Detection of structural changes and mechanical properties of light alloys after severe plastic deformation

V A Krasnoveikin¹,², A A Kozulin¹ and V A Skripnyak¹
¹ National Research Tomsk State University, Lenina ave. 36, Tomsk 634050, Russia
² Institute of Strength Physics and Materials Science of Siberian Branch Russian Academy of Sciences, pr. Akademicheskiii 2/4, Tomsk 634055, Russia
E-mail: volodia74ms@yandex.ru

Abstract. Severe plastic deformation by equal channel angular pressing has been performed to produce light aluminum and magnesium alloy billets with ultrafine-grained structure. The physical and mechanical properties of the processed alloys are examined by studying their microstructure, measuring microhardness, yield strength, and uniaxial tensile strength. A non-destructive testing technique using three-dimensional X-ray tomography is proposed for detecting internal structural defects and monitoring damage formation in the structure of alloys subjected to severe plastic deformation. The investigation results prove the efficiency of the chosen method and selected mode of producing ultrafine-grained light alloys.

1. Introduction
Over the past decades, intensive efforts have been made to develop new technologies with the use of severe plastic deformation (SPD) methods for the production of ultrafine-grained (UFG) and nanostructured (NS) Al- and Mg-based light alloys. This has provided a qualitative increase in the efficiency of automotive and aerospace engineering, ship building, medical implants and equipment, power generation systems, and sports gear. The main aspects of the SPD influence on the properties of structural metals and alloys have been noted by many researchers [1-4]. They consist in the refinement of the material internal structure to UFG and NS states, due to which the physical and mechanical properties of the material change and the material hardens. The hardening effect after SPD shows up as an increase in microhardness, yield strength and ultimate tensile strength, a change in the degree of specimen elongation to failure, and an increase in durability and fatigue strength [4, 5]. There are different methods of SPD in metals and alloys that can be performed in local zones, on surfaces, and in the bulk of the material [4, 6-9]. The most common method is equal channel angular pressing (ECAP) that allows changing the structure in bulk specimens. A correctly selected scheme and an optimal pressing mode should provide uniform microstructure refinement in alloys without the formation of micro- and macrodefects and without cracking of the specimen material.

The major goal of this study is to define the effect of SPD on the physical and mechanical properties of the Russian light structural alloys such as aluminum alloy of grade 1560 and magnesium alloy of grade Ma2-1, which are the closest analogues of the known alloys 5083 and AZ31, respectively. These alloys are widely used in aviation, automotive and space industries. By changing their physical and mechanical properties, it becomes possible to optimize structural elements and to increase the service efficiency under various operating conditions owing to the reduction in the weight of parts and structures.

2. Materials and methods
Prismatic billets (measured 8x8x40 mm³) for processing and subsequent investigation were prepared from bars of hot rolled coarse-grained alloys 1560 (chemical composition: 91% Al – 6.12% Mg – 0.59 Mn - ~ 2.29% other impurities) and Ma2-1 (chemical composition: 93.65% Mg – 4.36% Al - 1.33%
Zn – Mn ~ 0.66% other impurities). The main billet axes coincided with the bar rolling direction. The billets were subjected to SPD by ECAP to produce UFG structure using a collapsible steel die (figure 1a) with the channels intersecting at an angle of 90 degrees without fillets. The given die geometry provides the maximum plastic strain accumulation in the material in one pass, equal to 115%. The pressing was carried out using route $B_c$ (figure 1b) in which the billet is rotated by 90 degrees about the longitudinal direction (LD) in every next pass. The chosen ECAP scheme was assumed to be the most suitable for producing a homogeneous UFG structure within the specimen and to provide isotropic mechanical properties [4]. In order to eliminate defects in the ECAPed billets of the both alloys, optimal pressing modes were selected by changing the governing experimental parameters. Pressing was carried out on a universal servo-hydraulic testing machine INSTRON 40/50-20 equipped with an oven which permits a fine adjustment of pressing speed and force up to 50 kN.

![Diagram](image)

**Figure 1.** General view of the collapsible die (a) for ECAP, and (b) schematic drawing of route $B_c$, here values LD, ND and TD are longitudinal, normal and transvers directions, respectively.

This paper reports the X-ray tomography results on the detection of internal and surface defects in the alloys after processing. The quality of the processed billets was assessed by non-destructive testing using the microfocus X-ray system YXLON Y. Cheetah [10,11].

A series of tests were performed on the processed prismatic billets to examine the physical and mechanical properties of the studied materials, including microstructural analysis, microhardness measurements, and uniaxial tensile testing of flat specimens.

The texture and microstructure of the material before and after processing were observed by EBSD analysis using a scanning electron microscope Tescan Vega II LMU equipped with an EBSD device. The specimen surface was prepared for the investigation by mechanical polishing and ion etching using an ion slicer EM-09100. The obtained data were analyzed with the licensed HKL-Channel 5 software.

The mechanical texture of alloys after SPD was examined on an optical inverted microscope Olympus GX. The gage part of the flat specimens produced for uniaxial tensile tests was chemically etched for optical microscopy.

The Vickers microhardness (HV) measurements were conducted according to ISO 6507-1:2005 on the lateral surface of the prismatic billets in different regions along the pressing axis using a microhardness tester HMV G21ST Shimadzu. The measurements were taken under a load of 0.5 N and a dwell time of 10 s. Over 100 measurements were made for each specimen. The specimen surfaces were prepared by standard techniques using mechanical grinding and polishing to a defect-free mirror finish.
Flat dumbbell specimens for tensile testing with the gage part measured 10 mm in length, 1.6 mm in thickness, 3 mm in width, and with a 2.5 mm shoulder radius were cut out from the ECAPed prismatic billets along the pressing axis by electric discharge machining. Uniaxial tensile tests were conducted on a servo-hydraulic testing machine INSTRON 40/50-20 with the strain rate 0.001 1/s and test temperature 298 K. The tensile loads applied to the specimens were recorded with an accuracy of 0.05% using a load cell Dynacell.

3. Results and Discussion
While selecting optimal pressing modes for the studied materials, some crucial governing parameters were experimentally revealed which must be taken into account. The first parameter is the temperature, which must be 473 K for aluminum alloy and 523 K for magnesium alloy. The second parameter is the pressing speed, which was equal to 15 mm/min. The third parameter is friction. Friction between the billets and the die channel walls was minimized using a high-temperature lubricant on the basis of mineral oil and molybdenum disulphide [12]. With the above parameters, the processed material preserves its continuity without cracking.

Figure 2. Prismatic billets before and after pressing: (a) – as-received; (b) – after processing by the optimal ECAP mode; (c) – fragmented specimen after processing by the non-optimal ECAP mode.

The fragmented billet depicted in figure 1c is an example of using a non-optimal pressing mode. Cracks that cause the billet fragmentation are generated at an angle of 45 degrees to the longitudinal direction along the maximum shear strain direction in the channel intersection zone. The billet processed in the optimal mode (figure 2b) shows no visible signs of cracking.

Figure 3. Voxel model of prismatic billets subjected to SPD: isometric projection (a); front section (b); left (c) and right (d) longitudinal section.

Modern X-ray tomography methods were applied to evaluate the internal structure, porosity, the presence of microcracks and voids in the bulk of the ECAPed billets without visible defects. X-ray tomography provides a 3D image of an object in the form of a voxel model that allows viewing an
image of any surface or internal section with micron accuracy [10]. Figure 3 shows a voxel model of the prismatic billet with a crack after ECAP processing. The model is shown in an isometric projection and three sections. The surface defect viewed as a small step is a crack propagating from the surface into the bulk. The billets which had such defects were considered unsuitable for further investigation and were rejected. The X-ray tomography results confirmed that if SPD is performed in the optimal mode, the structure of the material after processing is free from macro- and microdefects.

Figure 4. Grain size distribution in aluminum alloy 1560: (a) – as-received material; (b) – after 4 ECAP passes.

Figures 4 and 5 demonstrate grain size distribution histograms, where $d_g$ is the grain size in µm, $N$ and $n$ is the total number of measurements and the number of grains of a certain size, respectively. Microstructural studies have shown that aluminum alloy 1560 in the as-received state exhibits a grain size distribution within a wide range from 2 to 400 µm, with the average grain size 50 µm. The grain size distribution in the as-received magnesium alloy Ma2-1 ranges from 2 to 60 µm, with the average grain size 18 µm. Since the billets were cut out from hot rolled bars, the grain structure has a well-defined direction. Coarse grains are elongated along the rolling direction and clusters of fine grains are adjacent to them, together forming bimodal structures.

Figure 5. Grain size distribution histograms in magnesium alloy Ma2-1: (a) – as-received material; (b) – after 4 ECAP passes.
After four ECAP passes in the selected optimal mode, a more homogeneous UFG structure with an average grain size of 3 and 7 µm is formed in the central part of aluminum and magnesium alloy billets, respectively.

Figure 6 illustrates the averaged experimental data obtained in Vickers microhardness (HV) measurements on the studied materials before and after ECAP. Here, the value \( \Delta d/d_0 \) is the ratio between the distance from the measurement area to the billet edge \( \Delta d \) (mm) and its total length \( d_0 \) (mm). Statistical analysis for all results was carried out by sampling and averaging the obtained data, with the determination of the confidence intervals.

Experimental results indicate that ECAP leads to an increase in microhardness in the entire specimen volume, but there is inhomogeneity in the axial direction. After four ECAP passes, the microhardness increases by on average 40–60% as compared to its initial value for the both materials. The maximum microhardness reaches 1550 and 786 MPa, with the initial value not exceeding 1000 and 560 MPa for the aluminum and magnesium alloys, respectively. The microhardness in the head ends of aluminum specimens is somewhat lower (up to 10%) than the average value. The microhardness deviation from the average values in the central specimen part both along the longitudinal axis and in the transverse direction is within 10%.

Uniaxial tensile test results are represented as stress-strain curves in figure 7. It is seen that after four ECAP passes the yield strength of aluminum alloy 1560 increases from 150 to 270 MPa and its ultimate tensile strength increases from 320 to 460 MPa, while those of magnesium alloy increase...
from 150 to 200 MPa and from 250 to 290 MPa, respectively. The maximum elongation to failure under uniaxial tension increases from 0.17 to 0.24 for magnesium alloy and decreases from 0.24 to 0.17 for aluminum alloy. These data indicate that the strength characteristics in the bulk of the both studied alloys increase during SPD by ECAP.

The serrated flow curve for uniaxially tensile aluminum alloy is indicative of serrated flow occurring due to the Portevin-Le Chatelier effect. The serrated profile is not observed in the entire plastic flow region. It begins with 6% plastic deformation in the as-received alloy, and then the beginning of serration is shifted to higher strains up to 10% in the processed alloy with UFG structure.

The stress-strain curves for magnesium alloy in the as-delivered state and after ECAP are smooth.

4. Summary
The ECAP temperature and speed were determined experimentally for aluminum alloy 1560 and magnesium alloy Ma2-1. An optimal processing mode was selected to produce specimens without defects in the entire volume. A non-destructive testing technique on the basis of X-ray tomography was proposed which enables the detection of defects such as cracks and voids both on the surface and in the bulk of prismatic billets.

The investigation results showed that for SPD of light alloys 1560 and Ma2-1 by multipass ECAP four passes are enough to produce a homogeneous structure in the specimen bulk with an average grain size of 3 and 7 μm, respectively. The alloys processed in this mode assume an ultrafine-grained structure.

It was found experimentally that SPD of the studied alloys increases the yield strength and short-term strength. The maximum elongation to failure under uniaxial tension increases for magnesium alloy but decreases for aluminum alloy. The microhardness of the both alloys after four ECAP passes increases by 40–60%.

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