Structure Property Correlation in Ultrafine Grained Copper Processed by Equal Channel Angular Processing

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Abstract

Ultra fine grained copper is produced by equal channel angular processing (ECAP) routes.Multiple passing through the ECAP die reveals high grain refinement in first few passes.Transmission electron microscopies in to the samples reveal a grain refinement of 150 µm to 500 nm sized grains after 4 passes. Mechanical properties measuerement shows that the severe plastic deformation has improved the structural strength to 410 MPa. Structure property study confirms the Hall petch strengthening mechanism operational during the deformation process. The effect of initial microstructral inhomogeniety is investigated and it is found to have marginal influence in the grain refinement of the multiple pass ECAP. Process modeling study using finite element models reveal the developed shear strain and stress fields during the deformtion process.

Introduction

ECAP process is the most viable forming process to extrude material using specially designed channel dies without substantial change in geometry resulting in ultra fine grained (UFG) materials [1,2,3]. The possibility to produce massive UFG specimens via severe plastic deformation makes them attractive for engineering applications and creates new opportunities to explore their specific properties in comparison with ordinary coarse grain materials using the standard specimens. Equal-channel angular pressing (ECAP) [2,3] is a technique that allows us to achieve extremely large imposed strains through intensive simple shear in bulk samples. The properties of the materials are strongly dependent on the plastic deformation behaviour during pressing that is governed mainly by die geometry (a channel angle, corner angle and preform geometry), material properties (strength and hardening behaviour), and process variables (temperature and lubrication). However, many modern engineering applications require a rather sophisticated combination of physical and mechanical properties.Therefore joining aspect of the UFG materials is of high technological importance.Bulk nano crystalline materials are of high interest because of its electro chemical characteristics. Here the focus of the study is on the process dependence on the fine grained structure formation during Equal Channel Angular Pressing (ECAP).Copper being high conducting(electrical/thermal) has been the unique choice for many thermal electrical application.Different grades of copper has been developed for these special purpose application like electrical connections, trolley wire, electrode tips, thermal tiles etc.Recently there has been increased interest on the UFG properties of these copper processed using ECAP routes.Here the investigation carried on the commercial pure Oxygen Free High Conductivity (OFHC) copper.

Materials and Processing methods

OFHC copper rod with 9.8 mm diameter is used as deformable material for the study. Extruded OFHC copper (99.99% pure) rods of 10mm diameter were annealed at 6000C for 2 hours in vacuum to obtain a hardness of 78 HV and a grain size of 100-150 µm.In order to study the effect of initial inhomogeneity in microstructure cold drawn OFHC rod (9.8 mm Dia) passed through a wire drawing die set (fig. 1a).The cold drawn OFHC samples were annealed at 600 C for 2 hr resulting in a hardness of 86 HV. Cold drawing in the billet resulted in a microstructure with 150 µ elongated grains at the center of the rod, and

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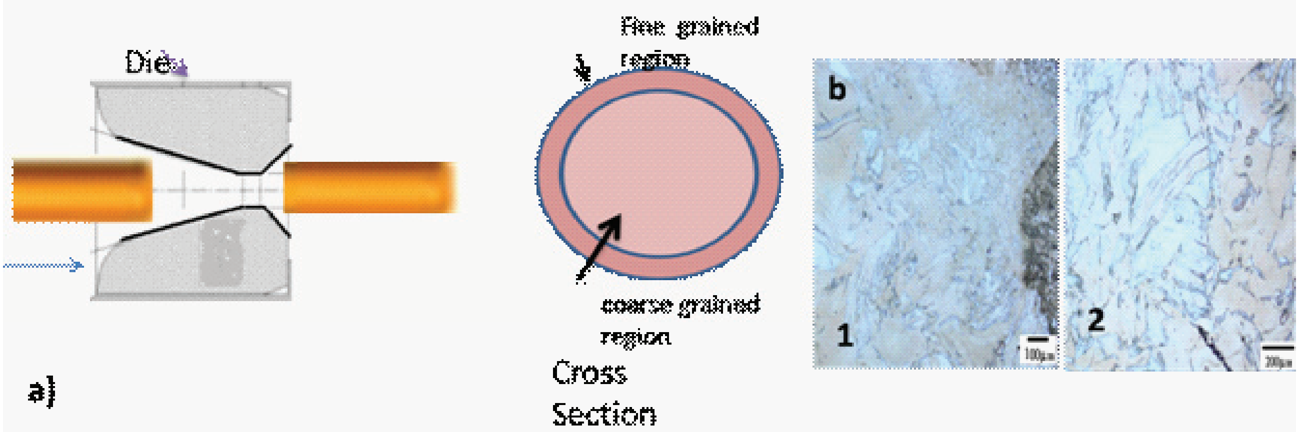

Fig. 1. a) Cold wire drawing schematic b) Microstructure after wire drawing b_1 fine grained region b_2 coarse gained region

80-100 µ sized grain at the outer 2 mm layer of the rod as shown in figure 1b.The cold drawing resulted in finer grain sized microstructure in the periphery and coarse grains towards the centre of the rod. ECAP Multiple pressing was performed around 1 to 8 times with 1mm/s velocity at room temperature via route Bc (the billet is rotated 90 °C CW around the longitudinal axis between consecutive passes) using a die set with circular channels that intersect at an angle _ = 105° and an outer arc angle _ = 30° as shown in figure 2(a_c).

With the exception of the entry and exit points, the channel diameter was uniformly 10 mm. The diameter was slightly enlarged at the entry and exit points to permit easy reinsertion of the sample in the channel. The initial samples of OFHC were cut into billets of 7 cm length and 9.8 mm diameter that permitted a loose fit in the channel. Pressing was carried out using a H-13 tool steel plunger guided by a hydraulic press.MoS2 spray was used as the lubricant. The tolerance of the plunger was kept extremely low to prevent material from flowing between the walls of the channel and the plunger.

Mechanical property data at room temperature was obtained from all ECAPed specimens from micro hardness measurement as well as compression testing, using universal testing machine (Shimatsu 250 UTM). The Vickers micro hardness was

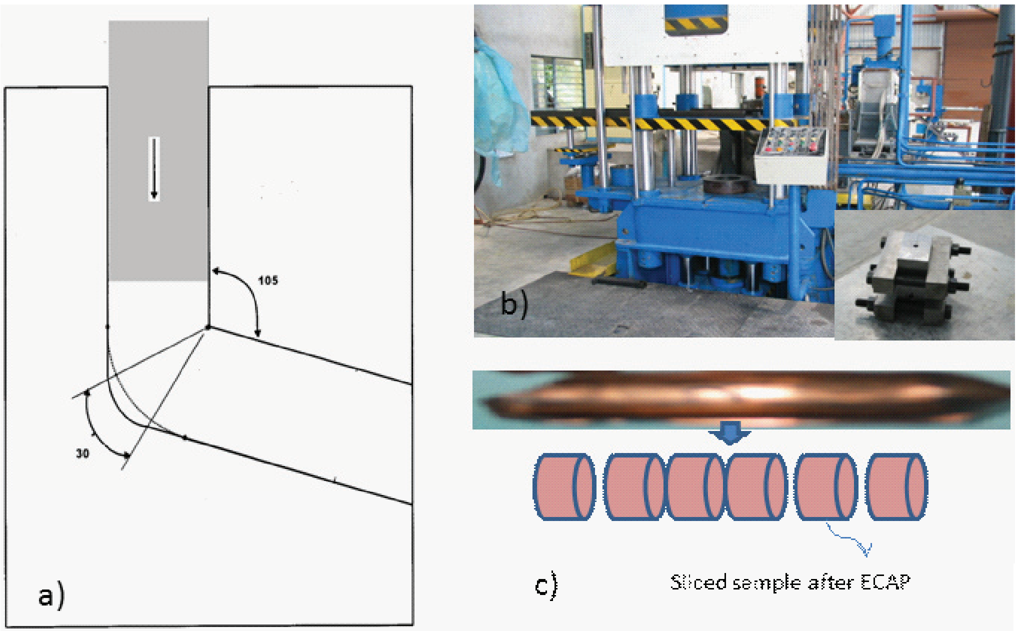

Fig. 2. a) Schematic of ECAP die b) Ecap die and press set up c) Ecaped sample

Table 1.
Calculated grain sizes from the Hall–Petch relationship

References	k (GPa • 10-4 m1/2)	d (grain size)
Feltham and Meakin [6]	3.53	740nm
Armstrong [7]	1.1	71nm
Haouaoui M et al.[8]	2.34	307nm
Gourdin and Lassila [9]	2.78	459 nm
Andrade et al. [10]	0.56–2.6	402 nm
Merz MD and Dahlgren SD[11].	4.3	1.1 µm
Wang and Murr [12]	5.80	2 µm

measured on the plane perpendicular to the working direction in the as fabricated ECAP state after different number of pressings through the dies and after annealing under various conditions. The applied load was 500 g, and the loading time was of 15 s. The average values of at least five successive measurements were calculated. Grain size determination of the ECAPed samples was carried out through several micro-graphs taken from optical and Transmission Electron Microscopes (TEM).

Mechanical Testing

Fig. 3a shows the Vicker's hardness behaviour of OFHC copper as a function of number of passes. As can be clearly seen, hardness saturates at eight passes. There is reasonable agreement between different researchers [4, 5]. The saturation is connected to the lower limit of the grain size achieved by ECAP, which is in the range of 200 to 500 nm. If one applies the Hall–Petch relation, one can infer a grain size from the value of the yield stress values (fig.3b). There is some variation in the Hall–Petch slope in the literature, and the values are presented in the table 1. The results of compression tests are presented in Fig. 2b. The two-pass sample showed a significant jump in strength over the initial sample. The rise in strength with subsequent passes was not as significant as it was for the first two passes. This is suggestive of the fact that the most of grain refinement happens in the first few passes only.ECAP yielded a steep decline in the grain size down to 200_500 nm on the first few passes while additional passes simply increased the fraction of grains in this size range.

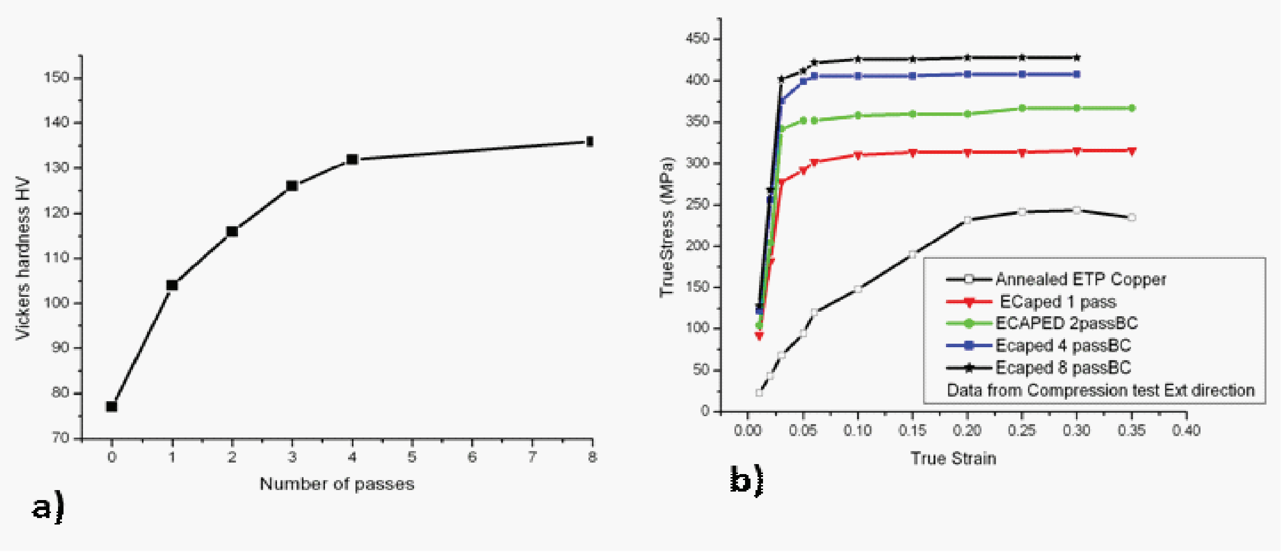

Fig. 3. Mechanical properties a) hardness evelution as function of number of passes b) Flow stress properties from compression test

Microstructure

The microstructure of the unprocessed annealed Cu shown in Fig. 4a, consists of equiaxed grains with size of 100- 150 µm and some twinning. The microstructure details of the ECAP samples processed from one to eight passes through the die are illustrated in Fig4b-f. It is evident the significant elongation of the grains has taken place since the first pass. Samples were examined by TEM as well as optical microscope in the transverse directions (fig 2d-f). As can be seen from the transverse microstructures, the grains are fairly equiaxed and a significant amount of grain breakdown process has taken place in the first two passes. The initial grain size is 150 µm while after four passes a significant fraction of the grains is in the range of 200- 500 nm . With subsequent passes, the amount of grains in the ultra fine range increases.These observations are in good agreement with published results [5,13].

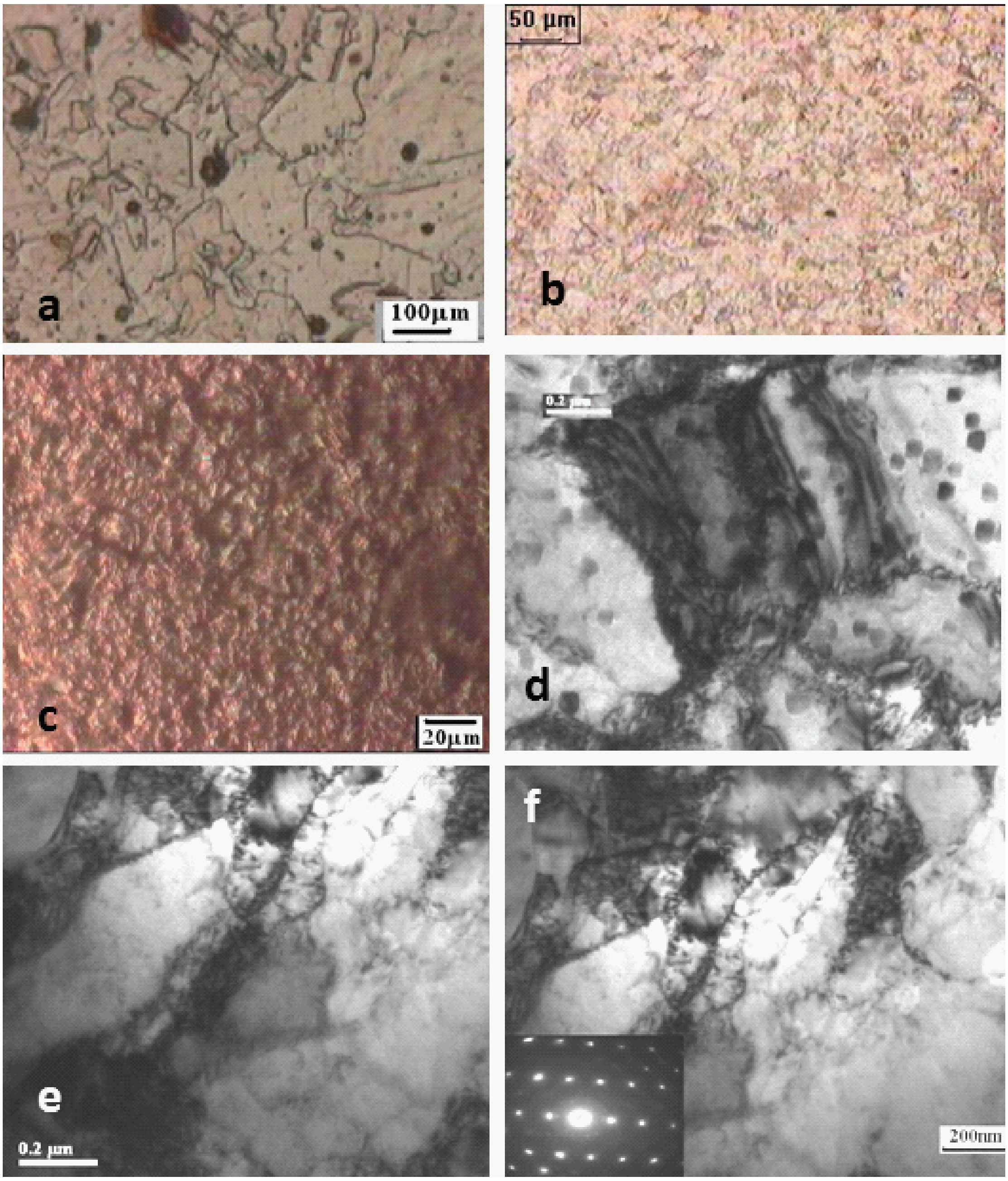

Fig. 4. Microstructure of cross section billet after successive passes ECAP Bc. a)Initial annealed OFHC b)after 2Pass Bc, c)after 4 pass Bc d) TEM after 2 pass Bc e) TEM after 4 Pass Bc f) TEM after 8 Pass Bc

Effect of Initial inhomogeneity in microstructure

Initial inhomogeneity was introduced by wire drawing the sample through a die set reaching 9.8 mm rod. The outer surface region shows the effect of cold work showing a break down grain structure with less than 80 μm sized grains, while the core region shows a grain size range of 150-200 μm.The microstructural examination of this two region after successive Ecap pass shows (fig.5a1_a4 &fig.4b1_b4) that the initial inhomogeneous microstructure are essentially eliminated after two passes. As it is evident form the hardness measurement fig 6 effect of initial state of specimen (grain size inhomogeneity).

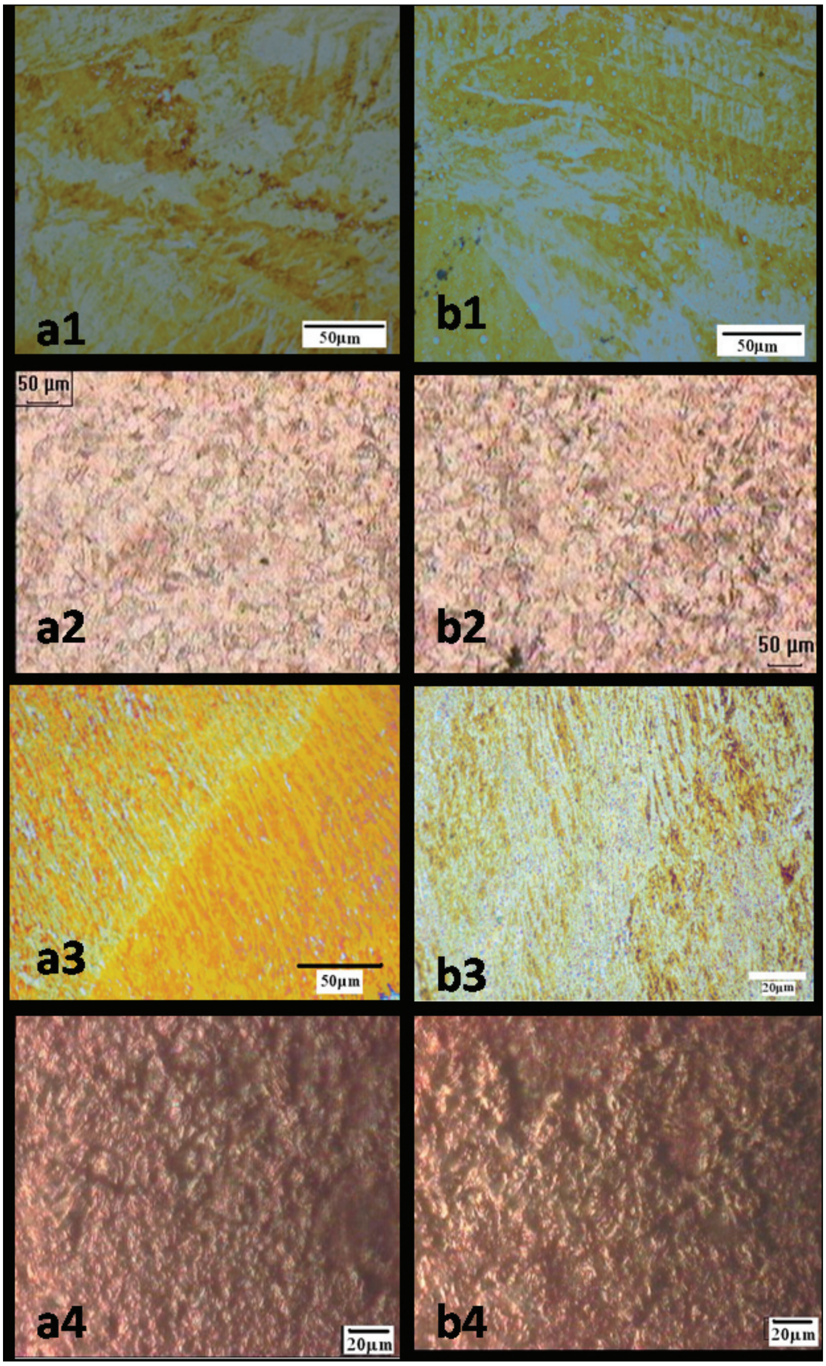

Fig. 5. (left) Effect of initial inhomogeneity in microsctructure a1,a2, a3,a4 fine grained outer region after 1pass, 2pass, 3pass,4pass BC ECAP . b1,b2,b3&b4 coarse grained inner region after 1pass, 2pass, 3pass,4pass BC ECAP

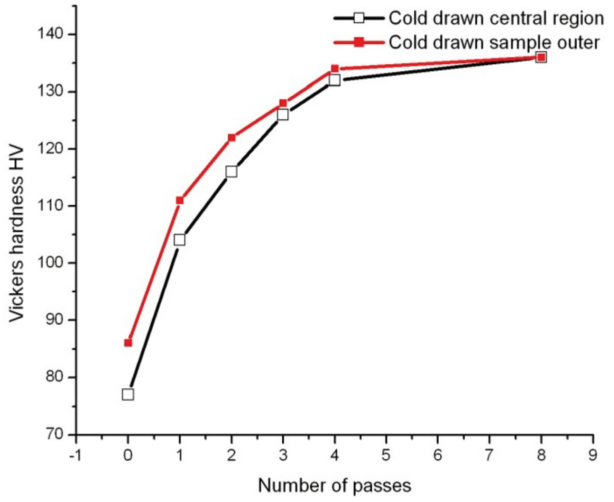

Fig.6 (below). Hardness variation as function of number of pass measured from two regions

Deformation analysis using FEM

DEFORM finite element code was used for carrying out elastic plastic analysis of the deformation process. Here, ECAP process is simulated for a stroke length of 60 mm with elastic and plastic properties obtained from a standard tensile test of OFHC samples. A 9.8 mm OD, and 60 mm long rod geometry and mesh model were used for extrusion analysis. The ECAP process is simulated with the press punch moving at speed of 1 mm/s with the die wall giving stable contact surface. This gives the simulation frame work for studying the flow of the rod material along the 105° channel. As shown in figure 7, the deformation flow has resulted in a corner gap at the 300 angled corner of the die. The strain of 0.891 at angle location is in good agreement with the one dimensional calculation of shear strain 0.88. A peak stress of 125 MPa is developed at the corner showing plastically deforming regions.A lower stress of 40 MPa shows axial pressure requirement of less than 4 ton during the ECAP. Experimental data showed a 3.5 ton maximum load during the process.The stress strain field results are found to be in good agreement with one dimensional equation estimations [2,14].

Conclusion

1. The processes of severe plastic deformation ECAP are effective for the production of ultra fine grained microstructures of OFHC pure copper. A significant increase in the mechanical strength has been observed after subjecting the material to ECAP.

2. Grain breakdown process take place in the first two passes resulting in grain of 200-500nm, whereas the subsequent passes the amount of grains in the ultra fine range increases.

3. The effect of initial state of specimen (grain size inhomogeneity) did not affect the mechanical response significantly. After 3passes, the microstrustucture become homogeneous across the cross section of the Ecaped samples.

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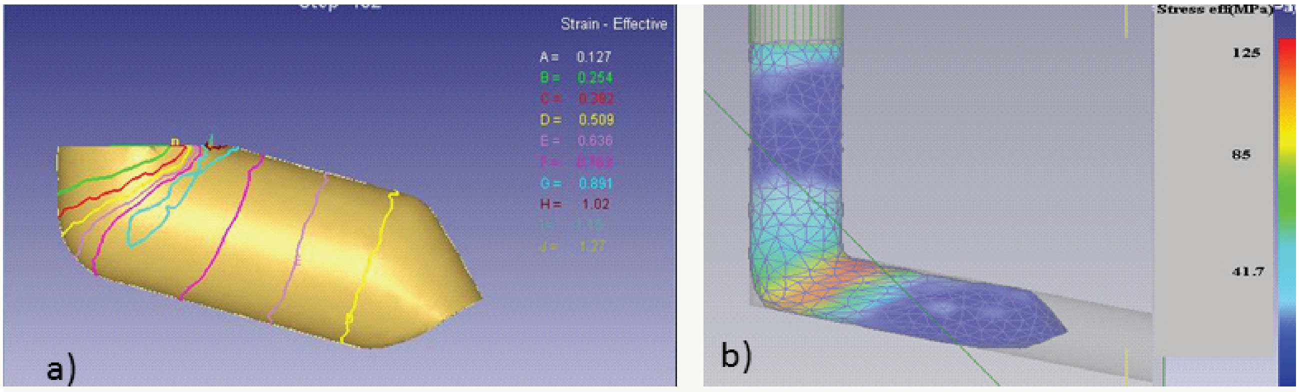

Fig. 7. FEM results a) effective Strain b) Effectve Stress

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