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In-Crystal Dislocation Behaviour and Hardness Changes in the Case of Severe Plastic Deformation of Aluminium Samples

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Abstract: The presence of dislocations significantly modifies the mechanical properties of crystalline solids. Severe plastic deformation (SPD) and the most used SPD process – the Equal Channel Angular Pressing (ECAP), affect the multiplication and localized accumulation of dislocations. This research is related to the observation of dislocation pile-up and significant reduction of the crystalline grain size caused by severe deformations in the ECAP process of the widely used aluminium material (Al 99.5%). Because of its lightweight, the application of Al 99.5 % can pose a challenge for the aviation and space industry, especially since its mechanical properties limit its application. Improving these mechanical properties can extend its applicability in cases of demanding constructions as well as influence the final product cost. As a confirmation of SPD in-fluence on mechanical properties, material hardness has been examined and described. Dislocation monitoring is enabled using the light and electron microscopy and AFM (Atomic Force Microscope) device. A numerical simulation of the Equal Channel Angular Pressing process using the ABAQUS software package determined the representative area of the most severe deformation.

Keywords: aluminium (Al); atomic force microscope (AFM); equal channel angular pressing (ECAP); hardness; severe plastic deformation (SPD)

1 INTRODUCTION

Each real crystal contains defects or irregularities in the crystal structure that can be dynamic or static. While dynamic defects, errors, irregularities of the crystal structure are caused by excitations of the crystal structure, static defects occur during the construction of the crystal structure or later processes such as mechanical deformations, heating, radiation, and others. Static defects can be point, line, surface or volume defects or irregularities. Many macroscopic properties of crystals can only be explained by the existence and change in the concentration of defects in crystals. From the perspective of mechanical properties, the dislocation is the most important crystal defect. It has been recognized that dislocations are the primary carriers of plastic deformation in crystalline solids, leading to ductility that makes metals workable, and through their manipulation and entanglement, hardening that makes the same metals stronger. [1].

Due to the action of external forces a glide of many dislocations occurs, which then results in a slip and leads to plastic deformation in crystalline solids. It can be thought of as sliding or successively moving one plane of an atom over another on so-called slip planes. This is uniquely defined as a plane containing both a line and a Burgers dislocation vector. Further deformation occurs either by more movement on existing planes of atoms or by creating new slip planes. The slip planes are usually the planes with the highest atom density, with the slip direction being the shortest translation of the vector. Often this direction is the one in which the atoms are closest. The sliding results in the formation of steps on the crystal surface. The direction of the slip is necessarily always parallel to the Burgers dislocation vector responsible for the sliding. The glide of one dislocation across the plane of the slip to the surface of the crystal produces a surface slip step equal to the Burgers vector [2].

The dislocation which is sliding through the crystal structure occurs within a single crystal. The remaining dislocations or grain boundaries present an obstacle for the movement of the dislocations. To overcome such obstacles, a large amount of energy and a very high force is required, which, in practice, prevents such movement. Therefore, during cold plastic deformation, dislocations accumulate within the grain and at the grain boundaries, thus making difficult further plastic deformation and increasing the strength of the deformed material.

Many scientific studies since the 1940s, up to now, relate to the experimental study and numerical calculation and simulation of the behaviour of dislocations within the crystal structure of metallic materials.

One of the first calculations can be dated back to 1940, when Burgers suggested that if an array of moving dislocations were stopped, a large local stress concentration would result [3]. This was the genesis of the dislocation pile-up, a concept which has proven to be helpful for analysing work hardening phenomena. Rosenfield and Hahn (1968) [4] have calculated the time dependent positions of dislocations emitted from a source into infinite homogenous medium and the positions of a moving array suddenly confronted with an obstacle. In 1969 Kanninen and Rosenfield [5] researched the numerical calculation of time dependent formation of single-ended dislocation pileups from the sequential emission of dislocations from the stress activated source. In 1974 Turnen [6] presented a method of numerical calculation for time dependent behaviour of several curved dislocations in a crystal. Devincre and Condat (1992) [7] made 3D computer modelling of plastic flow and model validation of a 3D simulation of dislocation dynamics. Also, several other authors were dealing with 3D computer modelling of dislocation movement in plastic deformations (Zbib 1998, Wang 2001, Yashiro 2006) [8-10].

Some techniques provided the possibility of a visual investigation of dislocations and their interactions in materials such as electron microscopy and X-ray diffraction measurement systems. Scientific research which relies on these techniques was conducted by Shen, Wagoner, and Clark (1987) [11] and Krause, Sylla and Oriwol (2016) [12].

This research work is related to the observation of dislocations pile-up caused by severe deformations of the aluminium material and, at the same time, a significant reduction of the crystalline grain size. Dislocation monitoring is enabled using the AFM (Atomic Force Microscope) device. Severe plastic deformation was performed using the ECAP (Equal Channel Angular Pressing) procedure. The ECAP procedure, as a method of achieving severe plastic deformation finds its application mostly for the hardening of titanium, copper, and aluminium alloys. ECAP application in the field of material hardening and several new interventions in the basic form of ECAP process has been described in scientific papers by Chia-Nan Wang (2016) [13] and Öğüt (2021) [14]. In this research, ECAP process has been applied on Al 99.5 % samples. This is a light weight, widely used, cheap material. Because of its light weight, the application of Al 99.5 % can pose a challenge for the aviation and space industry, especially because its mechanical properties limit its application. Improving these mechanical properties can extend its applicability in cases of demanding constructions as well as influence the final product cost.

2 SEVERE PLASTIC DEFORMATION

Since grain boundaries present an obstacle to the movement of dislocations, the fine-grained crystalline also results in an increase in the strength of the material. Hence, the modification of grain size can enable to design materials with desired properties. Physical, mechanical, and chemical properties can benefit greatly from the reduction of grain size [15, 16]. One of the possible ways for the microstructural refinement of metals is Severe Plastic Deformation (SPD).

The modern SPD technology originates from the work done by P.W. Bridgman who developed the techniques for materials processing through a combination of high hydrostatic pressure and shear deformation [17]. Severe plastic deformation leads to exceptional grain refinement of the material without introducing any significant changes in the overall dimensions of a specimen or workpiece. Materials produced by SPD techniques have grain sizes in the range of (50–1000) nm.

One of the most interesting, most frequently researched and most frequently used methods of severe plastic deformation (SPD) is Equal channel angular pressing (ECAP). This method has been proven to improve the mechanical properties of commercially pure metals, alloys, and composites [18, 19]. ECAP, like other SPD procedures, achieves extreme reduction of crystal grains by specific deformation of the material and brings their size to the nano level (even below 100 nm). Such an effect cannot be achieved or approximated by conventional heat treatment or conventional plastic processing. Most research on the angle extrusion process is devoted to relatively soft metals with Face centred cubic (FCC) crystal structure, such as aluminium and copper. However, ECAP finds its application in the case of processing more complex alloys and solid metals for which the number of slide planes along which their deformation takes place is limited [20].

2.1 ECAP Material Treatment

Plastic deformation by the ECAP process is achieved by extruding the material to be processed through two channels of the matrix that intersect at a certain angle, usually 90° [21-22]. The cross-sections of the channels are identical to the cross-section of the sample of material being processed and may be square or circular in shape. At the intersection of these channels there is a shear zone in which a large shear deformation is introduced. The amount of shear deformation is mainly defined by the geometry of the tool (inner and outer angle). A sample of the material is processed by metal removing operations to bring it to a measure where it fits the dimensions of the matrix channel before the sample is subjected to the ECAP process. The ECAP process can be repeated several times, which introduces greater plastic deformation into the workpiece without changing its dimensions.

In this specific case of experimental research work, an ECAP tool was designed (Fig. 1). The inlet and outcome channels of the die intersect at 90° (Fig. 2). They have identical, square cross sections, 14 mm in size. The ECAP process is conducted using high-capacity hydraulic press modified to slow down a ram speed. The obtained pressing speed is approximately 10 mm/s. Initial process temperature is 20 °C. Lubrication is provided using a homogeneous mixture of graphite and molybdenum disulphide. Two ECAP passes were conducted with so-called A route - when the orientation of the specimen remains unchanged after each pass. The initial material state is a cold rolled profile with an initial cold deformation of 15 %. Before the last step of cold deformation, it has been hot formed. Treated material is aluminium 99.5 %.

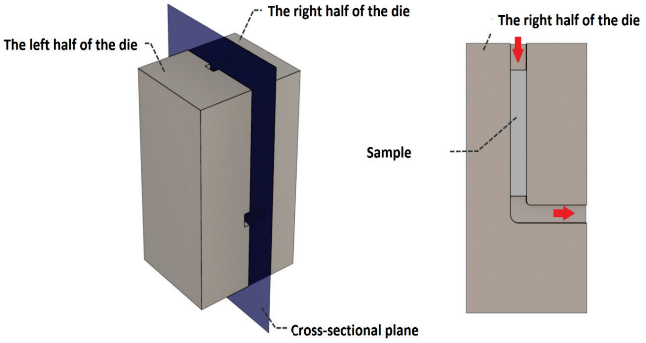


Figure 1 A scheme of the ECAP tool used in experimental research

To explore deformation status of the treated sample a numerical simulation of ECAP process was performed using Abaqus simulation software [23]. To determine the area of the highest severe plastic deformation influence, the first pass was observed. The same cross section is used in experimental monitoring of the second pass.

A three-dimensional case of the ECAP process was studied. The tool consists of the left and right halves of the die, which are symmetrical around the plane in which they are joined (Fig. 2). Process modelling is simplified with several assumptions which do not impair the accuracy of the results, but only shorten the time required to perform the numerical analysis. The first assumption is that the entire tool and the sample itself are symmetrical around the joining plane; the possibility of setting a boundary condition of symmetry around that plane was used. In this way, the number of finite elements was significantly reduced in the discretization of the model. This directly means that the number of unknown parameters in the algebraic system of equations is also smaller, which in the end provides significant savings in the time required to solve the analysis, with unchanged results. However, to further simplify the three-dimensional analysis, the plunger was completely ejected and instead of it, an edge displacement condition of – 140 mm path along the *Y*-axis was placed on the upper surface of the 140 mm long specimen. The next step before running the simulation was to define the finite elements. Three-dimensional, first-order hexahedral finite elements were selected for this analysis, using the Lagrange formulation and reduced integration - C3D8R. The number of elements on the sample is 2880 and the number of elements on the matrix is 13159. Defined friction coefficient was 0.15. Flow curve was chosen from the ABAQUS database.

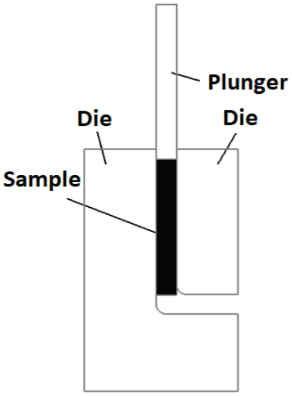


Figure 2 ECAP tool parts

According to the deformation scale, the largest equivalent deformation occurs at the cross-section A-A marked on Fig. 3. This cross section was used to examine structural changes in the sample material and changes in material hardness.

3 MATERIAL PROPERTIES – PRELIMINARY ANNOTATION

Two ECAP passes were performed on aluminium samples, using A route (the orientation of the specimen remains unchanged after each pass). Each pass resulted with changes in grain size and mechanical properties. The mechanical property observed was the hardness of the material. According to the deformation scheme of the numerical simulation, the cross section with the largest equivalent deformation was used. Testing samples for grain size and hardness examination were made by the cross cutting of ECAP deformed samples. Evaluation of the sample microstructure was made using light and electron microscopy. Equipment used in research was light microscope Olympus GX51F-5 with DP-25 CCD camera and image analysing software and scanning electron microscope VEGA TESCAN TS5136LS, Brno, Czech Republic with integrated SE and BSE detectors. The Figs. 4a), 4b) and 4c) present microstructure obtained by severe plastic deformation in ECAP procedure. Experimental results showed that ECAP deformation resulted in the grinding of crystalline grain. Grain size changes are presented in Fig. 4. After one ECAP pass the average grain size is 60 % smaller, and after two ECAP passes it is 87 % smaller than the initial grain size.

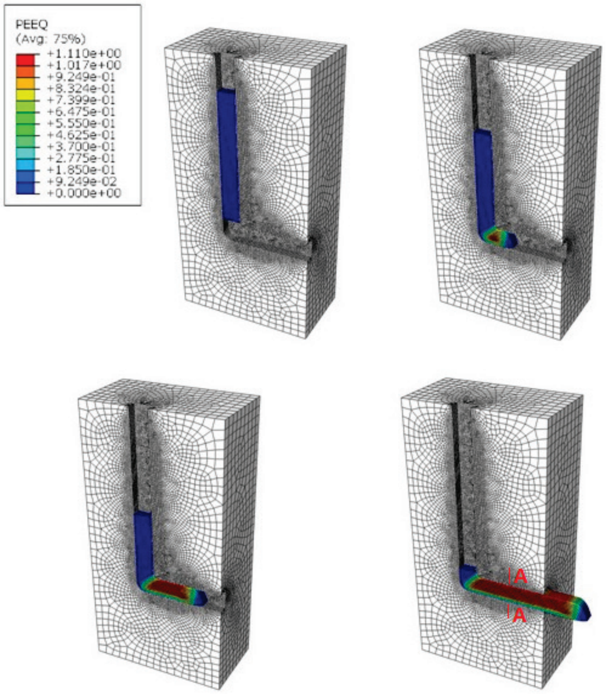
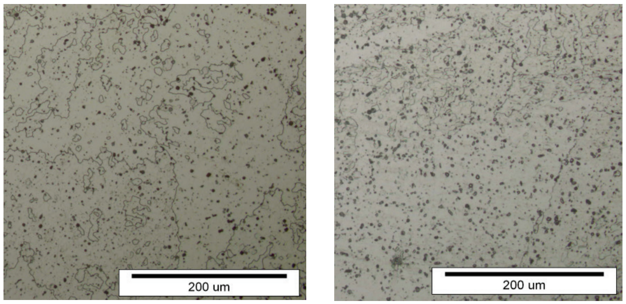
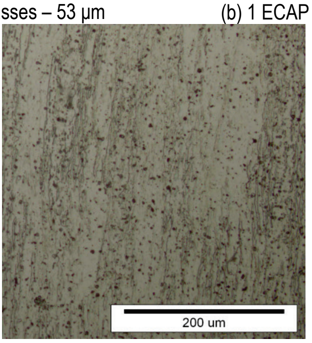


Figure 3 Equivalent plastic deformation after the first pass



(a) 0 ECAP passes – 53 μm (b) 1 ECAP pass – 21 μm



(c) 2 ECAP passes – 7 μm

Figure 4 Photo of cross section microstructure for three different deformation statuses. Grain size is calculated using the Linear Intercept Method.

Furthermore, besides changes in the crystal structure of the material, changes in mechanical properties have also occurred. The observed mechanical property was hardness, expressed as HV. It can be seen from the graph in Fig. 5 that the hardness after one ECAP pass has been increased by 50 % and after two ECAP passes by 110 % relative to the initial state of the material. Such hardness behaviour confirms the great influence of ECAP processes on the mechanical properties of the treated material.

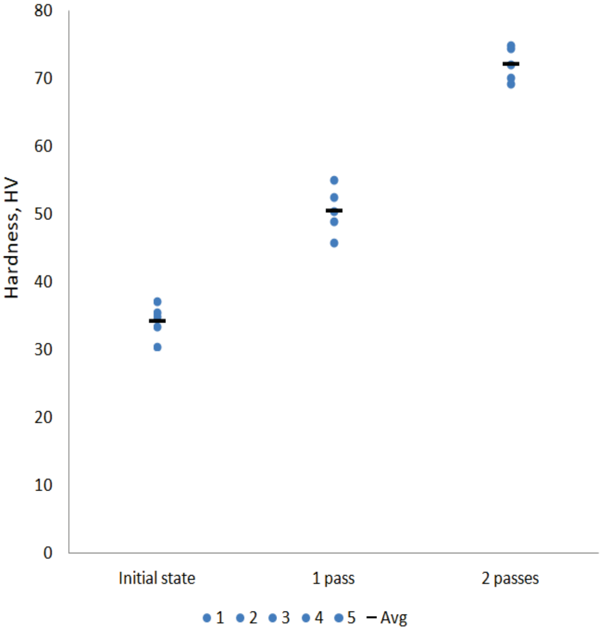


Figure 5 Hardness changes related to the number of ECAP passes

4 AFM INSPECTION OF CROSS SECTION

Dislocations on samples in origin state and after ECAP were detected using the AFM. All observations were taken on the surfaces with the largest deformation, marked as cross-section A-A (Fig. 3). Samples were prepared before scanning to adequate size 14 × 14 × 2 mm and polished using a 9 µm diamond paste. Surface analysis was conducted using the Oxford instruments model MFP-3D Origin atomic force microscope (AFM). AFM scanning was conducted using the AC mode (tapping mode). The tapping mode is a favourable inspecting mode for inspecting the materials whose surface is easily damaged. Contrary to the contact mode, where the tip is in constant contact with the surface, in the tapping mode the cantilever oscillates slightly below its resonant frequency causing lightly "taps" of the tip on the inspected surface.

The size of the investigated area spans 20 × 20 µm. Figs. 6, 7, 8, 9 and 10 show microstructures of aluminium samples. Also, reconstructed 3D images of surfaces for each sample are shown.

Arrows in the Figs. 6-10 mark dislocation areas. In the case of the initial state of the material that the dislocations are arranged relatively correctly within the structure. With the increase of the total deformation through one or two ECAP passes, the dislocations become broken and are irregularly distributed within the structure of the material. In the case of the two ECAP passes broken dislocations are pilled-up inside the small grain.

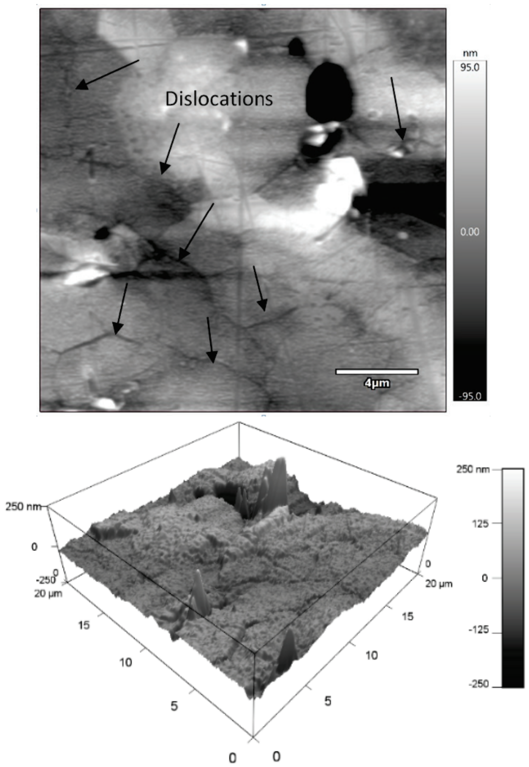


Figure 6 Microstructure of 99.5 % Al sample in origin state (before ECAP)

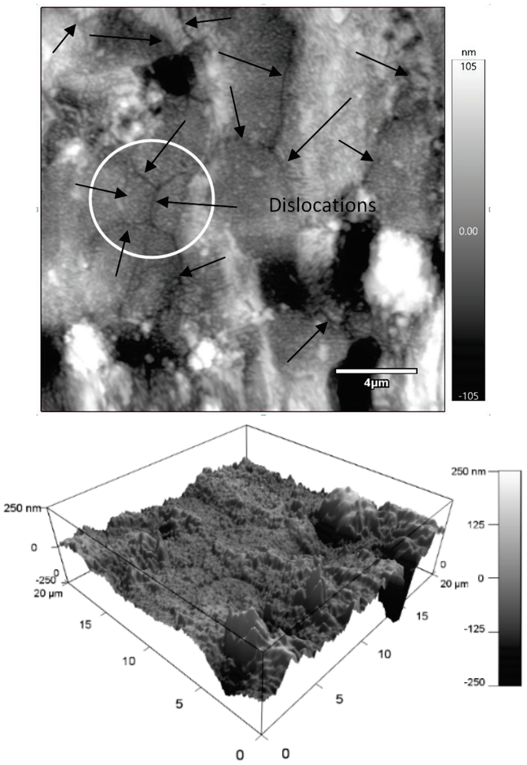


Figure 7 Microstructure of 99.5 % Al samples after 1 pass through ECAP

Since dimensions of the investigated area are 20 × 20 µm, the length and width are smaller than the average grain diameter of the initial material state. In the case of one ECAP pass the investigated area dimensions correspond to the average grain diameter, and in the case of the two ECAP passes the investigated area dimensions are three times the grain diameter. The interior of the grain is thus clearly shown. It should be considered that in Fig. 7 and 8, some of the grain boundaries must be located within the observed area, with significantly more grain boundaries in the case of the two ECAP passes – Fig. 9. Those grain boundaries relatively disturb the correct view on dislocations because dislocations are located and pilled-up within the crystal grain. A different type of magnification of the detail marked on Fig. 10 gives a better perspective into dislocation pile-up caused by the Severe Plastic Deformation.

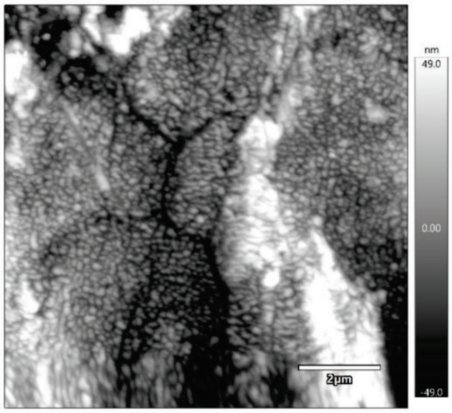


Figure 8 Detail from the microstructure of 99.5 % Al samples after 1 pass through ECAP marked with the white circle in Fig. 7 (note the different magnification)

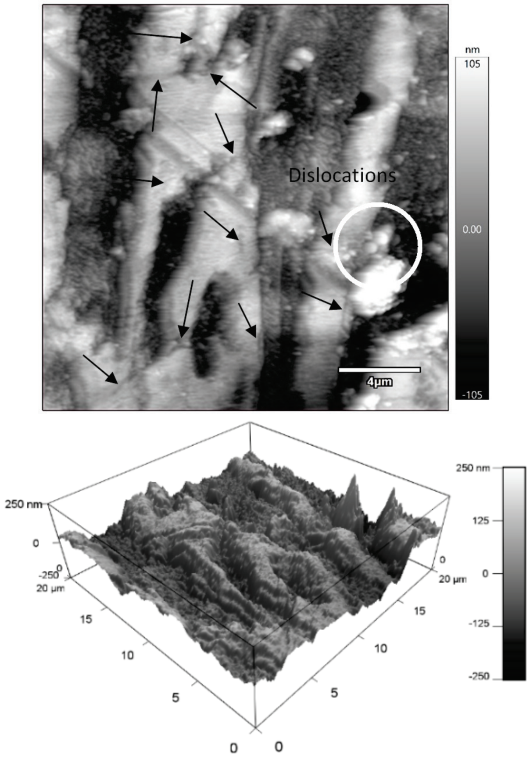


Figure 9 Microstructure of 99.5 % Al samples after 2 passes through ECAP

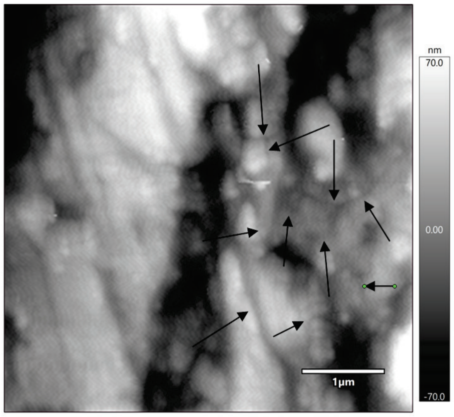


Figure 10 Details from the microstructure of 99.5 % Al samples after 2 passes through the ECAP marked with the white circle in Fig. 9 (note the different magnification)

To give a better visualization of the dimensions of individual structures encountered within the crystal structure, they are shown via diagram in Fig. 11. According to the diagram, the observed dislocations are within dimensions 10^{-5} - 10^{-8} m.

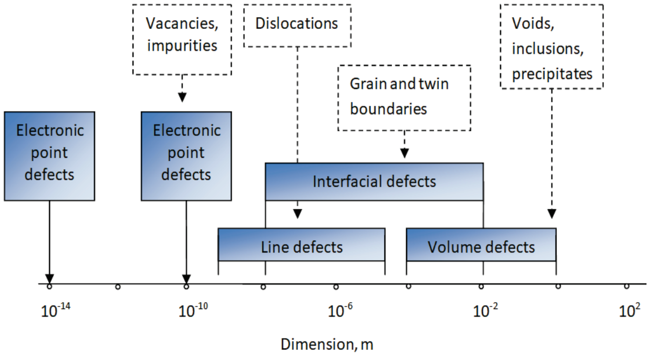


Figure 11 Dimensions of different lattice defects

5 CONCLUSION

The presence of dislocations can significantly modify the properties of crystalline solids. They are present in every crystalline material to a greater or lower extent. From the perspective of mechanical properties, the dislocation is the most influential crystalline defect. The greater the number of dislocations within the single grain, the greater the strength and hardness of the metallic material. Severe plastic deformation causes the diminishing of grain size, the increase of the number of dislocations and their accumulation within the smaller grain. In that way it influences the changes in mechanical properties of the material.

Through the presented research on the influence of the ECAP process on the structure of the aluminium Al 99.5 % material and consequently on its mechanical properties, the following can be concluded:

- After one ECAP pass the average grain size is 60 % smaller, and after two ECAP passes it is 87 % smaller than the initial grain size.
- The hardness after one ECAP pass has been increased by 50 % and after two ECAP passes by 110 % relative to the initial state of the material.

- The AFM observation showed that in the case of the initial state of the material the dislocations are arranged relatively correctly within the structure. With the increase of the total deformation through one or two ECAP passes, the dislocations are broken up and are irregularly distributed within the structure of the material. In the case of the two ECAP passes the broken dislocations are pilled-up inside the small grain.

Viewed from above it is clear that the ECAP process significantly changes and improves the structure and properties of the metallic material. Improving the mechanical properties of aluminium can extend its applicability in cases of demanding constructs as well as influence the final product cost.

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