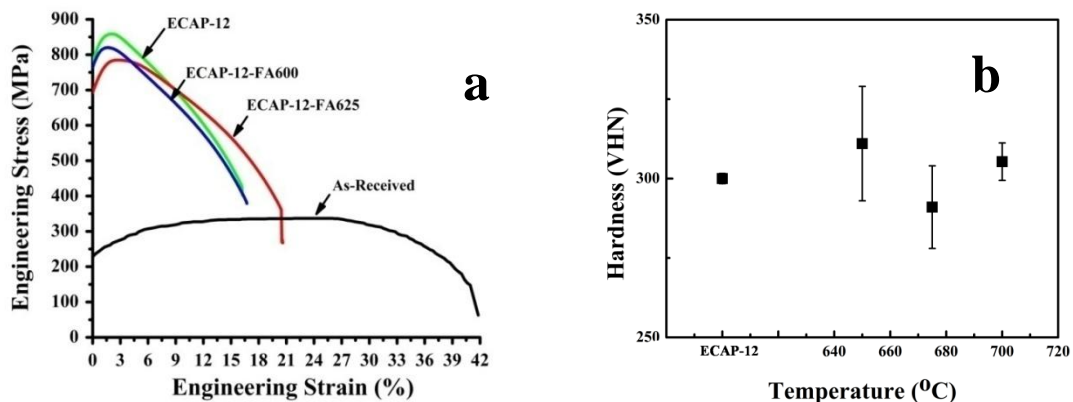


Figure 7.11: Engineering stress-strain curves of IF steel after ECAP-12 and ECAP followed by flash annealed at 600, 625°C compared with a received material (b) variation of hardness of ECAP-12 with annealing temperature.

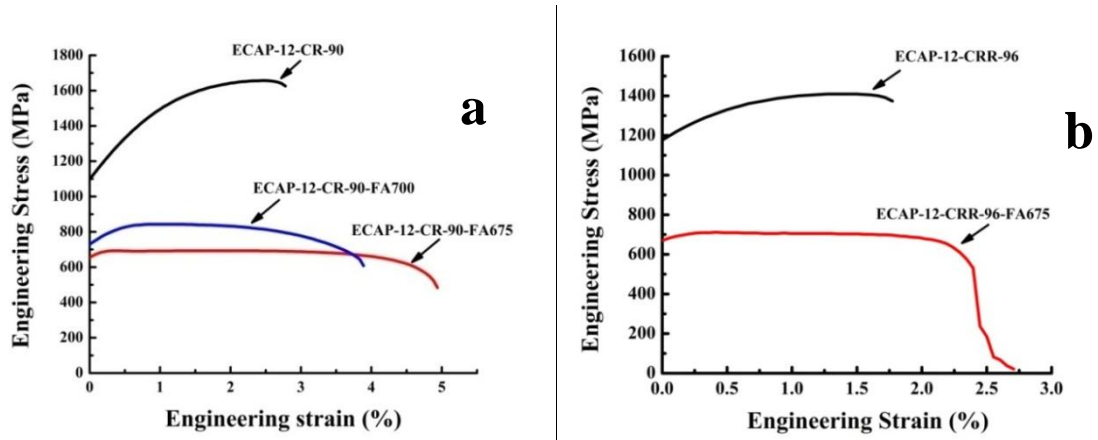


Figure 7.12: Engineering stress-strain curves of IF steel after ECAP-12-CR-90-FA675, ECAP-12-CR-90-FA700, (b) ECAP-12-CRR-96 FA675.

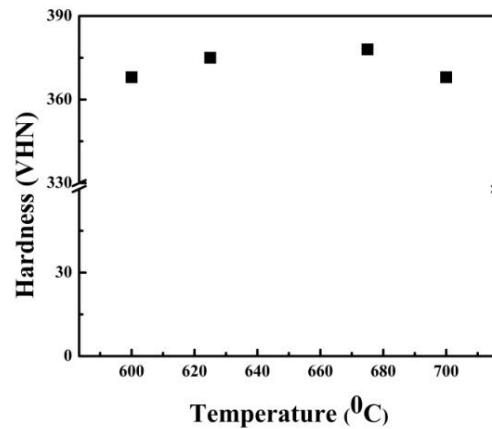


Figure 7.13: Variation of hardness of ECAPed ($\epsilon_{vm}=21$) IF steel with flash annealing temperature.

7.3 Discussion

Recovery observed after flash annealing at 823K (550°C), 873K (600°C), 923K (650°C) of ECAP-3 is attributed to rearrangement of dislocations and formation of cell structures (as observed in Figure 7.1a,b,c and Figure 7.2a) as morphology of microstructure is not changed but most of the grains remain elongated in shape and hardness decreases consequently. In case of ECAP-3-FA675, abnormal subgrain growth occurs. The discontinuous subgrain growth is because of high angle boundary mobility for

small misorientations. Those subgrains which are more misoriented than the average gives rise to discontinuous subgrain growth. Holm et al. [Holm et al. 2003] have proposed that abnormal subgrain growth is nucleation for recrystallization as recrystallized nuclei arises from recovered subgrain structure. Hardness drops significantly at this stage due to rearrangement and annihilation of dislocations (Figure 7.10). The process of abnormal subgrain growth is truncated by onset of recrystallization [Humphreys 1995] which is observed after flash annealing at 973K (700°C) (Figure 7.2b) and results in rapid decrease in hardness (Figure 7.10). Further annealing of ECAP-3 at 998K (725°C) leads to coarsening of fine grains (Figure 7.1f, 7.2d). Coarse grains grow through grain boundary migration by consuming smaller grains as coarse grains have relatively lower grain boundary curvature [Cawthorn 1996]. Even though grain growth is observed at this temperature but effect of deformation is visible as dislocations as well as cells are present (Figure 7.2c). Annealing at 1023K (750°C) brings uniform coarsening (Figure 7.1f and 7.2d) with sharp grain boundaries. Hardness at temperature range decreases and becomes minimum (Figure 7.10a).

Annealing of ECAP-12 sample at 873K (600°C) leads to inhomogeneous microstructure as both ultrafine grains and elongated grains are observed. Thus, this temperature is referred to as recovery temperature. Tensile strength variation also conform the effect of recovery as strength and elongation are comparable to that of deformation state. Increasing temperature of annealing to 898-923K (625-650°C) of ECAP-12 sample, abnormal grain growth is observed (Figure 7.3b, 7.4a) with large fraction of equiaxed grains which are supposed to be new recrystallized grains. Abnormally large grains are formed by consumption of new recrystallized grains. When

tensile testing is done for 898K (625°C) annealed material, strength decreases marginally and elongation increases (Figure 7.11a). Microstructure of ECAP-12-FA675 consists of uniformly coarse grains (Figure 7.4b).

On flash annealing of ECAP-21 at 873K (600°C), recovery is observed with increased subgrain size that leaves the hardness value constant similar to that of deformed state. After the flash annealing of ECAP-21 sample at 898K (625°C) and 948K (675°C), the increase in hardness (Figure. 7.13) could be observed even though the grain size is more than the later case (Figure 7.5b,c, 7.6b,c). Increase in strength due to annealing is observed by many authors [Torrents et al. 2010, Greer et al. 2005, Huang et al. 2006, Kamikawa et al. 2009]. Hasnaoui et al. [Hasnaoui et al. 2002] has suggested by molecular dynamic simulation that after processing, grain boundaries of nano-crystalline materials are often in a nonequilibrium state and annealing results in grain boundaries and triple-junction regions to approach towards equilibrium. This process strengthens the material and reduces the ductility. Further rise in flash annealing temperature for ECAP-21 to 973K (700°C), the grain size increases to a large value (Figure 7.5d) leading to the decreasing trend in hardness (Figure 7.13).

Flash annealing of ECAPed samples followed by cold rolling or cryorolling increases the grain size (Table 7.1) more than that of their respective ECAPed state. This increase in grain size is due to high mobility of boundary at annealing temperature [Humphreys 1995]. The release of elastic stored energy has a combined effect of decrease in lattice strain/microstrain (Table 7.2) and the relaxation of long range elastic stresses associated with cell walls. Every et al. [Every et al. 1974] have reported that the release of elastic stored energy is not only related to the decrease in dislocation density but also

to the relaxation of long range stresses. However, there is residual microstrain within the material due to incomplete annihilation of dislocations after flash annealing. The presence of equiaxed grains in the microstructure (Figures. 7.7b,c, 7.8e) of ECAP-12-CR-90-FA675 and ECAP-12-CRR-96-FA675 samples indicate that the steel is partially recrystallized. The HAGBs form during annealing (Table 7.2, Figures 7.9a,b) through boundary migration and regeneration of the subgrain boundaries by local re-orientation of dislocations into low energy configurations.

The flash annealing of ECAPed and cold rolled IF steel reduces both yield strength as well as the ultimate tensile strength (Figure 7.12) due to the increased subgrain size, grain size and partial recrystallisation whereas the total elongation is partly recovered due to reduced defect density, released microstrain and elastic stored energy. Hardness of respective samples also confirm above result. However the strength of the flash annealed steel is still 2-3 times the strength of as-received coarse grained material (Table 7.3) as the grain size of the former is two orders of magnitude finer than the later (Table 7.1).

7.4 Summary

The bimodal grain size distribution in ultrafine-grain range has been successfully achieved in IF steel processed by equal-channel angular pressing followed by flash annealing. The recrystallization temperature of ultrafine-grained IF steel decreases with increase in equivalent strain from low to intermediate strain but it increases at high equivalent strain due to lower defect density. The temperature of abnormal grain growth in IF steel decreases with increase in equivalent strain from low to intermediate strain but it increases at high equivalent strain. The hardness of severely

deformed IF steel can be maintained upto 923K (650°C) at least for short duration of 300 seconds. When UFG IF steel samples processed by ECAP at $\epsilon_{vm}=12$ followed by cold rolling/cryorolling to >90% reduction in area and flash annealed at 948K (675°C), the materials are partially recrystallized. The increased subgrain size, the grain size and reduced residual lattice strain all lead to lowering of the hardness and the 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 coarse-grained as-received steel even though at that condition the yield strength is maintained 2-3 times higher than that of the as-received steel.