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Methods of fabricating metals for nano-technology

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Abstract. The paper gives an introduction to nanostructuring techniques used for industrial fabrication of bulk nanocrystalline metals – basic materials utilized in shaping nanoscale structures. Nanostructured metals, called nanometals, can be produced by severe plastic deformation (SPD). We give an expert coverage of current achievements in all important SPD methods and present future industry developments and research directions including both batch and continuous processes.

In the laboratories of both WUT and UOS we have developed industry standard equipment and machinery for nanometals processing. Utilizing the latest examples from our research, we provide a concise introduction to the field of mass production of nanometals for nanotechnology.

Key words: ultra-fine grain structure, severe plastic deformation, ECAP (equal channel angular pressing), grain refinement, aluminium alloys.

1. Introduction

For engineers, the fundamental attraction of nanometals is their ability to decrease the dimensions of mechanical devices. In fact, the developments in all important fields of micro- and nanotechnology depend on availability of suitable materials. Also successful shaping methods of engineering nanocomponents are required for building MEMS and nanodevices.

The most challenging issue is to merge the manufacturing process with material fabrication. The nanoscale synthesis of functional nanomaterials gives huge possibilities in this area. The self-assembly, non-traditional lithography, templated growth and biomimetics are some of the potential technologies [1]. In such processes, nanoparticles, delivered in the form of nanotubes, nanopowders, quantum-dots, and biomaterials are staked into final product in a designed way. The most challenging problem is the cost of making many of the raw components for functional nanomaterials. The cost of producing these materials often exceeds $700/g and makes potential products economically infeasible. The time required for performing any engineering work at nano scale is also considerable for methods based on the material synthesis. Another major challenge is the lack of chemical, morphological, or mechanical stability in many novel nanomaterials. These often lead to spatial distortions, suboptimal thermal behaviour, reduced mechanical response and poor electrical properties, all of which undermine the overall system performance.

An alternative approach is to employ bulk nanomaterials and traditional shaping methods while constructing systems and devices at the micro and nano scales. However, conventional metallurgy can not supply metals featuring grains substantially smaller than characteristic dimensions of the engineering micro-components. This and the encouraging economic forecasts for nanometals cause great interest in the development of new fabrication techniques, with mass-production scale-up capabilities and low-cost.

The technologies claiming bulk capability include electrodeposition and crystallisation of initially amorphous metals. However, the two main competing technologies are compaction and sintering of nanopowders and severe plastic deformation of bulk metals. The latter process avoids the presence of impurities or porosity typical of powder metallurgy. It involves generation of a very large plastic strain in coarse-grain bulk metals using one of the newly developed metal forming processes. The most popular one is equal-channel angular pressing (ECAP). In this process, a billet is pressed through a segmented channel to introduce a large shear strain without any concomitant change in the cross-sectional dimensions of the billet [2,3].

The aim of our paper is to present experimental results of ECAP trials for different tool configurations and demonstrate feasibility of the applied tool design methodology in the industrial context.

2. Grain refinement

At room temperature, the yield stress of metallic materials increases with the decreasing grain size. This is known as the Hall-Petch relationship. One of the possible techniques, used to obtain small grains in metals, consist in applying a large level of plastic strain to a course-grain precursor. A simultaneous accumulation of localised dislocations and increase in the lattice misorientation are responsible for crystal subdivision and subsequently developed submicroscopic grains. However, some researches reveal that, particularly in pure metals, there exists a limit below which reducing the grain size further results in shifting in the deformation mechanism into a different yet unknown mechanism of plastic flow.

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Strain hardening, which is defined as a change in flow stress with strain, is caused by the interaction of dislocations with each other and other defects. The traditional picture of plastic deformation behaviour and its mechanical response (i.e. hardness increase due to work hardening) seem to be no longer valid while reducing grain size far into submicron scale. For example, in nanocrystalline Cu, deformation behaviour falls into two patterns. Using molecular dynamic simulations, there has been found [4] that, if the crystal size is small enough (in Cu it is for the grain size from 5 nm to 50 nm), grain boundary sliding starts to dominate the deformation behaviour. This result was recently confirmed in nanocrystalline Ni experimentally [5]. It was found that electrodeposited nanocrystalline Ni, subjected to plastic strain, did not build up a residual dislocation network.

Taking into account the recent literature announcements on deformation behaviour, one can split polycrystalline metals into the following grain size regimes. For sizes greater than 1000 nm, traditional mechanisms determine deformation (coarse-grained polycrystalline metals). In the range from 1000 nm down to 30 nm, disordered grain boundaries begin to dominate the mechanical behaviour (ultrafine-grained polycrystalline metals). This transition becomes much more evident below 100 nm. At smaller scales, the atomic sliding at grain boundaries increases, leading to virtually no further work hardening of the plastically deformed metal. The introduced classes of metals have been shown in Fig. 1, revealing the type of mechanical response and characteristics of dislocation activity. The supposed grain size, d_g, for the change in the deformation mechanism, was indicated with a number of tick marks.

Data shown in Fig. 1 have one important implication for grain refinement in metals via plastic strain. Plastic working remains efficient in terms of producing small grain sizes only in the range down to 30 nm for most metals and their alloys. Concluding further, one can state that, employing metal forming, it is possible to fabricate only so called ultra-fine grained (UFG) metals, not even touching desirable nanometals. However, new findings on the mechanism of plastic flow of nanometals could shed more light and open the gate for metal forming also in this range. For now, the nanometals' territory has been restricted to new synthesis-based methods of materials fabrication.

There are a lot of peculiarities affecting the grain refinement obtained by severe plastic deformation. Alloys are more responsive to intensive straining than pure metals, which results in finer grains [6]. This effect, however, requires the application of larger strain. The rate of grain refinement can be increased by the presence of coarse second-phase particles [7]. UFG metals exhibit exceptionally high strength and reduced, but reasonable ductility. As stated in Fig. 1, they cannot go through strain hardening and are prone to flow localisation during forming. However, by choosing appropriate parameters of the grain refinement process, it is possible to remove or delay the plastic instabilities. For example, a thermo mechanical process, involving cryogenic forming to suppressing dynamic recovery followed by short annealing allowing an abnormal grain growth, led to a composite-like microstructure consisting of two populations of different size grains. This resulted in much improved ductility of the processed copper material [8]. Usually researches aiming at grain refinement do not take into consideration the influence of strain rate. Dislocations generated under static conditions of deformation run through small grains without virtually any disturbance in their movement. Dislocations move with a constant rate which depends only on the mechanical properties of metal. So performing high speed working is the only way to increase probability of collisions between dislocations and achieving a higher rate of strain hardening [9].

The above mentioned issues are only a few examples of the potential of plastic working in engineering of metals microstructures. The process parameter interactions should be studied further and the traditional picture of the metal forming technology should be revised in the light of latest findings on grain refinement.

It is worth emphasising that all metallic materials respond to severe straining in basically the same way. Technically, nanostructure can be achieved locally by a number of processes like cold rolling [10] or friction wear. However, these processes are difficult to control in terms of grain refinement in the whole deformed slug and there is no room for the manipulation of grain structure efficiently. First time, it was achieved in a controllable way, was due to an equal channel angular extrusion (ECAE) process introduced by Segal in the 1970s [11]. ECAE, also known as ECAP, gave an impetus to the development of the whole range of similar processes which led to establishing a new discipline of metal forming – SPD methods.

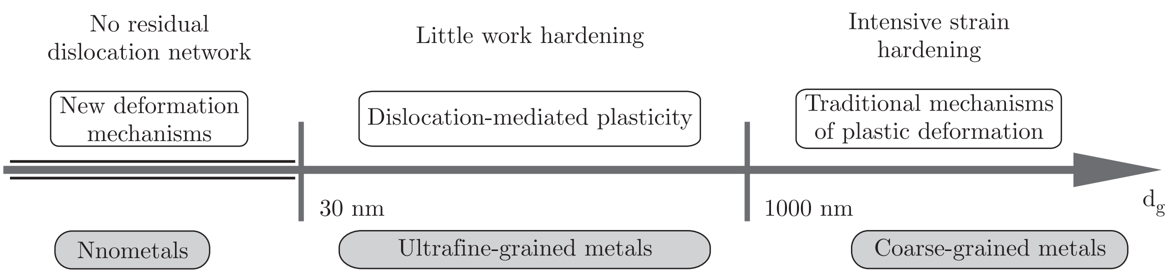
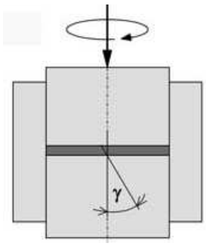
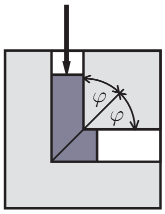
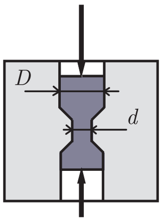
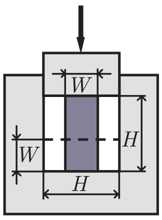
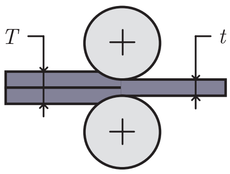
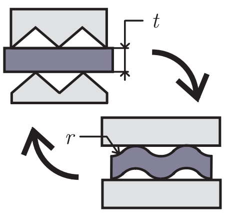
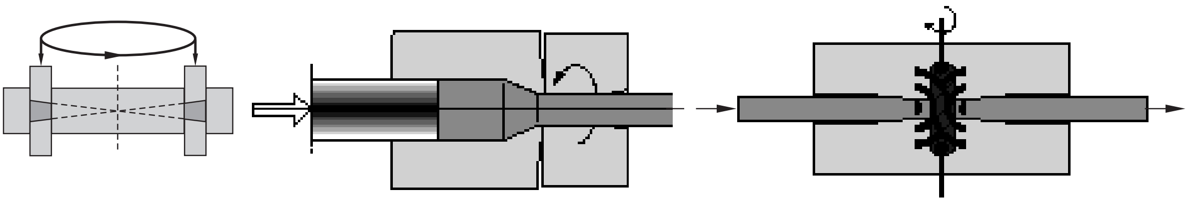


Fig. 1. Classification of polycrystalline metals according to grain size

Table 1
SPD processes developed for grain refinement

Process name	Schematic representation	Equivalent plastic strain
High-pressure torsion (HPT), Valiev, 1997		$\varepsilon = \frac{\mathrm{tg}\gamma}{\sqrt{3}}$
Equal channel angular processing (ECAP), Segal, 1977		$\varepsilon = n\frac{2}{\sqrt{3}}\cot\varphi$
Cyclic extrusion-compression (CEC), J. and M. Richert, Zasadziński, Korbel, 1979		$\varepsilon = n4\ln\left(\frac{D}{d}\right)$
Multiaxial forging (MF), Ghosh, 1988		$\varepsilon = n\frac{2}{\sqrt{3}}\ln\left(\frac{H}{W}\right)$
Accumulative roll-bonding (ARB), Saito, Tsuji, Utsunomiya, Sakai, 1998		$\varepsilon = n\frac{2}{\sqrt{3}}\ln\left(\frac{T}{t}\right)$
Repetitive corrugation and straightening (RCS), Zhu, Lowe, Jiang, Huang, 2001		$\varepsilon = n\frac{4}{\sqrt{3}}\ln\left(\frac{r+t}{r+0.5t}\right)$



(a) (b) (c)

Fig. 2. Different applications of twisting in SPD processes: HPT of rings (a), cyclic twist extrusion (b), friction stir welding (c)

3. Methods of SPD

The amount of plastic strain produced by classical metal forming operations is often limited because of the material or tool failure. Compressive forming conditions are preferable to hamper void nucleation, growth and coalescence which leads to ductile fracture. In some sequential processes such as rolling or drawing large reductions of the material thickness can be achieved. However, the shapes produced by these processes are not bulk enough to be used for further conversion into products. Thus new metal forming processes capable of generating very large or severe plastic deformation (SPD) without major change in the billet geometry have been developed. Originally the term SPD was defined as "intense plastic straining under high imposed pressure" [12]. Recently, it covers all metal forming processes which are based on simple shear and/or repetitive reversed straining and tend to preserve the initial shape of the billet. A survey of SPD methods can be found in numerous publications including a critical survey [13], testimony of developments [14], and materials engineering approach [15]. SPD processes, which were originally proposed for grain refinement, are listed in Table 1. The way of calculating plastic strain, which is the main process parameter, has been quoted in the last column. For HPT it is a continuous relationship. However, in most cases the required strain will be accumulated in a recurring way, depending, for a single pass, on tool characteristics (ECAP, CEC), billet dimensions (MF, ARB) or a combination of both (RCS).

The recent years have brought some new developments which can be split into two categories: redevelopment of originally invented SPD techniques or equipment of existing metal forming processes with intense shearing. Inventive use of rotational tool movement, essential for performing uninterrupted shear straining in torsion, can serve as an example. Fig. 2 shows a few rotating tool configurations (proposed by Polish inventors) which add new features into the grain refinement process. Erbel's tool [16] reduces strain gradient in the radial direction, Korbel's approach [17] enables processing long products in a cyclic twist forward extrusion, and Litwiński's method [18] turns friction stir welding into a continuous grain refining process.

From a customer point of view, categorising which takes into account the form of the product, i.e. slug, rod, wire, hoop, band, sheet is of some importance. Most techniques enable processing a finite portion of metal. Some of them, usually derived from well established metal forming methods, work with narrow continuous billets. ARB is the first SPD process which deals with the material in the sheet form while retaining sheet thickness. Also RCS has such capability. Erbel's tool (Fig. 1a) is capable of producing UFG rings. Developing SPD methods towards production of tailored products made of UFG metals would be relatively easy. However, it has not been tried since the market for nanometals remains in an embryonic phase [19]. The key to wide commercialization of UFG materials is to lower their processing cost and waste. It is believed that it can be achieved through continuous processing [20]. Continuous SPD processes, based on popular metal forming operations, e.g. wire drawing [21] and continuous extrusion [22] are illustrated in Fig. 3.

Recent achievements in the severe plastic deformation technology are outlined in [23]. The paper concludes that processing community must address several significant technological challenges before the wave of promised new applications can appear. One of those challenges is that many preprocessing (i.e. grain refining) and post-processing (i.e. shaping) techniques are reaching their limits for applications involving the high strength nanostructured materials. At present, the fundamental SPD pre-processes listed in Table 1 are still mostly employed for grain refinement, however, they are being improved and made robust for an industrial use. Among them, ECAP has become prominent over the years. ECAP is based on straining a material by simple shear, which is performed in a die at the intersection of two connected channels. The angle of this intersection usually ranges from 45° to 65°. Despite the apparent simplicity of the process, microstructure evolution in ECAP is quite complex and depends on the process parameters used. Also defect generation can occur by an improper use of ECAP. The influence of process parameters on microstructure evolution is outlined in [24]. ECAP has the biggest potential for scaling up since development of microstructure after pressing does not depend on the initial size of the sample nor the location within the sample [25]. Processing larger sizes of bulk metals will certainly involve significant investment in tooling. However, taking into account the above mentioned facts it is worth to discuss new trends in ECAP development.

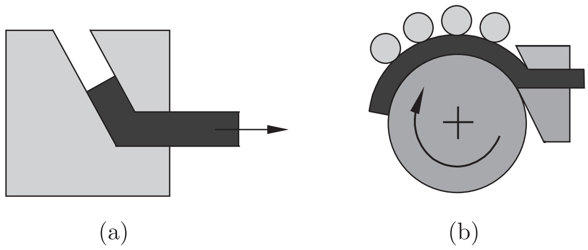


Fig. 3. Continuous SPD processes derived from metal forming technology: equal channel angular drawing ECAD (a), continuous confined strip shearing CONSHEARING (b)

4. ECAP developments

Being the most popular process, ECAP has experienced several alterations or modifications. To deal with friction Segal invented movable walls in the channels [26] (Fig. 4a). Stecher and Thomson [27] (Fig. 4b) introduced a die equipped with a cylindrical surface in the form of a rotatable roll to avoid friction. Markushev et al. [28] patented an unequal channel extrusion in which the billet changes its cross-section in the outlet channel (Fig. 4c). This kind of modification of the outlet channel will increase the process force and decrease the tool life. Using small slenderness of the billet or increasing the angle between consecutive channels in the die can help reduce the force.

Most small design alterations have only a limited impact on improving the processing conditions. Thus modifications which improve the performance of the ECAP method are of considerable importance to industry. An idea how to increase the billet's deformation during one pass in the die, giving additionally a possibility of immediate input of the slug with reduced cross-section for the next pass in an S-shape channel [29], was shown in Fig. 5a. This scheme is equivalent to so called route C of ECAP which involves rotating the billet about its axis by 180° between each pass in the classical two channel's die. A two-turn, U-shape channel [30], presented schematically in Fig. 5b, is equivalent to route A realised outside the die (no rotation of the billet). Using two-turn channels doubles the strain produced in one pass and increases the productivity of ECAP. In order to obtain homogeneous microstructures of equiaxed grains separated by high-angle boundaries, route B_C (rotation by 90°) should be used [31]. A square channel, with two-turns and all angles equal 90°, configured to realise route BC in die, is drawn in Fig. 5c. Fig. 5d shows a three-turns channel for a similar configuration, which was used for processing commercial purity aluminium [32]. Due to a complicated geometry of ECAP channels, the dies enabling this geometry were designed as segmented, prestressed tools. Tool prestressing is the methods routinely used in the metal forming technology. Tool design and making is the well developed sector which should facilitate industrial implementation of ECAP, even in the case of complex ECAP dies. The tool issues will be addressed later in the following sections.

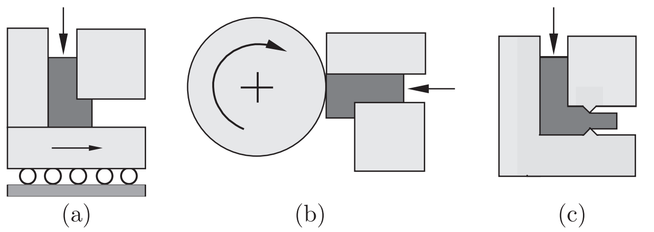


Fig. 4. Modifications of ECAP: movable wall (a), rotatable roll (b), profiled output channel (c)

Manipulation of billets between consecutive passes of ECAP may cause problems in industrial situations. There is, however, a method of reducing the number of such manipulations. It can be achieved not only by increasing the number of channel turns in a die but also by increasing the number of inlets and outlets in a die and the number of corresponding punches. Fig. 6a shows a cross-like configuration of channels with the billet material subjected to simple shear in the central hub at the channel crossing [33]. The channels are presented to the appropriate punches by rotating the die before each deformation cycle. The deformation scheme realised is that of route A with the leading end of the billet altered before each cycle. The billet is left in the die until the whole program of deformation is finished. The ECAP die showed in Fig. 6b features the same concept realised in a stationary die and the channels orientated in three dimensions [34]. Thus the deformation scheme is that of B_C with the leading end of the billet altered before each cycle. A similar die design realising route B_C without the leading end alteration [35] is shown in Fig. 6c. In this design, the billet passes each channel only once being successively pushed by the appropriate punches.

The ECAP procedures presented in Fig. 6 are in fact multi-stage metal forming processes realised in a single progressive die. Development and maintenance of shaping operations performed at one stand is very common for metal forming technology. It even seems reasonable to merge fabrication of UFG metals with shaping products in the near future. The driving forces for the development of SPD are: availability of machines (variety of presses), knowledge of tool design (e.g. prestressed dies) and good practice in billet treatment (lubrication techniques).

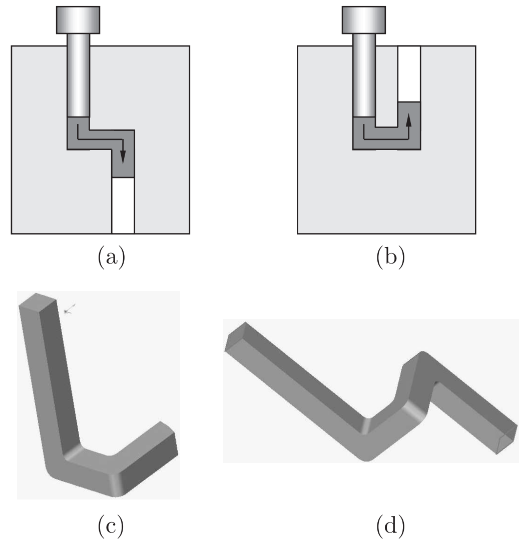


Fig. 5. Channel designs for multi-turn ECAP: 2-turn with route C in die (a), 2-turn with route A in die (b), 2-turn with route B_C in die (c), 3-turn with route B_C in die (d)

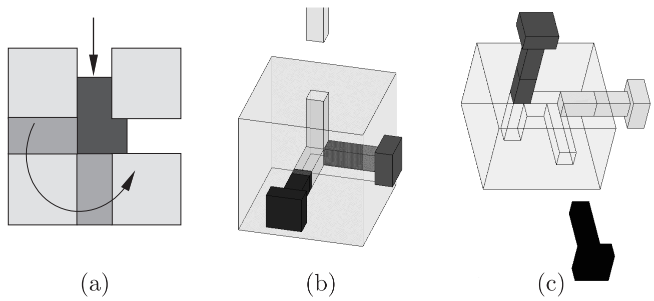


Fig. 6. Multi-punch ECAP procedure: with rotating die (a), with multi-pass channels (b), with one-pass channels (c)

5. ECAP process in practice

Batch SPD processes are in fact multi-operation metal forming processes with the same main SPD operation repeated many times until a total value of equivalent strain, required for desired grain refinement, is reached. Rules for designing the accompanying operations, necessary for fluent fabrication of UFG metallic material, are pretty much common with the standard metal forming practices. Designers must consider the economy of fabrication, which requires fewer forming opera-

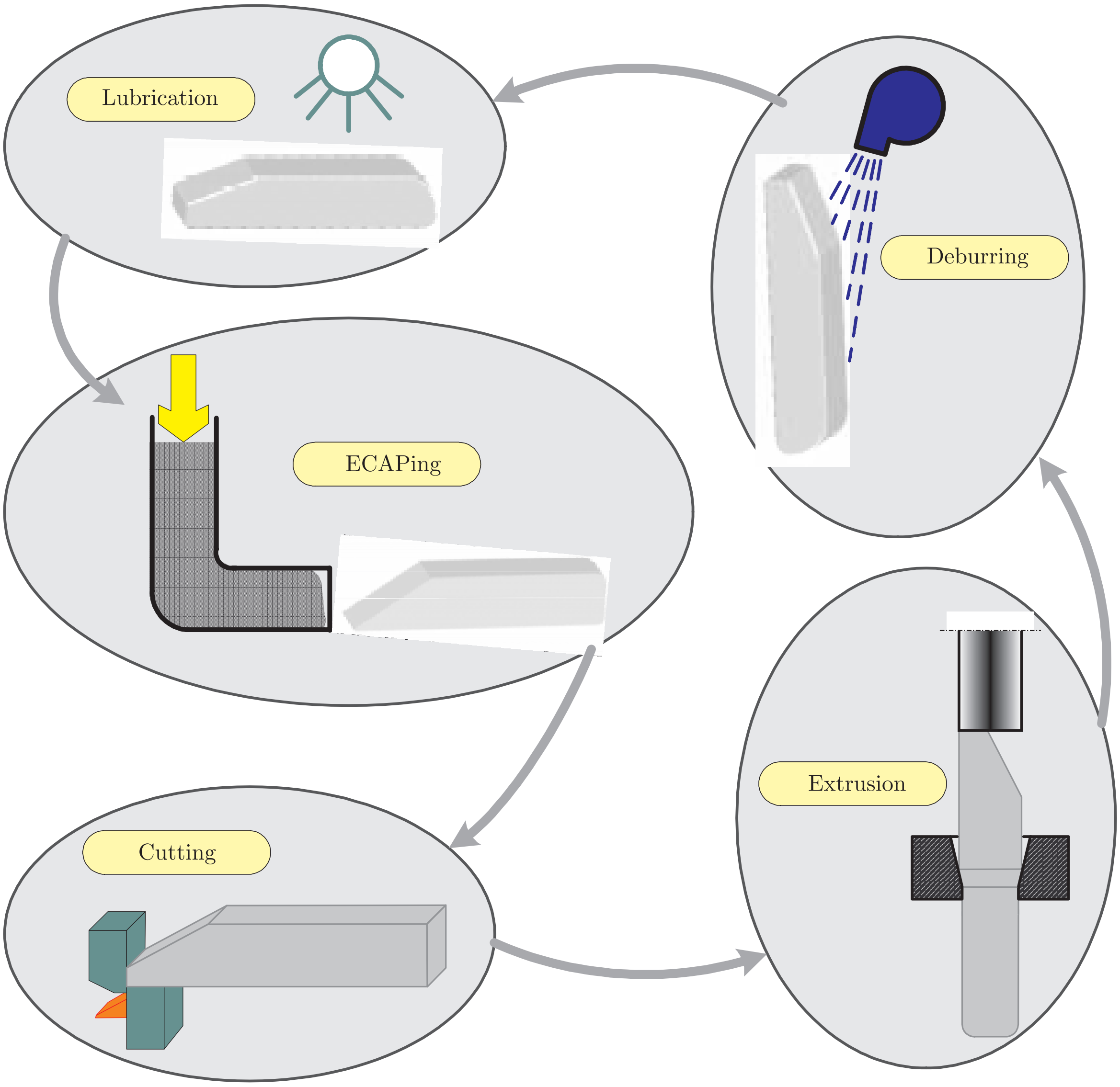


Fig. 7. Technological scheme of ECAP sequence for square billets

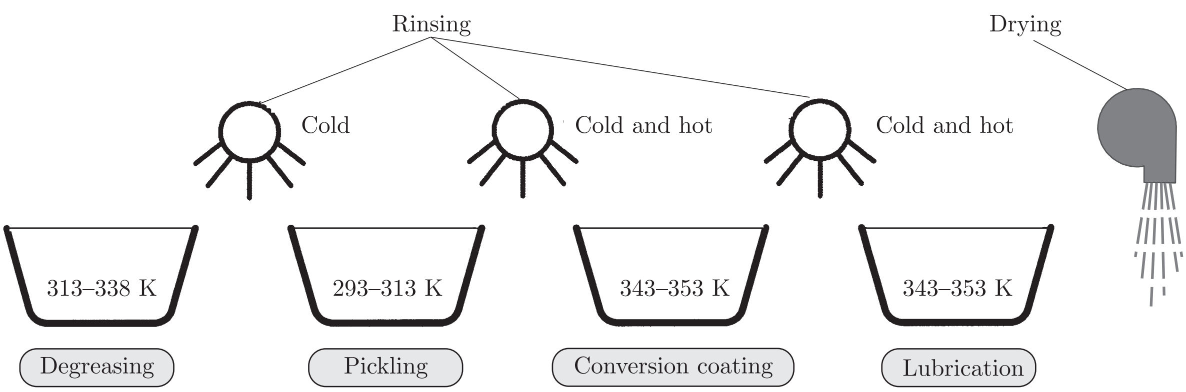


Fig. 8. Immersion process for generation of lubricated conversion coatings to facilitate SPD processes of metallic materials

tions, a simple maintenance of the tool set, and a proper selection of tool materials and process ancillary equipment.

Based on the gathered experience, we bring below a few recommendations for cold and warm batch ECAP processes. These processes were introduced and successfully performed at both WUT and UOS.

5.1. Processing sequence. The first step in design is to choose the billet and determine how to manufacture it from raw material. Usually the billet material is sheared from wires or bars. General rule is that the billet diameter is smaller than its height. Sometimes the billet must be preshaped. This could be the case when the ECAP die has a square cross-section. The required change/reduction in the cross-section area can be achieved by cold forward extrusion. This operation introduces initial strain hardening which can be of some value for subsequent grain refining.

Sometimes, between the ECAP operations, an additional reduction in the cross-section area would be required for putting back the billet into the inlet channel. The cross-sectional dimensions of billets can be reduced via machining or using a metal forming operation, like open die extrusion.

The ECAP process is known for a substantial deformation of the billet ends. Tipped bits have to be removed as they can disturb the work of the punch which pushes the billet in the inlet channel. Also sharp edges have to be made blunt before performing the next SPD operation.

Figure 7 shows schematically a single loop of a batch ECAP process capable of producing square billets of UFG metal.

5.2. Lubrication. In SPD processes, like in any other metal forming processes, friction influences tool wear and the loads necessary for plastic deformation. Therefore, adequate lubrication is essential. Different lubrication techniques may be selected depending on the processed metallic material, SPD operation, and processing condition. For ECAP, a suitable lubrication layer consists of conversion coating which is subsequently treated with a solid lubricant. Usually lubrication is a chemical process consisting of several bathes, as shown in Fig. 8.

Another factor to consider in a lubrication system is the selection of hard coating of dies. An optimal combination of metal-lubricant-coating system was validated experimentally using a developed friction testing rig before being adopted for ECAP. The efficiency of lubrication and the magnitude of friction were judged by comparing the process forces in a forward extrusion process, which was carried out by applying the strain level equivalent to that in ECAP.

5.3. Heating. For ECAP, performed at elevated temperatures, billets must be preheated. The usual industrial practice is to heat the billet material in a furnace. When a required temperature is reached the billet is transferred to the die. SPD processes are carried out far below the recrystallisation temperature, hence, heating in the die becomes common practice. The prerequisite is to keep temperature far from the tempering temperature of the tool steel used.

5.4. Tools. The tools play an important role for trouble-free ECAP operation. Tooling for the batch ECAP consists of a punch and die set, coupled with a guide system.

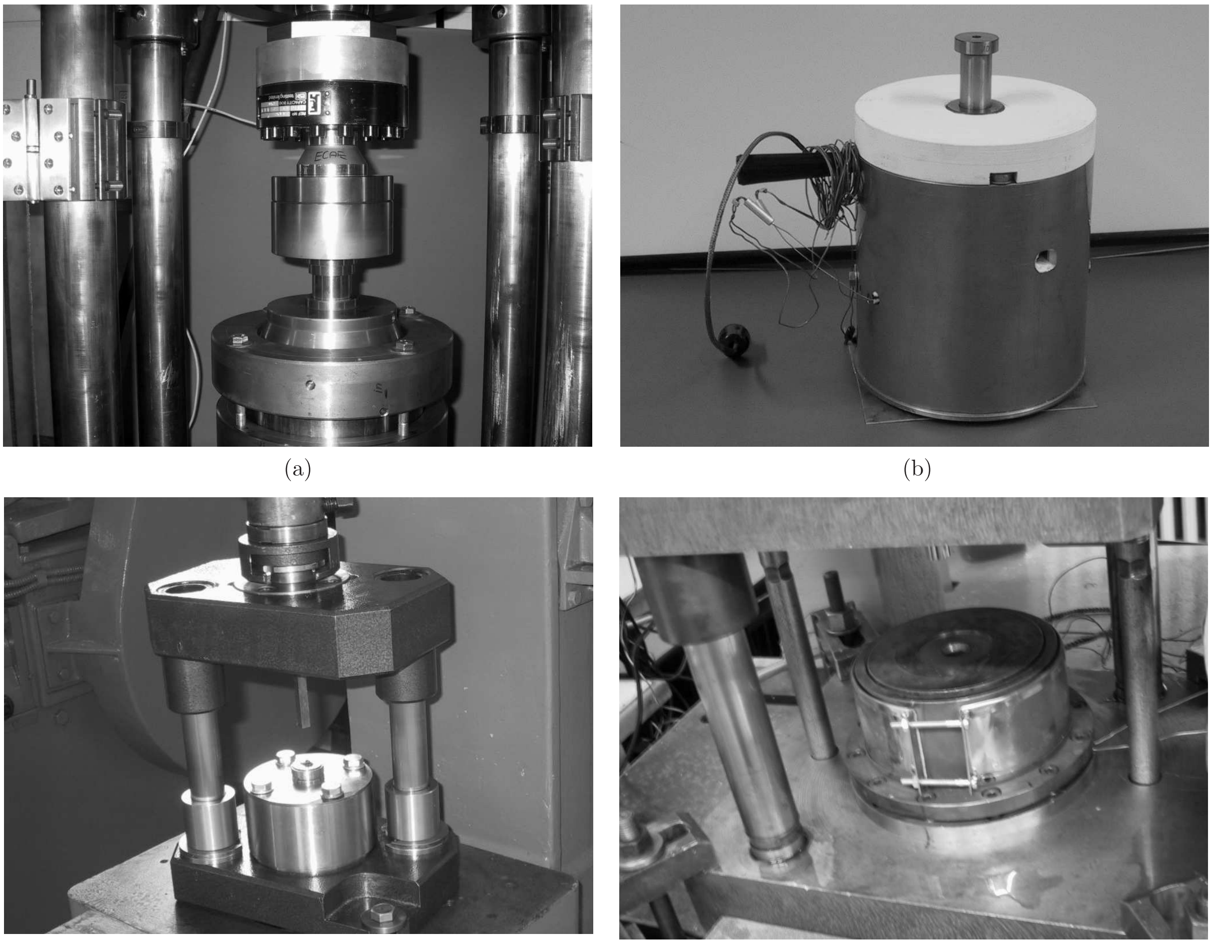
Punches should be made as short as possible with no sudden variations in sections. Dimensions at the working end should have sufficient entry clearance to enter into the inlet channel. The value of this clearance depends on the channel width and the expected punch pressure. Generally, one or two shrink rings are used for prestressing the segmented die inserts. Design concepts of ECAP dies are usually patented. Fig. 9 shows the billets pressed in the dies invented in WUT (Fig. 9a) and UOS (Fig. 9b).

(a)

(b)

Fig. 9. Billets pressed through 2-turn square (a) and 3-turn round channels of ECAP dies (b)

5.5. Eqiupment. ECAP and other related operations are performed on hydraulic presses. Fig. 10 shows tool setups for cold ECAP, warm ECAP, reduction of billet cross-section area and friction testing for validation of lubrication procedures. The rigs were equipped with force/stroke (or temperature if necessary) instrumentation. Recordings of force versus stroke were used for revealing any process disturbances, e.g. breakdown of the lubricant layer.



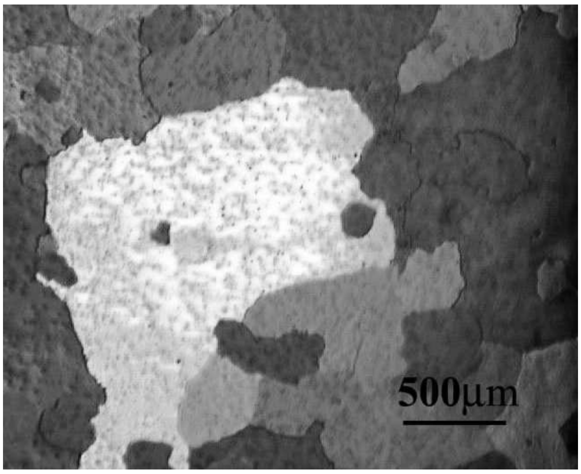
(a) (b) (c) (d)

Fig. 10. ECAP and ECAP related rigs for: cold ECAP (a), warm ECAP (b), billet area conversion (c), friction testing (d)

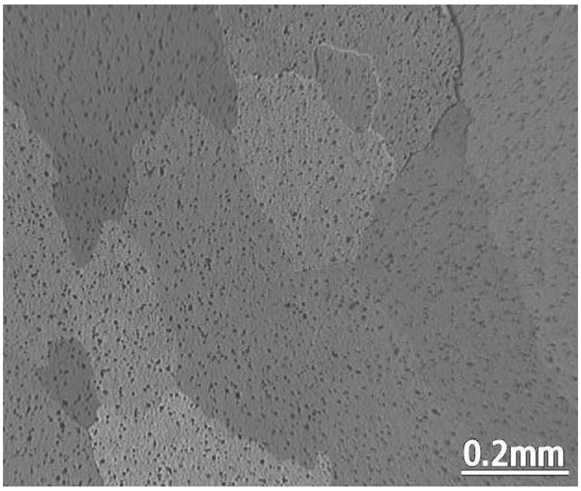
6. Fabricated nanometals

Using SPD, it is possible to fabricate submicron scale structures in variety of metals. Nowadays aluminium plays an important role in mechanical engineering. This is the main material for aircraft structures and increasingly in other means of transport. Nanometals can be used in these sectors in the form of different fasteners which exhibit high strength and good toughness. Joints involving aluminium alloys are prone to galvanic corrosion when in contact with other metals having a different galvanic potential. The driving force for this corrosion is a potential difference between the materials. Hence, fasteners should be made of material similar to that of the whole construction. Al-Mg based alloys are known as an optimal material for fasteners for joining aluminium parts. Since they are not precipitation hardened alloys, the only way to achieve required hardness is to strain harden them together with grain refinement, which simultaneously retains ductility. Despite this obvious application, SPD-based nanostructured metals have been first used in the electronics industry. Here, sputtering targets for various PVD coating processes have been made of the nanostructured low-alloyed aluminium and copper. Significant benefits to performance are claimed such as better process control, greater coating efficiency and cost reduction. Honeywell Electronic Materials has already commercialised a sputtering target production process based on ECAP [36].

Addressing the above mentioned market opportunities, commercial purity aluminium (1070) and a commercial Al-Mg-Mn alloy (5083) were selected for ECAP processing at WUT and UOS. The initial microstructures of these materials are shown in Fig. 11. To reduce the tooling cost, a relatively small cross-section of billets was chosen. In both cases, $8\times8\times48$ mm billets were used for demonstration of the nanometal mass production capability. Additionally, a square cross-section enables easy design and making of dies. The process forces are also reduced by this small cross-section area resulting in acceptable tool loading, even in the case of 3-turn die design with a relatively long channel. To achieve the strain required for sufficient grain refinement, the process was carried out repeatedly. The working procedure was always the same: after pressing the first billet down the inlet channel, the following billets were used to push the preceding ones along the channel and finally out of the die; the billet was rotated by 90° between the passes to conform to in-die rotation (route $\mathrm{B_C}$ was kept for all trials). Some pictures of the processing results are shown in Fig. 12.

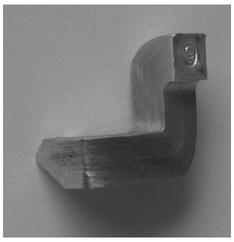


(a)



(b)

Fig. 11. Initial microstructure of metals used for demonstration of industrial scale grain refinement: 99.7% purity aluminium alloy (1070) (a), Al-Mg-Mn non heat-treatable aluminium alloy (5083) (b)



(a)

(b)

(c)

Fig. 12. Aluminium billets processed using ECAP facilities developed in WUT and UOS: cold pressed billet inside a 3-turn die (a), cold pressed billet leaving a 3-turn die (b), warm pressed billet leaving a classical ECAP die (c)

In the case of 1070 alloy, cold pressing in a 3-turn ECAP die was used to accumulate the strain of $3 \times 1.15 = 3.45$ in one pass. The 5083 alloy, processed with an ECAP die featuring classical design, was heated up to the temperature higher than 453 K to avoid material fracture during processing.

7. Identification of properties

Industrial adoption of nanometals will certainly require developing general material specifications, especially for structural and high performance applications. However, at the present stage of EACP development, each time a new material is processed, its mechanical behaviour and structural evolution is verified. Mechanical properties are evaluated in tensile tests under static conditions. Usually, the material data evaluated are: the 0.2% proof stress $\mathrm{R_e}$, ultimate tensile stress $\mathrm{R_m}$, reduction area at fracture Z, and elongation to failure $\mathrm{A_5}$. In metallurgical tests, grain refinement is documented, mostly using TEM micrographs. High grain boundary misorientation angles are the major concern while investigating structural evolution.

In both WUT and UOS, a small tensile specimen technique was adopted to evaluate mechanical properties of the material before and after an ECAP process. 10kN Hounsfield H10KS and ZWICK Rel 2061 testing machines were equipped with specially designed jaws to grip specimens cut directly from ECAP'ed billets. Tensile specimens with 2.5 mm diameter and 1:5 diameter-to-length ratio were strained at room temperature with the ram speed of 0.5 mm/min, giving the initial strain rate of about 6×10^{-4} s^{-1}. Figure 13 displays the tensile response of the initial 1070 material as well as the same material cold strained to $\varepsilon = 6.9$ in a 3-turn ECAP die.

The initial yield strength of Al 1070, subjected only to two passes in our ECAP die, has been tripled while its tensile strength doubled. The total elongation has been halved and, judging from a small distance between the yield point and the onset of localization point, the uniform strain is largely reduced. As illustrated in Fig. 14, these trends establish quickly during the first pass so that the second pass does not change much in terms of strength and ductility.

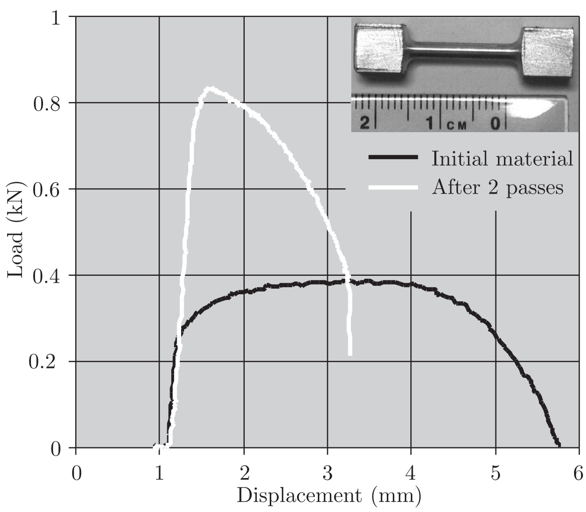


Fig. 13. Tensile test curves for Al 1070 before and after cold ECAP straining to $\varepsilon = 6.9$ in 3-turn die

Figure 15 shows the evolution of tensile strength and ductility of 5083 Al-Mg-Mn alloy subjected to ECAP at elevated temperature. A similar evolution of strength was observed in the case of this hard aluminium alloy as it was for the soft one. However, a significant decrease in ductility was recorded. This time, the billets were pressed to a total strain of $\varepsilon = 9.2$. It is worth noticing that the mechanical properties achieved at the end of ECAP were close in value to those obtained just after reaching half of the maximum strain. At this smaller strain, Al 5083 could possess the strength sufficient for many applications and, at the same time, decent ductility.

Judging the microstructure, we have generally registered a 500 times reduction in grain size. The microstructure evolution of aluminium alloys subjected to ECAP at WUT and UOS was presented elsewhere [32,35].

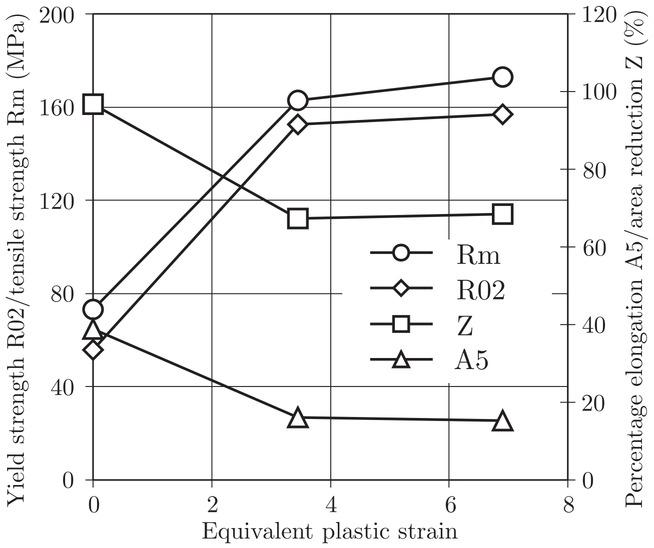


Fig. 14. Evolution of tensile strength and ductility in Al 1070 for one and two passes carried out in a 3-turn die

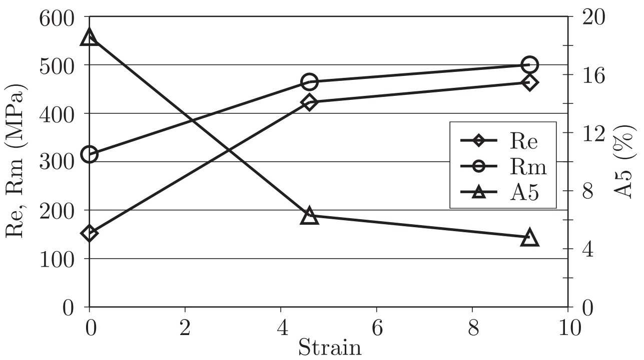


Fig. 15. Evolution of tensile strength and ductility in Al 5083 subjected to warm ECAP at 453 K

8. Summary

Dislocation activity in nanocrystalline metals must be fundamentally different to polycrystalline metals. However, for some range of grain sizes, plastic straining has capability for substantial grain refinement. Thus the metal forming technology, which is normally associated with shaping metal billets into products, can be used to change coarse grain metals into ultra fine (submicron size) grain metals. A new bread of metal forming processes used for this purpose is referred to as severe plastic deformation (SPD) processes. A popular batch process of this kind is ECAP, in which the high deformation levels are obtained merely by performing successive passes. In order to increase its productivity a multi-turn version of ECAP was introduced.

Recent achievements in ECAP technology were practically tested under conditions of mass production in press-shops at WUT and UOS. We have developed a series of single and multi-turn dies to process UFG metals efficiently. Cold and warm batch processes were successfully performed for demonstration of quick grain refinement in different aluminium alloys. Multi-turn ECAP dies survived intensive work, confirming their suitability for a large scale, efficient and cost effective production. These results support the forecasts that nanomaterials are expected to be one of the earliest practical results of nanotechnology to appear in commercial applications.

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