EQUAL CHANNEL ANGULAR PRESSING (ECAP) FOR HIGH-PERFORMANCE METALLIC MATERIALS

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ABSTRACT: The Equal Channel Angular Pressing (ECAP) technology is one of the industrially most relevant method of severe plastic deformation (SPD). SPD is the only technically viable, direct method for production of bulk, nanostructured metallic materials by means of large plastic deformation. The material deformation during ECAP is characterized by a i) multidimensional deformation under ii) enhanced hydrostatic pressure and iii) to a large extent by the preservation of the original shape of samples. In principle, metallic material in shape of cylindrical bolts is multiple pressed through a die consisting of two equal channels with a set angle of intersection under enhanced hydrostatic pressure without changing the cross-section area of the ingot. That fact enables extremely high deformation (by repeated pressing) of even hard-to-deform materials.

The ECAP facility at RHP-Technology in Wiener Neustadt, Austria consists of a variety of dies with angle of intersection 120°, 105° and 90° suitable for cylindrical bolts with diameters of 12 mm, 15 mm, 20 mm, 25 mm , 30 mm and 40 mm and total sample length up to 120 mm (limited by press' stroke). Besides standard, single ECAP a unique Double-ECAP die – with two areas of deformation – can be used for processing of selected materials. All dies can be operated at room temperature. Some dies can be cooled down to -70°C or heated up to 500°C. The temperature of the processed workpiece can vary between -196°C and 900°C. The hydraulic press enables pressing forces up to 700 kN. The process can be carried out in manual, semi-automatic or fully automatic (robotized) operating modes.

The effects of different ECAP process parameters on the microstructure and (mechanical) properties will be highlighted in detail for a broad range of materials such as aluminium and magnesium alloys.

KEYWORDS: Equal Channel Angular Pressing (ECAP), Double ECAP (D-ECAP), Low Temperature ECAP (LT-ECAP), Mg alloys, Al alloys.

Introduction

Equal Channel Angular Pressing (ECAP) is a process, where cylindrical billets of the starting material are repeatedly pressed through a channel that has one or more angles (and thus forming areas).

Under extremely high hydrostatic pressure, this leads to a high degree of plastic deformation, a strong grain refinement in the material and thus to an improvement of the mechanical properties, especially to increase the strength. At RHP single ECAP tools and as well novel double ECAP tools are available, that can improve properties of metals and alloys in a very effective way.

Mechanical tests with different deformation rates, namely on the one hand very slow (a factor of 1000 slower than in a standard test) and on the other hand very fast (a factor of 1000 faster, test duration only about 0.5 seconds) provided an in-depth understanding of the behaviour of the material under mechanical loading and served as a starting point for the tensile tests and fatigue strength tests.

In special cases of e.g. biomedical applications, the tests can also be performed in simulated body fluid (SBF).

ECAP can be used with a single bending channel, but also multi-bending channels like Double ECAP (D-ECAP) have interesting advantages and effects on the material properties, as well as heated and cooled setups. One setup investigated is low temperature ECAP (LT-ECAP) presented in the past, also highlighted in this paper.

Experimental: Magnesium alloy

In recent years the scientific and economic interest in biodegradable materials, especially in certain Mg-based alloys intended to be used as temporary medical implants, has grown rapidly [1,2]. These alloys are particularly suitable for osteosynthesis implants which are absorbed by the human body after fulfilling their purpose [5,6] due to local corrosion processes acting on the highly non-noble metal in the environment of the human body [3,4]. Therefore, a second so-called

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explanation surgery is prevented, being beneficial for both patients and the healthcare system. In the present study a lean, highly biocompatible Mg–Zn–Ca alloy was investigated. The alloying elements zinc and calcium were chosen since they are essential minerals or trace elements already existing in the human body [7]. It should be emphasized that no rare earth elements were used as alloying elements as they may enrich in organs [8] and therefore may result in at present unknown long-term side effects in the human body.

The lean MgZnCa alloy developed in the group (ZX00) [9] was produced in several steps (casting, heat treatment, extrusion) specially adapted to the alloy composition. The low content of alloying elements results in a desirably slow degradation rate in the body and ensures excellent biocompatibility. However, due to the low concentration of alloying elements, ZX00 alloy is less strong than alloys with higher Zn and Ca content.

To compensate for this, ECAP was used. The starting material as well as the material optimized by ECAP were subsequently analysed and characterized using different methods with the aim of providing the most comprehensive and quantitative description possible of the in vitro degradation behaviour of the MgZnCa alloy as a function of microstructure.

In the mechanical tests performed, it can be clearly seen, that the slower the slope of the load, the lower the strength and the higher the ductility of the ZX00 Mg alloy gets. This behaviour is not unusual and means, that an implant made of Mg-Zn-Ca can withstand higher short-term loading, for example during an impact or fall, than under continuous loading.

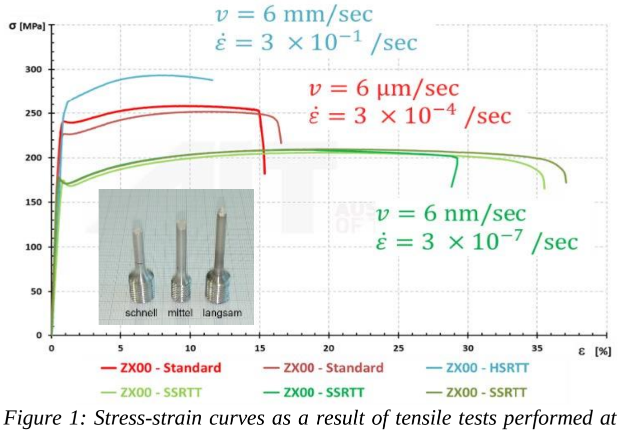

Figure 1: Stress-strain curves as a result of tensile tests performed at different deformation rates.

The degradation tests consisted of immersion tests in body-like fluids, i.e. stress-free degradation tests, as well as static and dynamic tests under various loads - including dynamic continuous loading.

These tests allowed the formulation of rate equations about the degradation process, which formed the basis for computer simulation models. In future, these findings should help to ensure, that medical implants manufactured from the biodegradable Mg alloy can be optimally designed and applied.

With the aid of optical deformation and strain analyses of the samples, it was possible for the first time to obtain important data on how the strain and stress states change locally during the degradation process. This is of essential importance regarding the future applicability of MgZnCa alloys for the stabilization of bone fractures. While in the ideal case the fractured bone gains more and more stability until complete healing, the implant must ensure and take over the still missing stability during this delicate period. It is not only the volume of the degrading implant that is decisive, but also the structure and strength, which determine whether the implant adequately fulfils the task of bone stabilization while losing its load-bearing capacity over time.

The degradation rates of the ZX00 alloy in its initial state and after ECAP were measured by means of immersion tests in SBF at body temperature. The individual samples are placed in their own containers filled with SBF, in which the hydrogen produced during degradation (corrosion) can be read. The amount of hydrogen gas produced in a given time indicates how much magnesium has dissolved. To keep the temperature constant, all containers are placed in a heated water bath. The results of the measurements, which took a total of 3 weeks, show that ECAP has no significant effect on the degradation rate of ZX00. The test rig used to determine the degradation rate in SBF under static load and slowly increasing stress was also used to determine the fatigue strength in air as well as in SBF. These measurements proved to be very time-consuming. Not only can the measurement time take up to 6 days - the preparations require perfect coordination - from the preparation and warming up of the SBF, programming of the tests to the mechanical setup - of the tests. The tests were carried out - as usual - up to the break of the specimen or up to 10 million cycles. They show the strength increasing effect of ECAP and increase of fatigue strength from 130 MPa to about 210 MPa.

Since both mechanical and corrosive properties of Mg alloys are highly dependent on grain and precipitate structure, the influence of ECAP deformation on the formation of these microstructural features was investigated. For this purpose, the material in its initial state and after one and after two ECAP pressings was investigated by Electron Back Scattered Diffraction (EBSD), Energy Dispersive X-ray Analysis (EDX) and Focused Ion Beam (FIB) - High Resolution Scanning Electron Microscopy (HRSEM) methods.

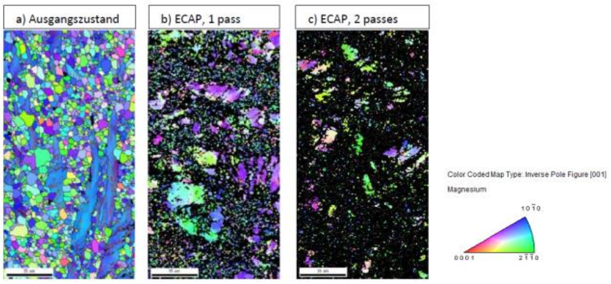

Figure 2: Color-coded EBSD microstructure images (see legend bottom right) of the Mg alloy ZX00 in the initial state (a) and after deformation (b) 1 ECAP cycle, (c) 2 ECAP cycles

From the results, the lower degradation rate of the cyclically loaded compared to unloaded specimens is related to better adhered and denser interface layers, which can be linked to a type of surface passivation.

Experimental investigated results and rate equations can describe a phenomenological approach based on the Arrhenius equation for modeling reaction rates as a function of temperature and concentration of corrosive species.

Double Equal Channel Angular Pressing (D-ECAP)

A newly developed D-ECAP (results compare [9]) die with two angels of 90◦ in the channel [11,12] was used, schematically shown in Fig. 3. The equivalent strain per pass in the D-ECAP die is approximately 2 and the arrangement of the channels leads to an inherent processing route BC [13]. It is important to note that the strain achieved with D-ECAP is significantly higher than in case of conventional ECAP dies with only one deformation zone.

Moreover, an enhanced back-pressure at the first angle is resulted from the deformation at the second deformation zone and due to overall increased friction. This is favorable for deforming rather brittle materials like Mg alloys [10,11,12].

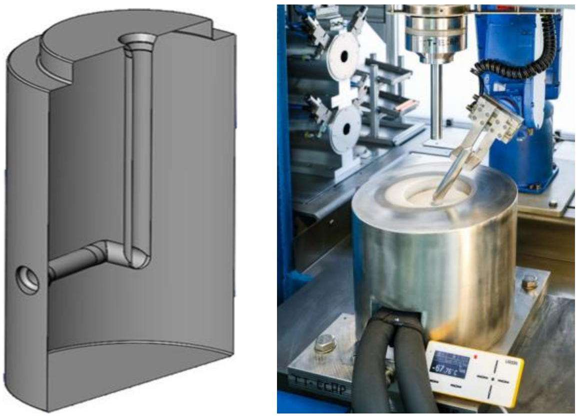

Figure 3: left: Section view through a design drawing of the core of the D-ECAP tool showing the channel with two angles of 90◦ each. Outside this core, a heating jacket is installed to adjust the desired process temperature. The samples are pressed through the tool by a plunger from the top. Right: Foto of the ECAP tool in the Press.

Within this study different D-ECAP process temperatures from 350 ◦C down to 220 ◦C were used. Before the first pass, all samples were kept in the preheated die for 2 min to reach the desired processing temperature.

In cases where more than one D-ECAP pass was performed, route BC was used by rotating the samples by 90◦ after each pass. The bolts were lubricated with graphite-based paste; the processing speed was 2 mm/s. Samples were either processed for one or more passes at a constant temperature or the temperature was decreased between the successive passes. The reason for the latter is that the passes at higher temperature increase the workability of the Mg alloy thereby allowing further passes at lower temperature [14].

Microstructure after D-ECAP

Exemplary optical microscopy images of ZX00 after D-ECAP processing are shown in Fig. 4 (cross-sections) Large, non-recrystallized grains are still present in the microstructure. However, their number and size are both strongly decreased, depending on the D-ECAP processing parameters. Bands of small, newly-formed grains run through the larger grains, as can be seen in both sections. The elongated shape of the large grains is still visible after one pass while with increasing number of passes these grains get more and more fragmented.

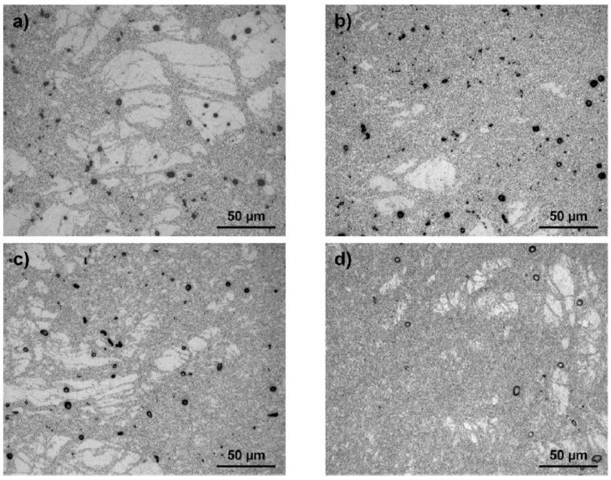

Figure 4: Microstructure as observed on the cross-sections of samples processed by D-ECAP. The respective process parameters are a) one pass at 300 ◦C, b) two passes at 300 ◦C, c) one pass at 280 ◦C, d) two passes at 280 ◦C. All images were taken at the center of the samples after etching.

Fig. 5 shows high-magnification optical microscopy images of areas containing mainly small, recrystallized grains with equiaxed shape. Individual grains can be distinguished even if their size is in the range of only one micron. It can be clearly seen that processing at 280 ◦C (Fig. 5b) led to a smaller grain size than processing at 300 ◦C (Fig. 5a).

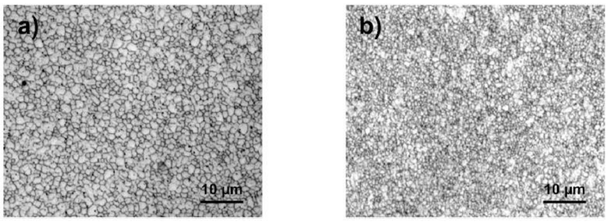

Figure 5: High-magnification micrographs taken on cross-sectional planes after a) 2 D-ECAP passes at 300 ◦C and b) 2 passes at 280 ◦C.

Grain sizes and values of the area fractions of large grains obtained after D-ECAP processing at various temperatures and various numbers of passes are summarized in Fig. 6. It can be seen that the grain size decreases with decreasing D-ECAP temperature while the area fraction of large grains decreases with increasing number of passes.

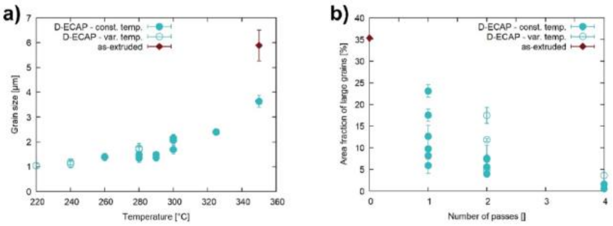

Figure 6: a) Grain size as a function of the temperature of the D-ECAP process; b) area fraction of large grains as a function of the number of D-ECAP passes. Full symbols indicate samples where the D-ECAP process was conducted at constant temperature while open symbols represent results obtained on samples where the process temperature was varied between the passes.

On the other hand, the grain size does not depend on the number of passes and the area fraction of large grains shows no clear correlation with the process temperature. Only in case of one single D-ECAP pass, there is a slight tendency for a lower area fraction of large grains with higher processing temperature.

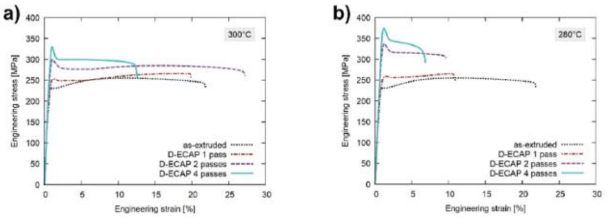

Figure 7: Exemplary tensile stress-strain curves of samples in the as extruded condition as well as processed by D-ECAP at 300 ◦C (a) and 280 ◦C (b) for different numbers of passes.

Exemplary results of tensile tests are shown in Fig. 7. It can be seen, that the initial, extruded ZX00 alloy exhibits some strain hardening and an ultimate tensile strength of more than 250 MPa. D-ECAP increases the tensile strength. Furthermore, in case of a higher D-ECAP temperature, the elongation at fracture can be increased as well. A common feature independent of the process temperature is the occurrence of a yield point phenomenon after two or more D-ECAP passes. This can lead to the result that the maximum tensile strength (which is the highest stress reached in a tensile test and may not necessarily be identical with the ultimate tensile strength as defined according to EN ISO 6892–1) is in some samples already reached immediately after yielding.

Grain sizes between 1.3 and 1.5 µm were achieved by D-ECAP processing at 280 ◦C. Even smaller grain sizes can be obtained at lower temperatures, however, with the risk of crack formation. The area fraction of the larger grains of the bimodal grain size distribution was decreased strongly with increasing number of D-ECAP passes.

By varying the processing parameters, it is possible to obtain different microstructures and thereby tailor the mechanical properties. The hardness is strongly increased by decreasing the size of the small, recrystallized grains while the tensile strength is more dependent on the remaining area fraction of the large grains. The elongation at fracture can be kept high or even increased if a certain fraction of large grains remains in the material.

Thus, the mechanical properties can be optimized and tailored by DECAP within a certain range. For example, a very high tensile strength of more than 370 MPa can be achieved in combination with 7% elongation at fracture. Alternatively, a moderately increased tensile strength of about 300 MPa in combination with an increased elongation at fracture of 26% can be obtained. The mechanical properties of the present lean and thus highly biocompatible Mg–Zn–Ca alloy after D-ECAP are comparable to those of Mg alloys with significantly higher alloying levels processed by conventional ECAP.

Low Temperature ECAP

For investigating material questions for low temperature ECAP behaviour, the core topic was the development of an ECAP infrastructure, which enables the forming at low temperatures (down to -70°C). This is closely linked to the development and definition of the optimal process parameters as well as the elaboration of corresponding process descriptions. From the scientific point of view, an attempt was made to obtain experimental data on the mechanisms of the plastic deformation of Al and Mg alloys as well as of the materials with order-disorder transformation under the conditions of ECAP deformation at low temperatures. For the cooling of the ECAP tool, a commercial low-temperature cooling unit was purchased and equipped with the necessary control units. The setup of the machine is shown in Figure 1 (right), Figure 7 exhibits the developed low-temperature cooling jacket for a stable temperature niveau of +/-2°C at all set temperature levels.

Figure 7: Cooling jacket before soldering; finished cooling jacket with the ECAP die.

Experimental: Al 7075 – LT-ECAP

The effectiveness of ECAP forming was investigated using low-temperature ECAP pressing followed by structural and mechanical investigations on three groups of materials: 1) Al alloys 2) Mg alloys and 3) alloys with a segregation gap of Au-Cu type. The microstructure was characterized by scanning electron microscopy (SEM). The mechanical characteristics were determined by hardness measurements and strength and ductility by standardized quasi-static tensile and compression tests. For this study some examples on Al7075 alloy (AlZnMgCu1,5) are given as results from this study of low temperature ECAP investigations.

Figure 8: Al alloy samples after ECAP prepared for Tensile testing

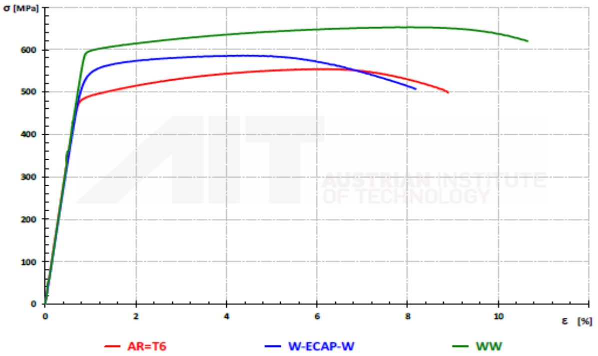

Figure 9: Al7075 comparison of stress-strain curves of three conditions (AR: as received T6, WW: thermal treated without ECAP, W-ECAP-W: thermal treated – ECAP – thermal treated)

Low temperatures cause thermally activated (diffusion-controlled) cross-sliding processes of the dislocations as well as annihilation processes during plastic deformation are strongly restricted. This leads to a reduction in the number of dislocations in the case of face-centered cubic (kfz) metals such as aluminum, and enables the plastic forming of highly plastic forming of high-strength, brittle and therefore difficult-to-form kfz Al-based high-performance alloys. This effect is well documented in the literature for uniaxial cryogenic rolling or multidirectional forging, where the goal was to confirm for cryogenic ECAP. Cryogenic forming has also been technically applied to aluminium sheet for instance.

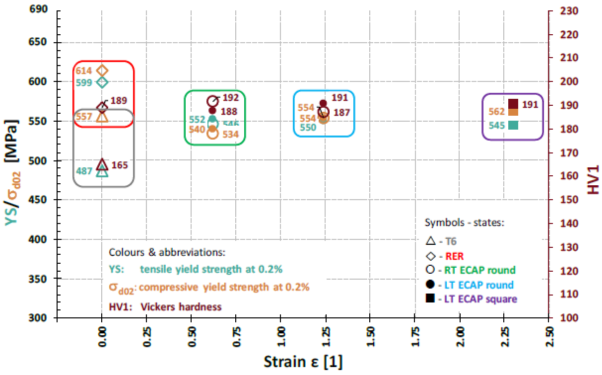

Figure 10: Tensile tests (tensile strength, YS) and compression tests (compressive strength at 0.2% compression, d02) on Al7075

Similar experiments like for Al alloys were performed using Mg alloys, this study is ongoing and will be presented in future publications.

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