EBSD and TEM Analysis of Copper Processed by Severe Plastic Deformation

UCSD Jacobs | Materials Science and Engineering

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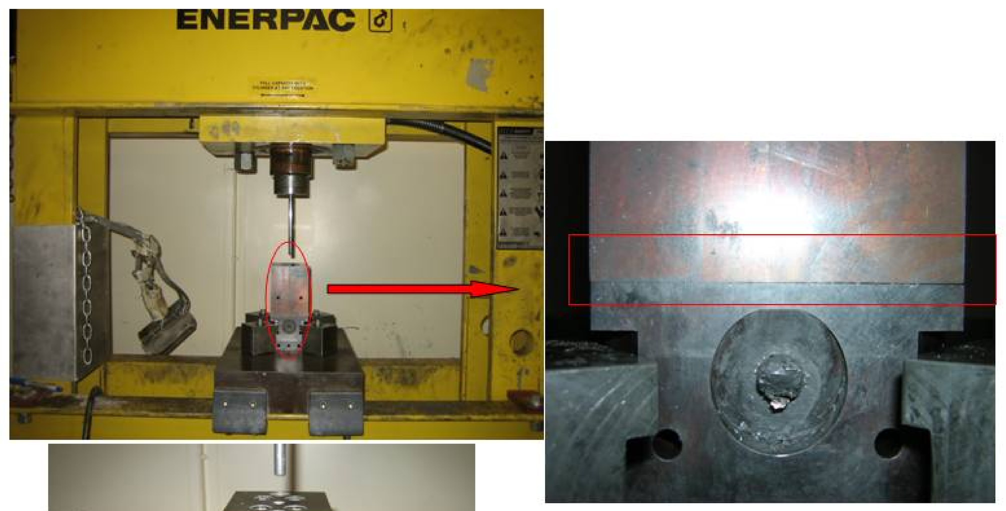
Abstract

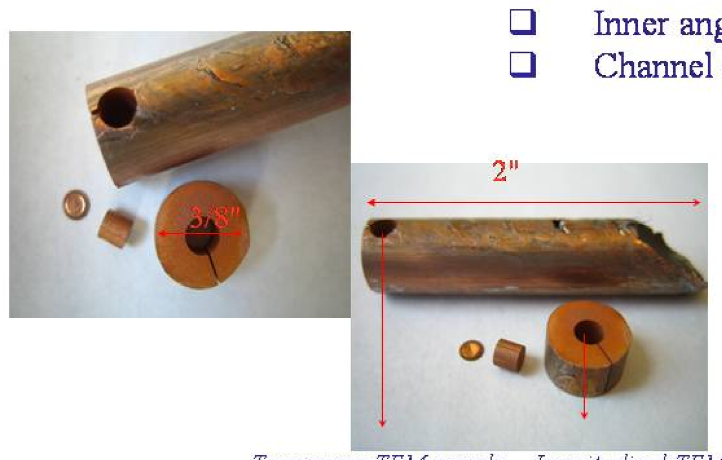
Equal Channel Angular Pressing (ECAP) as a variant of severe plastic deformation has been established as an effective process for producing ultra-fine grain size (<1 μm) in conventional metals and alloys. In the present work, ECAP has been carried out on commercially pure Cu to study the effect of die angle, rotation scheme and number of passes. TEM and EBSD analysis techniques have been used to characterize the microstructure.



"Horizontal Split Die": replaceable lower piece with changing channel angle.

Die specifics -

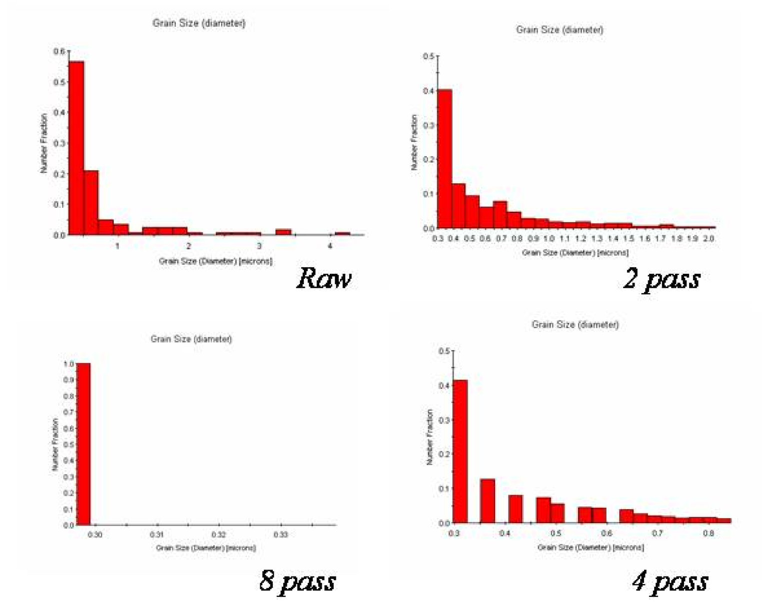
- H-13 tool steel: 6'× 4'× 2'
- Outer arc: smooth 20º
- Inner angle: 102º and 90º
- Channel diameter: 3/8"



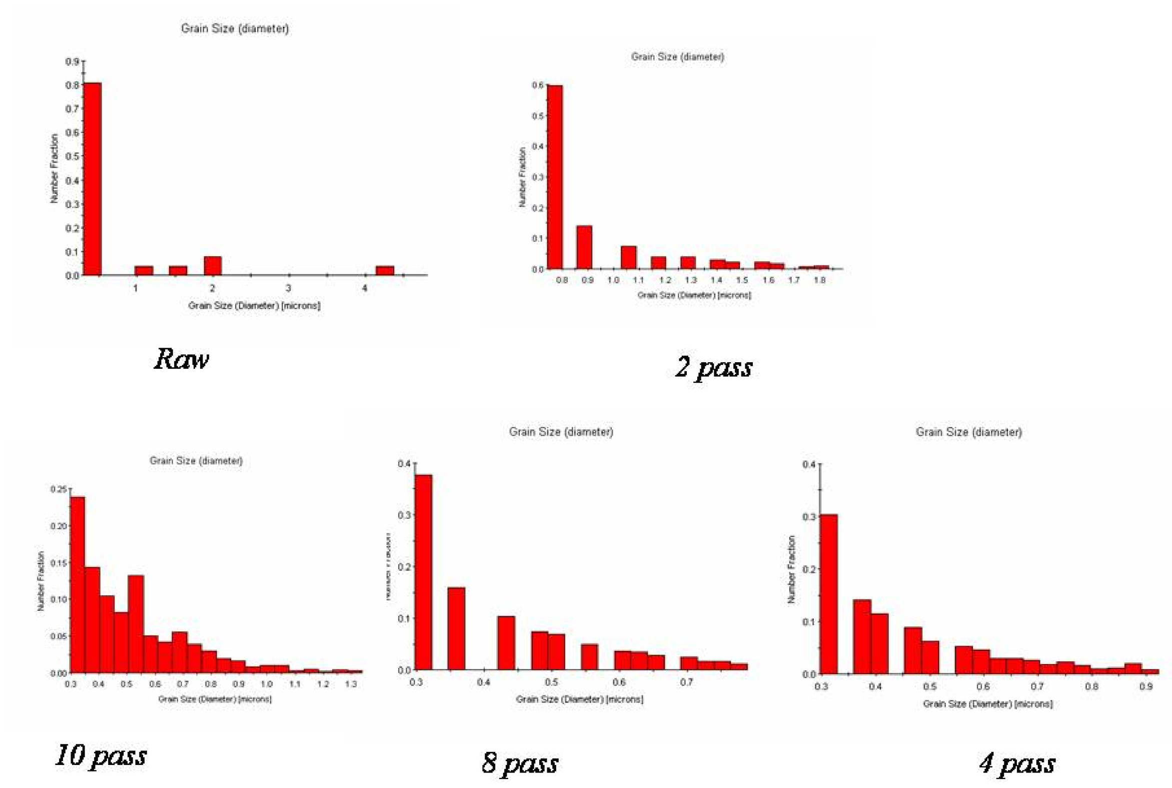
Transverse TEM sample Longitudinal TEM sample

Experimental Specimens

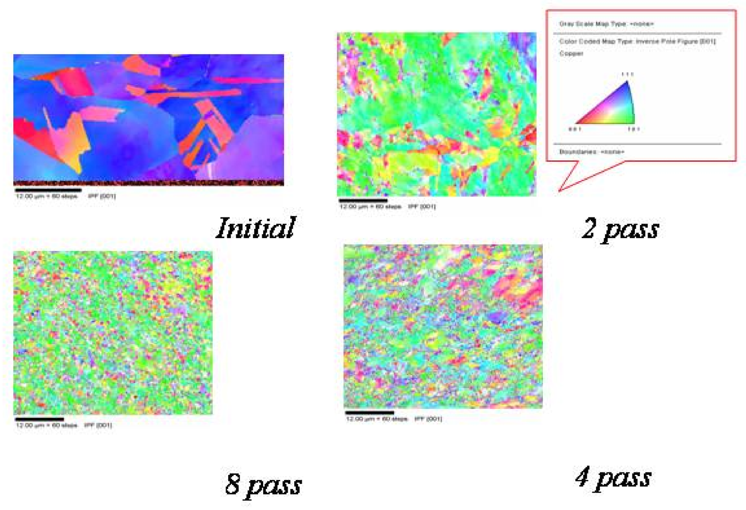
	Number of passes								
	Route B_C							Route A	Route C
90º die	1	2	3	4	6	8	10	8	8
102º die	1	2	3	4	6	8	-	8	8



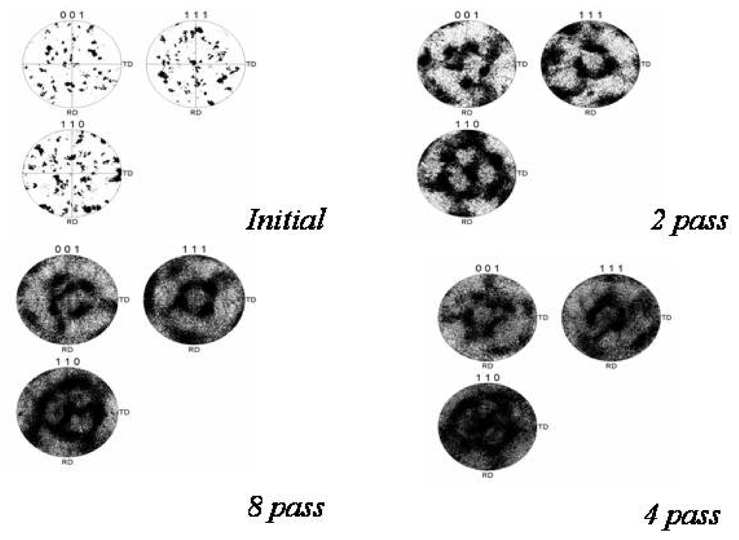
Grain size distribution of samples with different number of passes for 102^0 die using route B_C



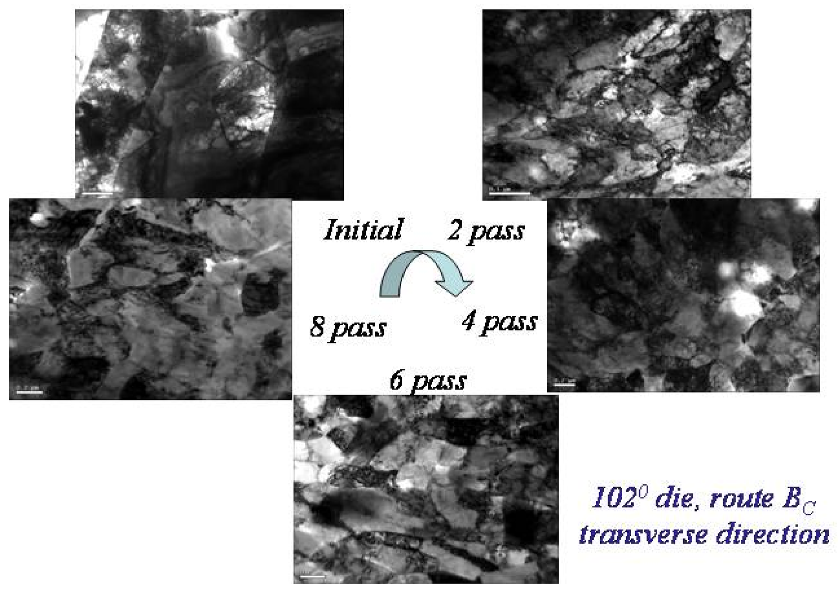
Grain size distribution of samples with different number of passes for 90^0 die using route B_C.



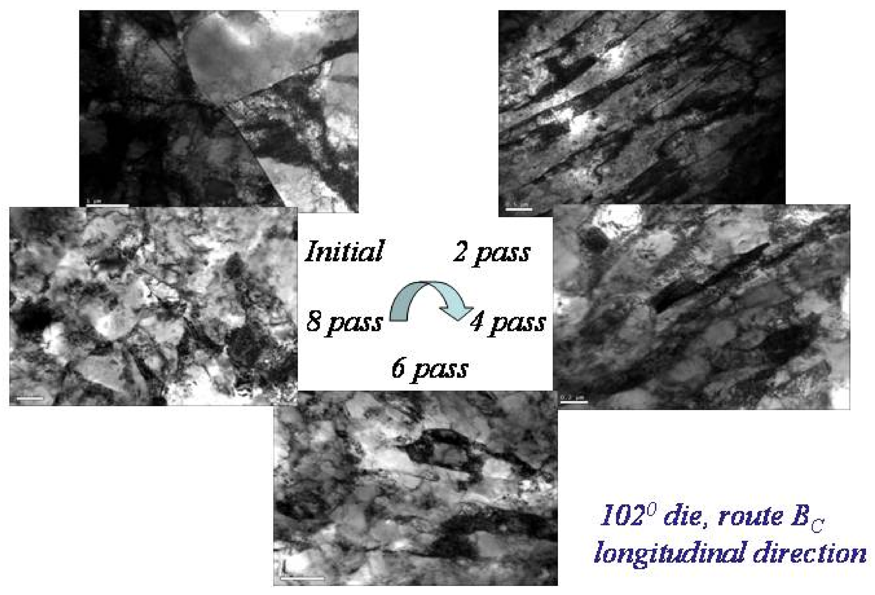
Inverse Pole Figure(IPF) from EBSD examination of 102^0 samples processed by route B_C



Pole figures from EBSD examination of 102^0 samples processed by route B_C.



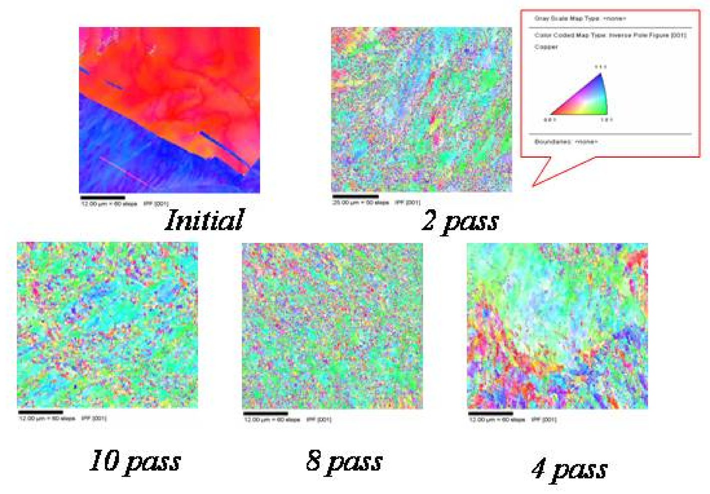
102^0 die, route B_C transverse direction



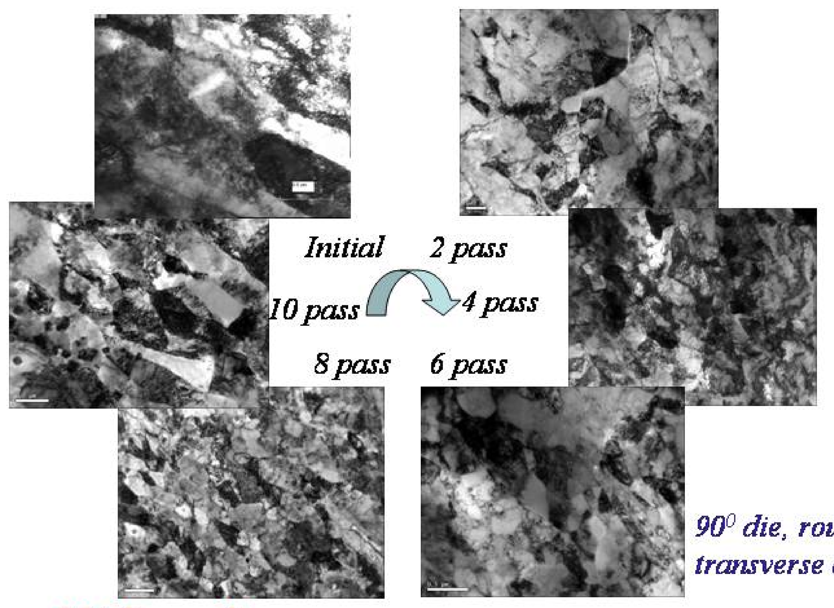
102^0 die, route B_C longitudinal direction

102º die results:

The initial grain size is a few microns. The initial texture ranges from (110) to (111) with the bigger grains having either (100) or (111) orientation. This can be clearly seen as blue and red grains in the Inverse Pole Figure (IPF). A large fraction of the grains refined to ultra-fine size in the first two passes. The texture evolves to (101) after 2 passes (indicated by green color in the IPF) . Almost all the grains break down to sizes less than 1 μm after 2 passes (60% grains < 0.5 μm). Further passes make the (101) texture stronger (indicated by increasing presence of green color and lead to a more equiaxed distribution of grains. Additionally, all the grains in the micron range are broken down to ultra-fine size in higher number of passes. After 8 passes, almost 100% structure is ultra-fine.



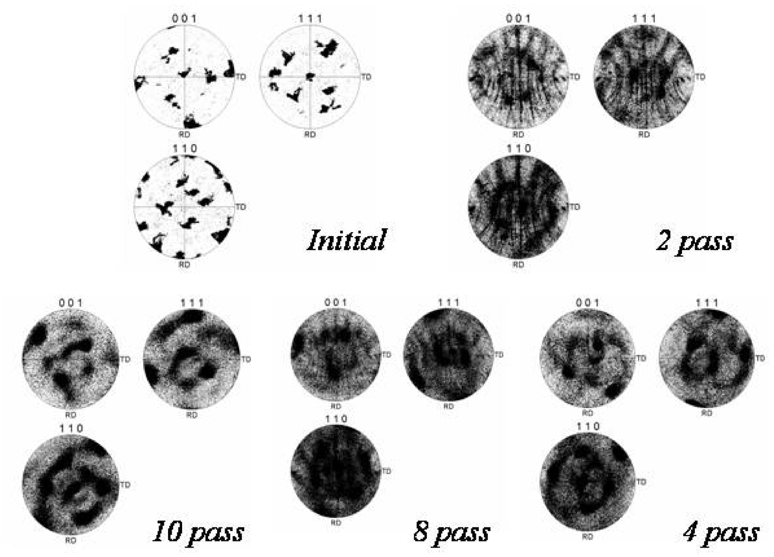
IPF from EBSD examination of 90^0 samples processed by route B_C



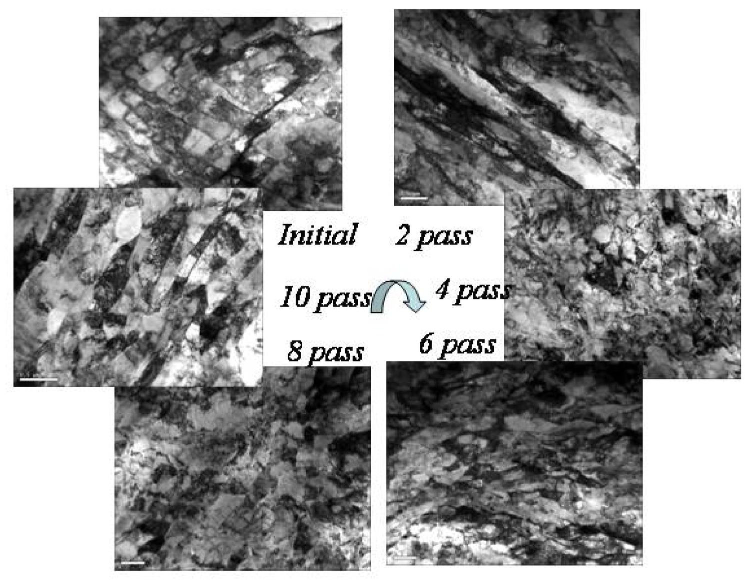
90^0 die, route B_C transverse direction

90º die results:

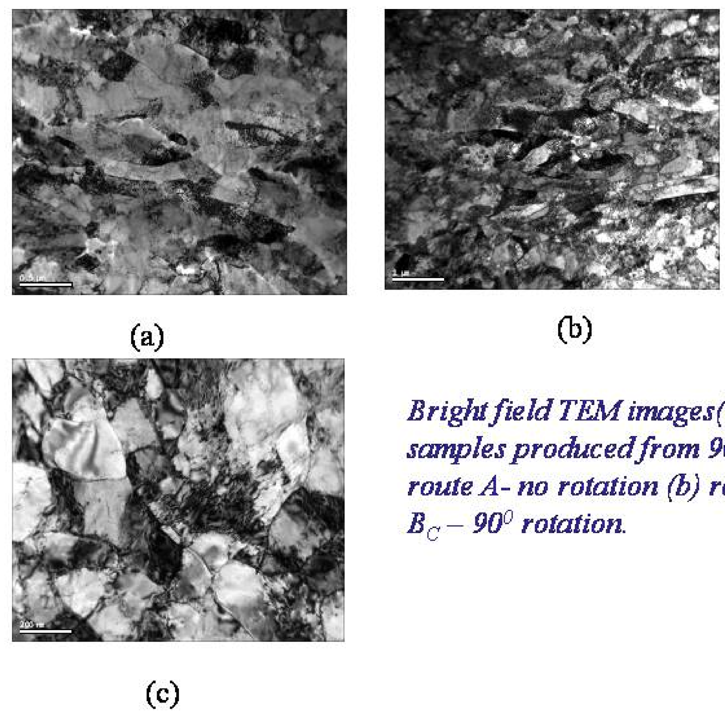
The evolution of the microstructure, specially texture seems to be similar in the samples produced by 90º die as compared to the 102º die. Difference existed in the microstructure of the raw sample to begin with. The 90º raw sample had grain size roughly twice as large as the grain size in the raw sample of 102º die.



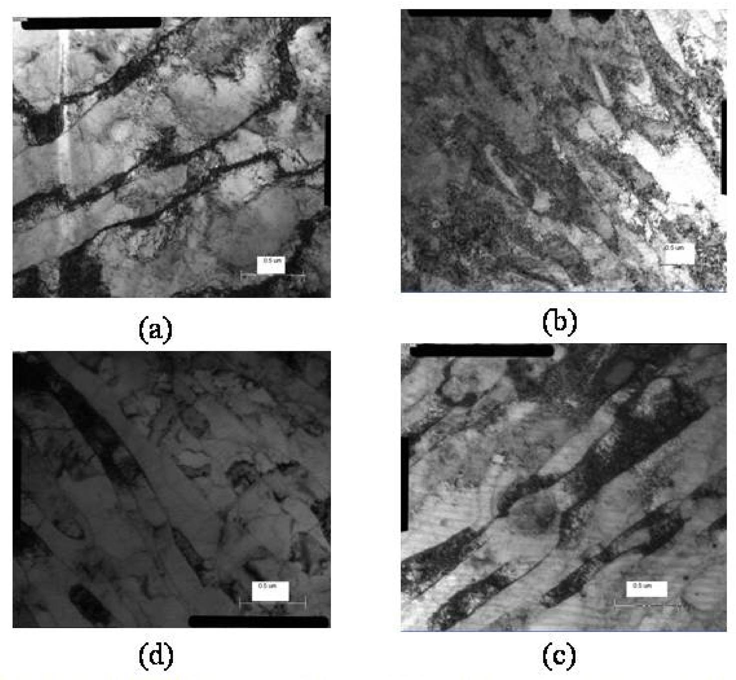
Pole figures from EBSD examination of 90^0 samples processed by route B_C.



90^0 die, route B_C, longitudinal direction

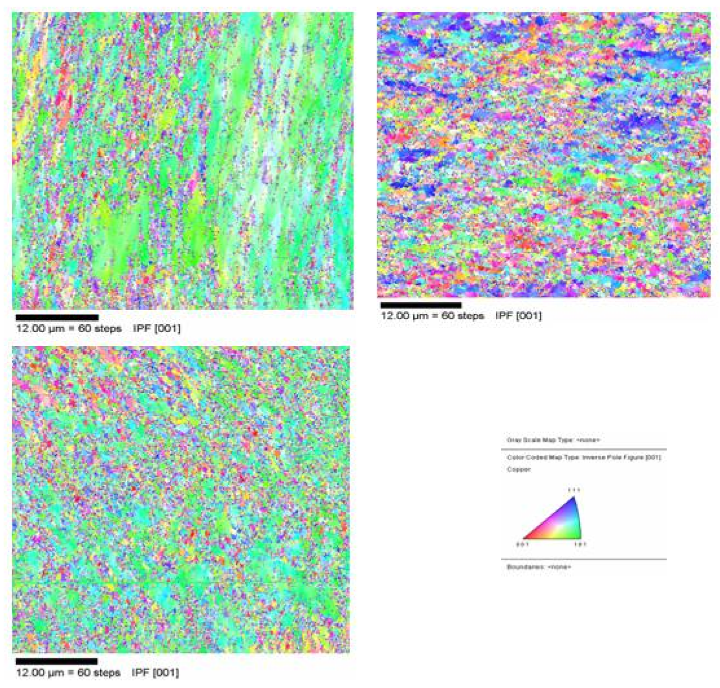


Bright field TEM images(extrusion axis) of 8 pass samples produced from 90^0 die using three routes (a) route A- no rotation (b) route C- 180^0 rotation (c) route B_C – 90^0 rotation.

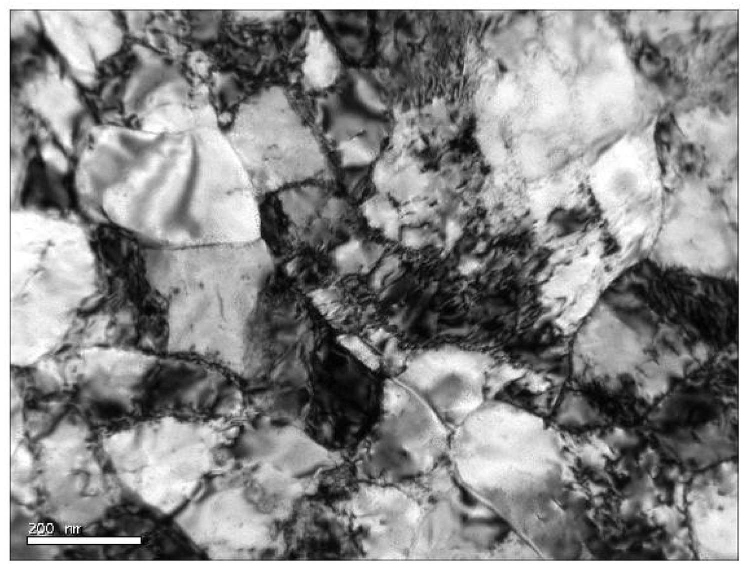


Bright field TEM images (longitudinal direction) of samples with (a) initial (b) 1 pass (c) 2 pass (d) 3 pass using 90^0 die, route B_C.

Comparison of the results produced by different rotation schemes- route B_C, route A and route C showed that route A and C produced elongated grains while route B_C resulted in a more equiaxed microstructure. The texture evolved to (101) in both route B_C and route A while it resulted mainly in (111) in the case of route C.



IPF from EBSD examination of samples processed by 90^0 die using different rotation scheme.



Bright field TEM micrograph of a 8 pass (route B_C) sample showing inhomogeneous deformation within a grain and neighboring grains. Dislocation cells and sub grains can be observed in some grains.

For samples processed by few passes, we observed difference (sometimes huge) in the microstructural features of grains lying in close vicinity. Additionally, within a grain an inhomogeneous distribution of microstructural deformation was also observed. For example, a small area of a particular grain was found completely dislocation- free whereas the other showed a high density of dislocation. The inhomogeneity in distribution of deformation could be related to misorientation within the grain. While deformation progresses, dislocations rearrange themselves to form dislocation cells leading subsequently to the formation of subgrains. The subgrains, on additional passes form finer grains. Grains with lower or no dislocation density remain less affected. This could be the reason that the final microstructure, after a few passes, show an inhomogeneous distribution of grain size.

Conclusion:

ECAP is an effective process for producing ultra-fine microstructure. The bulk of grain refinement seems to take place in the first few passes (2 passes). These are among the key steps in understanding the mechanism of grain refinement. In the present work, the results obtained from the 102º or 90º die did not show any significant difference in the evolution of the microstructure as a function of number of passes. Change in texture is strong in the first few passes. For our case, texture ranged from (110) to (111) in the raw sample and it tends to (101) in the final structure. Route B_C is more effective than route A and route C for producing equiaxed microstructure. Texture evolution is dissimilar for route C as compared to route A and route B_C. We observed inhomogeneity in deformation from one grain to the other and even within a grain. From our understanding, this inhomogeneity in deformation could lead to an unequal distribution of grain size in the final microstructure.