

## Ten Years of Severe Plastic Deformation (SPD) in Iran, part I: Equal-Channel Angular Pressing (ECAP)

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**Abstract:** The superior properties of ultrafine-grained materials fabricated by severe plastic deformation (SPD) have attracted the attention of many researchers around the world. Among the top-ranked countries that are active in this field, Iran is interesting because of the late beginnings of SPD in this country and, subsequently, the highest rate of growth in the number of publications during the last decade. The first Iranian work published in the field of equal-channel angular pressing (ECAP) goes back to 2007, meaning that SPD research covers a period of only about ten years. Nevertheless, since that time there has been an increasing growth rate in the number of Iranian publications dealing with ECAP and especially the introduction of new methods based on ECAP and simulation of the method. The present overview is designed to summarize the main contributions from Iran in the field of ECAP processing. Interestingly, the main contribution of Iranian researchers in ECAP is focused on simulation/modelling and the introduction of new methods of SPD based on ECAP.

**Keywords:** Severe plastic deformation, Equal-channel angular pressing, Ultrafine-grained materials, Mechanical and physical properties.

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### 1. Introduction

In the last 30 years, severe plastic deformation (SPD) has been considered as one of the best methods for fabrication bulk nanostructured and ultrafine-grained materials [1]. In this period, several methods were introduced for imposing SPD on various materials with a major emphasis on equal-channel angular

pressing (ECAP). Based on Thomson Reuters Web of Science, around 4800 researchers from more than 70 countries were involved in investigating and developing the various SPD methods. Although the first use of the ECAP technique dates back to 1981 [2], the use of this method among Iranian researchers has only begun since 2007 [3-6]. Despite the late start of SPD research in Iran, this country is now ranked 8<sup>th</sup> in the world, 4<sup>th</sup> in Asia and 1<sup>st</sup> in the Middle East according to the total number of publications (NP). Analysis shows that Iranian scientists have published more than 470 documents (>5.5% of the total documents in this field). The initial work on SPD processing in Iran started with several studies on ECAP at Sharif University of Technology [3-9], Shiraz University [10-14], Tehran University [15-23] and Isfahan University of Technology [24,25]. Regarding ECAP research, Iran occupied the 6<sup>th</sup> position in the world and the 3<sup>rd</sup> in Asia from 2007 to 2017. The sharp rise in the number of total publications in the field of SPD by Iranian scholars gained this country the 4<sup>th</sup> place in the world in 2016 regarding the total NP in that year. Today, Iran is regularly ranked as having one of the highest NPs in the field of SPD in the world. Furthermore, the rank of Iran using the total number of citations, the average number of citations and the h-index is 12<sup>th</sup>, 17<sup>th</sup> and 13<sup>th</sup> in the world, respectively. This low rank of Iran, compared to its rank based on NP, may be due to the somewhat lower importance of research conducted in Iran and/or the low visibility of Iranian studies in SPD. However, it is interesting to note that the fraction of the proceeding papers published by Iranians is about 200-300% lower than that of other countries. Therefore, Iranians, unlike most others, have not widely attended conferences.

Iran also experiences a relatively lower number of collaborations compared with other countries and this can be attributed to two major reasons. The first reason is the low amount of research funding in Iran which is generally insufficient to cover conference fees. The second and most important reason is Iran sanctions which, since 1979, has led to numerous embargos and limitations [26]. The consequence of these sanctions on the Iranian scientific community has been discussed in detail in the literature [27].

Referring to the above statistics and analyses, the importance of a study on the history and achievements in the field of SPD in Iran is needed due to the following factors. First, in 2016, Iran was ranked 4<sup>th</sup>, 2<sup>nd</sup> and 1<sup>st</sup> in the field of SPD (totally), and the specific SPD methods of equal-channel angular pressing (ECAP) [2] and accumulative roll bonding (ARB) [28], respectively. Second, since the collaboration and exchange of results and discussions between Iranian scholars and researchers from other countries are relatively low, at least by comparison with many other countries, an introduction to the flourishing of Iranian science after 10 years of SPD in this country will be of interest for all scientists in the SPD field as well as providing valuable information for scientists in many other disciplines. It is important also to know how a developing country like Iran has had the highest growth rate in a new and advanced field like SPD. Third, some new methods and many combinations and modifications of SPD methods have been invented or introduced by Iranian scholars. Until now, there are no review articles to classify and describe these methods and innovations. In a set of publications in the current journal which will be published sequentially, the critical achievements in the field of SPD methods will be reviewed with special emphasis on the studies conducted in Iran. Each of these articles is dedicated to an individual topic, while the current article concentrates on ECAP.

Equal-channel angular pressing (ECAP), named also as equal-channel angular extrusion (ECAE), was first introduced in 1981 by Segal et al. [2] in the former Soviet Union. The principle of this method is very well described in [29] by Valiev and Langdon.

The principle of ECAP processing, and especially the overall implications of this processing technique, were reviewed previously by several researchers including Valiev et al. [30] in 2006, Beyerlein and Tóth [31] in 2009, Figueiredo and Langdon [32] in 2012, Kawasaki and Langdon [33] in 2015 and Valiev et al. [34] in 2016. To avoid repetition, the present section is focused specifically on those aspects that have not been sufficiently reviewed to date in an attempt to provide a historical summary of the

achievements of Iranian scholars in ECAP with emphasis on the developments and innovations in this field since 2007.

## 2. ECAPed Materials in Iran

As is apparent from Table 1, in Iran a variety of materials have been deformed by ECAP including aluminum alloys, copper alloys, steels, titanium alloys and magnesium alloys as well as different metal matrix composites. Among all of these materials, aluminum alloys account for about 40% of the publications in this field and they have attracted considerable attention. About 15%, 13%, 11%, 9% and 6% of the publications are focused on metal matrix composites, Mg alloys, steels, Cu alloys, and Ti alloys, respectively.

One of the stimulating applications of ECAP is its usage in the production of bimetallic rods which was introduced by Eivani and Karimi Taheri [34] in 2007. They showed that by using ECAP, a visibly sound joint is obtained which has an acceptable shear interface strength between the inner aluminum core and the outer copper layer (see Fig. 1).

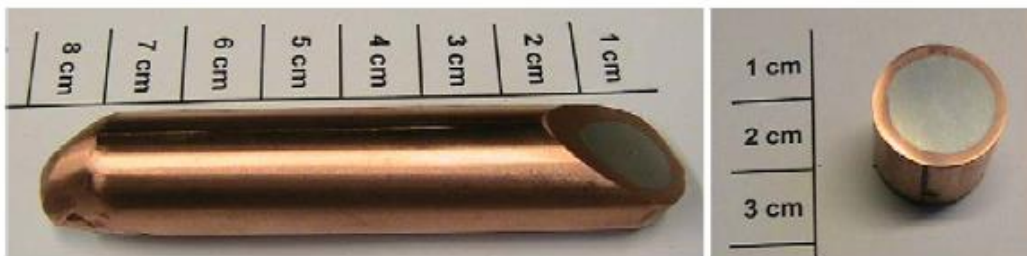


Fig. 1. Views of the joint produced with one pass of ECAP results in the production of copper sheathed aluminum rods, left) side view, right) front view [34].

Table. 1. Summary of the used materials, process parameters and the goal of the ECAP process in the Iranian published works

Material	Aim	Die parameters	No. of passes	Route	Temp.	Ref.
<b>Aluminum and its alloys</b>						
Commercial purity aluminum	Grain refinement	$\Phi = 90^\circ$ $\Psi = 0^\circ$	8	B <sub>C</sub>	RT	[13,14]
Commercial purity aluminum	Grain refinement	$\Phi = 90^\circ$ $\Psi = 20^\circ$	8	B <sub>C</sub>	RT	[35]
Commercial purity aluminum	Grain refinement	$\Phi = 90^\circ$ $\Psi = 20^\circ$	8	B <sub>C</sub>	RT	[36]
Commercial purity aluminum	Texture investigation	$\Phi = 90^\circ$ $\Psi = 20^\circ$	2	A, B <sub>A</sub> , B <sub>C</sub> , C	RT	[24,25,37]
Commercial purity aluminum	Fatigue design	$\Phi = 90^\circ$ $\Psi = 15^\circ$	8	B <sub>C</sub>	RT	[38]
Commercial purity aluminum	Enhancement of strain distribution uniformity	$\Phi = 90^\circ$ $\Psi = 15^\circ$	8	A	RT	[39]
Commercial purity aluminum	effect of ultrasonic vibration during ECAP	$\Phi = 90^\circ$ $\Psi = 15^\circ$	1	-	RT	[40]
Al (A356)	Post semi-solid processing	$\Phi = 90^\circ$ $\Psi = 20^\circ$	4-1	B <sub>C</sub>	NR-RT	] 15,18,20,21,4 [1
Al (A356)	Effect of heat-treatments	$\Phi = 90^\circ$ $\Psi = 20^\circ$	1	-	RT, HT	[42]
Al (A356)	Effect of reheating and Partial Re-melting	$\Phi = 90^\circ$ $\Psi = 20^\circ$	8	A	RT	[43]
2014 Al alloy	Predicting the critical pre-aging time	$\Phi = 90^\circ$ $\Psi = 20^\circ$	4	B <sub>C</sub>	RT	[44]

Table 1 Continued.

2024 Al alloy	Effect of post aging	$\phi = 90^\circ$ $\Psi = 22.5^\circ$	2	B <sub>C</sub>	RT	[45]
6061 Al alloy	Fatigue design	$\phi = 90^\circ$ , $\Psi = 15^\circ$	4	B <sub>C</sub>	RT	[38]
6061 Al alloy	Grain refinement-Effect of dynamic ageing on the mechanical properties	$\phi = 90^\circ$ $\Psi = 10^\circ$	4	B <sub>C</sub>	100°C, 150°C, 200°C	[46]-[47]
6061 Al alloy	Influence of Friction	$\phi = 90^\circ$ $\Psi = 20^\circ$	1	-	RT	[48]
6061 Al alloy	Enhancement of corrosion resistance-Tensile properties and impact toughness-Improving homogeneity	$\phi = 90^\circ$ $\Psi = 20^\circ$	4-4-2	C	RT	[49]-[50]-[51]
6061 Al alloy	Effect of post cold rolling on hot deformation-Modeling the Hot Ductility	$\phi = 100^\circ$	2	C <sub>x</sub>	RT	[52]-[53]
6061 Al alloy	Effect of aging (processing Temp.) on homogeneity	$\phi = 90^\circ$ $\Psi = 10^\circ$	4	B <sub>C</sub>	100°C, 150°C, 200°C	[54]
6061 Al alloy	Effect of post bake hardening	$\phi = 90^\circ$ $\Psi = 0^\circ$	8	NR	NR	[55]
6061 Al alloy	Investigation of machinability	$\phi = 120^\circ$ $\Psi = 0^\circ$	10	A, B <sub>C</sub>	RT	[56]
6061 Al alloy	Hot and Cold Tensile Behavior	$\phi = 110^\circ$ $\Psi = 20^\circ$	4	B <sub>C</sub>	RT	[57]
6063 Al alloy	Effect of precipitation hardening	$\phi = 90^\circ$ $\Psi = 20^\circ$	4	B <sub>C</sub>	RT	[58]
6063 Al alloy	Fatigue behavior	$\phi = 90^\circ$	6	A	200°C	[59]
6082 Al alloy	Strengthening study and aging treatment	$\phi = 120^\circ$ $\Psi = 0^\circ$	1	-	RT	[60]
6082 Al alloy	Effect of pre, during and post aging	$\phi = 90^\circ$ $\Psi = 0^\circ$	1	-	RT, HT	[61]
6082 Al alloy	Effects of ECAP+ Shot Peening process (SP) on the fatigue properties	$\phi = 90^\circ$ $\Psi = 20^\circ$	4	B <sub>C</sub>	NR	[62]
7075 Al alloy	Predicting the critical pre-aging time- Texture investigation	$\phi = 90^\circ$ $\Psi = 20^\circ$	4	B <sub>C</sub> - A, B <sub>C</sub>	RT	[44]- [63]
7075 Al alloy	Effect of pre, during and post aging	$\phi = 90^\circ$ $\Psi = 20^\circ$	4	B <sub>C</sub>	Different Temperatures	[64]
7075 Al alloy	Effect of ECAP on aging-texture evolution	$\phi = 90^\circ$ $\Psi = 20^\circ$	3-4	B <sub>C</sub>	RT, 120°C- RT	[65]-[66]
8006 Al Alloy	Effect of semisolid post heat treatment	$\phi = 90^\circ$	1	-	NR	[67]
Al-1.4Si-0.9Cu-0.9Mg-0.6Fe-0.1Mn	Effect of dynamic strain aging on hardness and electrical resistivity	$\phi = 90^\circ$ $\Psi = 20^\circ$	1	-	RT, 50°C, 100°C, 150°C, 200°C	[68]
Al-1.8Fe-0.95Si	Post semisolid processing	$\phi = 90^\circ$ $\Psi = 20^\circ$	1	-	RT	[69]
Al-6Mg	Grain refinement	$\phi = 120^\circ$	4	A, B <sub>A</sub> , B <sub>C</sub> , C	RT	[70]
Al-1Mg	Tailoring grain size and work hardening	-	7	B <sub>C</sub>	-	[71]
<b>Copper and its alloys</b>						
commercially pure copper	Fatigue design	$\phi = 90^\circ$ $\Psi = 15^\circ$	4	B <sub>C</sub>	RT	[38]

Table 1 Continued.

commercially pure copper	Effect of post-rolling-A comparison with TE	$\phi = 110^\circ$ $\Psi = 20^\circ$	4	B <sub>C</sub>	RT	[74-72]-[75]
commercially pure copper	Effect of various strain rates-Fatigue behavior	$\phi = 90^\circ$	8	A	NR	[76]-[77]
OFHC copper	Texture investigation	$\phi = 120^\circ$ $\Psi = 20^\circ$	10	A, B <sub>30</sub> <sup>-</sup> B <sub>60</sub> , B <sub>C</sub>	RT	[78]-[79]
OFHC copper	Grain refinement	$\phi = 120^\circ$	20	A, B <sub>30</sub> , B <sub>45</sub> , B <sub>60</sub> , B <sub>C</sub>	RT	[80]
Various brass and bronze alloys	Investigation of shear localization and segmentation	$\phi = 90^\circ$ $\Psi = 20^\circ$	1	-	RT, 350°C	[81]
<b>Steels</b>						
18Ni maraging steel	Grain refinement	$\phi = 90^\circ$ $\Psi = 0^\circ$	4	B <sub>C</sub>	RT	[16,17,19,82]
Fe-0.22C-2.0Si-3.0Mn	Martensitic transformation	$\phi = 90^\circ$ $\Psi = 0^\circ$	2	B <sub>C</sub>	RT	[83,84]
Fe-17.6Ni-8.3Co-4.5Mo-0.7 Ti martensitic steel	Grain refinement	$\phi = 90^\circ$ $\Psi = 0^\circ$	4	B <sub>C</sub>	RT	[85]
Fe-10Ni-7Mn	Cold roll Vs. ECAP	$\phi = 90^\circ$ $\Psi = 0^\circ$	4	B <sub>C</sub>	RT	[86]
Fe-22Mn-3Si-Al austenitic TWIP steel	Feasibility of RT ECAP processing of TWIP steel	$\phi = 120^\circ$ $\Psi = 45^\circ$	1	-	RT	[87]
commercially pure Fe	Compare ECAP with cold rolling and drawing by AFM investigations	NR	4	A	RT	[88]
Commercial 304 SS	Grain refinement	$\phi = 90^\circ$ $\Psi = 20^\circ$	3	B <sub>C</sub>	RT	[89]
AISI 316L austenitic stainless steel	Corrosion and biological behavior	$\phi = 105^\circ$ $\Psi = 20^\circ$	8	B <sub>C</sub>	350°C	[90,91]
<b>Titanium and its alloys</b>						
commercially pure titanium	Corrosion behavior	$\phi = 90^\circ$ $\Psi = 20^\circ$	8	B <sub>C</sub>	450°C	[92]
Commercial purity titanium	Grain refinement-Recrystallization kinetics-Effect of post rolling on grain refinement- Electrochemical and cellular behavior	$\phi = 105^\circ$ $\Psi = 20^\circ$	10	B <sub>C</sub>	250°C	[97-93]
Ti-6Al-4 V	Effect of ultrasonic vibrations on the ECAP process	$\phi = 120^\circ$ $\Psi = 20^\circ$	4	B <sub>C</sub>	RT	[98]
<b>Magnesium and its alloys</b>						
AZ31 Mg alloy	Effects of rare earth elements and Ca additions	$\phi = 90^\circ$ $\Psi = 20^\circ$	4	B <sub>C</sub>	200°C	[99,100]
AZ31 Mg alloy	Grain refinement	$\phi = 90^\circ$ $\Psi = 20^\circ$	4	B <sub>C</sub>	200°C	[101]
AZ31 Mg alloy	Grain refinement	$\phi = 90^\circ$ $\Psi = 20^\circ$	4	B <sub>C</sub>	230°C	[102]
AZ31 Mg alloy	Correlation between shear punch and tensile measurements	$\phi = 90^\circ$ $\Psi = 20^\circ$	4	B <sub>C</sub>	230°C	[103]
AZ31 Mg alloy+Al-3Ti-0.15C master alloy as grain refiner	The grain refinement efficiency of Al-3Ti-0.15C master alloy	$\phi = 90^\circ$ $\Psi = 20^\circ$	4	B <sub>C</sub>	230°C	[104]
AZ80 Mg alloy	Grain refinement	$\phi = 90^\circ$ $\Psi = 20^\circ$	6	B <sub>C</sub>	220°C	[105]
AM60 Mg alloy	Fatigue behavior-Grain refinement	$\phi = 90^\circ$ $\Psi = 20^\circ$	6	B <sub>C</sub>	220°C	[106,107]
AM30 Mg alloy	Grain refinement	$\phi = 90^\circ$ $\Psi = 20^\circ$	4	B <sub>C</sub>	275°C	[108]

Table 1 Continued.

Mg-12Li-1Zn alloy	Superplasticity	$\Phi = 90^\circ$ $\Psi = 20^\circ$	2	B <sub>C</sub>	200°C	[109]
GZ31magnesiumalloy	Superplasticity	$\Phi = 90^\circ$ $\Psi = 20^\circ$	2	B <sub>C</sub>	280°C	[110]
<b>Metal Matrix Composites</b>						
AZ31 magnesium alloy+ Al <sub>2</sub> O <sub>3</sub> nano-particles	Grain refinement	$\Phi = 90^\circ$ $\Psi = 20^\circ$	4	B <sub>C</sub>	230°C	[102]
Cu-1.1 wt-% Al <sub>2</sub> O <sub>3</sub> +Al <sub>2</sub> O <sub>3</sub> particles	Softening behavior-Effect of strain path	$\Phi = 120^\circ$ $\Psi = 60^\circ$	1-2	-A,B,C	RT	[111,112]
Al+ $\alpha$ -Al <sub>2</sub> O <sub>3</sub> nanoparticles	Fabrication (consolidation)	$\Phi = 90^\circ$ $\Psi = 20^\circ$	4	B <sub>C</sub>	200°C	[115-113]
Al-4.5%Mg +10Vol% SiC <sub>p</sub>	High strain rate superplasticity	$\Phi = 90^\circ$ $\Psi = 20^\circ$	8	B <sub>C</sub>	RT	[116]
AA6061-5% SiC <sub>p</sub>	Effect of Aging	$\Phi = 120^\circ$ $\Psi = 60^\circ$	2	A	RT	[117]
PureAl+ nickel based(90%Ni-10%Cr)wires	Flow behavior	$\Phi = 90^\circ$ $\Psi = 20^\circ$	2	A	RT	[118]
<b>Bimetals</b>						
Al-Cu bimetal	Fabrication	$\Phi = 90^\circ$ $\Psi = 45^\circ$	2	A, C	350°C	[3]
AA1100- commercially pure copper bimetal	Diffusion bonding (fabrication)	$\Phi = 120^\circ$ $\Psi = 0^\circ$	1	-	RT+HT	[119]
Al-Cu bimetal	Fabrication	Various $\Phi$ s and $\Psi$ s	1	-	RT+HT	[120]
Al-Cu bimetal	Cold-Welding and Diffusion-Bonding	$\Phi = 120^\circ$ $\Psi = 0^\circ$	1	-	RT+HT	[121]
<b>Others</b>						
Sn-lwt.% Bi alloy	Creep and superplasticity	$\Phi = 90^\circ$ $\Psi = 20^\circ$	4	B <sub>C</sub>	RT	[23]
Sn- 5 wt.% Sb	Superplasticity	$\Phi = 90^\circ$ $\Psi = 20^\circ$	4	B <sub>C</sub>	RT and HT	[122,123]
Commercially pure aluminum powder	Fabrication (consolidation)	$\Phi = 90^\circ$	1	-	200°C	[124,125]
NiTi shape memory alloys	Feasibility of RT ECAP processing of NiTi shape memory alloys-study martensitic stabilization and recrystallization kinetics-Effect of post-deformation annealing	$\Phi = 90^\circ$ $\Psi = 20^\circ$	2-1-1	C	RT	[129-126] - [130,131]

RT: room temperature, HT: high temperature, NR: not reported;  $\Phi$ = channel angle,  $\Psi$ =outer corner angle

### 3. Processing by ECAP

In this section, some applications on ECAP, which are not well described in several earlier review articles [29,132-135], are discussed.

#### 3.1. ECAP as a Strain-Induced Melt Activation (SIMA) process

Semi-solid metal processing is an alternative for conventional casting routes due to its lower cycle time, the possibility of increasing die life, the potential for a reduction in porosity and solidification shrinkage, improvements in the mechanical properties and so on [136]. Using this method, semi-solid billets with a spheroidal rather than a dendritic microstructure are formed to manufacture near net shaped products at the semi-solid temperature, referred to as a temperature between the liquidus and solidus. The first and the most important step in semi-solid processing is the preparation of semi-solid billets. Strain induced and melt activated (SIMA) is a good method to produce feedstocks, especially for active metals. In general,

SIMA consists of two steps, a strain-induced step and a melt-activated step. For the strain-induced step, different processes such as rolling, forging, upsetting and extrusion can be used. The main restriction of all of these methods is the limitation in the degree of plastic deformation which is more critical for “hard to deform” materials like Mg and Ti alloys. In order to overcome the difficulties of plastic deformation in conventional methods, Jiang et al. [137] used ECAP as a SIMA process for semi-solid processing and concluded that it was valuable to include a strain-induced step. This new SIMA is a good method to prepare a semi-solid of the AZ91D alloy owing to its desirable microstructure with fine spheroidal grains and its high mechanical properties of formed components at both room and elevated temperatures. Shortly after this research, Liang et al. [138] showed that two passes of ECAP could provide sufficient strain to achieve fine and globular grains for the semi-solid forming of an AZ91 alloy. At the same time, Ashouri et al. [15], investigated the microstructural changes of an aluminum A356 alloy fabricated by semi-solid processing using ECAP. They proposed a model for the semi-solid structure formation of ECAP specimens as shown in Fig. 2. The initial as-cast dendritic microstructure is changed to an elongated shape with a banded structure after ECAP. During reheating, the dendrites start to disintegrate due to the penetration of the liquid phase into the high-energy grain boundaries. By increasing the holding time, the connected and agglomerated recrystallized microstructure disintegrates and grain spheroidization and coarsening are activated. Due to the Gibbs-Thomson effect, the sharp edges of the deformed dendrites with lower melting points change gradually to globular shapes. It is interesting to note that other Iranian scholars, Meidani et al. [18], had already reported the formation of globular structure by ECAP and isothermal treatment of a semi-solid aluminum A356 alloy. Similar reports for semi-solid processing of aluminum A356 alloy, [21,41,43] and aluminum A8006 alloy [67,69] were published by Iranian scholars.

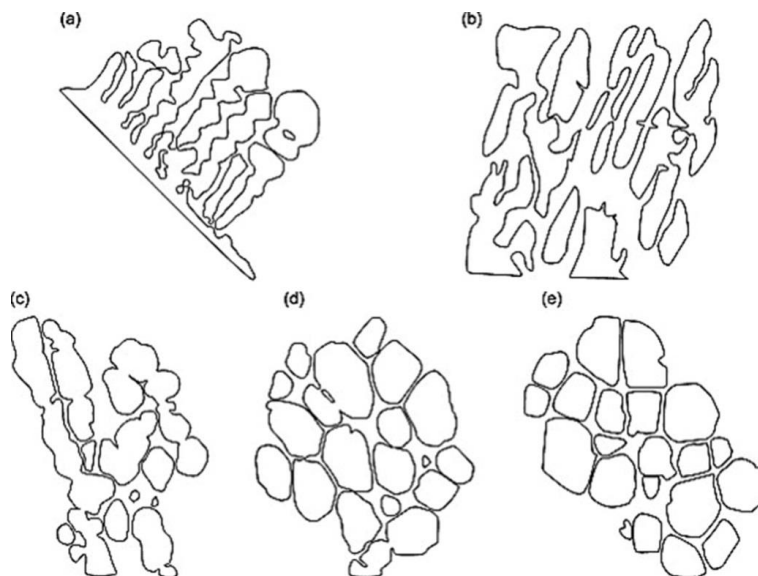


Fig. 2. Schematic illustration of microstructural evolution of the as-cast, deformed and reheated semi-solid structure formation of ECAPed specimens: (a) as-cast, (b) 4 passes of ECAP, (c) 5 min reheated, (d) 10 min reheated and (e) 20 min reheated [15].

The flow-plane of an A356 aluminum alloy billet after equal channel angular pressing in the semisolid state is shown in Fig. 3. As observed, the dendritic solid phase is inclined to the horizontal X-direction over a range of angles from  $20^\circ$  to  $60^\circ$  in an anticlockwise rotation. The regions A and B show different degrees of deformation apparently due to different constraints during ECAP. In region A, second phase particles have been distributed in a uniform matrix which could be dynamically recrystallized during hot deformation. On the other hand, the dendritic arms have been fragmented in region B.

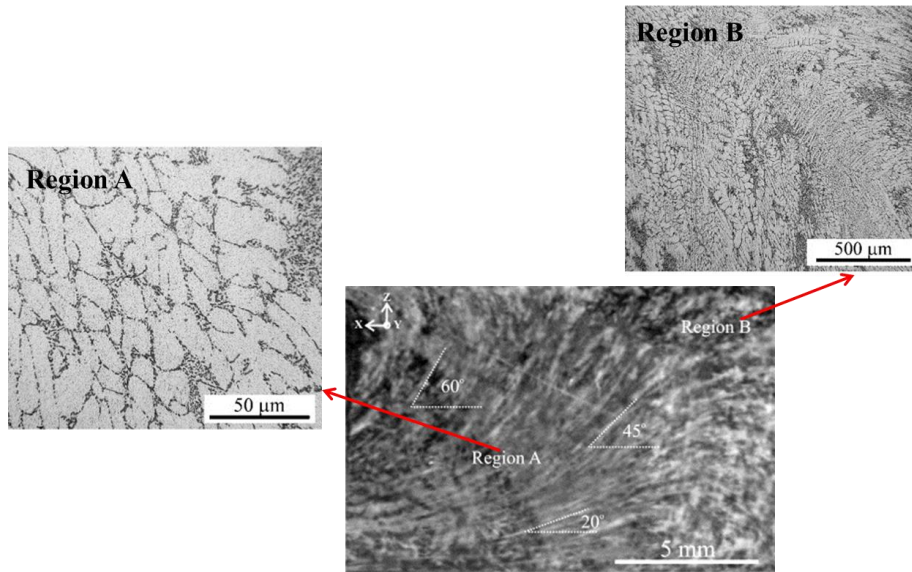


Fig. 3. The flow-plane of an A356 aluminum alloy billet after equal channel angular pressing in the semisolid state (extracted and rearranged from [18]).

### 3.2. ECAP as a consolidation method

Although ECAP is generally used for microstructural refinement in solid metals, it may be used also for the consolidation of metallic powders. The mechanism for powder consolidation by ECAP is the deformation of the particles which is different from sintering in which the main mechanism is diffusion as shown in Fig. 4 [139]. Usually a layer of oxide that covers the surface of metal particles prevents their bonding with each other. In conventional sintering processes, the compacted particles are heated up to elevated temperatures and held from a few minutes to a few hours. Atoms then diffuse through the surface or the interior of the particles to reduce the surface energy and, as a result, the bonding and densification are achieved. Therefore, during sintering, a density increment happens because the gap between the particles is filled. Since the material flow is limited during sintering, a complete elimination of pores and full densification are almost impossible. Nevertheless, the oxide layer is fractured during SPD consolidation and this leads to a direct contact between the metal surfaces. In addition, the material flow fills any gap between the particles and helps to achieve the full density. In this case, no diffusion is required for bonding. Therefore, the processing can be carried out at lower temperatures and consolidation is obtained instantaneously as the particles are deformed.

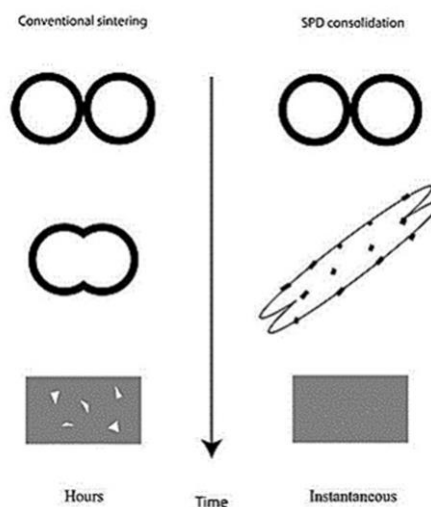


Fig. 4. Schematic illustration of particle consolidation in conventional sintering (left column) and SPD consolidation (right column) [139].



Xiang et al. [140] were the first researchers to use ECAP for the consolidation of metallic powders and they were able to consolidate an Al-2024 alloy powder by ECAP at 300°C up to a maximum of 3 passes. A pre-cold isostatic pressing (CIP) of the powder was a prerequisite for the process. However, cracks were formed on the specimen surface even after a single pass of ECAP. In 2000, Matsuki et al. [141] showed that the use of a cap (Fig. 5a) prior to ECAP prevented the surface cracking after ECAP processing. The CIP sample of Al-2024 alloy powder (18 mm in diameter and 35 mm in length) was put into a 2024Al cap (Fig. 5b) and was then consolidated by ECAP at 300°C without surface cracking (Fig. 5c). Successful ECAP consolidation has been performed on several powders including Al [142], 6061 Al [143], 2024 Al [141,144,145], Mg [146,147], AA4032- CNT composite [148], WC+Co[149,150], Ti [142], Ti-6Al-4V [126,151], titanium silicide ( $Ti_5Si_3$ ) [152], Cu [153,154], Cu-Ta alloys [155], amorphous Al ( $Al_{89}Gd_7Ni_3Fe_1$  and  $Al_{85}Ni_{10}Y_{2.5}La_{2.5}$ ) [156], amorphous Cu ( $Cu_{50}Ti_{32}Zr_{12}Ni_5Si_1$ ) [157], carbon nanotube (CNT)-metal matrix composites [158,159], Al-Si [160], Al- $Al_2O_3$  [161,162], Al-AlN [163], Vitreloy 106a ( $Zr_{58.5}Nb_{2.8}Cu_{15.6}Ni_{12.8}Al_{10.3}$ )+W [160], Ti-SiC[164], Ti-Al [165,166], Al- $Y_2O_3$  composite [167], AA7075- $ZrO_2$ [168] and Al- $Al_2O_3$  [169].

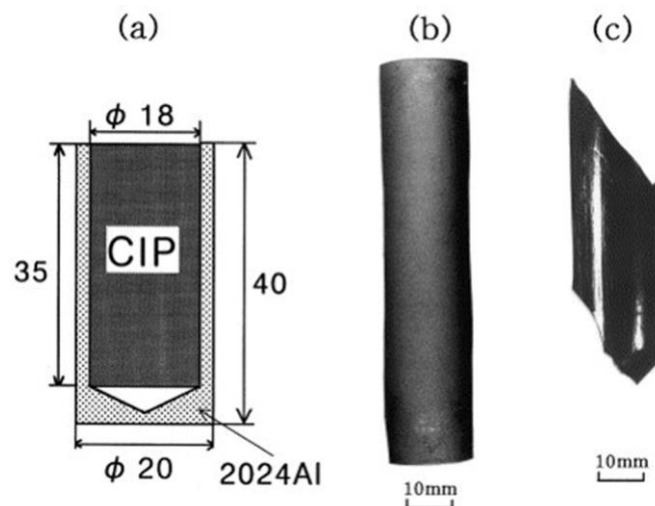


Fig. 5. Specimen configurations to prevent surface cracking before and after ECAP of CIP Al 2024 alloy powder: (a) the cross-section of the CIP specimen with a 2024 Al cap, (b) the CIP specimen before pressing, and (c) the consolidated alloy after a single pass of ECAP at 300°C [141].

In 2005, Xia and Wu [170] used back-pressure equal-channel angular consolidation (BP-ECAC) to consolidate pure Al particles at a low temperature of 100 °C. They concluded that applying a back-pressure (BP) to the front of the samples on the exit channel of ECAP is preferable to ECAP without back-pressure due to the achievement of full density and good bonding after only a single pass. This serves to eliminate the pre-compaction step and can use loose particles. BP was also used in different studies to consolidate a wide range of powders such as Al [139,171-173], Mg [174,175], Al matrix composites [176], Al- $Al_2O_3$  composite [177], Al-C nanocomposites [178], Cu+ $MgB_2$ [154,179], Ti [180], Ti-6Al-4V [181,182] and TiN-Ti composites [183]. The application of PB needs some additional equipment, such as a double action pressing machine that has the capability of controlling the forward- and backward-pressure [184], and the process tends to be time-consuming and expensive.

To overcome these problems, Paydar et al. [11] suggested that the extrusion process designed in the second channel of the ECAP die could provide the required back-pressure to achieve almost fully dense samples. The most important feature of this method, named ECAP-FE, was that two processes, ECAP and FE, were carried out subsequently in a single tool (Fig. 6a) that led to the elimination of additional equipment. These researchers also showed that the consolidation of pure aluminum powder at 200 °C

produced by ECAP-FE with an extrusion ratio of 10 could achieve a density comparable with that of fully dense bulk aluminum. In addition, using an Arrhenius equation, they calculated the self-diffusion coefficient of Al during ECAP and ECAP-FE. In a condition when no load had been applied, the back-pressure created during ECAP-FE increased the self-diffusion coefficient of Al by a factor of 3.8 relative to its value during simple ECAP. The increase in the self-diffusion coefficient of Al during ECAP-FE had a significant effect on the pore closure and improved the mechanical properties of the workpiece.

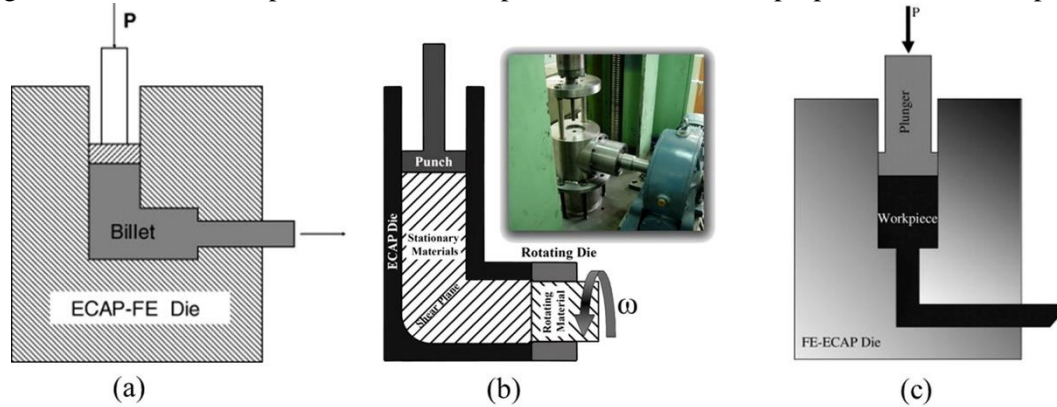


Fig. 6. Schematic view of the combined methods based on ECAP to consolidate powders: (a) ECAP-FE[11]; (b) T-ECAP [185]; (c) FE-ECAP [124].

Another combined method which was used to consolidate powders is torsional-equal channel angular pressing (T-ECAP) [185] which is shown in Fig. 6b. Consolidation of pure Al particles showed that after three passes of T-ECAP, the maximum density was achieved. Mani et al. [185] investigated the pore shape change and closure during T-ECAP by using metallographic inspections. It was shown that the porosities changed from an elongated shape/morphology in the ECAP stage into a nearly spherical one in the rotating stage during the T-ECAP. Despite the similarities between the T-ECAP and ECAP-FE, there are two main differences between these processes: the feasibility of the T-ECAP process and the requirement for using cans in T-ECAP prior to processing. Furthermore, the rotating part of the T-ECAP needs additional equipment which tends to be costly. Therefore, it seems that ECAP-FE is more applicable than T-ECAP but it needs more detailed studies to clarify the different aspects of these two processes.

Although ECAP was usually used as an alternative technique for conventional methods like compaction plus sintering and extrusion to consolidate powders, in some studies it was used as a post-process in order to refine the microstructure and enhance the mechanical properties. In 2008, Paydar et al. [124] consolidated Al particles successfully using ECAP after an extrusion channel in a single die, referred to as forward extrusion-equal channel angular pressing (FE-ECAP) which is shown in Fig. 6c. A good bonding with superior mechanical properties was achieved after FE-ECAP compared to FE. The yield strength for the consolidated air atomized commercially pure aluminum powder with an average particle size of 45  $\mu\text{m}$  was respectively 142 and 124 MPa for the ECAP-FE [125] processes processed at 200°C. There was a similar behavior for the ultimate tensile strength and hardness measurements. The fabrication of Al-10 vol% SiC<sub>p</sub> composites by FE and FE-ECAP at 550 °C resulted in an increase in the ultimate tensile strength from 136 MPa for FE to 148 MPa for FE-ECAP [186,187]. As illustrated in Fig. 7, the bulk material resulting from FE-ECAP and ECAP-FE techniques possesses high strength and excellent ductility which are respectively comparable and much superior to those achieved by conventional extrusion. Dislocation strengthening is the most important mechanism responsible for the high strength of these workpieces.

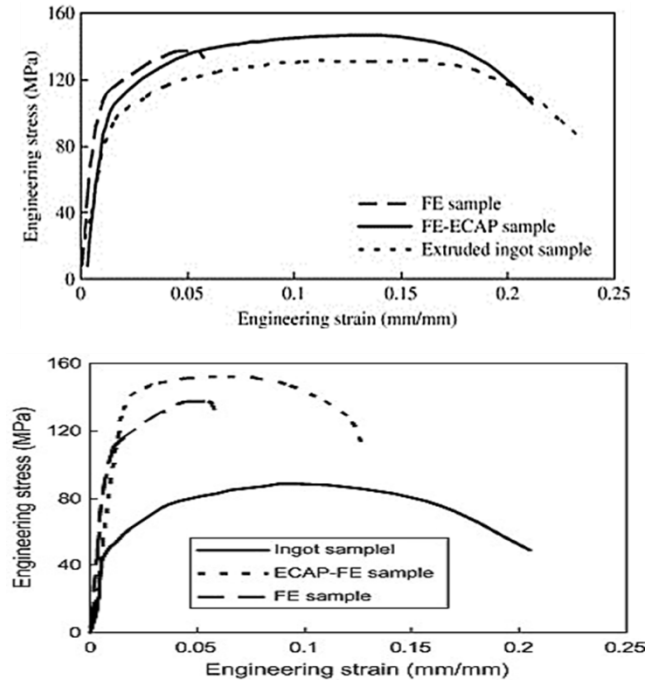


Fig. 7. Engineering stress-strain curves of the ingot, FE, and FE-ECAP [10] and ECAP-FE [11] consolidated pure aluminum powders.

In 2010, Nagasekhar et al. [188] compared the deformation flow, strain homogeneity, and load requirements of FE-ECAP and ECAP-FE with those of individual FE and ECAP processes using finite element analysis. Fig. 8 shows the effective strain variation across the width of a cross-section of the studied specimen from top to bottom. The effective strain is more uniform in the ECAP process. They also concluded that the strain distribution in the combination processes, FE+ECAP and ECAP+FE, was dominated by the deformation behavior and strain distribution of the FE process. Therefore, by comparing FE-ECAP to ECAP-FE, it can be concluded that ECAP-FE is more effective than FE-ECAP in order to consolidate powders because it imposes higher strains and results in a more uniform strain distribution and better mechanical properties of the product.

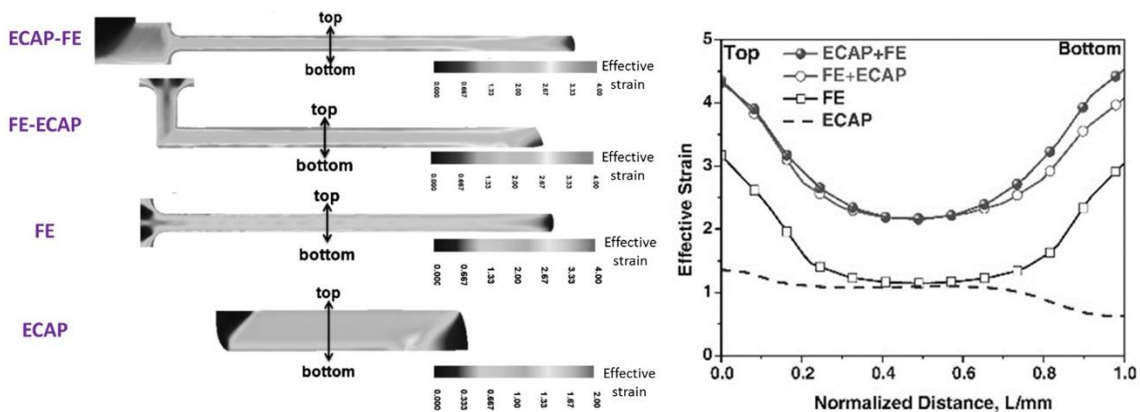


Fig. 8. Effective strain variation across the width of the cross-section from top to bottom in various deformation processes, ECAP-FE, FE-ECAP, FE and ECAP [188].

#### 4. Modeling and Simulation

Material modeling and simulation can help complement experimental results and enable researchers to predict the deformation behavior and the properties of materials. Since the primary analytical models [189,190] proposed to estimate the strain in ECAP, several methods have been used to model and to

simulate different aspects of the process. These include finite element modeling (FEM) [188,191-230], upper bound (UB) models [12,231-258], visco plastic self-consistent (VPSC) models [206,209,259-280], Monte-Carlo modeling [8,9,281-285] and artificial neural network (ANN) [286-291]. By using these strategies many different characteristics of ECAP and ECA pressed samples were predicted successfully including effective strain and strain homogeneity [189,190,233,248,266,292-299], processing parameters [12,210,213,231-258,287,300,301], microstructure [9,207,260,302-318], texture [198,209,221,260,262,276,303,311,315,319-333] and mechanical properties [312,322,324,334-343].

Taking a brief look at the publications in the field of ECAP, it can be noticed that about 13% of the records are involved in simulation and modeling. As shown in Fig. 9, this amount makes up almost 17% of all documents published in this field by Iranian researchers, which represents its great popularity among Iranian scholars. There can be a number of factual reasons for this issue. Firstly, Iran has not signed or ratified any of multilateral international copyright treaties. Therefore, Iranian researchers have access to a wide range of software packages and they have become experts in developing simulations. Secondly, Iran allocates fewer funds to research and has less access to advanced characterization methods. The easy access to software packages, therefore, means that simulations are cheaper than experiments.

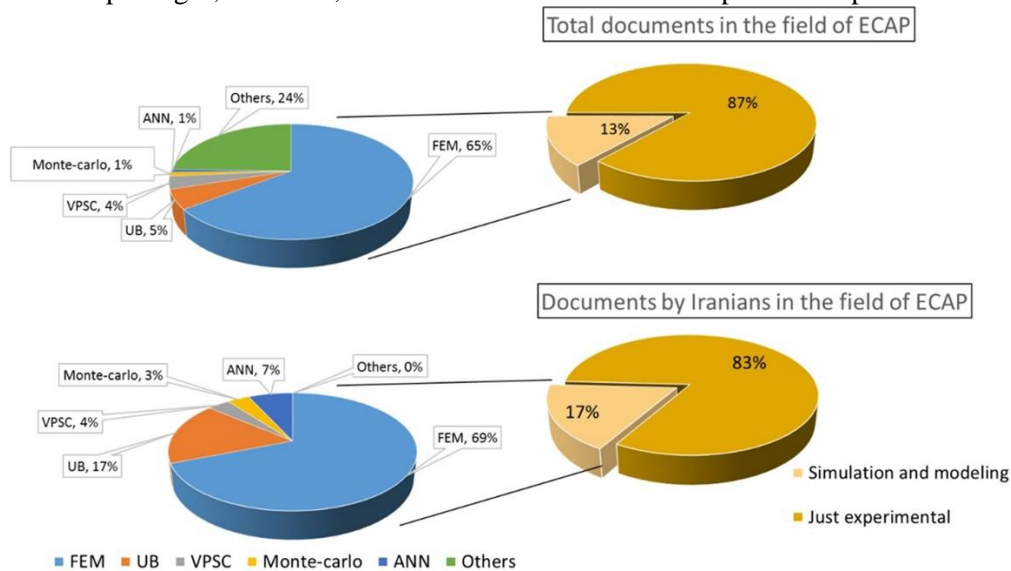


Fig. 9. Fraction of publications that are focused on modeling and simulation with respect to the total number of publications on ECAP as well as the contribution of various methods in modeling and simulation

As summarized in Table 2, most of the modeling and simulations by Iranian researchers were confirmed by experimental evidence. According to Fig. 9, about 69% of the documents published by Iranians on simulations and modeling have used FEM for simulation, while this amount is ~65% for the total documents. This shows that most FEM studies are simply case studies which are relatively easy to confirm. However, it is interesting to note that Iranian scholars are pioneers in both old and new modeling methods. Thus about 17% of the records of ECAP in this field have used an upper bound (UB) method (a basic analysis method) to investigate deformation behavior during ECAP. On the other hand, only 5% of the total publications in modeling and simulations are based on UB models (Fig. 9). More than 20% of all the research that used UB models to analyze ECAP was published by Iranians.

Parallel to the first theoretical studies in Iran to estimate the strain [4,344], a new configuration of ECAP nominated as the multi-stage ECAP was used to calculate the strain in ECAP, and the first UB models to analyze ECAP were presented. In the multi-stage ECAP die, the deformation zone was divided into a number of “sub-dies”, and each of them inserted a shear strain into the material. By this approach the total strain was calculated simply, and interestingly it was the same as the relationship proposed by

Alkorta and Sevillano [234]. Eivani and Karimi Taheri [239], applied a UB model in order to investigate the plastic deformation behavior of the material during ECAP. Although there were numerous reports that focused on the UB analysis of ECAP [232,234,236,237,345], before that time none of them had considered the effects of parameters such as friction, die angle and the outer curved corner angle on the mechanics of the process. Thus these works were the first to consider the effect of all the process parameters simultaneously.

In 2009, Reihanian et al. [245] used, for the first time, a new upper-bound approach based on Avitzur and Pachla [346,347] to analyze the ECAP. The same researchers, again for the first time, proposed a UB approach to analyze ECAP with circular cross-sections [12]. A short time after this work, Eivani et al. [348] proposed another UB model for circular cross-sections which considered the effect of the deformation passes. In 2010, in a collaboration between Iranian and Korean researchers, the effects of temperature and strain rate on the mechanics of ECAP at elevated temperatures were investigated by a UB model [251]. For this reason a parameter known as the velocity-modified temperature ( $\bar{T}$ ) was utilized to relate the temperature to strain rate. They used the same formulation as that proposed by Paydar et al. [12] but with the consideration of the effect of velocity-modified temperature. By using the UB analysis, the powers dissipated during the process and consequently the processing load of the process could be calculated. Also, it is possible to minimize the processing load by optimizing the processing parameters such as die angles and frictional forces.

One of the most important microstructural features of ECAP samples is their texture that affects other material behaviors such as strength, plastic anisotropy, formability, grain refinement, and fracture. Therefore, the simulation of texture evolution during processing has attracted the attention of many in the materials science community. To study the details of the developments in the simulation and modeling of texture, a comprehensive review of this field was published earlier [31]. Despite the wide range of articles on simulations and modeling of texture, there are only a limited number of reports by Iranian researchers [24,349]. Since these works were conducted with contributions from French and Canadian researchers, it is suggested that the lack of equipment, such as XRD and EBSD, is the main reason for the lack of studies in the simulation of textures in Iran.

Another aspect, which is important for all researchers, is to predict the mechanical properties of the materials after ECAP. Methods to predict the mechanical responses of materials after SPD processing based on dislocation kinetics are well reviewed by Vinogradov [350]. Regarding the dislocation-based approach, the premature strain localization in UFG metals and alloys manufactured by SPD is intimately related to the dislocation density evolution. In this report it was concluded that the uniform elongation is primarily controlled by the rate of dislocation recovery [351].

Table 2. Summary of the methods, their novelty and the aim of the simulations and models proposed by Iranian researchers.

Method	Novelty	Scope of research	Confirmation	Ref.
multi-stage ECAE dies	Simplification	Strain estimation	Previous models	[4,344]
Continuum mechanics	Introduce a new equation base on the logarithmic strain	Effect of die geometry on strain homogeneity and mean effective strain	Previous models	[22]
Upper bound analysis	Consideration of all the process parameters simultaneously	Effects of die geometry and friction coefficient on the total strain and extrusion pressure	Experiments and a previous model	[239]

Table 2 Continued.

Upper bound analysis	Consideration of the effect of dead metal zone formation	Effect of dead metal zone formation on strain and extrusion force	Experiments and a previous model	[7]
Upper bound analysis	Using rotational and linear velocity fields by the approach of Avitzur and Pachla [346,347]	Effect of constant friction factor and inner corner radius on the development and shape of PDZ was investigated.	Experimental results	[352]
Upper bound analysis	Analyze the circular cross-sections	Prediction of the size of the plastic deformation zone and the relative extrusion pressure	Experimental results	[12]
Upper bound analysis	A solution for circular cross-sections (axi-symmetric channels)	Effect of deformations passes on the extrusion pressure	Experimental results	[348]
Upper bound analysis	Using a Bezier-shaped curve to define a general streamline	To introduce a more general and much improved formulation with respect to previous works	Previous experiments and models	[246,295]
Upper bound analysis	Theoretically consider the effects of temperature and strain rate on plastic flow behavior during the ECAP	Investigation of the plastic deformation and dynamic strain ageing behavior of Al-6082 alloy	Experimental results	[251]
A flow line model	consider the outer curve of ECAP die wall	Predict the dislocation structure evolution and nano-structure	Previous experimental results	[353]
A flow line model	Propose a new flow line function	Determine the velocity field, the strain rate field, and, in general, the shape of the plastic deformation zone and also to simulate textures	Experimental results	[349]
A kinetic dislocation model + Monte Carlo algorithm	Combination of a flow filed model, kinetic dislocation model, and Monte Carlo algorithm	Prediction of the recrystallized microstructure	Experimental results	[9]
A Monte Carlo model	Modification by using Turnbull–Fisher nucleation rate model	To predict the microstructure evolution during annealing	Previous experiments	[8]
ETMB model [354]	Introduce more solidarity in the model	Investigation of flow softening of FCC materials during SPD	-	[355]
Simulations by VPSC model	-	Multi pass simulation of texture	Experimental results	[24]
FEM simulation	-	Influences of die parameters (and material properties) on deformation by equal channel angular pressing with parallel channels (ECAP-PC)	Experimental results	[356]
FEM simulation	-	Investigation of deformation behavior of pure Al	Experimental results	[357]
FEM simulation	Propose channel angular deformation (CAD) as a substitute for the traditional direct extrusion	Test different CAD profiles to find the especial geometries of CAD which could be used as an alternative for direct extrusion effectively	-	[358]
FEM simulation	Design ECAP die based on the optimum strain behavior	Selection of ECAP parameters based on strain distribution uniformity	Experimental results	[39]
FEM simulation	using Gurson model [359]	Modeling of densification of aluminum powder in tube through multipass ECAP at room temperature	Experimental results	[360]

Table 2 Continued.

FEM simulation	A new specimen and punch modification has been proposed to reduce the head and tail zone effects of ECAPed billet	Fabricate materials with least wastage, higher ES magnitude and better strain dispersal homogeneity	Experimental results	[361]
FEM simulation	superimposing ultrasonic vibration during ECAP	Propose a more realistic approach to simulate ultrasonic-assisted metal forming to investigate the effect of ultrasonic vibrations on the forming conditions in ECAP p	Experimental results	[362,363]
FEM simulation	-	Damage prediction of AA7025 alloy during ECAP	Experimental results	[364]
FEM simulation	A Comparison Between Numerical and Analytical Modeling of ECAP	Investigation of the strain, strain rate and load of processing for AA6101 aluminum alloy	Previous UB analysis	[365]
FEM simulation + RSM + ANOVA	The first study on optimization of ECA rolling parameters	Obtain the optimum values of the ECA rolling parameters	Experimental results	[366]
FEM simulation and ANN Modeling	-	Study the deformation behavior of AA2024 alloy	Experimental results	[286,287]

VPSC: Visco plastic self-consistent; ETMB: (Estrin–Tóth–Molinary–Bréchet); FEM: finite element method; ANN: artificial neural network; RSM: response surface methodology; ANOVA: analysis of variance;

## 5. Special Characterization

The main objective of ECAP is to refine the microstructure in order to achieve exceptional mechanical and physical properties. Therefore, the first aim of most of researchers is to investigate the microstructure and texture of the processed sample that was well reviewed in numerous reports [29,31,132-134,350]. As summarized in Table 1, many different aspects of ECAP samples include microstructure, texture, mechanical properties, fatigue behavior, superplasticity, creep and corrosion. To avoid repeating the reports already published in earlier reviews, it is sufficient to address only some novel achievements in the characterization of ECAP samples by Iranian researchers. These achievements are those that result in a progress in this field, thereby attract the attention of other scholars.

### 5.1. Microstructural inhomogeneity during ECAP

When the deformation is ideal during ECAP, the stress exhibits a uniform distribution in the cross-section of the billet and consequently a homogenous strain distribution is expected [351]. Because of the homogeneous strain distribution, a uniformity of microstructure and mechanical properties in ECAP specimens is essential. However, ideal material flow during ECAP is far from a reality. Therefore, investigations of the microstructural uniformity during ECAP are very important. This research theme was the aim of the first study in Iran on ECAP processing which was conducted on commercially pure Al billets. In this study, the microstructural homogeneity through the section parallel to the pressing direction (transverse direction (TD)) of ECAP-processed samples was investigated from 1 to 8 passes through route B<sub>C</sub> [13]. In 2005, Xu et al. [367] investigated the microstructural homogeneity of the ECAP-processed pure Al billets through the perpendicular section to the pressing direction (extrusion direction (ED)) using

microhardness measurements and TEM observations. From the TEM observations, they concluded that in the central region, the elongated grains and the equiaxed grains each occupied the same area fraction of  $\sim 50\%$  while the elongated grains were more prevalent in the vicinity of the lower surface occupying an estimated area fraction of  $\sim 90\%$ . For the elongated grains in the central region shown in Fig. 10, the average widths and lengths were  $\sim 1.3$  and  $\sim 5\mu\text{m}$ , respectively. The size of the elongated grains in the periphery was identical to that of the central region. They also reported that the hardness distribution was inhomogeneous after one pass with a lower hardness in the periphery, but the distribution became essentially homogeneous with additional passes.

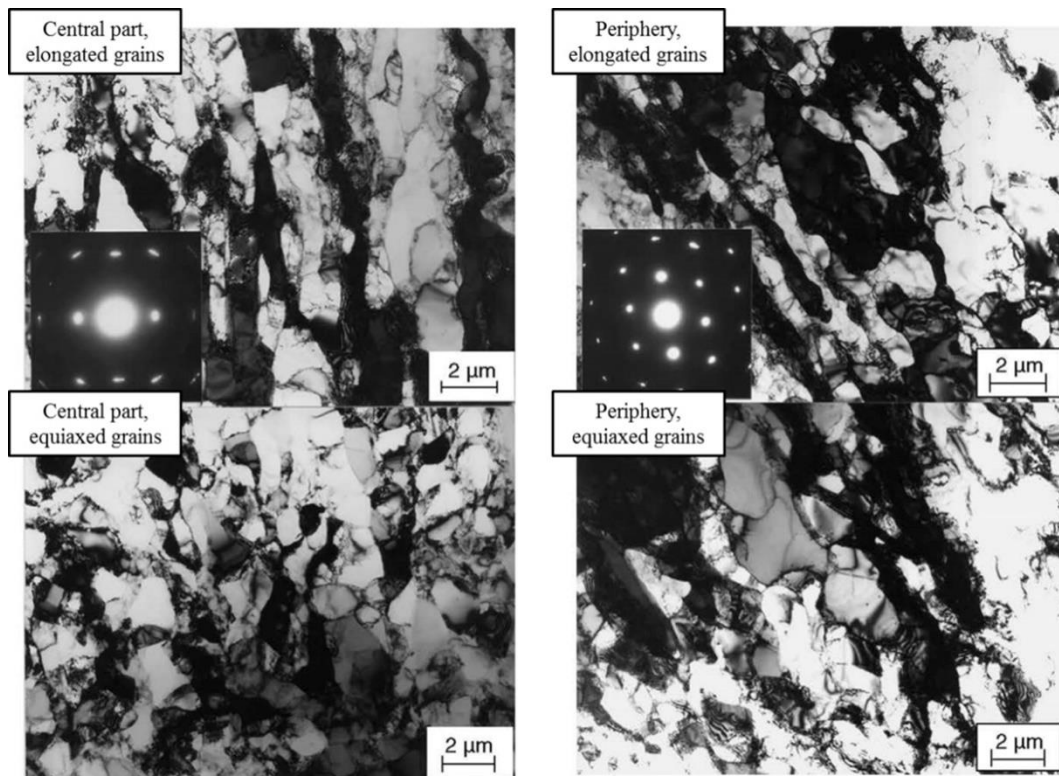


Fig. 10. TEM image of the microstructure in the central and peripheral part of the pure aluminum sample after ECAP through one pass at room temperature [367].

In 2008, using EBSD scans, Reihaniann et al. [13] showed that for all employed passes (from 1 to 8 passes) the fraction of high-angle boundaries ( $f_{HAGBs}$ ) and the mean misorientation angle ( $\theta_m$ ) had their highest values at the center of the sample through the TD-section while the grain size was the lowest. In addition, by measuring from the center to the periphery, the grain size increased while  $f_{HAGBs}$  and  $\theta_m$  decreased gradually. For example, Fig. 11 shows the orientation color maps and boundary misorientation maps taken from the center and periphery of the TD section. The basic microstructural parameters such as mean boundary spacing including all boundaries ( $d^{All}$ ),  $\theta_m$  and  $f_{HAGBs}$  were measured from the center to the periphery of eight ECAPed samples. Therefore, it was concluded that the microstructure after ECAP processing was inhomogeneous through both the ED and TD sections. However, the microstructure became more uniform by increasing the number of passes.



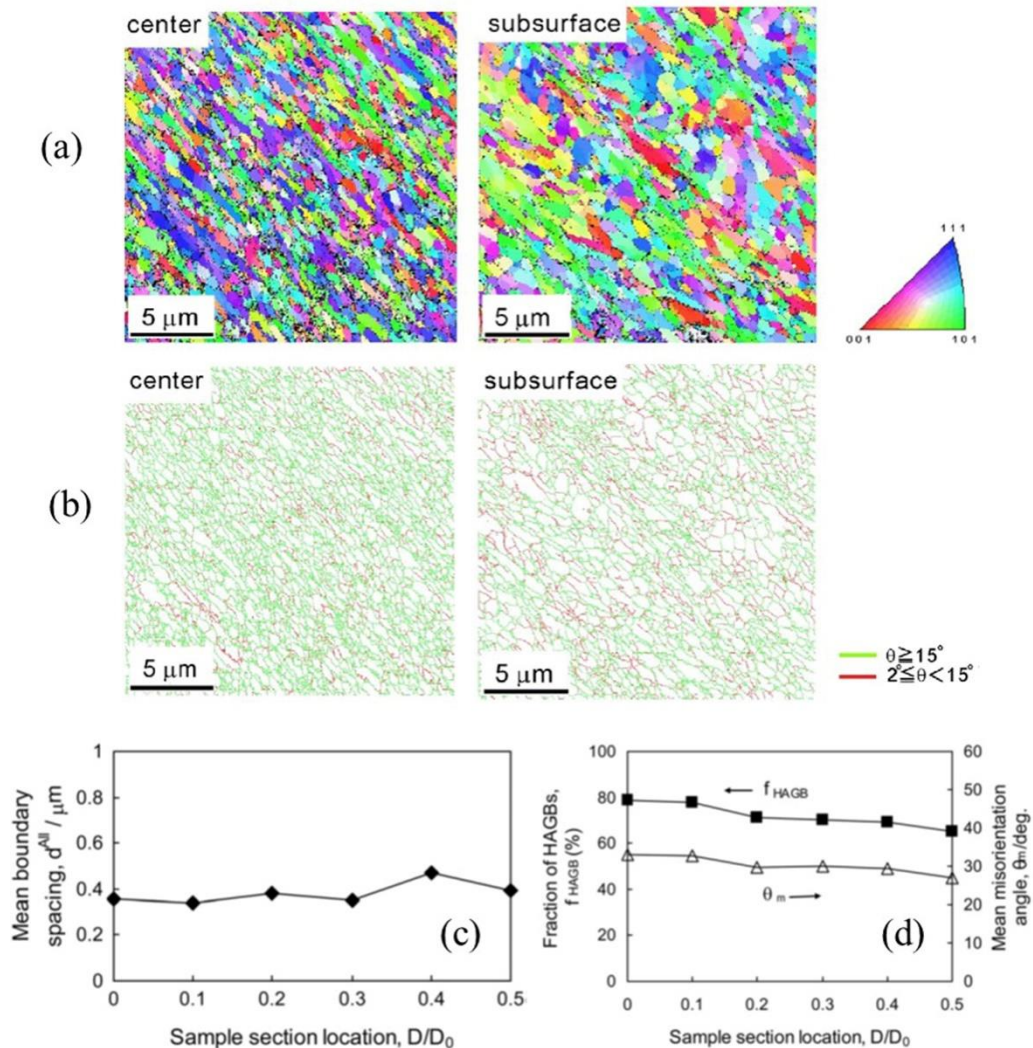


Fig. 11. (a) EBSD orientation color maps and (b) the corresponding boundary maps taken from the center and periphery of transverse direction (TD) section of pure Al subjected to eight passes of ECAP, (c) mean boundary spacing and (d) fraction of high angle grain boundaries and the mean misorientation angle through the TD section of the same sample (all images extracted from [13]).

## 5.2. Superplasticity

The phenomenon of superplasticity implies that a polycrystalline material deformed in tension at low stresses exhibited neck-free elongations of many hundreds of percent prior to failure. This was first reported explicitly by Pearson [368] in 1934. He observed a remarkable tensile elongation of about 1950% in a Pb–Sn eutectic alloy. After more than eighty years of research in this field, many aspects of this phenomenon have been clarified as reported in several review articles [369]. To achieve superplasticity in conventional polycrystalline metals, a small and stable grain size and a relatively high testing temperature (higher than one-half of  $T_m$ , the absolute melting temperature) are required [370]. Therefore, ECAP samples are good candidates for superplastic forming (SPF) [369] in which large volume components with curved and complex shapes are produced due to the very small grain sizes achieved by ECAP. In 2016, Kawasaki and Langdon [371] provided a review to explain the exceptional superplastic flow in a series of UFG aluminum and magnesium alloys after ECAP and HPT. Although more than 400 documents reported the superplastic behavior of UFG materials fabricated by ECAP, the number of publications in this field by Iranians is less than 10 articles. Thus while superplasticity was the aim of 8% of the total ECAP studies, this value was reduced to 3% for those articles published by Iranian researchers.

Most of the reports on the superplasticity of alloys after ECAP have used conventional tensile tests. However, in 2008 Mahmudi et al. [23] employed localized indentation to investigate the superplastic indentation creep behavior of the fine-grained Sn-1%Bi alloy at room temperature ( $T > 0.6T_m$ ) fabricated by ECAP by measuring the stress exponents and strain rate sensitivity (SRS) indices of the alloy after ECAP. In this respect, they used an equation obtained in 1992 [372] to relate the time dependent hardness ( $H_V(t)$ ) to other parameters as in

$$H_V(t) = \frac{\sigma_0}{(C_4 \dot{\epsilon}_0 t)^m} \quad (1)$$

where  $\dot{\epsilon}_0$  is the strain rate at a reference stress  $\sigma_0$ ,  $m$  denotes SRS index,  $C_4$  is a constant,  $T$  is the temperature and  $t$  is the dwell time. Therefore, by plotting  $\ln(H_V)$  against  $\ln(t)$  at a constant temperature, the SRS index can be calculated.

Another method that was used to measure SRS of ECAP alloys is impression testing techniques [122], a modified version of the indentation creep method. In the impression testing technique, the impression depth is recorded during the dwell time after pushing a flat-bottomed cylindrical punch into the test specimen under a constant load. Consequently, the penetration rate or impression velocity can be obtained. A suitable alternative method for conventional tensile tests is shear punch testing (SPT). In SPT, the sample is clamped between two die halves and a flat cylindrical punch is driven through it. Mechanical properties, ultimate shear strength, and shear elongation values can be obtained by plotting shear stress against normalized displacement obtained by SPT. In 2013, Mahmudi et al. [373] used SPT to evaluate the superplastic behavior for the first time. Based on their proposed procedure, it is possible to calculate the SRS index ( $m$ ) through the following relationship:

$$m = \left( \frac{\partial \ln \tau}{\partial \ln \dot{\gamma}} \right)_{\tau} \quad (2)$$

in which  $\tau$  is the shear stress and  $\dot{\gamma}$  is the shear strain rate. Consequently, the slope of a log-log plot of shear stress against shear strain rate gives the SRS index. For instance, in Fig. 12 the shear yield stress was plotted on a double-logarithmic scale against the shear strain rate at various test temperatures for an Mg–Li–Zn (LZ121) alloy containing 11.8 wt% Li and 0.8 wt% Zn after ECAP and extrusion. By increasing the temperature, the strain rate dependence of the shear stress for the fine-grained ECAPed material deviated from linearity toward a typical sigmoidal shape having three distinct regions: I, II and III (Fig. 12a). Due to Eq. (2), the slope of the curves in any of these regions gave the corresponding SRS index ( $m$ ). The maximum values of 0.30 and 0.45 were achieved for the SRS index in the intermediate strain-rate region II for the temperatures of 523 and 548 K respectively. It is noteworthy that  $m$ -value increased as the temperature increased from 473 to 548 K. Therefore, it was established that the SPT, as an easy-to-perform method, is capable of evaluating the shear superplastic behavior of different materials by measuring the SRS index particularly when the material is only available as small test pieces such as those usually obtained by severe plastic deformation processes.

A summary of superplasticity data for different materials after ECAP investigated by SPT can be found in Table 2 in another report [374]. Comparing SPT with other testing methods, it was shown that SPT provides a convenient method for measuring the SRS indices [373]. The main advantage of SPT is its ability to use small thin test pieces such as those usually obtained through SPD processing.

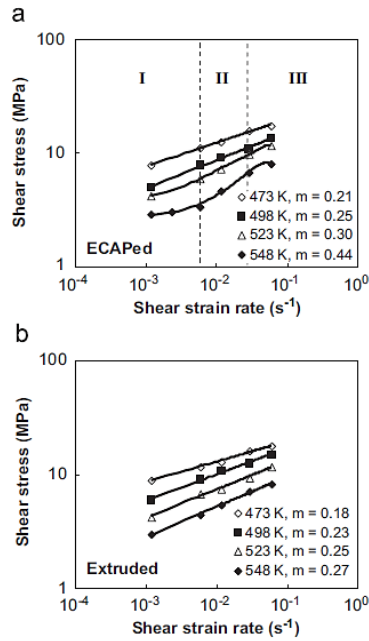


Fig. 12. Shear stress as a function of shear strain rate for (a) ECAPed, and (b) extruded Mg–11.8 wt% Li–0.8 wt% (LZ121) at various temperatures [375].

5.3. Corrosion

Corrosion in polycrystalline materials is strongly affected by their grain size. In a review on the corrosion of UFG materials produced by SPD, Miyamoto [376] concluded that grain refinement by SPD does not sacrifice the overall corrosion resistance and in many cases, SPD can improve it compared to coarse-grained counterparts. For example, it was reported that the corrosion resistance of ultrafine-grained Fe-Cr alloy fabricated by SPD was much higher than that of a conventional grain size [375]. SEM micrograph and atomic force microscopy images of the surfaces of as-received -grained (CG) annealed and nanocrystalline (NC) samples of an Fe-Cr alloy after electrochemical potentiodynamic polarization tests are shown in Fig. 13. As can be seen, the CG sample was severely affected by localized corrosion (pitting corrosion), while in the NC sample, the localized corrosion had declined. As can be seen, the surface of the nanostructured sample shows a uniform and smooth morphology after corrosion test, while the CG sample includes the deep porosities resulted from the localized corrosion.

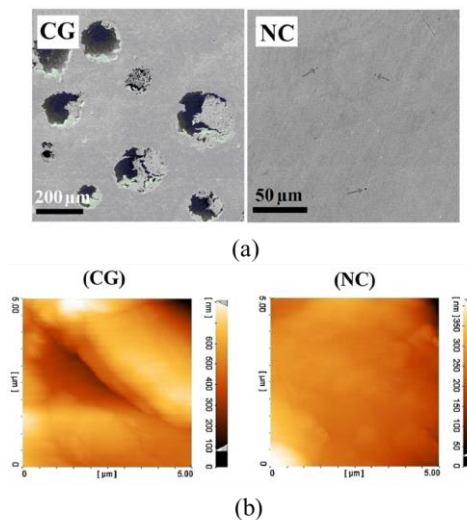


Fig. 13. (a) SEM micrograph, and (b) Atomic force microscopy images of the surfaces of coarse-grained (CG) and nanocrystalline (NC) samples of 316L stainless steel after implementation of polarization tests [377].

The obtained high corrosion resistance is not only due to the grain size reduction but also due to the formation of non-equilibrium grain boundaries during SPD. For example, in Fe-Cr system/alloy, the Cr diffusion to the surface is accelerated by the high-density non-equilibrium grain boundaries and it stabilizes the passive layer in a corrosive environment [378]. Figure 14 demonstrates schematically the difference between the grain structure, the grain boundary density, and the native oxide layer of CG and NC materials. The nanostructured sample possesses a higher grain boundary density, which proves the higher density of the native oxide layer on the surface of the nanostructured sample in comparison with its coarse-grained counterpart. The native oxide layer of the metal is considered as a protective layer (resistance to corrosion) against the metal reactions in the corrosive environment. Therefore, the corrosion resistance of the nanostructured metals is much greater in comparison to that of the coarse-grained ones [377].

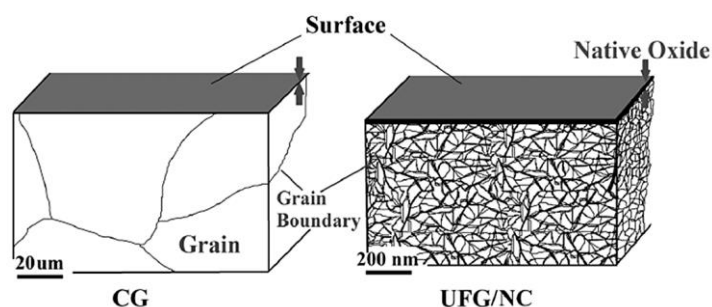


Fig. 14. Schematic representation of the differences of coarse-grained (CG) and (NC) samples in terms of grain structure, density of grain boundaries, and native oxide layer on the metal surface. UFG, ultrafine-grained [377].

Previous studies suggest that the corrosion resistance can be enhanced by SPD for materials with alloying elements of passivity formation such as Mg-Al, Cu-Al, etc. More than 60 reports have been published that investigate the corrosion behavior of different materials after ECAP and it is important to note that Iranian scholars are active in this field. According to Table 1, the corrosion resistance of 6061 Al alloy [23], 316L austenitic stainless steel [90,91] and commercially pure titanium (CP Ti) [92] was investigated by Iranian researchers.

The corrosion resistance of CP Ti was shown to be slightly improved as a result of applying ECAP. In 2009, Hoseini et al. [92] investigated the effect of texture on the corrosion properties of commercially pure titanium processed by ECAP. They showed that the effect of texture on corrosion is strong because the basal plane is most resistant to corrosion. Earlier, Blawert et al. [379] tried to correlate texture to the corrosion properties in magnesium fabricated by Physical Vapour Deposition (PVD). However, the texture change between different deposition processes was too small to be visible in the corrosion behavior. Following the work of Hoseini et al. [91], there have been some publications that investigate the effect of texture on corrosion behavior in Mg [380-382] and Ti [383]. From all of these publications, it can be concluded that the higher electron working function of atoms in the basal plane due to the high atomic coordination of the basal plane, leads to the highest resistance to dissolution. However, it is hard to draw general conclusions on corrosion resistance because of the dependency of the texture on the ECAP processing routes [376].

## 6. Summary and Conclusion

The fabrication of ultrafine-grained materials by ECAP is one of the main and basic topics in the SPD research community. The first work on ECAP in Iran was published in 2007. Since that time, the contribution of Iran to this research field has increased continuously. The main contribution of Iranian researchers in ECAP is focused on simulation/modelling. This can be attributed to the fewer research

funds and the limited access to advanced characterization methods. In addition, the topics of the published works indicate Iranian researchers' remarkable interest in introducing new methods of SPD based on ECAP. This tendency has placed Iran as the first ranked country for developing new SPD methods based on ECAP. There is no doubt that the attention of Iranian researchers will be shifted to other topics of ECAP process in the future if the opportunities for Iranian scientists to collaborate with other countries are improved, sufficient funds for characterization are provided and laboratories with advanced characterization tools are constructed in Iran.

**Acknowledgments:** The financial support of Shahid Chamran University of Ahvaz and Shiraz University is gratefully appreciated. The work of one of us was supported by the European Research Council under ERC Research Grant No. 267464-SPDMETALS (TGL).

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## ده سال تغییر شکل پلاستیک شدید در ایران، بخش اول: فشردن در کانال های هم مقطع زاویه دار

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**چکیده:** خواص فوق العاده مواد فوق ریزدانه ساخته شده توسط روش های تغییر شکل پلاستیک شدید، توجه محققان زیادی را از سراسر جهان به خود جلب کرده است. در بین کشور های تراز اول در این زمینه، ایران به دلیل دارا بودن رشد بسیار سریع در یک ده ی گذشته علی رغم ورود دیر هنگام به این عرصه، از اهمیت ویژه ای برخوردار است. اولین تحقیق در ایران در سال 2007 و در زمینه روش فشردن در کانال های هم مقطع زاویه دار انجام شده است که نشان دهنده ی عمر نزدیک به ده ساله این روش ها در ایران می باشد. با این وجود، از آن زمان، تعداد مقالات ایرانیان در این زمینه و به ویژه در معرفی روش های جدید بر پایه ی فشردن در کانال های هم مقطع زاویه دار به شدت رشد کرده است. در این مقاله ی مروری سهم ایران در روش های بر پایه ی فشردن در کانال های هم مقطع زاویه دار خلاصه شده است. جالب است که نقش عمده ی ایران در شبیه سازی و مدل کردن فرایندها و معرفی روش های جدید بر پایه ی فشردن در کانال های هم مقطع زاویه دار می باشد.

**واژه های کلیدی:** تغییر شکل پلاستیک شدید، فشردن در کانال های هم مقطع زاویه دار، مواد فوق ریز دانه، خواص فیزیکی و مکانیکی.