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Ph.D. Thesis

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Anisotropy of Mechanical Properties and Formability of Ultrafine-Grained Plates Made of Aluminium Alloys

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“Ultrafine grained plates with low anisotropy and the capabilities to deep drawing and superplastic deformation at high strain rates”

„Płytki o strukturze ultra drobnoziarnistej, małej anizotropii, zdolności do głębokiego tłoczenia i odkształcenia nadplastycznego przy dużej szybkości odkształcenia”

Abstract

Ultrafine-grained materials (UFG), defined as polycrystals characterized by grain size below 1 μm, feature new and often superior properties in comparison to conventional coarse-grained (CG) ones. In particular, they exhibit significant enhancement of mechanical strength, which however is often accompanied by a drastic loss of ductility. A great number of basic research has been carried out on UFG materials, mainly focusing on microstructure changes during processing, mechanisms of grain refinement and characterization of basic properties (hardness measurements and tensile tests), yet they are still not widely commercialized, which can be attributed to a lack of applied research, small sizes of manufacturing batches, as well as limited ductility.

A crucial step toward the commercialization of UFG materials is conducting applied research focused on actual forming processes. For that reason, this dissertation focuses on producing UFG materials in a form of a plate, as this shape is suitable for further sheet forming processes, and evaluating their formability. In the thesis, it is assumed that the isotropic properties of UFG plates and proper external conditions, i.e. strain rate and temperature, will make it possible to increase ductility and obtain formability at least equivalent to the material of conventional grain size. It should be noted that every processing route leading to producing a sheet or plate results in a certain degree of anisotropy, which can be defined as various mechanical behavior depending on the testing direction. Such anisotropy can result in various faults of the formed objects, such as earing and excessive thinning in certain directions. Thus, the objective of the thesis is to manufacture UFG aluminum alloy plates characterized by low anisotropy, improve their ductility and subject them to forming tests.

To produce plates, two techniques have been used, i.e. Incremental Equal Channel Angular Pressing (I-ECAP) and Hybrid Processing composed of multi-turn Equal Channel Angular Pressing followed by upsetting. I-ECAP has been used for processing AA1050 and AA3003, while Hybrid Processing for AA5754. The manufactured plates have been evaluated in terms of microstructural homogeneity, crystallographic texture, mechanical properties isotropy, planar and normal anisotropy, strain hardening ability, strain rate sensitivity, and formability.

The obtained results indicate that both processing methods lead to UFG microstructure and isotropic mechanical properties. For all the materials investigated, parameters describing anisotropy, mean r-value, are in a range typical for fcc materials, yet planar anisotropy, Δr, is smaller for plates after I-ECAP than for plates processed by any other methods described in the literature. This is attributed to the reduction of crystallographic texture intensity. Furthermore, even though in a tensile test conducted at room temperature, the metals show limited ductility, it is possible to enhance it significantly by increasing temperature, even to 82% of total elongation, facilitated by reduced activation volume and diffusion processes. As a formability trial, the cupping test was conducted at elevated temperature, resulting in an even two-fold increase of cup height for UFG metal in comparison to CG one.

Keywords: ultrafine-grained, aluminium, anisotropy, ductility, formability, microstructure, strain rate sensitivity, strain hardening

Streszczenie

Anizotropia właściwości mechanicznych i zdolność do formowania ultradrobnoziarnistych płytek ze stopów aluminium

Materiały ultradrobnoziarniste (ang. ultrafine-grained, UFG) definiowane jako polikryształy o wielkości ziarna poniżej 1 μm charakteryzują się one nowymi, i często lepszymi, właściwościami w porównaniu z materiałami o konwencjonalnej wielkości ziarna (ang. coarse grained, CG). Wyróżniającą cechą materiałów UFG, której zawdzięczają swoją popularność, jest bardzo wysoka wytrzymałość mechaniczna, której jednak często towarzyszy utrata plastyczności. Materiały UFG były obiektem intensywnych badań podstawowych, głównie dotyczących zmian mikrostruktury podczas procesów wytwarzania, mechanizmów rozdrobnienia ziarna oraz charakterystyki podstawowych właściwości (poprzez pomiary twardości i w próbie rozciągania). Materiały UFG wciąż nie są szeroko skomercjalizowane, co można przypisać małej ilości badań aplikacyjnych, niewielkim rozmiarom partii produkcyjnych, a także ograniczonej plastyczności.

Istotnym krokiem w kierunku komercjalizacji materiałów UFG jest prowadzenie badań skoncentrowanych na rzeczywistych procesach formowania. Dlatego też niniejsza rozprawa koncentruje się na wytwarzaniu materiałów UFG w postaci płytek, nadających się do dalszych procesów kształtowania i ocenia ich podatność na formowanie. W pracy założono, że właściwości izotropowe płyt ultradrobnoziarnistych oraz odpowiednie warunki zewnętrzne, tj. szybkość odkształcenia i temperatura, pozwolą na zwiększenie ciągliwości i uzyskanie odkształcalności co najmniej równoważnej materiałowi o konwencjonalnej wielkości ziarna. Należy zaznaczyć, że każda droga przeróbki plastycznej prowadząca do wytworzenia blachy lub płyty skutkuje również pewnym stopniem anizotropii, którą można określić jako różne właściwości mechaniczne w zależności od kierunku badania. Anizotropia może skutkować różnymi wadami formowanych obiektów, takimi jak tworzenie się „uch” czy pocienianie ścianek w określonych kierunkach. Dlatego też celem pracy jest wytworzenie ultradrobnoziarnistych płyt ze stopów aluminium charakteryzujących się niską anizotropią, poprawienie ich ciągliwości oraz poddanie ich próbom formowania.

Do uzyskania mikrostruktury UFG płyt zastosowano dwie techniki, tj. przyrostowe przeciskanie przez kanał kątowy (ang. Incremental Equal Channel Angular Pressing, I-ECAP) i metoda hybrydowa składająca się z wielozakrętowego przeciskania przez kanał kątowy (ang. multi turn ECAP, mt-ECAP), po którym następuje spęczanie. Metodą I-ECAP przetworzone zostało aluminium AA1050 i stop AA3003, natomiast metodą hybrydową stop AA5754. Wytwarzane płytki zostały scharakteryzowane pod względem jednorodności mikrostrukturalnej, tekstury krystalograficznej, izotropii właściwości mechanicznych, anizotropii płaskiej i normalnej, umocnienia odkształceniowego, czułości na szybkość odkształcania oraz zdolności do formowania.

Uzyskane wyniki wskazują, że obie metody przetwarzania prowadzą do uzyskania mikrostruktury UFG i izotropowych właściwości mechanicznych. Dla wszystkich badanych materiałów średnia wartość współczynnika Lankforda r mieści się w zakresie typowym dla materiałów o strukturze RSC, a anizotropia płaska, Δr, jest najmniejsza dla płyt po I-ECAP w porównaniu do płyt przetwarzanych innymi metodami opisanymi w literaturze. Przypisuje się to zmniejszeniu intensywności tekstury krystalograficznej. Ponadto, mimo że w próbie rozciągania w temperaturze pokojowej, badane materiały wykazują ograniczoną ciągliwość, możliwe jest jej znaczne zwiększenie, nawet do 82% całkowitego wydłużenia, poprzez podwyższenie temperatury, dzięki zmniejszeniu objętości aktywacji i zachodzeniu procesów dyfuzji. Jako próbę odkształcalności przeprowadzono test tłoczenia w podwyższonej temperaturze. Wykazano, że materiał UFG charakteryzuje się nawet dwukrotnie większą wysokość miseczki w porównaniu z CG.

Słowa kluczowe: ultradrobnoziarnisty, aluminium, anizotropia, plastyczność, zdolność do formowania, mikrostruktura, czułość na prędkość odkształcenia, umocnienie odkształceniowe

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1. Introduction

Ultrafine-grained materials (UFG), defined as polycrystalline materials characterized by a grain size below 1 μm, have attracted considerable interest for over 30 years. Due to their refined grain size, they feature new and often superior properties in comparison with conventional coarse-grained (CG) materials. A lot of basic research has been conducted on UFG materials, mainly to investigate relationship between microstructure and properties by means of a detailed characterization of microstructural and mechanical properties. A substantial amount of work has focused on designing an optimal microstructure for enhancing mechanical strength, achieving high ductility despite extreme strengthening, or acquiring superplastic properties. Yet the amount of applied research has been limited, which can be seen as a reason for the poor commercialization of UFG materials. Additionally, the small manufacturing batches and the geometry of the billets hinders the development of UFG products. Severe Plastic Deformation (SPD) methods, which are the most efficient for fabricating substantial quantities of UFG materials, are suitable for bulk processing; however, the billets are usually in the form of small rods or disks.

Increasing the application potential of UFG materials can be achieved either by upscaling the process, which necessitates constructing machinery of high power and larger processing tools, or adjusting the SPD methods to processing billets in shapes suitable for further technological processes, i.e. sheets or plates. From the technological point of view, metal working seems advantageous, as it is used for large scale manufacturing. The right selection of processing technology is essential, as each manufacturing method and processing route results in certain characteristic features that affect the microstructure of the material and define its properties. The majority of available SPD methods designed for sheet or plate processing are strongly directional in nature, which results in anisotropic mechanical properties in the sheet's plane. This in-plane kind of anisotropy exhibits different mechanical properties in various testing directions, which has an impact on the quality of the formed parts. The primary reason for the anisotropy is the crystallographic texture created during the sheet manufacturing process.

A crucial step towards the commercialization of UFG materials is to conduct applied research focused on actual forming processes in order to verify whether UFG materials are suitable for metal working. Until now, the number of such studies has been very limited. However, those available indicate that, even though UFG materials are characterized by substantially lower ductility than CG ones in uniaxial tensile tests, their performance in more complex strain states under which most metal working processes are conducted results in often comparable or sometimes even better formability.

The aim of this dissertation is to manufacture UFG aluminum alloy plates, conduct technological forming trials, and improve the ductility of the plates. In order to achieve this, two techniques are used, i.e. Incremental Equal Channel Angular Pressing, and hybrid processing composed of multi-turn Equal Channel Angular Pressing followed by upsetting. The manufactured plates are evaluated in terms of microstructural homogeneity and crystallographic texture, mechanical properties isotropy, planar and normal anisotropy, strain hardening ability, strain rate sensitivity, and formability.

2. Ultrafine-grained materials

2.1 Definition and general features

UFG materials can be defined as polycrystalline materials characterized by a very small grain size, below 1 μm, which feature equiaxed grains with a dominant fraction of high angle grain boundaries (HAGBs) [1]. This last feature is crucial as makes it possible to achieve new functional properties, such as enhanced electric conductivity, magnetostriction, thermoelectricity or biocompatibility. A summary of studies on functional properties can be found in review articles [2–4]. Refining grain size not only alters functional properties; it also has a tremendous effect on mechanical properties, as it causes a great increase in tensile strength at room temperature, makes it possible to achieve superplasticity at elevated temperatures, and alters fatigue properties [2]. Materials can be manufactured using two groups of methods: bottom-up and top-down. The first group is based on the concept of joining nanoparticles in such processes as consolidation or deposition. In the second group, known as Severe Plastic Deformation (SPD), the existing bulk material is subjected to severe strain in order to refine grain size. The majority of bottom-up methods based on deposition processes are particularly effective in producing nanoscale grain in particles or thin layers, but their application is limited due to the small volume of material produced. More effective in terms of volume are processes based on consolidation. In order to produce UFG metals at a larger scale for applications such as structural parts, SPD techniques are considered the most efficient, but are usually detrimental to ductility. The two main SPD methods are described in Section 2.4.

The most outstanding feature of UFG materials is theri superior mechanical strength, typically 3-8 times higher than that of conventional materials [2]. This is mainly attributed to their reduced grain size. The relationship between yield stress σ_y and grain size d for UFG materials can be described by Hall-Petch relationship, as in Eq. 1, where σ_0 and K_{HP} are constants for a given material. What follows from this equation it that the smaller the grain size, the higher the yield stress. The relationship between flow stress and grain size, for the experimental data and model, is presented in Fig. 1 a for various UFG metals with a faced centered cubic crystal structure. This trend is true up to a certain grain size, as for nanomaterials it can break down, as can be noted in Fig. 1 for aluminium (marked with a blue line), whose flow stress decreases below a grain size of 0.02 μm.

$$\sigma_y = \sigma_0 + K_{HP} d^{-1/2} \qquad \text{Eq. 1}$$

The superior strength mainly stems from the small grain size, but also from such microstructural features as the formation of nanoclusters, segregations, nanotwins and dislocations substructures [2]. The increase in mechanical strength is usually accompanied by reduced ductility, which will be further discussed in Section 2.2.

Apart from superior strength, some UFG metals feature other attractive mechanical properties, i.e. superplasticity, which is defined as the ability of a polycrystalline specimen to achieve very high strain, exceeding elongation of 400% in tension [5]. Superplastic properties can be achieved when, grain boundary sliding becomes the dominant flow process. Strain rate varies inversely with grain size to a power of 2, which basically means that small grain sizes of SPD processed materials could be suitable for achieving superplastic flow at extremely high strain rates. Additionally, superplastic flow is connected with temperature and strain rate, so a reduction in grain size should make it possible achieve significant elongations at relatively low temperatures and high strain rates. An example of superplastic tensile Zn-Al specimens with a maximum elongation of 1800% are shown in Fig. 1 b.

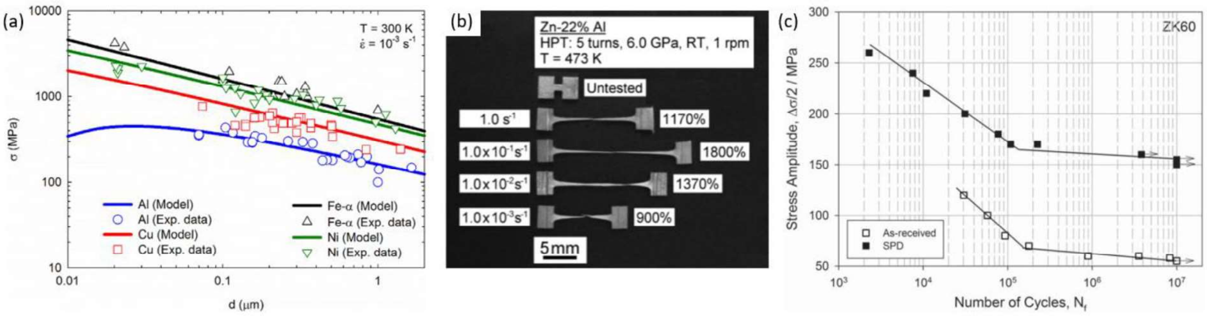

Fig. 1 Flow stress σ in grain size d function of various UFG metals [6] *(a) and superplastic tensile specimens after deformation* [7] *(b), change of fatigue behavior of magnesium alloy before and after SPD processing* [8]

A UFG microstructure also affects fatigue behavior, which is generally determined by strength and ductility. High-cycle fatigue (HCF) is dependent on the resistance of the material to crack initiation, i.e. fracture strength, while low-cycle fatigue (LCF) by crack propagation, i.e. ductility. Since SPD processing usually increases mechanical strength and decreases ductility, HFC is improved, while LFC decreases. However, cases have been reported where by refining grain size it was possible to improve both HCF and LCF, as for magnesium alloy, which at the same time showed enhanced strength and ductility, which translated into fatigue properties [8], as shown in Fig. 1 c.

2.2 Trade-off between strength and ductility of SPD processed materials

SPD processed metals feature a significant enhancement of mechanical strength, which is accompanied, however, by a loss of ductility. Numerous studies report on the relationship between strength and ductility, which is summarized in Fig. 2a [9] and which plainly indicates that there is a certain tradeoff between those properties; for the majority of metals it is impossible to achieve high values of both simultaneously. This great loss of ductility is even more clearly visible when evaluating uniform deformation, as presented in Fig. 2b. The majority of SDP-processed materials undergo necking at a uniform elongation below 5%, which is a significantly lower value than for CG materials. This sudden loss of ductility is visible at the early stages of SPD processing, at strain ε=1-2, and can sometimes be restored after further processing. As strain localization - i.e. exceeding uniform elongation - is present in the early stages of deformation, the material loses its strain hardening ability in comparison with its CG counterparts.

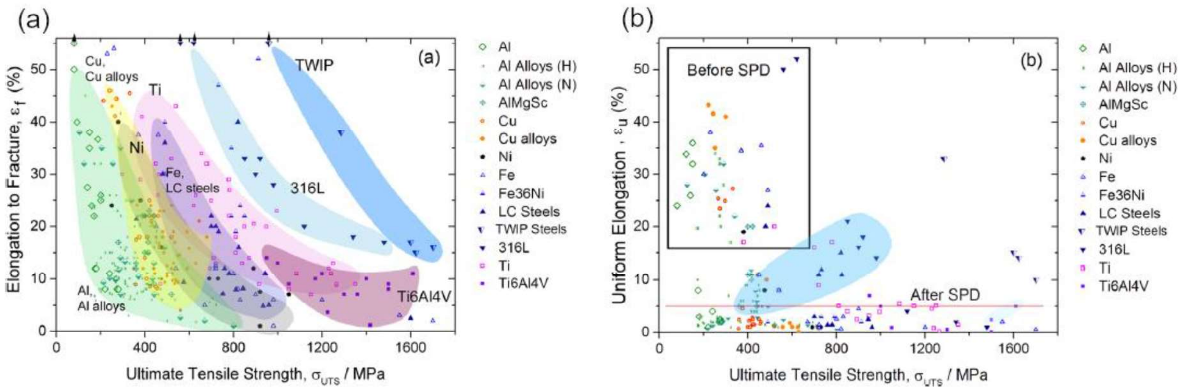

Fig. 2 Elongation to fracture (a) and uniform elongation (b) in a function of ultimate tensile strength of various UFG materials processed by SPD [9]

The inadequate ductility of UFG materials can be attributed to three causes: artifacts from processing, force instability in tension and crack nucleation or instability propagation [10]. The microstructural features of UFG materials and the resulting deformation mechanisms limit the material's ability to accumulate plastic deformation. This results in a rapid localization of strain, i.e. necking, which leads to a relatively fast material failure. This manifests as a rapid decrease in stress right after reaching a maximum value in engineering stress-strain curve, as shown in Fig. 3a, which is in clear contrast to the curve obtained for the CG material, where stress gradually increases, reaches a plateau and slowly decreases after achieving maximum stress at significant elongation. From Fig. 3b it is obvious that, even at the initial stages of SPD processing, necking occurs at a small strain rate in comparison with the CG material.

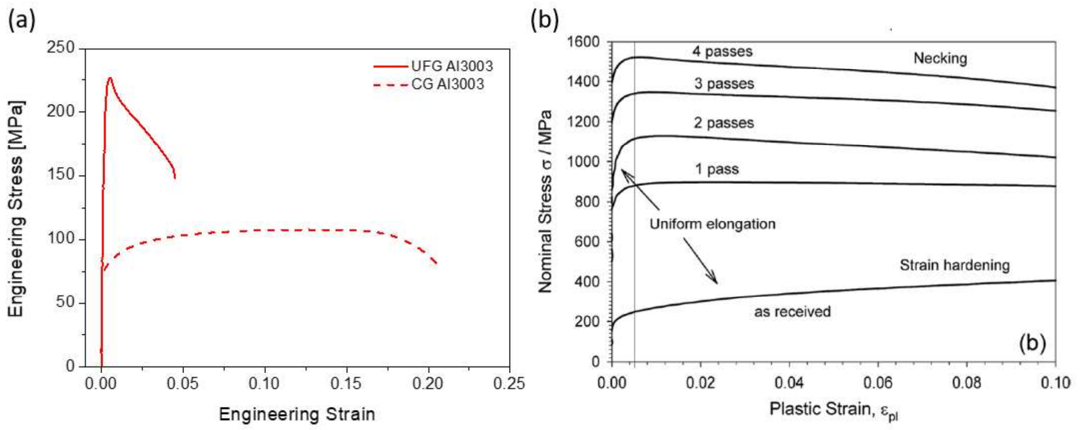

Fig. 3 Engineering stress-strain curves for UFG and CG aluminium alloy (author's results) (a), nominal stress vs plastic strain of 316L stainless steel (b) [9]

In general, ductility depends on two factors: strain hardening (SH) capacity and strain rate sensitivity (SRS); these can be described using two criteria: Considére (Eq. 2) and Hart's (Eq. 3), which define necking. The Considére criterion is based on the assumption that, if a material has a very high yield strength, rapid necking can only be compensated for by high SH capacity, whereas the Hart's criterion modifies this by adding an additional parameter, i.e. the SRS coefficient m, indicating that low SH capacity can be compensated for by a high value of m. Based on those criteria, it can be concluded that enhancing the ductility of a material is possible by increasing SH and/or SRS.

$$\left(\frac{\partial\sigma}{\partial\varepsilon}\right)_{\dot{\varepsilon}} \leq \sigma \qquad \textit{Eq. 2}$$

$$\frac{1}{\sigma}\left(\frac{\partial\sigma}{\partial\varepsilon}\right)_{\dot{\varepsilon}} - 1 + m \leq 0 \qquad \textit{Eq. 3}$$

According to Eq. 3 the ductility of a material is dependent on SH and SRS. For FCC materials, at room temperature, parameter m usually has values very close to 0, which increase together with temperature. Therefore, at RT, ductility is mainly controlled by SH, which can be defined as the ability of the material to strengthen during deformation. However, the ability of UFG materials to accumulate dislocations is very limited, as they are rapidly absorbed by grain boundaries. As a consequence, there is a challenge: how to enhance ductility while maintaining high mechanical strength and this topic has drawn significant attention from the scientific community.

2.3 Attempts to improve strength/ductility balance

2.3.1 Approaches to enhance strain hardening by microstructure design

Strain hardening can be defined as the ability of a material to strengthen during deformation, since during straining, dislocations are generated and as they block each other's motion, higher stress is needed to deform the sample. However, dislocations are not only generated, but also annihilated, which counteracts SH. The rate of generation and annihilation is called the recovery rate. The Kocks-Mecking-Estrin (KME) model [11] assumes that the kinetics of plastic flow are determined by dislocation density. This model is based on two assumptions: i.e. that the shear stress τ is dependent on the total dislocation density ρ, plastic shear strain rate $\dot{\gamma}$ and absolute temperature T (Eq. 4), and that the kinetic equation for the dislocation density is expressed by Eq. 5,where $\dot{\rho}^{+}$ is the rate of dislocation accumulation and $\dot{\rho}^{-}$ of dislocation annihilation.

$$\tau = \tau(\rho, \dot{\gamma}, T) \qquad \text{Eq. 4}$$

$$\dot{\rho} = \dot{\rho}^{+} + \dot{\rho}^{-} \qquad \text{Eq. 5}$$

Microstructural features govern SH ability, since the generation and accumulation of dislocations in a material depends on such features as grain boundary types and grain size [12]. Grain size plays an important role in SH, as for nanocrystalline materials, grain size is smaller than the mean free path of mobile dislocations, and therefore dislocation after bowing out from the grain boundary is not expected to interact with other dislocations inside the grain, but to travel to another grain boundary [7]. The type of boundaries is also important, since grain boundaries can act as sources and sinks of dislocations. LAGBs are partially mobile and can take part in dislocation annihilation. Additionally, LAGBs are thermally and mechanically unstable, which makes them susceptible to take part in recovery processes. Therefore, introducing a high fraction of HAGBs, which are stable, as well as decreasing the number of dislocations inside the grains, will suppress recovery processes and increase SH. Obtaining both high strength and high ductility in UFG metals is essential, and several approaches based on tailoring the microstructure have been developed to achieve this goal.

One of the proposed approaches is to create a metal with a high density of twin boundaries (TBs), which have been found to be effective in blocking dislocation movements and thereby increasing SH capacity. A microstructure containing a high fraction of TBs has been successfully developed in a thin foil as well as in a bulk material. In [13], a copper foil of

400 nm average grain size with a high density of growth twins was synthesized using pulse electrodeposition. Dislocations were proven to accumulate at the TBs, which effectively blocked their motion, leading to a mechanical strength 10 times greater than that of coarse-grained copper, and to a total elongation of 13%. The improvement of mechanical strength and ductility of Cu with a high density of TBs in comparison with nanocrystalline and coarse-grained copper is illustrated in Fig. 4 a. In [14], bulk copper-aluminum (15 at.%) alloy with a large number of TBs was produced using friction stir processing with rapid cooling. The processing introduced abundant annealing TBs due to the occurrence of dynamic recrystallization, and resulted in the acquisition of a high-strength material characterized by SH ability and enhanced ductility, i.e. a total elongation exceeding 15%.

Another approach is based on the concept of bi-modal grain size distribution, i.e. two populations of micro- and nano-scale grains [15]. In this concept, the nano-scale grains provide higher strength, while the micro-scale grains compensate for the limited ductility. An example of copper featuring various microstructure types is shown in Fig. 4 b, where curve A represents a metal with a homogenous UFG microstructure and curve C shows the tensile curve of a bi-modal one. This sample was characterized by a majority of nano-sized grains, i.e. below 200 nm, and only 25% of micro-sized grains. It was found that a significant number of dislocations were stored in the latter, which enhanced SH capacity. Due to the high fraction of significantly refined grains, high mechanical strength was maintained, more than two-fold in comparison with the coarse grained metal. Manipulating grain size populations, as well as their fraction and distribution offers a wide array of possibilities that have been studied extensively by many researches and are summarized in [16].

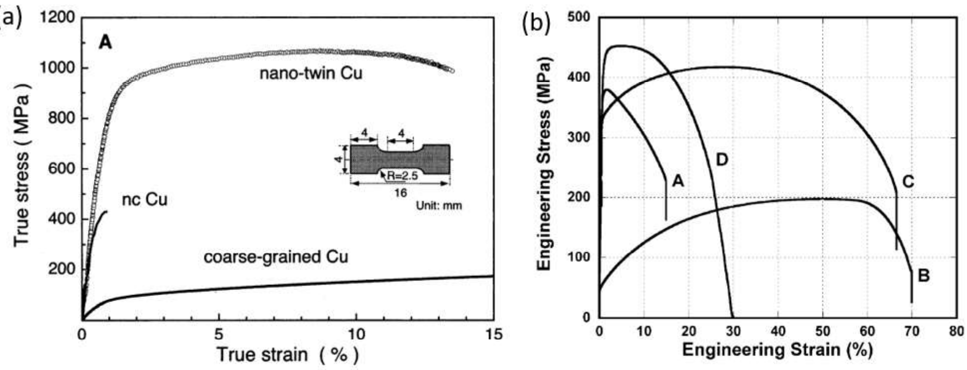

Fig. 4 Tensile curves of copper film with high density of TBs compared with nanocrystalline and coarse-grained counterparts [13] *(a), Cu with different microstructures; Cu after ECAP – A; coarse grained Cu – B; bimodal nanostructured Cu – C; Cu after ECAP, cold rolling at liquid nitrogen temperature and annealed – D* [15]

Introducing second-phase particles has also been shown to simultaneously increase strength and ductility, by increasing dislocation accumulation and resistance to dislocation slip. To achieve strengthening by second-phase particles, 7075 aluminium alloy was nanostructured by solution treatment followed by subsequent cryo-rolling and aging, which resulted in an average grain size of 100 nm and fine second-phase particles. A TEM investigation of the tensile coupons after tensile testing revealed that dislocations accumulated around the second phase particles and enhanced dislocation storage capability [17]. The effect of second-phase particles on the tensile curves is shown in Fig. 5 a, where materials with and without particles are designated NS+P and NS, respectively. The presence of second-phase particles led to a two-fold increase in ductility and higher mechanical strength.

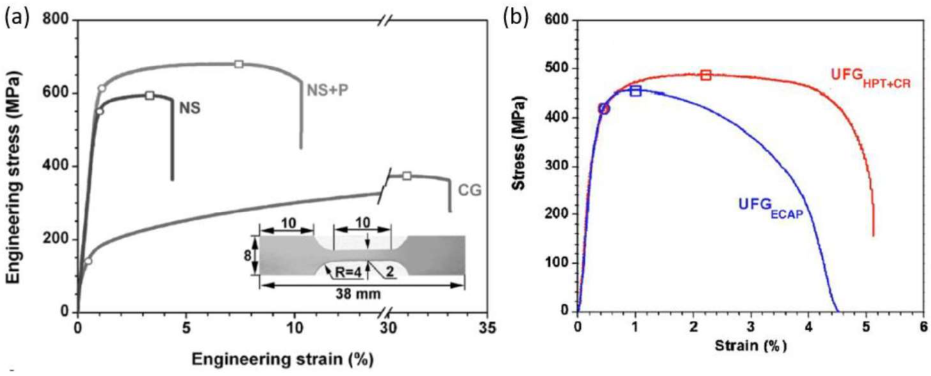

Fig. 5 Tensile curves of nanostructured aluminum (NS) and enhanced by second-phase particles (NS+P) [17] *(a) and copper characterized by LAGBs with a high density of dislocations (UFG_{ECAP}) and HAGBs with a limited number of dislocations (UFG_{HPT+CR})* [18] *(b)*

An enhancement in strength and ductility was also noted when a microstructure characterized by a high number of HAGBs and a low dislocation density was developed [18]. To observe the impact of such a microstructure, pure copper was processed by SPD techniques so as to acquire two opposed kinds - one characterized by a majority of LAGBs and a high density of dislocations inside grains, the other by a majority of HAGBs and a reduced number of dislocations. Tensile curves of those materials are shown in Fig. 5 b and marked as UFG_{ECAP} and UFG_{HPT+CR}, respectively. Sample UFG_{HPT+CR} is characterized by enhanced ability for SH, which led to greater elongation. It was suggested that this behavior is caused by the fact that HAGBs are more effective in blocking dislocations, while at LAGBs slipping dislocations can react with extrinsic ones, which results in their annihilation. Additionally, a lower initial dislocation density may allow for their accumulation during tensile strain.

Modifying stacking fault energy (SFE) is another proposed method. To investigate this phenomenon nanocrystalline Cu-Zn alloys with various SFE were produced and compared to pure copper, whose SFE is equal to 41 mJ/m^2 [19]. Lowering the SFE to 22 mJ/m^2 proved to increase dislocation and twin accumulation, leading to higher SH and ductility. However, decreasing the SFE further to 7 mJ/m^2 led to an extremely small grain size, i.e. 15 nm, which translated into increased mechanical strength and deteriorated ductility. Similar research was conducted on Pd and Pd-Ag alloy processed by HPT, and comparable results were obtained [20]. Lowering the SFE by adding alloying elements to pure metal made it possible to improve the strength/ductility balance of the alloy.

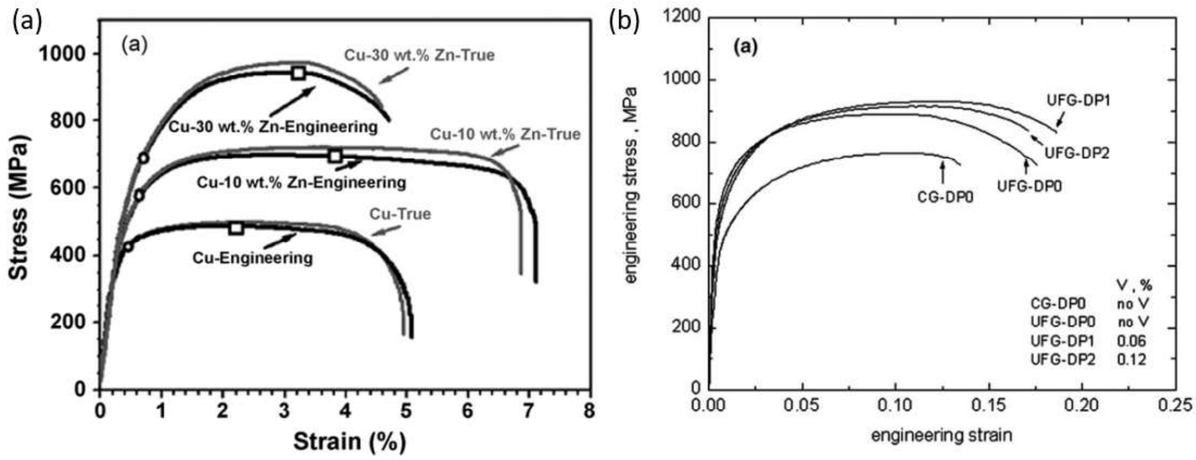

Fig. 6 Tensile curves of Cu-Zn alloy with various SFE; 41, 22 and 7 mJ/m^2 for pure Cu, Cu-10% Zn and Cu-30% Zn, respectively [19] *(a) and dual-phase carbon steel with various V content* [21] *(b)*

Establishing multiple phases is another possible way to increase both strength and ductility [21]. Low carbon steel with various content of V was processed by ECAP and heat treatment to produce a ferrite-martensite dual phase material. The tensile curves presented in Fig. 6 b indicate that the UFG samples, marked as UFG-DP0, UFG-DP1 and UFG-DP2; feature higher mechanical strength and ductility than the coarse-grained samples, as well as different SH behavior. Two crucial observations were made: mobile dislocations were present only in ferrite which was close to martensite, and the inter-martensite spacing for UFG materials was much smaller than for the CG samples. Due to the presence of those dislocations, mechanical strength and ductility were simultaneously enhanced. Similar results are reported [22] and it is concluded that overcoming early plastic instability leading to limited elongation is possible by creating a dual phase microstructure, such as ferrite-martensite or ferrite-bainite.

2.3.2 Strain rate sensitivity

SRS can be defined using physically based strain rate sensitivity S description as in Eq. 6 [23]:

$$S = \frac{k_B T}{v^*} \qquad \textit{Eq. 6}$$

where k_B is Boltzmann's constant, T is absolute temperature and v^* is activation volume. v^* is defined as the derivative of the activation enthalpy, as in Eq. 7, where ΔH(τ) is the activation enthalpy, τ is shear stress and $\dot{\gamma}$ is shear strain rate.

$$v^* = -\left[\frac{\partial \Delta H(\tau)}{\partial \tau}\right]_T = k_B T \left(\frac{\partial ln\dot{\gamma}}{\partial \tau}\right)_T \qquad \textit{Eq. 7}$$

In engineering, SRS is commonly described by parameter m, defined by Eq. 8

$$m = \frac{\partial ln\tau}{\partial ln\dot{\gamma}} \qquad \textit{Eq. 8}$$

By substituting Eq. 6 with Eq. 8, the following relationship is created between activation volume and m parameter [24]:

$$m = \frac{S}{\tau} = \frac{k_B T}{\tau v^*} \qquad \textit{Eq. 9}$$

This indicates that SRS is dependent on grain size and temperature. Therefore, to enhance ductility without reducing mechanical strength, it is possible to take advantage of SRS parameter m. For FCC metals, with increasing temperature, SRS increases, as it does with decreasing grain size, as shown in Fig. 7. The reason for this is a decrease in the activation volume, which can indicate active plastic deformation mechanisms. For aluminum, a rate-controlling mechanism for materials with traditional grain size ($d>\sim10^{-6}$m) is dislocation intersections, whereas for ultra-fine grained materials ($d\sim10^{-8}$ to $\sim10^{-6}$m) it is dislocation emission from grain boundary sources, or grain boundary shear promoted by dislocation pile-ups; nanocrystalline ($d<\sim10^{-6}$m) materials are dependent on grain boundary processes only [25]. However, those mechanisms are also dependent on strain rate and temperature, as at elevated temperature thermally-activated processes can take place.

Conventional FCC metals usually have an activation volume of 10^2-10^3 b^3, which is associated with dislocations cutting through forest dislocations. For UFG materials, the apparent activation volume usually slightly exceeds 10 b^3, due to from reduced dislocation interactions. The main rate controlling mechanism is no longer work hardening caused by forest dislocation intersections, but other mechanisms are present. The activation volume values observed for diffusion based processes, caused by rearrangements or defect movements within the grain boundary, are usually below 10 b^3.

As indicated in Eq. 9, activation volume is the denominator, which means the smaller its value, the larger the m value. Therefore, a decrease of activation volume leads to an increase in the SRS parameter m, which in turn enhances ductility. In the case of increasing v^* by reducing grain size, the strength of the material is simultaneously enhanced, as indicated by the Hall-Petch relationship.

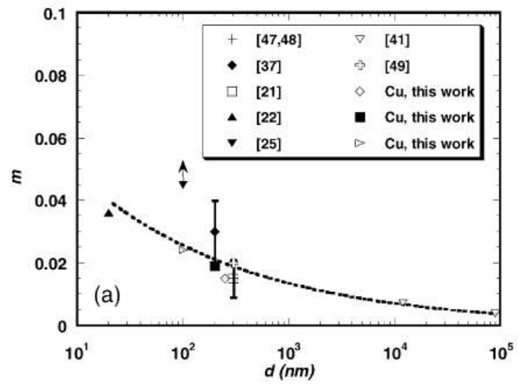

Fig. 7 SRS parameter m as a function of grain size [24]

2.4 Fabrication of UFG materials

UFG materials can be produced in relatively large quantities using SPD methods, which were first proposed in 1952 by Bridgman [5], when thin discs were compressed and subjected to torsional straining. A major advancement was achieved in 1981, when Segal et al. [26] proposed two intersecting channels of equal cross-section and proved that such geometry provides uniform deformation by simple shear, as shown in Fig. 8. The methods of SPD and bulk nanostructured materials have attracted a growing interest in materials science. A review paper concerning NSM by Ovid'ko [7] published in 2018 is one of the most cited in the world with more than 5,000 citations.

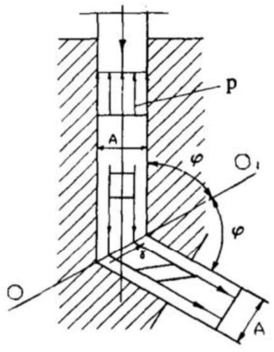

Fig. 8 Equal Channel Angular Extrusion proposed by Segal [27]

The method proposed by Segal later became the most recognized SPD method known as Equal Channel Angular Pressing (ECAP). It is based on the concept of two intersecting channels of the same cross-section, through which a metallic material is pressed and therefore subjected to large strains. As the shape of the specimen does not change during processing, one piece of material can be subjected to ECAP several times leading to extremely high strains. The strain is dependent mainly on the angle between the channels Φ, which is the highest for a right angle, in which case the imposed strain in one pass is 1.15. Aside from the number of passes, the specimens can be rotated between consecutive passes, which results in differences in the microstructure acquired as a result of the shearing patterns specific to each route.

In [28], it was proposed that grain refinement during ECAP is a consequence of the very high dislocation density introduced into the material, which leads to the formation of an intragranular structure composed of cell walls and low angles of misorientation. It is important to note that, as the strain increases, the cell wall thickness decreases as the dislocations are annihilated, resulting in an increasing number of dislocations of only one sign, which in consequence introduce grains separated by high-angle, non-equilibrium boundaries. This theory explains the increase in the average grain boundary misorientation with the number of consecutive passes. However, grain refinement does not continue infinitely, but saturates at some point. This is explained by extending the theory - by adding the relationship between slip and the formation of subgrain boundaries. Depending on the applied route, i.e. the sequence of rotations of the billet between consecutive passes, various shear planes are activated. The route that activates the highest number of shear planes, in which the billet is rotated by 90° after each pass - called B_C - leads to the creation of the most homogenous and equiaxed microstructure, as the shear planes interact with each other at the highest angle [29]. These shearing patterns resulting from the processing route are the most important factor influencing the material's microstructural features, including both grain morphology and misorientation angles.

The second most known processing method is High Pressure Torsion (HPT), in which a sample is subjected to torsional straining under high hydrostatic pressure. This method is mostly conducted using small disks, although some developments have been reported for cylindrical specimens [30] and rings [31]. The main idea of the process is shown in Fig. 9. A billet in a form of small disk is placed between two plungers, pressure is applied, and the material is torsionally strained by rotating one of the anvils. To stop the material from flowing between anvils, the disk is placed in a cavity in the tools.

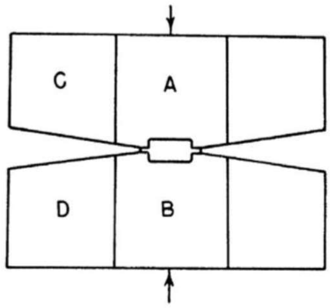

Fig. 9 Principle of HPT [32]

The equivalent von Mises strain imposed on the disk is expressed by Eq. 10, where N is the number of turns, r is the radial distance measured from the center of the disk and h is its initial thickness.

$$\varepsilon = \frac{2\pi N r}{h\sqrt{3}} \qquad \text{Eq. 10}$$

It follows from the equation that the strain varies depending on the distance from the center of the disk, where it equals to 0, and increases with distance towards the outer edge. However, since multiple rotations can be performed using HPT, it has been demonstrated that reasonably homogenous materials can be produced using this technique [33]. Additionally, HPT can produce a material having a smaller grain size and higher fraction of HAGBs than is possible using ECAP. The main disadvantage of HPT is that scaling up the process is difficult.

Despite their high efficiency in grain refinement, most SPD methods are designed for refining grain size in rods, bars or disks, whose shape imposes limitations on the possibilities of further processing and creating a final product. A well-developed group of conventional processing techniques, i.e. sheet forming processes, are designed for sheet or plate billets. Therefore, metal in sheet form would be more suitable and attractive for commercial use.

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3. Production of UFG plates and sheets

3.1 Processes based on rolling

3.1.1 Accumulative roll bonding

Among processes based on rolling Accumulative Roll Bonding (ARB) is the most popular and was first proposed by Saito et al. [34] in 1998 as a novel method for imposing a high plastic strain on bulk materials. The process is based on conventional roll bonding, a commonly used process in the plastic forming industry, in which two strips are placed on each other and joined together by rolling. The contact surfaces are chemically and mechanically treated in advance so as to enhance the bonding. The proposed development in the ARB process is that the bonded strips are cut in half in length, stacked on top of each other and bonded again, as shown in Fig. 10a. The process can be repeated many times, and results in a layered material with a UFG microstructure characterized by layers a few nm in thickness, as shown in Fig. 10b. The process must be carried out below the recrystallization temperature of the material, as excessive heating reduces the effect of the plastic working.

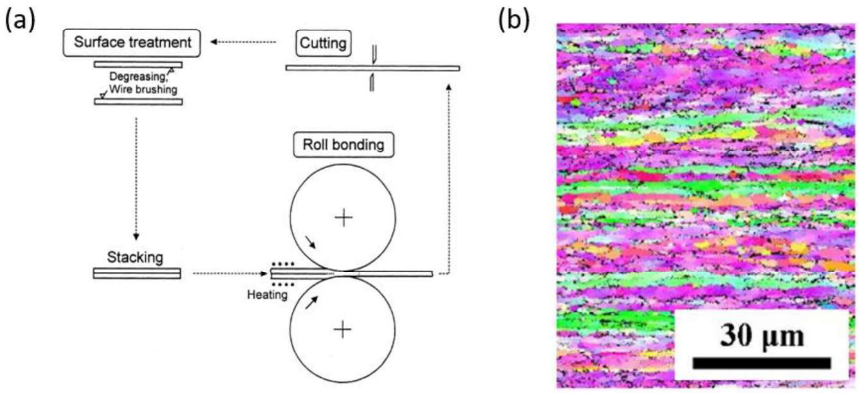

Fig. 10 Diagram of Accumulative Roll Bonding [34] *(a) and microstructure of 1199 after 4 ARB passes* [35] *(b)*

In ARB, two factors affect the sheet's properties:1) the diameter of the rolls and the reduction in thickness, through which shearing is introduced, and 2) the friction that exists between the rolls and the sheet, which only changes within a fairly thin surface layer. However, as the rolling is repeated and the sheets stacked on one another, a through-thickness strain gradient is created, which makes it difficult to produce a homogenous sheet [36]. During ARB, a typical rolling texture is created, i.e. containing a major copper and a minor brass component for face-centered cubic metals. Additionally, a rotated cube component typical of shearing is observed, although mainly in the surface area [37]. This texture results in a quite pronounced anisotropy, both normal and planar, as shown in Fig. 11 a and b, whereas the effect of the texture components on the r-value are presented in Fig. 11 c. Such an anisotropy can lead to such undesired effects in the parts formed as earing or uneven a thinning of the walls, which can limit ARB's potential for commercial applications.

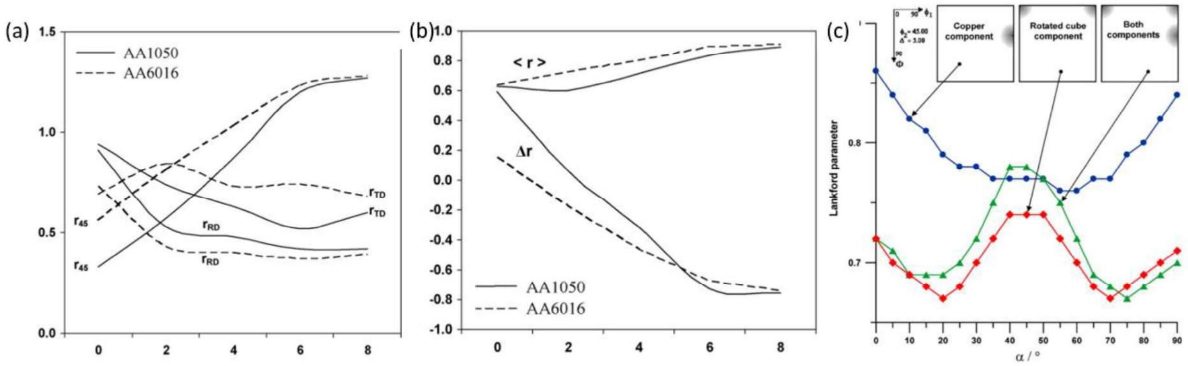

Fig. 11 r-value for RD, TD and 45 (a), planar anisotropy (b) and relationship between texture components and Lankford parameter (c) for ARB processed Al 1050 and 6016 sheets [36]

To reduce the texture intensity and anisotropy of the mechanical properties of ARB-processed sheets, cross rolling is employed, which involves introducing rotations between subsequent passes. The cross rolling not only reduces anisotropy, but also increases mechanical strength in comparison with unidirectional rolling, as shown in Fig. 12. It has been shown that in the course of cross rolling a rotated Brass texture is developed, resulting in similar tensile properties in the rolling and transverse directions [38]. This was attributed to the fact that changing the direction of the processing alters the strain path and permits the activation of a latent slip system. The critical resolved shear stress needed to activate dislocation sliding evolves during cross ARB [39].

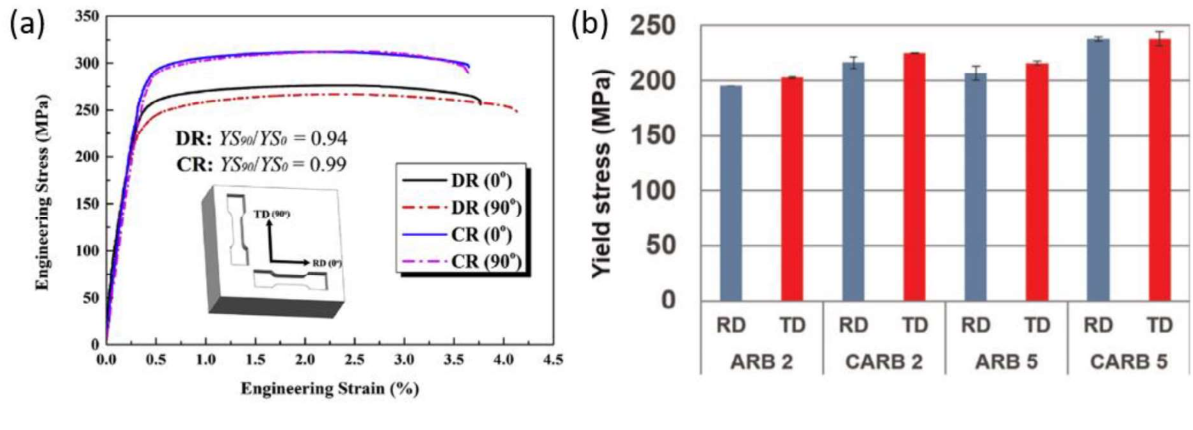

Fig. 12 Comparison of unidirectional rolling and cross rolling in ARB process in [38] *(a) and* [40] *(b)*

3.1.2 Equal Channel Angular Pressing combined with rolling

ECAP combined with subsequent rolling is a solution to manufacture UFG sheet simply. However, during such a process, a reasonably equiaxed microstructure crated during ECAP processing turns into a lamellar one, as depicted in Fig. 13. Additionally, it has been proven that such processing results in a significant increase in the share of HAGBs, which is attributed to dynamic recrystallization [41]. As rolling is the last processing step, a crystallographic texture is again typical for this operation Fig. 13 c.

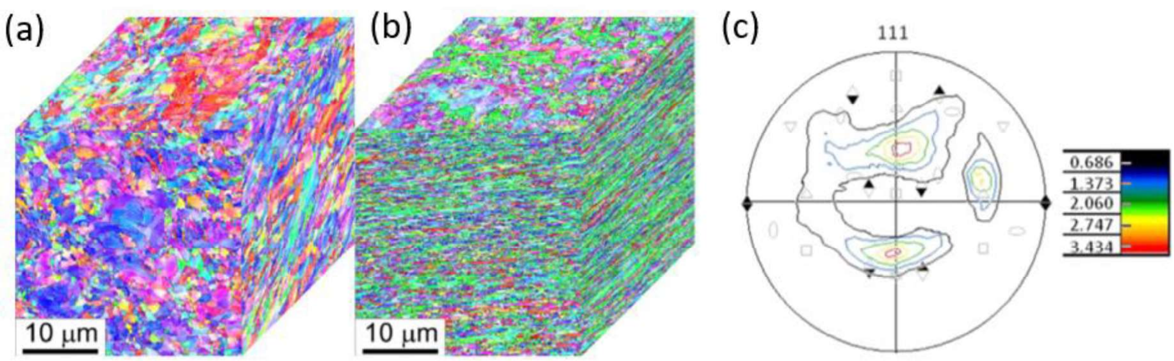

Fig. 13 Microstructure of specimen after ECAP processing (a) and subsequent rolling (b) [41] *and 111 pole figure of crystallographic texture after such processing* [42]

Cross rolling is also used to control the texture and properties of ECAP processed billets. In [43], copper billets were unidirectionally and cross rolled, which influenced the crystallographic texture as shown in Fig. 14. However, in that study, stress anisotropy was attributed to the microstructurally dependent anisotropy of critical resolved shear stress rather than texture strength.

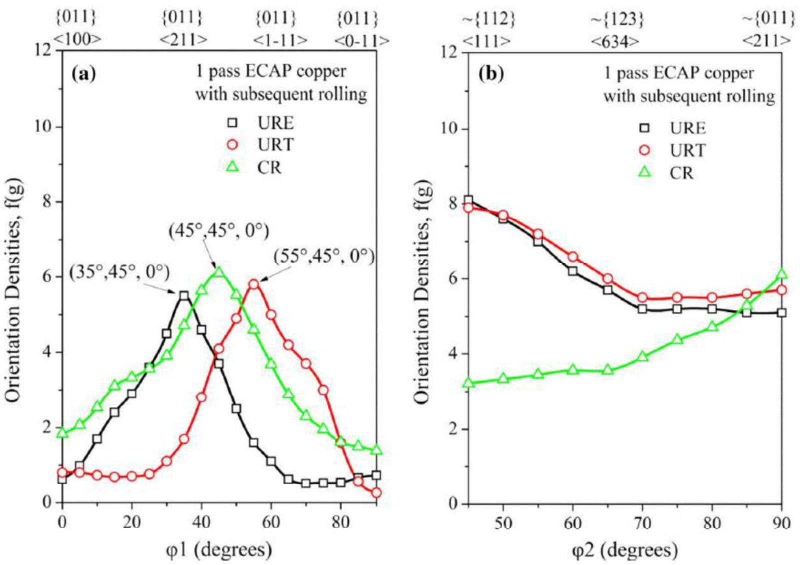

Fig. 14 Change in crystallographic texture depending on rolling direction [43]

3.1.3 High Pressure Torsion combined with rolling

High Pressure Torsion (HPT) is used to obtain a UFG or even nanoscale microstructure in small disks, as the specimens are subjected to a compressive force and concurrent torsional straining [33]. It was recently reported that subjecting disks after HPT to rolling makes it possible to manufacture long objects, improve the homogeneity of the microstructure of the disk, and improve its mechanical strength. As in other SPD processes followed by rolling, the grains become elongated and exhibit a microstructure typical of rolled materials; the crystallographic texture transitions from a shearing texture after HPT to a rolling texture, as in Fig. 15 [44].

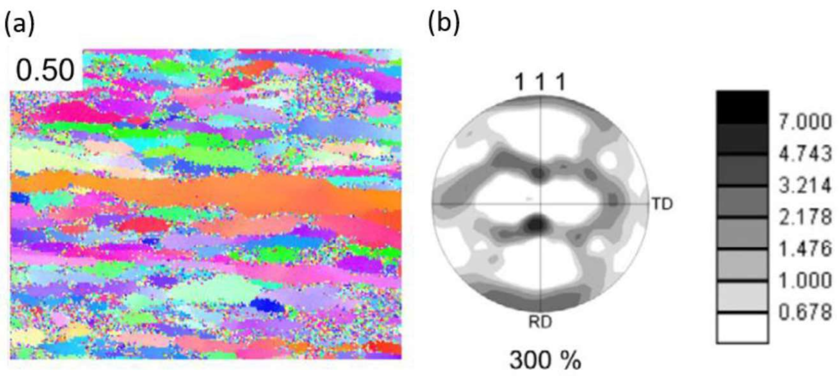

Fig. 15 Microstructure (a) and pole figure (b) of copper sample after HPT followed by rolling [44]

3.1.4 Other Methods

Apart from the methods described above, many others have been developed, such as a combination of Constrained Groove Pressing followed by rolling [45], Asymmetric Accumulative Roll Bonding [46], Asymmetric Rolling [47], Cryorolling [48], and Accumulative Roll Bonding followed by rolling [49].

3.2 Processes based on shearing

3.2.1 Conshearing, Continuous Confined Strip Shearing, Equal Channel Angular Rolling, ECAP-conform

Conshearing [50], Continuous Confined Strip Shearing [51] or Equal Channel Angular Rolling [52] and ECAP-conform [53] processes are variations of conventional ECAP, but characterized by continuous feeding of the material, thanks to which it is possible to process long billets. All the above methods are equivalent in principle, and differ only in technological solution of feeding or ECAP die geometry employed. The ability to process long billets is achieved using a combination of rolls that supply material into the ECAP tool, as shown in Fig. 16 a. Similarly to conventional EACP, it is possible to repeat the process several times for one billet, introducing rotations between passes, and to tailor the microstructure and properties in a similar manner. As shearing is the dominant strain mode, it is possible to acquire a crystallographic texture so as to obtain a significant fraction of {111}//ND, which increases formability. In [51], 1050 sheets were processed by Continuous Confined Strip Shearing, and it was possible to achieve such a texture, which consequently led to an r-value of 0.83 and quite a low anisotropy of 0.27, as presented in Fig. 16 b. Conshearing of 1100 also resulted in an increased fraction of <111>//ND and r-values up to 0.94 [50] or 0.94 [54].

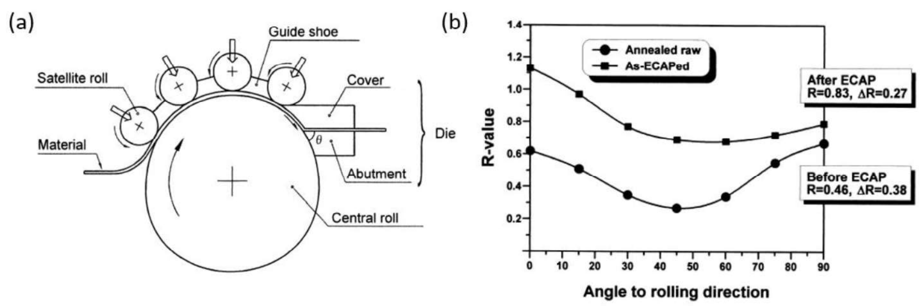

Fig. 16 Conshearing process [53] *(a) and changes in R-value in a function of angle to rolling direction for aluminium sheet after ECAR process* [51] *(b)*

Fig. 17 a shows the microstructure after continuous confided strip shearing, in which a fibrous structure with elongated grains at an angle of 72° to the processing direction are visible, which is compliant with shearing direction. As a result of processing, a strong shearing texture was created with components such as {111}<110> and {111}<112>, and {001}<110> rotated cube texture was also developed having reduced <110>//ND textures [55] (Fig. 17 b). This resulted in an enhancement of the r-value to even above 1, as shown in Fig. 17 c. However, due to the strong directionality of the process, the planar anisotropy also increased, which is unfavourable in terms of formability.

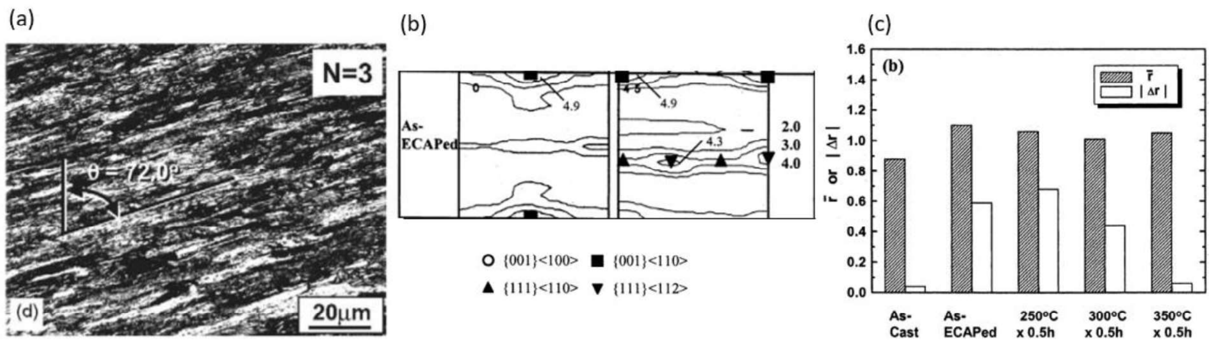

Fig. 17 Microstructure [56] *(a) crystallographic texture (b) and normal and planar anisotropy* [55] *(c) of 1050 processed by Continuous Confined Strip Shearing*

3.2.2 Modified ECAP for sheet processing

To reduce the number of processing steps necessary to manufacture UFG sheets, modified ECAP methods have been developed in which it is possible to insert a billet in the form of a plate by adjusting the die channel to a new geometry. However, a problem that arises from plate processing is that the force of friction becomes greatly elevated due to the larger surface remaining in contact with die. The technological solutions presented in Fig. 18 a and b are solid dies, in which friction is reduced by using a lubricant with high viscosity. Another design is shown in Fig. 18 c, namely Incremental Equal Channel Angular Pressing, where the processing tools are separated and the billet is deformed in small increments, so as to almost eliminate friction. This method is described in detail in Section 6.1.1, since it is used for processing the material discussed in this dissertation.

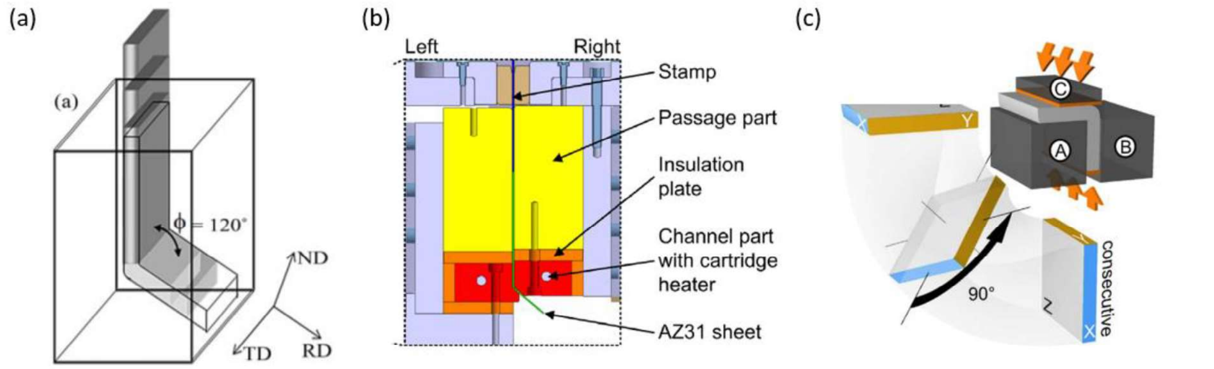

Fig. 18 Modified ECAP-based methods dedicated for sheet processing [57] *(a)* [58] *(b)* [59]*(c)*

3.2.3 Constrained Groove Pressing

Constrained Groove Pressing (CGP) [60] is a process in which a metal sheet is inserted between two asymmetrically grooved dies tightly constrained by a cylinder wall as in Fig. 19 a. The sheet is placed between the curved dies and then pressed. In the second pass, the sheet is pressed by flat dies, and so the previously deformed region is subjected to reverse shear deformation. The sheet is further processed in the same manner and can be rotated between subsequent passes.

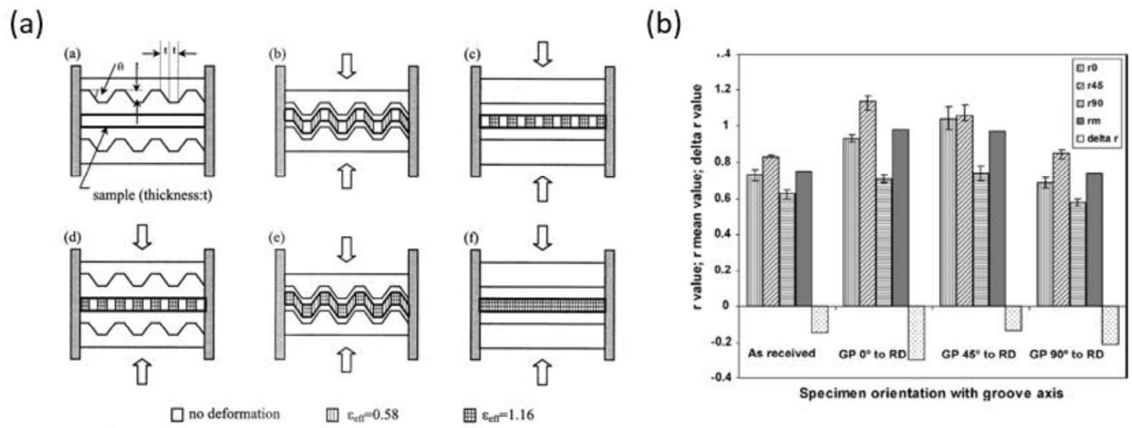

Fig. 19 Stages of Constrained Groove Pressing [60] *(a) and changes of r-value for various testing direction of the material after groove pressing* [61]

Another variation of CGP known as groove pressing investigated shown in [61] and was used in attempt to improve the r-value of the processed sheet. It was shown that by implementing certain rotations of the billet it is possible to acquire an r-value of around 1, as shown in Fig. 19 b. This improvement of r-value was also attributed to increased fraction of {111}//ND in comparison with the initial material, as shown in Fig. 20 a. Due to the CGP processing route, the microstructure of the plates is characterized by grains equiaxed in the sheet's plane, as presented in Fig. 20 b.

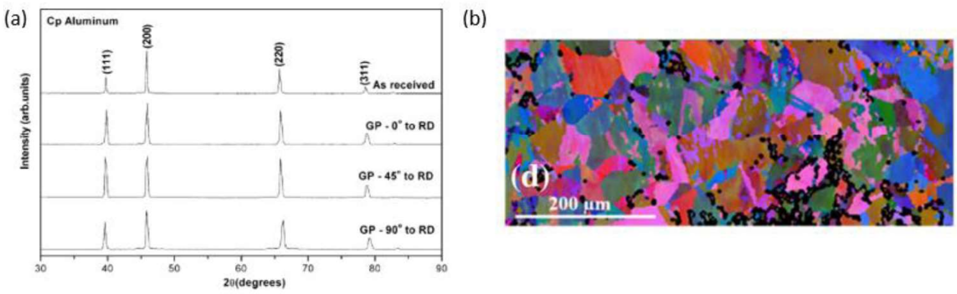

Fig. 20 Change of (111) peak intensity attributed to creating shearing texture [61] *(a) and microstructure of Cu-Zn CGP pressed sheet* [62]

3.2.4 Repetitive Corrugation and Straightening

During Repetitive Corrugation and Straightening (RSC) the workpiece is repetitively bent and straightened without changing the geometry [63]. This process is similar to CGP, the main difference being the tool geometry. The corrugation is carried out by tools like those shown in Fig. 21 a. It was proposed that this process could be easily redesigned so as to become a continuous process, as shown in Fig. 21 b; and indeed this was later developed [64].

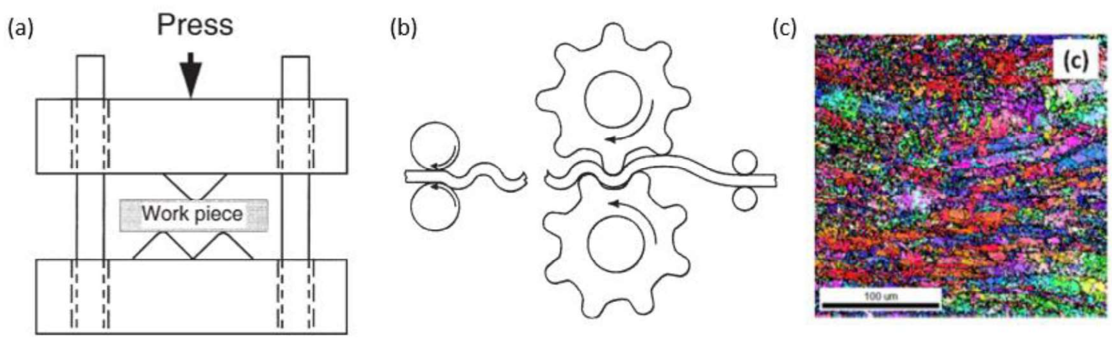

Fig. 21 Geometry of RCS tools (a), continuous RCS process (b) [63], *microstructure of Al-Li alloy processed by RCS (c)* [65]

Since it is possible to subject a billet to RCS many times and to rotate it between consecutive passes, a homogenous UFG microstructure can be obtained. An example of an Al-Li alloy UFG microstructure processed by RCS and characterized by elongated grains is shown in Fig. 21 c.

3.3 Anisotropy of Ultrafine-grained sheets and plates

A sheet demonstrates anisotropy when its properties measured in test pieces aligned at various angles to the processing direction display different values [66]. Additionally, those differences can be present in the plane of the sheet as well as through its thickness. The primary cause of anisotropy is the preferred orientation of grains [67]; this can more generally be described as a crystallographic texture. Such a texture is a consequence of the processing history of the workpiece, which is also the case for SPD processes. Depending on the stress state, SPD methods can be compared to conventional metal-forming processes, i.e. ARB introduces a rolling texture, while ECAP as a form of forging, introduces a shearing texture [68]. The extent of the sheet's anisotropy depends on the texture intensity, and influences the material's response to forming operations. Therefore, the desired anisotropy, or absence thereof, can be created by adjusting the crystallographic texture of the sheet.

The anisotropy of sheets is commonly expressed by Lankford parameter, also called r-value, which can be calculated during uniaxial tensile test of coupons cut at the direction of 0, 45 and 90° to the rolling direction (RD) of the sheet. The r_α-value, where α denotes the angle at which tensile coupon was cut, is a ratio of strain in the width (ε_w) and thickness (ε_t) in the coupon, as in Eq. 11. r_m parameter represents the mean value of the Lankford coefficient or normal anisotropy, and is defined by Eq. 12. Eq. 13 shows the planar anisotropy of a sheet, and Δr_p describes earing tendency (Eq. 14). The r-value is often used as a determinant of formability, as it describes the susceptibility of a material to thinning under tensile strain. According to Eq. 11, when r>1 the strain in the width is larger than in the thickness and the material can be stretched more extensively without failure. Therefore, the r-value shows the ability to resist thinning. r-value is also commonly used to describe the susceptibility to deep drawing, which is a good indicator of formability, as it clearly depicts not only the thinning tendency but also the anisotropy of plasticity, manifested as earing, i.e. an uneven rim of a deep-drawn cylinder. The influence of the severity of anisotropy on the formation of earing is shown in Fig. 22. From the point of view of deep drawing capability, materials characterized by an r-value >1 are desirable. However, in FCC metals is it difficult to achieve such values, since the cube texture, typical for cold-rolled and annealed sheets [61], is characterized by low r-value ~0.5 [69].

$$r_\alpha = \frac{\varepsilon_w}{\varepsilon_t} \qquad \text{Eq. 11}$$

$$r_m = \frac{r_0 + 2r_{45} + r_{90}}{4}$$ *Eq. 12*

$$\Delta r = \frac{r_0 - 2r_{45} + r_{90}}{2}$$ *Eq. 13*

$$\Delta r_p = r_{max} - r_{min}$$ *Eq. 14*

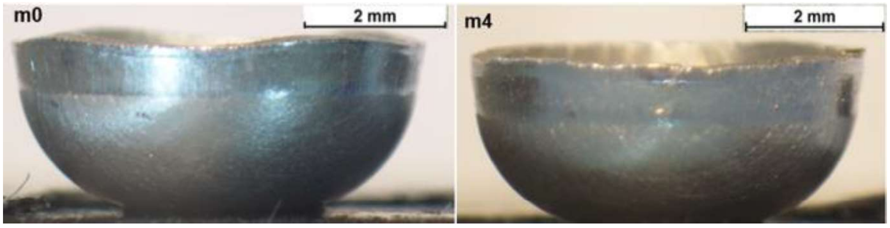

Fig. 22 Earing observed in a sheet presenting larger anisotropy (a) and reduced (b)

The anisotropic properties of sheet metal can be described using a crystallographic approach, according to which the deformation of a polycrystalline aggregate is dependent on the activation of slip systems specific to certain metals. In order to approximate the response of sheet metal to forming, it is necessary to describe the overall orientation of all the crystallites, which is done based on the orientation distribution of crystals, known as texture. There are various models available that predict the mechanical response of a polycrystal, of which one example is shown Fig. 23. The calculations were performed using the Continuum Mechanics of Textured Polycrystals method to predict the r-value of sheets with ideal orientations. It is apparent that strong anisotropy is observed for such orientations as cube {100}<001>, Goss {110}<001>, copper {112}<111> or S {123}<634>, which commonly occur in rolled sheets [70]. Additionally, those common orientations usually lead to r_m values below 1, which results in sheets prone to thinning.

It has been observed that a significant enhancement of r-value is possible when the grains axis <111> is oriented in parallel to the normal direction (ND) of the sheet. The creation of such a texture is impossible by rolling, yet it has been demonstrated that introducing shearing creates that possibility [71].

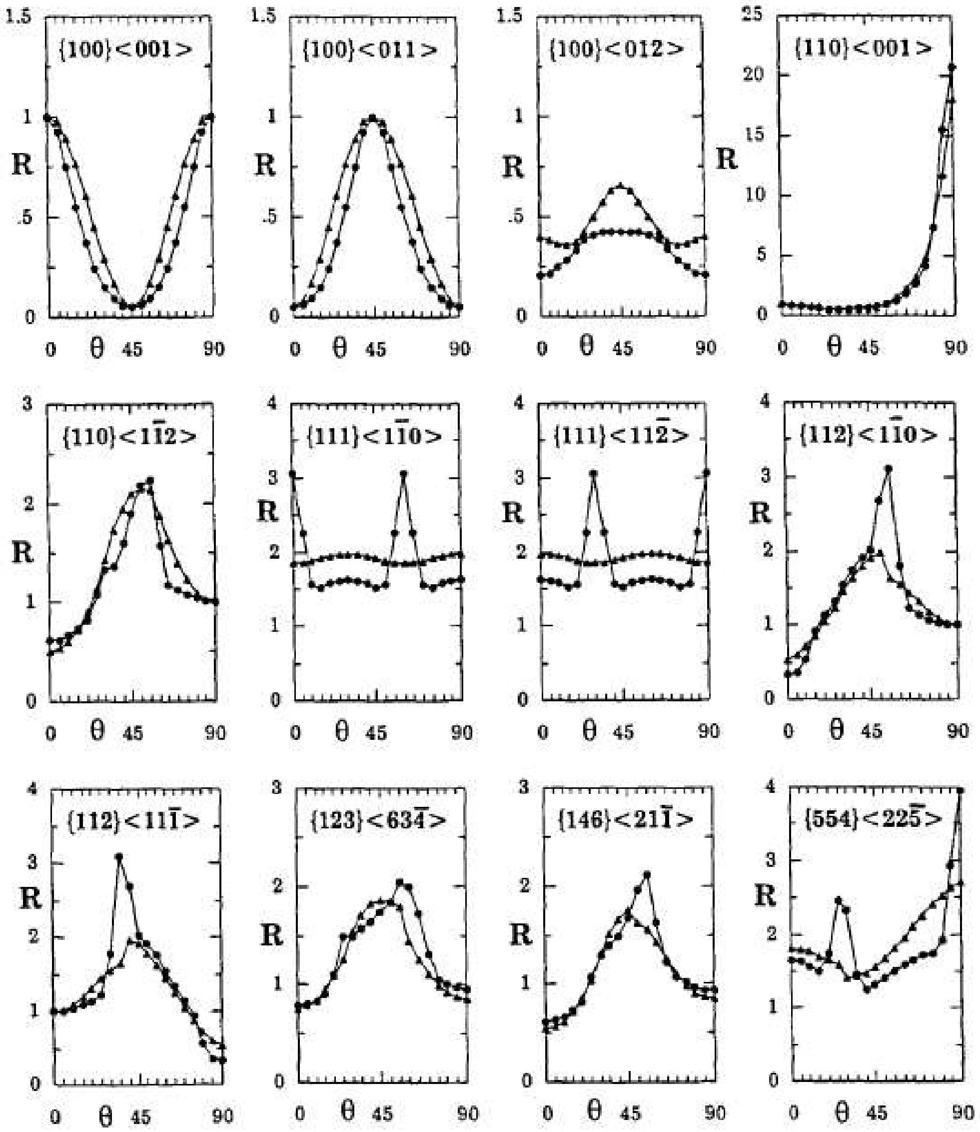

Fig. 23 Comparison of r-value predicted based on Continuum Mechanics of Textured Polycrystals method and experimental results for selected ideal orientations [70]

As crystallographic texture and r-value are dependent on the processing route, the manufacturing method is of key importance to producing a sheet with good drawability. Additionally, to manufacture a sheet with enhanced mechanical properties due to its having a UFG microstructure, it is necessary not only to design a process that makes it possible to induce the creation of a proper crystallographic texture and process a billet in the form of a sheet or plate, but also to refine the grain size. To meet this multitude of requirements, many different processes have been designed, as described above.

The methods of sheet or plate processing presented lead to an anisotropy of mechanical properties, as shown in Fig. 24, which presents normalized yield stress, YS, i.e. YS in a given sheet direction (rolling direction, RD; 45° to RD and 90° to RD) divided by YS in the RD. The majority of methods feature various values of normalized YS, indicating an in-plane anisotropy of mechanical properties. This indicates that there is a lack of methods designed for reducing anisotropy, and that developing such would be beneficial for the commercialization of UFG products.

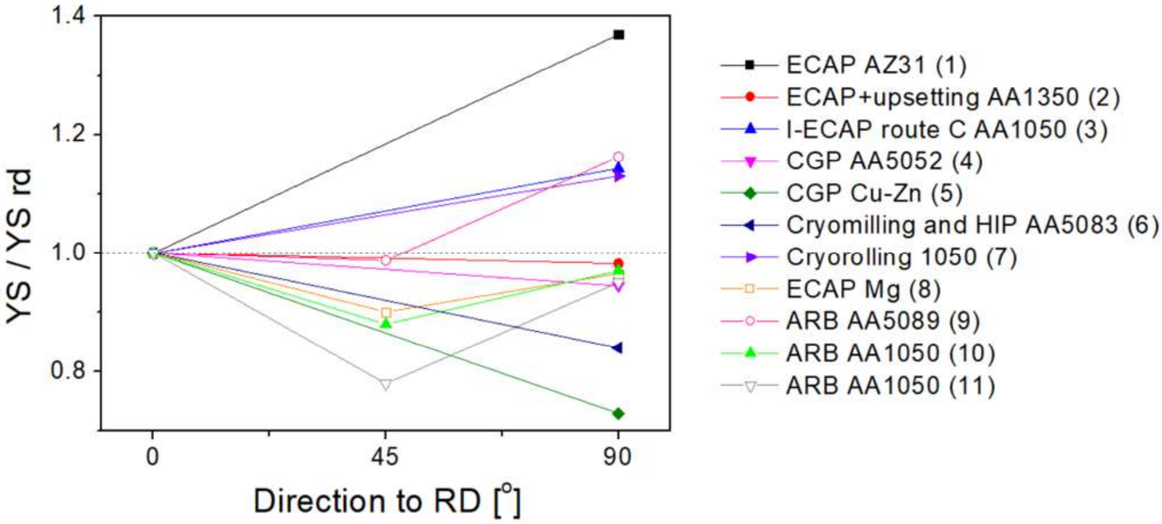

Fig. 24 Normalized YS as a function of direction to rolling direction (RD), (1)- [58]*; (2)-* [72]*; (3)- by courtesy of M. Orłowska; (4)-*[73]*; (5)-*[62] *; (6)-*[74]*; (7)-*[75]*; (8)-*[76]*; (9)-*[77]*; (10)-*[37]*; (11)-*[78]

4. Forming trials of UFG materials

The most common plastic forming processes are deep drawing, stretch drawing and bending, and each is conducted under various strain states, depending on the tools geometry and sheet thickness, so it is impossible to use one single parameter to describe the material's formability or choose one representative process that would cover every possible stress state. Evaluating ductility based on a uniaxial tensile test, the most common test for evaluating mechanical properties, as well as the total or uniform elongation values derived from it, is also misleading, because it represents only one particular stress state. UFG materials in general are prone to strain localization, and quickly fail during uniaxial tensile test, reaching low values of elongation. To represent a broader array of strain states, Forming Limit Diagrams (FLD) are used. These describe the behavior of sheet metal under various loading scenarios, from uniaxial tension to equi-biaxial [79]; these are presented graphically in a coordinate system of major and minor strains. Examples of FLD diagrams for UFG sheets compared with annealed CG material are presented in Fig. 25. A general trend is visible, i.e. a fully developed UFG microstructure makes it possible to achieve major strains lower than or a minor strain similar to the CG counterpart. However, it is worth noting that the difference in not as large as that in the ductility measured in a uniaxial tensile test.

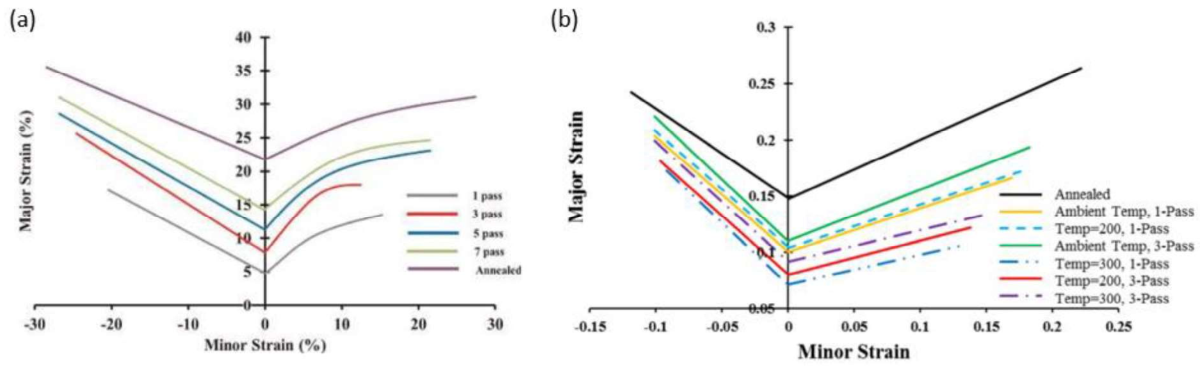

Fig. 25 FLD curves for 1050 processed by ARB [80] *(a) and 5083 processed by ECAR and annealed* [81] *(b)*

The available research concerning the formability of UFG materials mainly focuses on deep drawing, bulging tests or bending, examples of which are presented in Fig. 26. The research shows that it is even possible to acquire better results for UFG materials than for their CG counterparts, e.g. a higher drawing depth was achieved for UFG magnesium alloy in [58], and deep drawing was successfully performed on a UFG sheet, where the trial on CG failed [82]. Tests conducted for UFG copper also proved that a fine graine to increase formability [83,84], since a higher drawing ratio was achieved. This enhancement of formability was explained based on various phenomena. In [85] improved ductility was attributed to profuse microshear banding in biaxial stretching, which led to an increase in the amount of plastic deformation and to the activation of dislocation glide and grain boundary sliding. Another explanation provided in [86] suggests that, during stretching over a punch, unlike in uniaxial tensile tests, the onset of diffuse necking is not possible for some strain paths, and failure starts at the onset of local necking, which can be delayed depending on the geometric constraints. Additionally, it was implied that the UFG aluminum alloy examined was able to accommodate a high strain, which in biaxial stretching does not lead to immediate failure, but increases formability. Other studies indicate that improved formability can be traced back to the high density of grain boundaries in UFG high SFE material, due to which grain boundary-mediated plasticity phenomena such as grain migration and rotation are activated [87].

Despite the small amount of research available on the formability of UFG sheets or plates, the results seem promising. Developing processes capable of producing sheets or plates characterized by advantageous properties such as the ability to delay strain localization or low anisotropy should increase the number of possible applications of UFG sheets or plates.

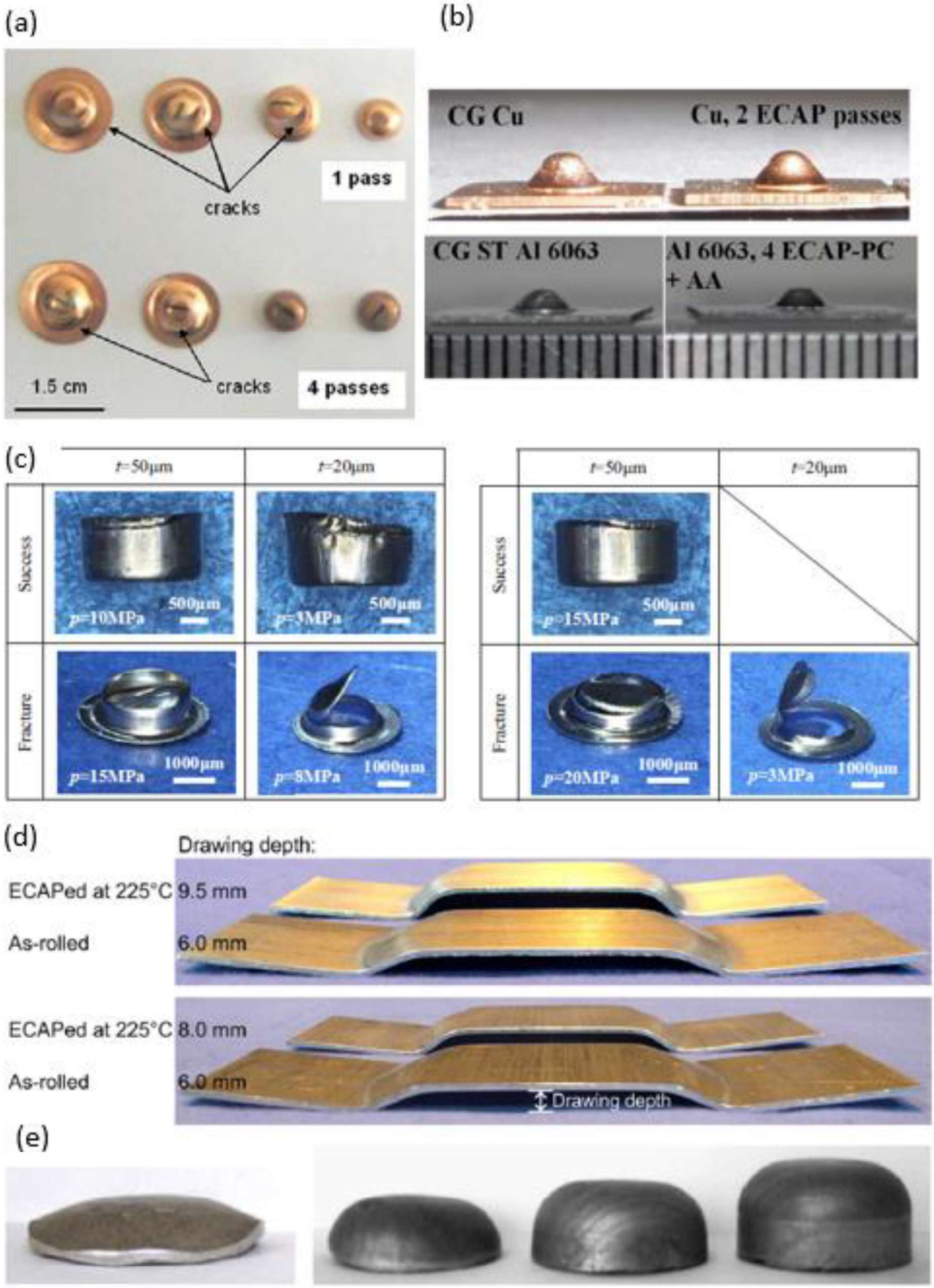

Fig. 26 Examples of forming test of UFG sheets: deep drawing of UFG copper sheet [83] *(a), small punch test of UFG copper and 6063 compared with CG counterparts* [84] *(b), deep drawing of UFG stainless steel (left) and CG steel (right)* [88] *(c), U-channel forming test of CG and UFG Mg alloy* [58] *(d) and deep drawing of CG (left) and UFG (right) AZ31 Mg alloy* [82] *(e)*

5. Objectives, hypothesis and scope of thesis

A substantial enhancement of the mechanical properties, a change in SRS and the activation of various deformation mechanisms are all possible by refining grain size to the ultrafine-grained regime. Developing such a microstructure in bulk materials of significant dimensions can be realized using Severe Plastic Deformation methods, which also allow the production of billets in the form of sheets or plates. Like any plastic deformation method, SPD techniques leave a certain crystallographic and morphologic texture in the processed billet, which influences its properties. Every processing route that leads to a sheet or plate results in a certain degree of anisotropy, which can be defined as various mechanical behavior dependent on the testing direction, usually designated with respect to processing direction. Such anisotropy can result in various faults in the objects formed, such as earing and excessive thinning in certain directions. Thus, the objective of the thesis is to **manufacture ultrafine-grained aluminum alloys plates characterized by low anisotropy and improved ductility, and to subject them to forming tests.**

UFG materials are characterized by limited ductility since, in uniaxial tensile tests, they often achieve only a few percent of total elongation. However, they display a strong tendency towards strain localization, which is particularly detrimental for UFG materials, causing an inability to accumulate plastic strain. Therefore, in order to evaluate the formability of UFG materials, it is necessary to perform trials under complex strain states that delay strain localization and material failure. An enhancement of ductility is also possible using approaches such as adjusting the microstructure by lowering the stacking fault energy, introducing a significant fraction of high angle grain boundaries, lowering the dislocation density, or changing the external conditions of the deformation processes, i.e. strain rate and temperature. Therefore, the hypothesis of this dissertation is: **an isotropic microstructure of ultrafine-grained plates together with proper external conditions, i.e. strain rate and temperature, will make it possible to increase ductility and obtain a formability at least equivalent to a material of conventional grain size.**

In order to accomplish this objective and prove this hypothesis, the research tasks are planned as set out in Fig. 27.

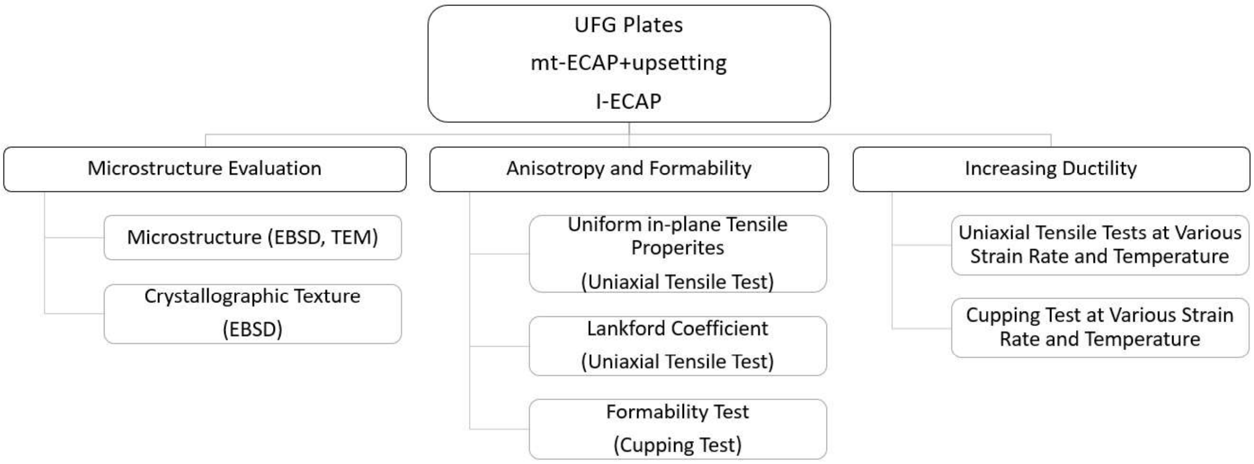

Fig. 27 Research tasks

6. Comments on the research results

The thesis is based on the following 6 publications:

1. M. Ciemiorek, W. Chromiński, L. Olejnik, M. Lewandowska, Evaluation of mechanical properties and anisotropy of ultra-fine grained 1050 aluminum sheets produced by incremental ECAP, Materials and Design, 130 (2017) 392–402, doi: 10.1016/j.matdes.2017.05.069
2. M. Ciemiorek, M. Lewandowska, L. Olejnik, Microstructure, tensile properties and formability of ultrafine-grained Al–Mn square plates processed by Incremental ECAP, Materials and Design, 196 (2020) 109125, doi: 10.1016/j.matdes.2020.109125
3. M. Ciemiorek, W. Chromiński, C. Jasiński, M. Lewandowska, Microstructural changes and formability of Al-Mg ultrafine-grained aluminum plates processed by multi-turn ECAP and upsetting, Materials Science and Engineering A, 831 (2022) 142202, doi: 10.1016/j.msea.2021.142202
4. Ductility and Formability of Al-Mn ultrafine grained plates processed by Incremental Equal Channel Angular Pressing
5. Ductility and formability of ultrafine-grained 5754 aluminium alloy under various strain rates and temperature
6. M. Ciemiorek, P. Bartkowski, W. Chromiński, L. Olejnik, M. Lewandowska, Forming Ability of Ultrafine-Grained Aluminum Plates Processed by Incremental Equal Channel Angular Pressing, Advanced Engineering Materials (2019), 1900473, doi: 10.1002/adem.201900473

The publications contain a detailed description and discussion of the obtained results; nonetheless, brief comments on the material processing and results is provided below.

6.1 Processing Methods

The first part of the research was to manufacture UFG plates suitable for further processing using metal working techniques, i.e. deep drawing, stamping or bending, aimed at increasing the possible applications of UFG materials. Based on the preliminary studies carried out within the author's master thesis and the technologies available at Warsaw University of Technology, two methods were used: I-ECAP and hybrid processing, based on mt-ECAP and upsetting. These were applied to two commercially available alloys, i.e. 3003 and 5754. Each alloy was processed by one of the available techniques due to technological limitations, namely machine capacity and the ductility of the chosen alloys. Additionally, research on the microstructure and mechanical properties of technically pure 1050 processed by I-ECAP, as a model material, is presented.

Each SPD operation was designed so as to acquire a microstructure that was homogeneous and as close as possible to isotropic, although these were determined by the specific characteristics of the process. The aim was to create a microstructure that would result in the plates having both isotropic mechanical properties and sufficient formability.

6.1.1 Incremental Equal Channel Angular Pressing

Incremental Equal Channel Angular Pressing is a method developed by Rosochowski and Olejnik [89]. It is continuous processing technique suitable for producing UFG metals. The method is based on conventional ECAP, and the similarities between the two process are shown in Fig. 28. In the conventional process, a billet is pushed through a rigid curved die. As the material crosses through the curve, marked in the figure by a dashed line, plastic deformation caused by simple shear occurs. During one pass, an equivalent strain of ε=1.15 is imposed. In order to obtain a UFG microstructure, higher strains are required, so the billet is usually subjected to several passes and is often rotated between them. The basic geometry of I-ECAP tools is basically similar, with the exception that the die parts are separated and can be moved; they are shown in Fig. 28 b. The billet is inserted into the channel, defined by the parts marked by A and B, and is then deformed by the reciprocating die C. The feeding stroke in one increment is expressed as distance a. During the feeding of the billet, the material has no contact with the tools, as clamp B can be moved to enlarge the cross section of the inlet channel, and so the force of friction is almost eliminated in this stage of the process. Shear deformation occurs when the reciprocating die moves towards the billet, which at this point is rigidly fixed in the inlet channel. Those two steps of feeding and deformation of the billet are repeated, which makes it possible to process long billets whose length is not limited by the die geometry, as is often observed in many SPD processes.

Stroke value is crucial in I-ECAP, as the speed of the process is dependent on this parameter, as well as on the homogeneity of the microstructure. If this value is not excessive, consecutive shear zones overlap each other, creating a homogenous microstructure. To determine the optimum stroke value, FEM simulations were performed (Fig. 28 c); they indicated that a stroke value equal to 10% of billet thickness results in a strain distribution similar to that of conventional ECAP processing, and provides a homogenous microstructure [90].

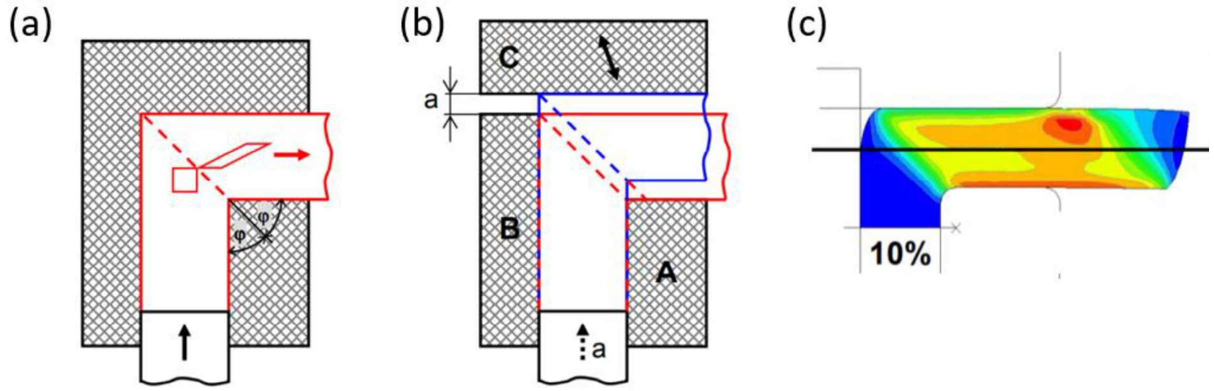

Fig. 28 Conventional (a) and Incremental (b) ECAP die, FEM simulation of I-ECAP processing with stroke value equal to 10% of billet thickness [90]

Several types of I-ECAP processing have been developed, such as double billet I-ECAP or I-ECAP suited for sheet processing. In double billet I-ECAP, the clamp is replaced by another billet, as shown in Fig. 29 a. In such a symmetrical operation, the two billets are processed simultaneously, which increases efficiency. Sheet or plate processing is possible using I-ECAP due to a significant reduction in friction thanks to the separation of the feeding and deforming stages and the fact that the billet is subjected to shearing gradually. Processing plates in conventional ECAP devices is either impossible, since the friction force is much higher due to the larger surface of the billet, or requires extensive lubrication. In order to process plates, it is necessary to modify the geometry of the tools so they fit the shape of a flat object, as shown in Fig. 29 b.

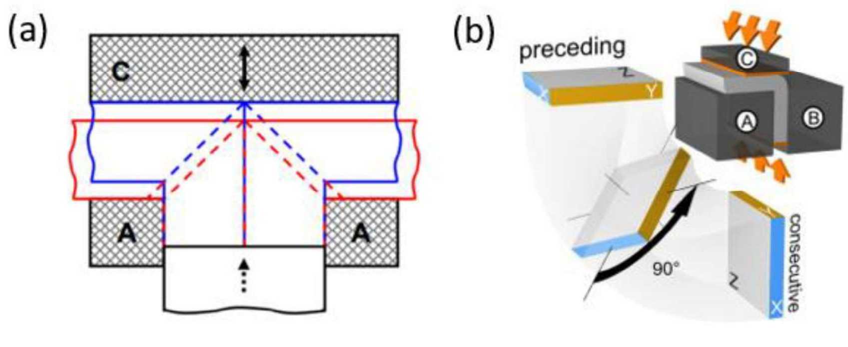

Fig. 29 Double billet I-ECAP [90] *(a) and I-ECAP adjusted to plates processing* [91] *(b)*

In I-ECAP operations, as in any conventional ECAP process, the billet can be rotated between consecutive passes. Depending on the plate geometry, various rotation scenarios can be implemented, e.g. rectangular plates can be rotated by 180° around the axis perpendicular to the sheet plane to fit the inlet channel, since one side is longer than the other. Square plates offer more possibilities for, being geometrically symmetrical, they can be rotated by 90°. Such a processing route imposes shear strain in four perpendicular directions.

6.1.2 Hybrid Processing: multi-turn Equal Channel Angular Pressing followed by Upsetting

Hybrid processing composed of two steps, i.e. multi-turn ECAP (mt-ECAP) and upsetting, a standard metal working operation, was proposed in [92]. An ECAP die with two curves was introduced in [93], and later the technique was developed by Rosochowski and Olejnik [94,95], who named it mt-ECAP. A diagram of a die with such a geometry is shown in Fig. 30 a. The main purpose of using two turns is to impose a larger strain in a single pass, thereby enhancing the efficiency of the process. When a billet passes through 2-turn ECAP, it first undergoes primary yielding, then unloading, in the middle of horizontal part of the channel, and is finally subjected to secondary yielding at the second curve, as illustrated in Fig. 30 b. It has been shown that such a process is more efficient at creating ultra-fine grains and high fraction of HAGBs, since shear occurs at two zones during a single pass [96].

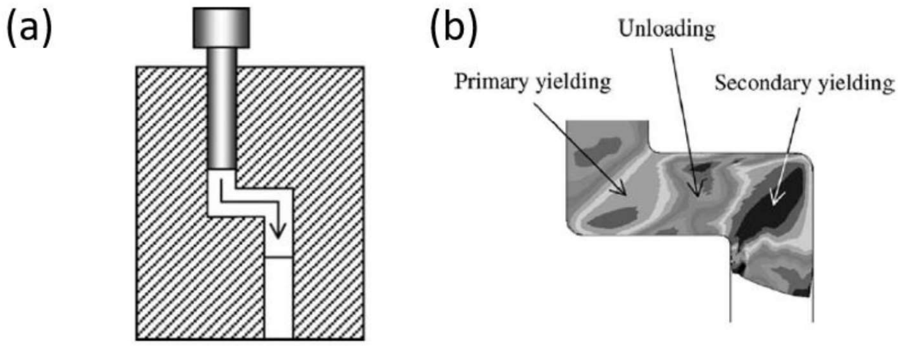

Fig. 30 Diagram of 2-turn ECAP (a) and distribution of equivalent stress (b) [94]

The upsetting process is a metal forming operation in which a compressive force is applied through dies [97], usually presses and flat dies. During the process, the billet is slowly squeezed, and its length is increased at the expense of its thickness, as shown in Fig. 31. Apart from the change in geometry, the process imposes strain, which causes microstructural changes in the billet.

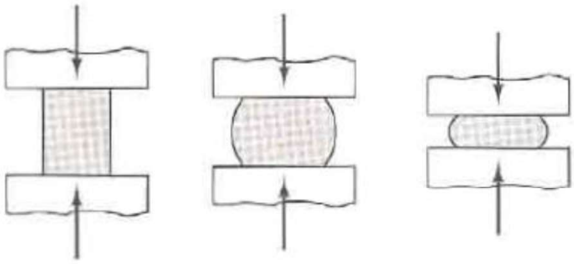

Fig. 31 Stages of upsetting process [97]

A combination of mt-ECAP and upsetting is a process designed for the efficient production of plates characterized by a UFG microstructure. mt-ECAP is efficient in refining grain size with fewer passes than conventional ECAP, whereas upsetting is a fast and simple operation for changing the geometry of the billet into a plate. Upsetting additionally results in higher accumulated strain, which greatly enhances the mechanical properties of the processed material.

6.1.3 Processing Routes of the Samples

Processing using SPD methods was performed courtesy of the group of Professor Lech Olejnik at the Faculty of Mechanical and Industrial Engineering, Warsaw University of Technology[1]. The upsetting process was performed at Research Network Łukasiewicz - Metal Forming Institute[2]. Three types of aluminium alloys were investigated: (1) technically pure aluminium 1050 as a model material, (2) 3003 representing a low strength alloy with Mn as a major alloying element, (3) 5754 representing a higher strength Al-Mg group of alloys. All the alloys were single phase, making grain refinement and plastic deformation the major strengthening mechanisms. Additionally, all of those materials are used commercially in the sheet forming industry. The materials for the investigation were supplied by Aluminium Konin-Impexmetal SA Granges Konin S.A[3].

The selection of alloys and processing techniques was influenced by the technological limitations of the available equipment. Alloy 5754 is characterized by high mechanical strength, and so it was necessary to conduct grain refinement at elevated temperature. The mt-ECAP tools were equipped with a suitable heating system, which made it possible to successfully process the billets. The I-ECAP processing equipment can process material at room temperature, so it was used for refining the microstructure in alloy 3003 and technically pure

[1] Website of prof. Lech Olejnik's group: http://lolejnik.eta.pl, access date: 13.04.2021
[2] Website of Research Network Łukasiewicz - Metal Forming Institute: https://inop.poznan.pl, access date: 13.04.2021
[3] Website of the company: http://impexmetal.com.pl, access date: 13.04.2021

aluminium 1050, both of which have lower mechanical properties. The processing routes of the metals were designed so as to create a homogenous microstructure and isotropic mechanical properties. Details on the processing parameters such as route, temperatureand equivalent plastic strain as well as billet geometry and dimensions are summarized in Tab. 1.

Tab. 1 Processing parameters of the investigated samples

Process	**I-ECAP**	**Hybrid Processing**
Alloy	1050 Al 99.5 – 0.5 others 3003 Al-(1÷1.5)Mn-0.7Fe-(0.05÷0.2)Cu-0.1Zn	5754 Al-(2.6÷3.6)Mg-0.4Si-0.4Fe-0.1Cu-0.5Mn-0.2Zn-0.3Cr
Billet geometry	Square plate 62x62x3 mm	mt-ECAP: Rod with square cross section 8x8 mm, length 40 mm mt-ECAP and upsetting: Oval plate 40 x 20 mm, 1 mm thickness
Processing route and conditions	I-ECAP passes with counterclockwise rotation by 90° around axis perpendicular to plate’s plane, room temperature 1050: 8 passes 3003: 4 passes	mt-ECAP: 4 passes, 2-turn die with 110° angle with rotations by 90° around rod axis, 150°C, extruded between passes upsetting: thickness reduction form 8 mm to 1 mm, room temperature
Strain after single operation	1.15	extrusion: 1.7 mt-ECAP and extrusion: 1.75 upsetting: 2.3
Equivalent plastic strain ε	1050: 9.6 3003: 4.6	Extrusion: 1.7 mt-ECAP and extrusion: 7 upsetting: 2.3 Total ε= 11

1050 and 3003 square plates of 62 mm side length and 3 mm thickness were processed using the I-ECAP technique. The plates were in condition H24 and 0/H111 for 1050 and 3003, respectively. The operation was conducted at RT, the billet was subjected to 8 and 4 passes with rotations around 90° around the Z axis, for 1050 and 3003, as presented in Fig. 32 a. Fig. 32 b shows 1050 processed plates, which are flat and characterized by a smooth surface. As the plate was processed 4n times, it was deformed symmetrically in four perpendicular directions, due to which it was possible to acquire equiaxed grains. The plastic strain imposed during one pass was 1.15, which resulted in a total accumulated strain of ε=9.2 and 4.6, for 1050 and 3003.

Fig. 32 Diagram of plate rotations (a) and processed plates (b)

The initial condition of 5754 aluminum alloy was a hot extruded rod of 20 mm diameter, which was cold extruded to a billet of sides 8 mm and length 40 mm, for further mt-ECAP processing, which imposed a strain of 1.7. The billet was also machined using the electro erosion technique to acquire the same geometry for the subsequent upsetting. The cold extruded rods were subjected to a hybrid processing composed of mt-ECAP followed by further extrusion and upsetting. mt-ECAP was performed at an elevated temperature of 150°C using a die with 2 angles intersecting at 110°, as shown in Fig. 33 a. Each billet was pressed 4 times, with a counterclockwise rotation of 90° around the rod axis. Additionally, after each mt-ECAP pass, the rod was subjected to extrusion due to the technological conditions. The aim of the extrusion was to shape the billet to perfectly fit the mt-ECAP channel. After 4 passes through the mt-ECAP and extrusion device, the billet was compressed in a single upsetting operation, which resulted in a plate of 1 mm thickness. Processed rods and a plate are shown in Fig. 33 b and c. The extrusion and upsetting was conducted at room temperature. Total plastic strain caused by the hybrid processing was 11.

Fig. 33 Die geometry with channel curved at 110 [98] *(a), mt-ECAP processed rods (b), UFG plate obtained by hybrid processing (c)*

6.2 Microstructure, mechanical properties and anisotropy of plates

This part of the thesis has been addressed in three papers. Two of them were about the I-ECAP processing of technically pure aluminum 1050 (‘Evaluation of mechanical properties and anisotropy of ultra-fine grained 1050 aluminum sheets produced by incremental ECAP,’ Appendix 1), and 3003 alloy (‘Microstructure, tensile properties and formability of ultrafine-grained Al–Mn square plates processed by Incremental ECAP,’ Appendix 2). The third paper concerned the effect of hybrid processing of 5754 aluminum plates (‘Microstructural changes and formability of Al-Mg ultrafine-grained aluminum plates processed by multi-turn ECAP and upsetting,’ Appendix 3).

6.2.1 Microstructural Characterization

The microstructural changes were evaluated based on the data obtained from the three perpendicular planes, i.e. X, Y and Z, which are presented schematically in Fig. 32 a. Detailed values of the equivalent grain size and aspect ratio are gathered in Tab. 2. -ECAP processing is extremely efficient in terms of creating an equiaxed isotropic microstructure with a high fraction of HAGBs. The equivalent grain size was ~500 nm for 1050 and 3003 aluminum, and the mean aspect ratios for all planes were 1.56 and 1.9, respectively. The acquired fraction of HAGBs was higher than in conventional ECAP processes, reaching 65, 70 and 63% for the X, Y and Z planes, respectively, for alloy 3003. Those microstructural features are attributed to the processing route which, through rotations and symmetricity, activated various shearing planes, leading to equiaxed grains and a high fraction of HAGBs. The microstructure of the plates after hybrid processing has different characteristics: the grains are strongly elongated in the Y direction and significantly larger in the Z plane than the X plane, which is reflected by the aspect ratio value of more than 4 for the Y plane and 2 for the Z plane. This is a consequence of compression having been imposed in one direction during the upsetting process. Nonetheless, a significant grain size reduction was achieved, i.e. ~400 nm, as well as a high fraction of HAGBs, especially in the X and Y planes, reaching 76% and 85%, respectively. The fraction of HAGBs in the Z plane was lower, at 48%.

Tab. 2 Microstructural parameters of plates processed by I-ECAP and hybrid method

		X plane	Y plane	Z plane
I-ECAP 1050	d_{LAGBs} [µm]	0.47	0.45	0.43
	AR_{LAGBs}	1.57	1.59	1.56
I-ECAP 3003	d_{LAGBs} [µm]	0.44	0.51	0.61
	AR_{LAGBs}	2.14	1.78	1.84
Hybrid Processing 5754	d_{LAGBs} [µm]	0.34	0.4	0.47
	AR_{LAGBs}	1.63	4.37	2.08

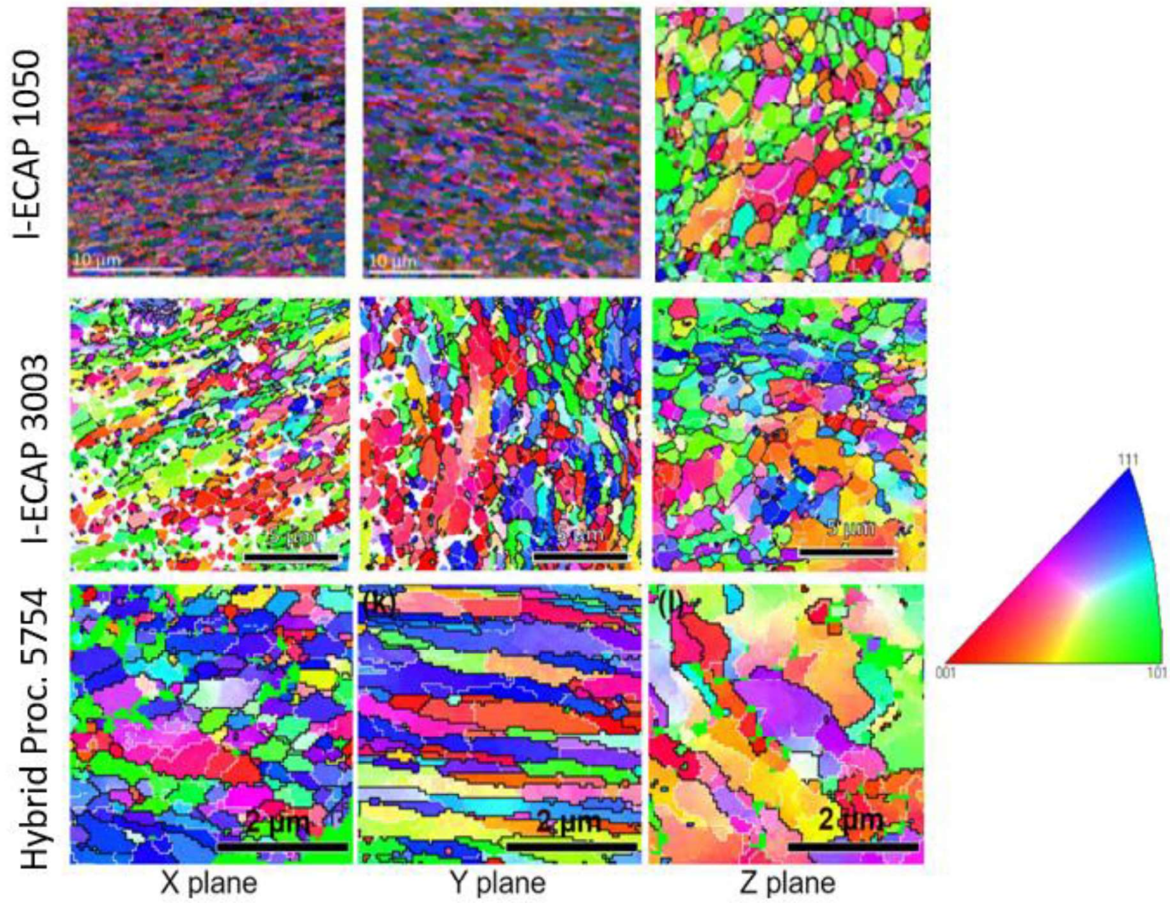

Fig. 34 EBSD maps of X, Y and Z planes of technically pure aluminum and 3003 and 5754 alloys processed by I-ECAP and hybrid method; note the various scale bars, microstructure of 1050 plane X and Y by courtesy of W. Chromiński

6.2.2 Mechanical Properties of UFG Plates

Tensile curves of UFG 1050, 3003 and 5754 plates and for the as-received materials are shown in Fig. 35. From each plate, tensile coupons were cut at an angle of 0, 45 and 90° to evaluate the anisotropy of the mechanical properties those planes. Since the initial condition of 1050 alloy was H24, i.e. work hardened and only partially annealed, the shape of the tensile curve is similar to that after I-ECAP processing. A different shape is typical for CG aluminum in recrystallized condition, as for 3003 and 5754 alloy, since in that case a gradual increase in stress is observed and maximum stress is reached at larger elongations, which indicates an SH region. The tensile curves for UFG aluminum alloys have a characteristic shape, as maximum stress is quickly reached and a sharp decrease in stress occurs, resulting in small elongations. The curves representing 1050 aluminum after I-ECAP show a more moderate decrease in stress, due to the higher ductility of the pure metal. For SPD processed alloys, mechanical strength increased more than two-fold, and was accompanied by a reduction in ductility by approximately five times. The I-ECAP processing of 1050 aluminum, which imposed a total equivalent strain of 9.2, resulted in an increase in UTS of 39% and YS of 26%. For 3003 alloy, where the total equivalent strain was 4.6, for the coupons cut in parallel to the final extrusion direction the parameters describing mechanical strength, i.e. YS and UTS, were 218 and 237 MPa, respectively, while for the initial material those values were 76 and 110 MPa. Total elongation was reduced to from the initial value of 19% to 4%. For 5754 alloy after hybrid processing, where the total equivalent strain was 11, the increase in mechanical strength was even more considerable: the YS and UTS were 435 and 452 MPa, while for the initial condition they were 144 and 204 MPa. Total elongation was reduced from 24 to 5%.

The plane anisotropy of the mechanical properties of I-ECAP processed samples was negligible, as differences in the YS and UTS values calculated for various testing directions were below 1%. Plates processed by mt-ECAP and upsetting featured a difference of 1% in YS and 2.5% in UTS, which are also insignificant. Based on the tensile curves, it can be concluded that UFG plates were characterized by close to isotropic mechanical properties in plane.

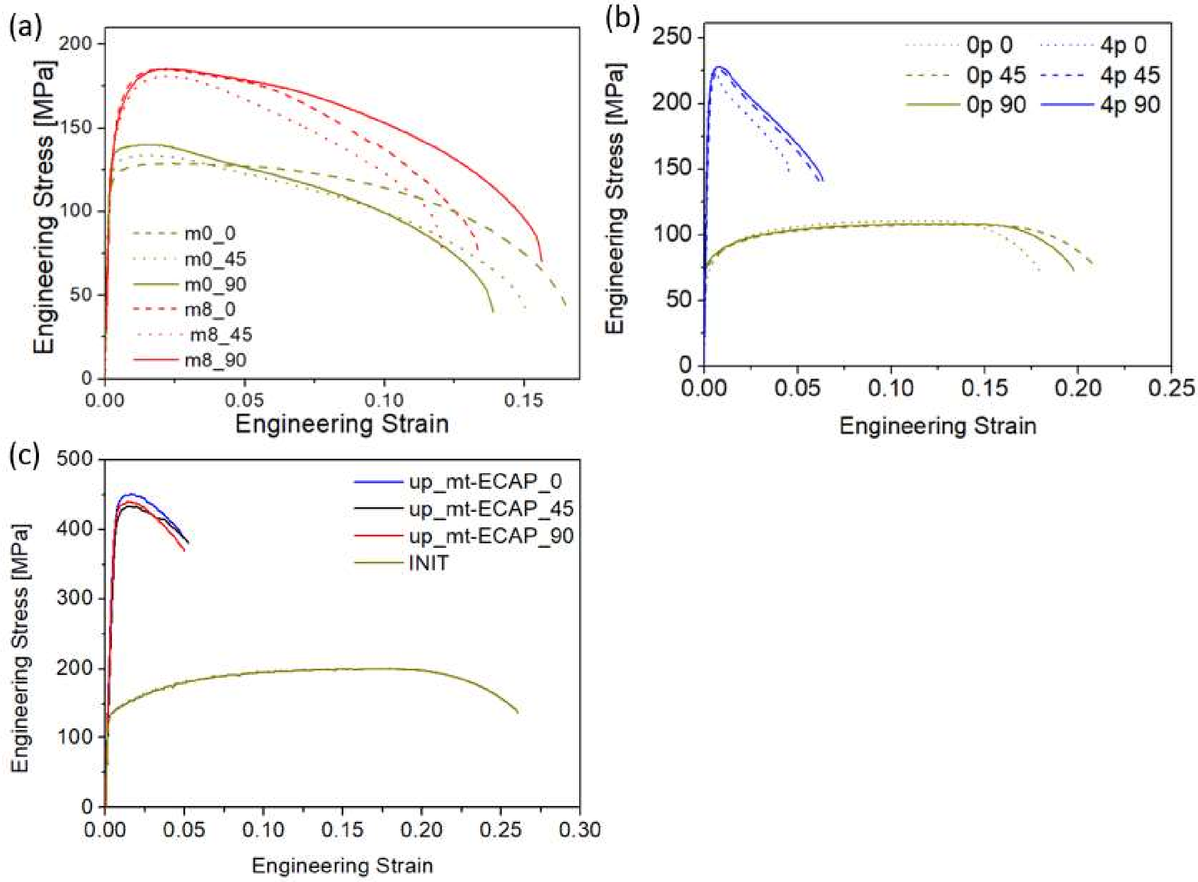

Fig. 35 Engineering stress - engineering strain of plates processed by I-ECAP, technically pure aluminium 1050 (a) and 3003 alloy (b) and hybrid method (c) cut at an angle of 0, 45 and 90° to last extrusion direction and initial material

6.2.3 Anisotropy

The values of mean r-value r_m, planar anisotropy Δr for the plates processed by I-ECAP and hybrid processing are shown in Fig. 36, where they are compared with those of plates obtained by other grain refinement methods. The mean r-values r_m for I-ECAP processed aluminum 1050 and 3003 and hybrid processing were 0.64, 0.59 and 0.5, respectively, which is typical for FCC materials. I-ECAP processing is particularly effective at reducing planar anisotropy, as Δr is equal to -0.04, while the mean r-value remains at a level similar to other methods. 5754 alloy plates after hybrid processing featured values of r_m and Δr, equal to 0.25, which are also comparable to other methods. However, it should be noted that this material was subjected to a particularly high strain, which resulted in a UTS of more than 440 MPa. Therefore, 5754 plates processed by mt-ECAP and upsetting feature parameters describing anisotropy similar to that achieved in other methods, but a substantially higher UTS. The values obtained indicate that both plates have the qualities of a product suitable for further processing by means of metal forming methods.

Anisotropy is related to the crystallographic orientation of grains, and so changes in it should be examined based on changes in texture. For the I-ECAP processed plates, it was concluded that the reduction in Δr from the initial state was caused by an overall decrease in texture intensity. The texture of the plates processed by the hybrid method was also proven to have become less intense, as the volume fraction of components contributing to a high r_m value, which also results in pronounced Δr, was diminished. This resulted in a relatively small Δr value, but also in a lower r_m. Apart from crystallographic texture, grain shape also contributes to anisotropy to some extent. The closer to equiaxed, the higher the r_m and the lower the Δr values. This trend was observed in the materials investigated, as discussed in detail in the three aforementioned papers (Appendices 1, 2 and 3).

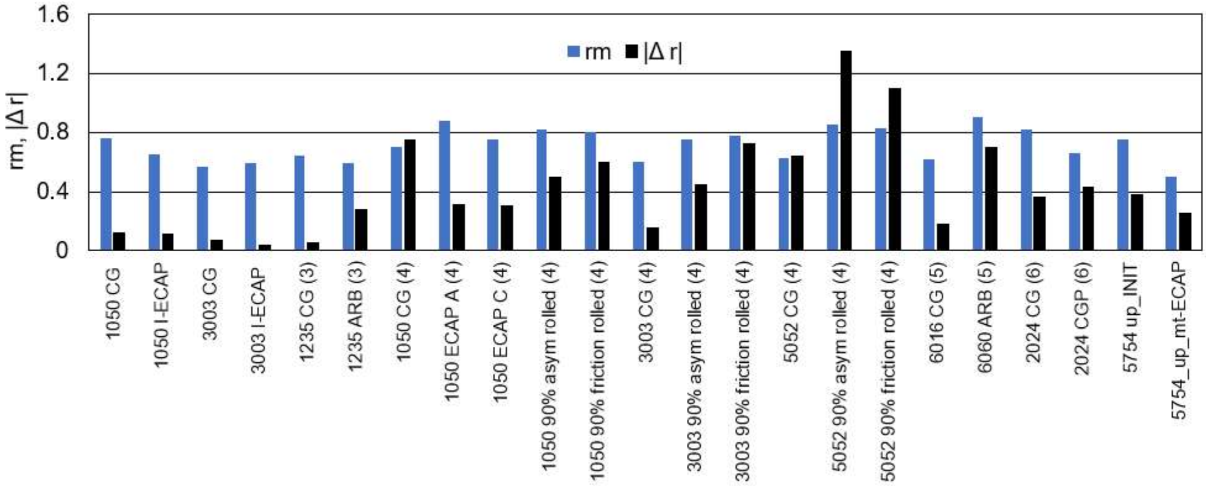

Fig. 36 Mean r-value and planar anisotropy for I-ECAP processed plates of 1050 and 3003, 5754 processed by Hybrid Method and others

6.3 Attempts to improve ductility and formability

The second part of the thesis focused on possible improvements in the formability of the manufactured plates by adjusting the conditions of the deformation processes. SH and SRS were investigated in various conditions of uniaxial tensile tests, i.e. temperature and strain rate. This investigation is described for 3003 alloy in the article entitled 'Ductility, Strain Hardening, Strain Rate Sensitivity and Formability of Al-Mn Ultrafine-Grained Plates,' Appendix 4, and for 5754 in 'Ductility and formability of ultrafine-grained 5754 aluminum alloy under various strain rates and temperature', Appendix 5. The major findings in this context are briefly summarized in two consecutive sections, 7.3.1 and 7.3.2, which concern the influence of temperature and strain rate on ductility (7.3.1) and the influence of temperature on the forming ability (7.3.2) of 3003 and 5754 plates

In addition to extensively investigating the formability of the I-ECAP processed plates, since that technique gives the best results in terms of isotropy, a detailed study based on cupping trials was performed on technically pure aluminum; itis described in ‘Forming Ability of Ultrafine-Grained Aluminum Plates Processed by Incremental Equal Channel Angular Pressing’, Appendix 6. The most important results are briefly described in section 7.3.3, where the effect of isotropy on forming ability is discussed.

6.3.1 Influence of Temperature and Strain Rate on Ductility

The influence of testing temperature on the mechanical properties of the plates is shown in Fig. 37. Fig. 37 a and b show engineering stress-engineering strain curves for CG and UFG metals, obtained at room temperature (RT) and at elevated temperatures (ET), which were 150°C and 200°C for alloys 3003 and 5754, respectively. The temperature was selected so as not to obtain excessive grain growth. Fig. 37 c and d illustrates the corresponding curves of the SH rate, θ.

Conducting tensile tests at ET led to an increase in total elongation for both the CG and UFG alloys, although the enhancement was more pronounced in the UFG samples. The data presented also indicate that, for UFG 3003 alloy, the increase in ductility was two-fold, while for UFG 5754 it was more than four-fold, and for the CG counterparts those values were 16 and 40%, respectively. Additionally, the flow stress was much lower at ET than at RT. Besides changes in total elongation, the shape of the tensile curves changed as well, and so did the SH rate. For the CG metals, the initial θ was higher at RT than at ET, and was characterized by a larger range of normalized stress, which corresponds to a larger uniform elongation (compared with tensile test curves). For alloy 3003 (CG) at RT, θ moderately decreased with increasing normalized stress until an ultimate tensile strength was reached. The θ curve at ET has a similar shape, but strain localized at a lower value than at RT. The CG 5754 alloy behaved similarly, except that the initial θ was higher. For the UFG materials tested at RT, the initial θ was substantially higher and its decrease steeper than for CG; the tensile curves quickly reach a maximum stress, shortly after which strain localization and fracture occurs. However, when tested at ET, θ decreased less rapidly than at RT, and ductility expressed as both uniform and total elongation, was enhanced.

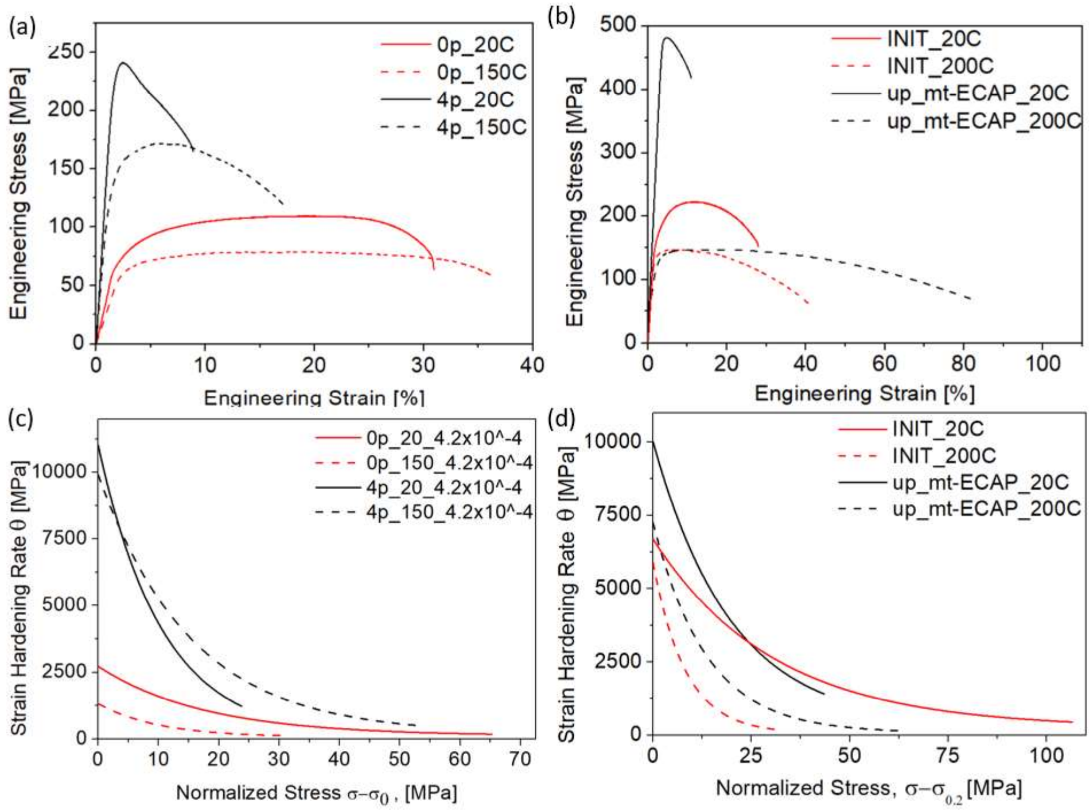

Fig. 37 Tensile curves for alloy 3003 initial material and I-ECAP processed plates (a) and 5754 alloy hybrid processed (b) and corresponding strain hardenig rate curves as a function of normalized stress (c) and (d), respectively

The increase in elongation at ET was attributed to enhanced SRS which, according to Hart's criterion, is one of the main factors contributing to ductility. Values of the m parameter as a function of temperature are shown in Fig. 38 a and b. For 3003 alloy, the m value increased from close to 0 to 0.13 at 150°C, and for 5754 to 0.21 at 200°C. The increase in the m parameter was caused by a reduction in the apparent activation volume, caused by grain refinement to the ultrafine regime, whose values are shown in Fig. 38 c and d. For the selected temperatures, the apparent activation volume was $24b^3$ for 3003 alloy and $13b^3$ for 5754. By reducing the apparent activation volume, mechanisms of plastic deformation other than those available for CG materials were activated. The activation volumes observed for diffusion-based processes are usually below $10b^3$, and so the values obtained for the UFG materials suggest that the deformation process was a combination of emission and propagation of dislocations along with thermally induced processes, which resulted in enhanced ductility.

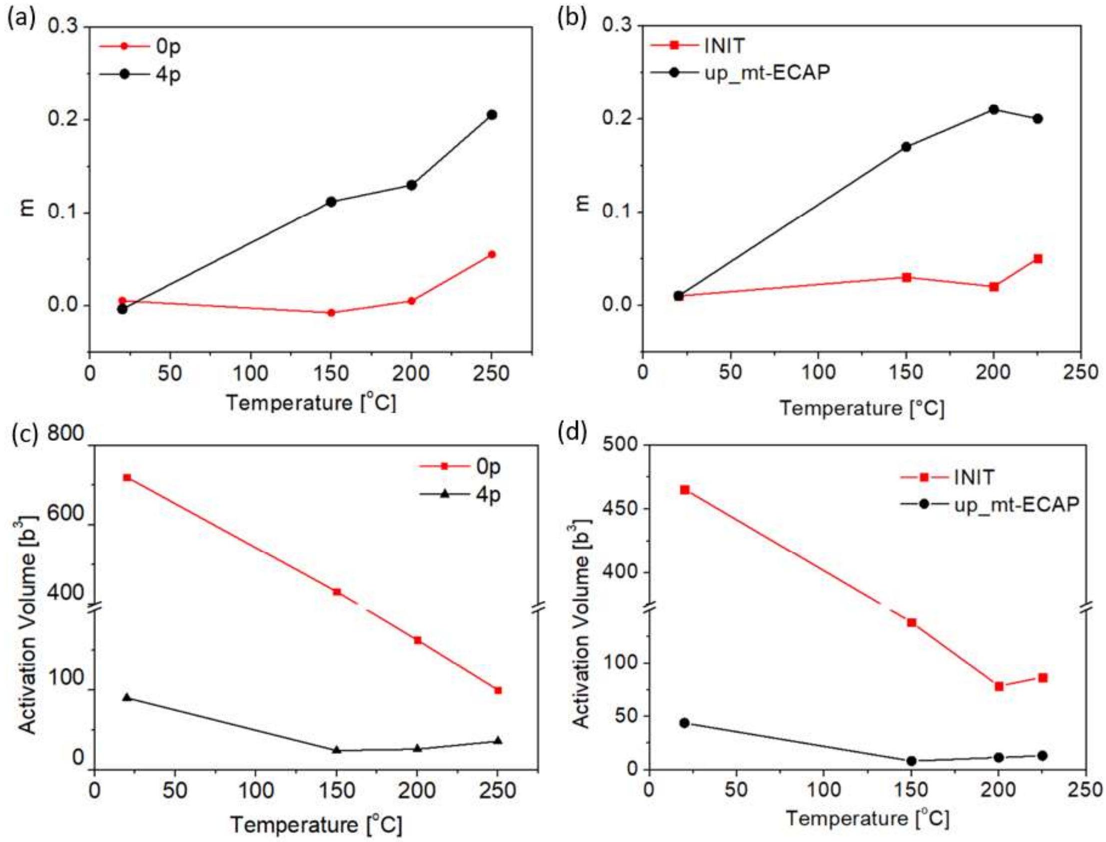

Fig. 38 SRS parameter m of alloy 3003 (a) and 5754 (b) and activation volume (c) and (d), respectively, as a function of temperature

6.3.2 Influence of temperature on forming ability

Fig. 39 a shows the results of the cupping test for 3003 and 5754 alloy plates at RT, 150 and 200°C, expressed as the height of the cup. With increasing temperature, the cup height increased for both alloys. That increase was especially pronounced for 5754 alloy at 200°C, which is consistent with the tensile test results, as at this temperature the total elongation was exceptionally exceptionally large. Fig. 39 b shows the increase in cup or part height, formed at RT and at 200°C, of the plates investigated (marked as I-ECAP, up_mt-ECAP and up_INIT) and of plates of CG 5754 alloy (marked as 6-11, based on literature data). The revealed increase is noticeably larger for hybrid-processed 5754 alloy, and is equal to 99%. The increase in cup height obtained for 3003 alloy, 62%, is also larger than most of the data presented here. This leads to the conclusion that, in UFG materials, by conducting a forming operation at ET it is possible to activate additional deformation mechanisms and achieve enhanced formability.

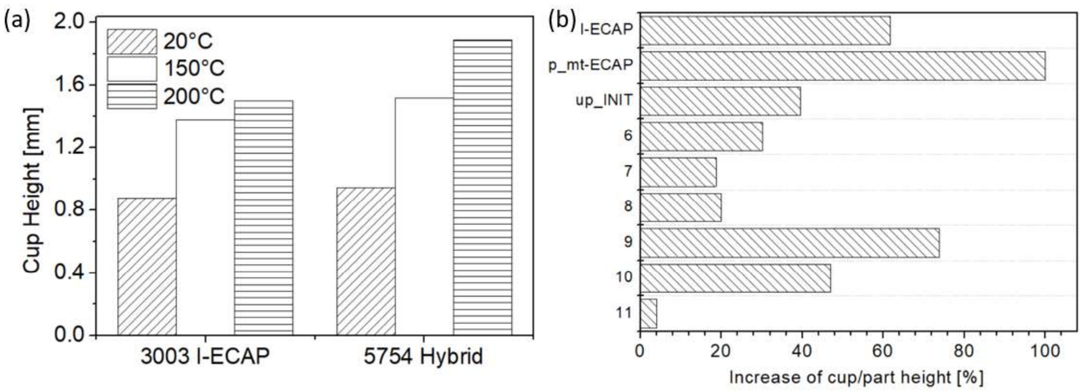

Fig. 39 Cup height of UFG 3003 and 5754 alloys investigated in the thesis, after deformation at various temperature (a) and increase in cup height formed at elevated temperature of 200°C in comparison with RT, with the exception that I-ECAPed 3003 alloy was formed at 150°C, for alloys investigated in the thesis and literature data on 5754 (samples 6-11) (b) [Appendix 3]

6.3.3 Effect of Isotropy of the Plates on forming ability

The above cupping test results do not evaluate the effect of isotropy on formability, and so cup drawing tests were performed on a model material, i.e. technically pure aluminium plates processed by I-ECAP. Fig. 40a, shows the results of cup drawing for CG and UFG aluminum, further referred to as m0 and m8; they show that it was possible to successfully draw cups from ø8 and ø9 mm blanks, regardless of grain size. Fig. 40 b shows a side view of cup drawn from blanks ø8 and ø9. The upper edge of the m0 cup is uneven, as earing occurred due to in-plane anisotropy, unlike the m8 samples. Those effects are consistent with the parameters describing anisotropy, i.e. initial mean r-value, r_m, and earing tendency, The values of Δr_p for the m0 samples were 0.78 and 0.82, and for the I-ECAP processed plate, m8, they were 0.65 and 0.13.

It can be concluded that reducing in-plane anisotropy of plates results in a mitigation of unwanted effects such as earing. Additionally, in the present case, developing a UFG microstructure, i.e. enhancing the mechanical properties, did not reduce formability in a cup drawing test in comparison with the CG material.

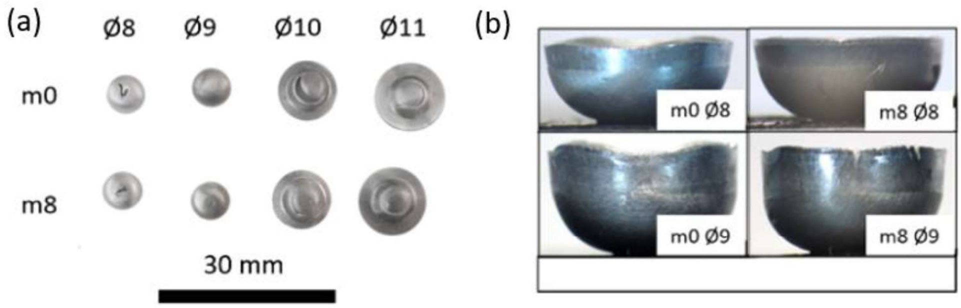

Fig. 40 Blanks drawn in cup drawing test of initial material m0 and after I-ECAP processing m8 view from top and (a) side (b)

7. Summary and concluding remarks

The research presented explores what constitutes a favorable microstructure of plates made of UFG aluminum alloys in terms of increasing their potential application through the use of sheet metal forming operations commonly used in the industry. Because UFG materials are mainly produced in the form of small rods or disks, which limits their possible applications, this thesis focused on SPD methods that allow the production of these materials in the form of thin plates.

The vast majority of studies on UFG materials fall into the category of basic research aimed at characterizing microstructure and mechanical properties, with only a few seeking to determine what properties could have wide application. To date, few researchers have dealt with the issue of formability, which is one possible reason for the slow commercialization of UFG materials. This could also be due to a lack of methods of producing semi-finished products in a form suitable for further shaping, which could be carried out by means of efficient processes of metal working of sheets or plates. Therefore, this thesis addresses two aspects related to the provision of materials attractive for commercial applications, namely, unconventional production of the material in the form of plates, and determining the material's ability to form in a complex deformation state.

Homogeneous microstructure, the type of crystallographic texture and isotropic mechanical properties all have a direct impact on a material's formability. Formability is usually determined using a uniaxial tensile test, which favors plastic strain localization, to which ultrafine grain materials are susceptible. The low values of elongation obtained in these tests suggest low plasticity, which, however, is largely the result of the quick localization of plastic deformation. Therefore, an important topic in this work is to draw attention to the fact that UFG materials, despite the small deformations obtained in the uniaxial tensile test, can be successfully shaped in operations in a state of complex deformation, and their susceptibility must be determined by additional tests. In this paper, it is demonstrated that, despite their significantly reduced plasticity defined in the uniaxial stress state, UFG materials in a state of complex deformation can exhibit as good or even better ductility than conventional materials.

Another factor that may increase our ability to shape UFG plates is adjustments of the external conditions in which the process takes place, i.e. temperature and strain rate. By adjusting these parameters, it is possible to control the thermally activated plastic deformation mechanisms, as well as the SH ability, so as to increase formability. It was proven that, by carrying out the operation of forming a UFG material at an elevated temperature, it is possible to improve formability (defined as the indenter stroke in the cupping test) by more than 100%, while mechanical strength remains up to twice as high as that of a conventional material. To date, there have been no studies in the world literature describing how deformation test conditions can be adjusted to improve the drawability of plates with a UFG structure; in this respect, this research is innovative. The available literature also lacks a detailed description of the SH capacity of such materials.

The outcomes of the research work and the analysis of the literature background are presented in 6 peer-reviewed papers, attached to the thesis. The detailed outcomes based on the acquired results are listed below.

1. Processing methods:
    - Hybrid processing, composed of mt-ECAP followed by upsetting, and I-ECAP are suitable for producing plates with an ultrafine-grained microstructure and isotropic mechanical properties. Achieving isotropic plates is possible using an appropriate deformation route, i.e. rotations of the billet between processing steps. Even though the last step of hybrid processing, i.e. upsetting, results in elongated grains, it does not cause in-plane anisotropy.
2. Microstructural features, mechanical properties and anisotropy:
    - Both proposed SPD methods are efficient in refining grain size to the UFG regime. The mean equivalent grain size obtained for the I-ECAP plates was ~ 0.5 μm with the fraction of HAGBs exceeding 60%, while for the hybrid processing those values were ~ 0.5 μm and 50%, respectively. Additionally, I-ECAP processing resulted in equiaxed grains.
    - A significant enhancement in mechanical strength was noted for all the materials investigated. The in-plane anisotropy of the plates was reduced, especially for the I-ECAP processed plates. The mean r-values were in a range typical for FCC materials, yet the planar anisotropy, Δr, was smaller for the plates after I-ECAP than for those processed by other methods. This was attributed to a reduction in the crystallographic texture intensity.
3. Improvement of ductility and formability:
    - It is possible to increase the ductility of UFG alloys 3003 and 5754 by reducing the strain rate and conducting tensile tests at an elevated temperature. By adjusting the

tensile test conditions, at ET total elongations of 82% and 16% for 5754 and 3003 alloys were achieved, compared with 5% at RT.

- The increase in ductility was attributed to enhanced SRS, which in turn was caused by reduced grain size, and therefore activation volume. The m parameter at ET was 0.11 for 3003 and 0.21 for 5754 alloy. The apparent activation volume expressed as a function of Burger's vector was reduced from several hundred for the CG materials to $13b^3$ for 5754 alloy and $24b^3$ for 3003 at ET.
- By conducting cupping tests at ET, it was possible to increase the cup height in comparison with RT by 99% and 62% for alloys 5754 and 3003, respectively, which is more effective than for CG materials.
- A major reduction in in-plane anisotropy caused a decrease in the earing phenomena in the 1050 plates deformed in a cupping test.

This thesis combines basic and applied research, and is aimed at increasing the range of potential applications of UFG aluminum plates. Recognition of the deformation path – microstructure – forming ability relationship will allow the SPD process to be designed consciously, and materials to be produced that are suitable for further metal forming operations.

Nonetheless, the issue of how strain rate and temperature affect deformation mechanisms requires further examination, as researchers do not fully agree on this matter. It is evident that thermal energy plays an important role in activating deformation mechanisms, but its exact influence has not yet been determined. A better understanding of the process would facilitate proper microstructure design for optimum results. Other issues that require attention are how to scale up the proposed SPD processes, and how to combine various alloys in the search for better ductility and strength.

8. List of appendices

Appendix 1

M. Ciemiorek, W. Chromiński, L. Olejnik, M. Lewandowska, Evaluation of mechanical properties and anisotropy of ultra-fine grained 1050 aluminum sheets produced by incremental ECAP, Materials and Design, 130 (2017) 392–402, doi: 10.1016/j.matdes.2017.05.069

Appendix 2

M. Ciemiorek, M. Lewandowska, L. Olejnik, Microstructure, tensile properties and formability of ultrafine-grained Al–Mn square plates processed by Incremental ECAP, Materials and Design, 196 (2020) 109125, doi: 10.1016/j.matdes.2020.109125

Appendix 3

M. Ciemiorek, W. Chromiński, C. Jasiński, M. Lewandowska, Microstructural changes and formability of Al-Mg ultrafine-grained aluminum plates processed by multi-turn ECAP and upsetting, Materials Science and Engineering A, 831 (2022) 142202, doi: 10.1016/j.msea.2021.142202

Appendix 4

Ductility and Formability of Al-Mn Ultrafine-Grained Plates Processed by Incremental Equal Channel Angular Pressing

Under review in Archives of Civil and Mechanical Engineering

Appendix 5

Ductility and formability of ultrafine-grained 5754 aluminium alloy under various strain rates and temperature, Materials Science and Engineering A, 848 (2022) 143375

doi.org/10.1016/j.msea.2022.143375

Appendix 6

M. Ciemiorek, P. Bartkowski, W. Chromiński, L. Olejnik, M. Lewandowska, Forming Ability of Ultrafine-Grained Aluminum Plates Processed by Incremental Equal Channel Angular Pressing, Advanced Engineering Materials (2019), 1900473, doi: 10.1002/adem.201900473

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