Evolution of the structure and properties of pure aluminum under severe plastic deformation

B K Rakhadilov, M K Kylyshkanov, M S Zhaparova
S. Amanzholov East Kazakhstan State University, 55 Kazakhstan st., Ust-Kamenogorsk 070004, Kazakhstan

E-mail: rakhadilovb@mail.ru

Abstract. The influence of severe plastic deformation (SPD) by equal-channel angular pressing (ECAP) on the formation of the structure and the mechanical properties of pure aluminum is studied. It is established that an ultrafine-grained structure is formed with an average size of structural elements of ~ 1.5 µm. X-ray diffraction studies have shown that SPD results in a broadening of interference lines with a decrease in their intensity, which indicates an increase in the imperfection of the structure and its high dispersion. SPD also results in a significant enhancement of microhardness, yield stress and ultimate tensile strength in aluminum.

1. Introduction
The study of the impact of the structure on properties of nanomaterials is of great relevance for materials science today [1]. The properties, inhered by the material in its usual coarse-grained state, undergo fundamental changes when a nanostructure is formed there. These materials are of primary interest in this regard [2]. For the last few years, material scientists have been showing their interest in ultrafine-grained (UFG) aluminum materials, where SPD is used for the structure refinement [3]. The formation of the UFG structure in these materials considerably improves their mechanical and physical properties, which can be widely applied. Currently, ECAP is one of the dominant approaches in the implementation of SPD. This method makes it possible to produce massive pore-free wrought semi products with ultra-fine grains with sizes ranging from tenths of microns for pure metals to dozens of nanometers for alloys and intermetallic compounds. The study of mechanical properties and structural-phase features influencing their formation in ECAP has been the subject of numerous studies [4].

In this view, the purpose of the study is to understand how SPD, performed by ECAP, affects the pure aluminium structure and its mechanical properties.

2. Experimental procedure
Pure aluminium rods (99.99 %) were employed as the test material. Aluminum rods were subjected to SPD by the ECAP method.

ECAP was carried out in a press mold at an angle of 90° for the intersection of working and output channels. Metal rods with a diameter of 20 mm and a length of 100 mm were pressed at a rate of 0.4 mm/s. The rods were subjected to ECAP at room temperature with a varying number of passes (from 1 to 12) rotating around their axis by 90° or 180° between the passes.
The structure of aluminium before and after ECAP was examined by optical microscopy using a Neophth-21 microscope and scanning electron microscopy using a JSM-6390LV microscope. Aluminum sheets have been polished in an electrolyte consisting of 20% HCl and 80% C$_2$H$_5$OH, at 20 °C and 25-35V prior to the study.

X-ray diffraction analysis was carried out on an X'Pert Pro diffractometer in Cu Kα- radiation to study the phase composition and crystal structure of the specimens.

Mechanical tests were carried out at room temperature on a Polyany type machine in accordance with the requirements of GOST 1497-87. During the tests, the sheet specimens were subjected to unaxial quasistatic tension till their rupture, meanwhile measuring the relative yield stress $\sigma_{0.2}$, the ultimate tensile strength $\sigma_{UTS}$, and the elongation to failure $\delta$. Specimens with a gauge size of 0.5×4×10 mm$^3$ were fabricated using electric spark cutting for these tests. Vickers microhardness was measured on a PMT-3 type machine at a load of 0.5 N for 10 seconds. The error of measurement was no more than 8%. A layer ~ 50 µm thick was removed from the surface of the specimen by mechanical grinding followed by electrolytic polishing.

3. Results and discussion

It was found out that in its initial state, aluminum consists of large grains with an average size of 80 µm (figure 1 a). Metallographic and electron-microscopic analyses have shown that ECAP results in a substantial refinement of pure aluminium. Subgrained fragments of ~ 4 and 1.5 µm were formed in the transverse and longitudinal sections after 8 and 12 ECAP passes (figure 1 b, c).

![Figure 1. Microstructure of pure aluminum a - in the initial state; b - after ECAP-8 passes, c - after ECAP-12 passes](image-url)

X-ray diffraction of coarse-grained and submicrocrystalline aluminum has shown considerable changes in the crystal structure after ECAP. As is known from [5], with increasing strain, along with a decrease in the grain size, the degree of imperfection of the structure also increases and the crystallographic deformation texture is formed. Figure 2 illustrates diffraction patterns of aluminum before and after 12 passes of ECAP. As can be seen, the reflexes (200) and (311) broaden, and their
intensity decreases. Broadening of the lines and a decrease in their intensity indicate an increase in the imperfection of the structure and its high dispersion.

![Diffraction patterns of aluminum before ECAP (a) and after 12 passes of ECAP (b).](image)

**Figure 2.** Diffraction patterns of aluminum before ECAP (a) and after 12 passes of ECAP (b).

The formation of ultrafine-grained structure of aluminum provides enhanced mechanical properties of the material. Figure 3 shows dependence of microhardness of aluminium on the number of steps of ECAP carried out with a rotation of the samples to 90° and 180° between the passes. As one can see, the ECAP results in an enhancement of the microhardness of pure aluminum depending on the number of steps, i.e. on the degree of deformation. One also can see that the specimens pressed with a rotation to 90° demonstrate a lower microhardness in comparison to that of the samples pressed with a rotation to 180°.

Thus, it is established that ECAP makes the microhardness of pure aluminum three times stronger.

![Dependence of the microhardness of aluminum on the number of ECAP passes with a rotation of samples to 90° (green) and 180° (brown).](image)

**Figure 3.** Dependence of the microhardness of aluminum on the number of ECAP passes with a rotation of samples to 90° (green) and 180° (brown).
After ECAP aluminum has improved yield limit and tensile strength (table 1). With an increase of the number of ECAP steps these properties increase too.

| ECAP mode          | $\sigma_{0.2}$, MPa | $\sigma_y$ MPa | $\delta$, % |
|--------------------|---------------------|----------------|-------------|
| Before ECAP        | 10                  | 45             | 50          |
| ECAP 4-passes      | 105                 | 137            | 7.5         |
| ECAP 8-passes      | 170                 | 191            | 12.5        |
| ECAP 12-passes     | 183                 | 214            | 14          |

The elongation to failure of the specimens drops down to 7.5% after 4 passes of ECAP and increase back up to 12.5% after 8 passes of ECAP and to 14% after 12 passes of ECAP. These changes are typical to UFG materials.

Thus, aluminium demonstrates mechanical properties which are typical of ultrafine-grained metals obtained by ECAP, i.e. enhanced strength properties, i.e. yield stress, ultimate strength and lower elongation to failure.

4. Conclusions

It was established that severe plastic deformation of pure aluminium by equal-channel angular pressing at room temperature leads to the formation of an ultrafine-grained structure with an average (sub)grain size of 1.5 µm.

It was revealed that as a result of ECAP, a significant hardening of pure aluminium occurs. As a result, the microhardness increased by a factor of 3, the UTS and YS increased by 4.7 and 18 times, respectively, with a simultaneous decrease in the elongation to failure up to 3.5 times.

References

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