The Formation of Grain Boundaries during Plastic Deformation of LiF Single Crystals

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ABSTRACT

The microstructural evolution of <100>-oriented LiF single crystals has been studied during plastic deformation at T=1073 K. It has been shown that plastic deformation leads to formation of low angle grain boundaries and their transformation to high angle ones. Further plastic deformation leads to the intensive grain boundary migration after some critical strain. The type of structural changes occurring in LiF single crystals is determined by the character of crystallographic slip. The reasons of critical strain existence and transition from "rotation "dynamic recrystallization to "migration" one are discussed.

INTRODUCTION

Plastic deformation of solid states leads to creation of new high angle grain boundaries [1]. This process is complex and consists of several stages. It is more convenient to analyse it by the example of an ionic crystal. At early stages of plastic flow networks of low angle grain boundaries are formed inside single crystal or initial grain interior. [1,2]. As strain increases smaller individual grains are formed by progressive misorientation of subgrains, without grain boundary migration [1,3,4]. This mechanism was called a "rotation "dynamic recrystallization (RDRX) [1]. In some cases in the region of high temperatures an extensive grain boundary migration starts after some critical strains (ε_{cr}=40-60%). This results in formation of coarse grains. This mechanism was called a "migration" dynamic recrystallization (MDRX) [1]. The transition from RDRX to MDRX was explained in the framework of the Lucke and Suwe theory for impurity-controlled grain boundary migration [1,3,5].

Due to this theory one can rather precisely calculate the temperature interval of MDRX occurrence. [3]. However, it turns out that to explain the existence of ε_{cr} is more difficult. The suggested MDRX mechanism [1] is based on classic concepts of grain boundary migration during deformation and is very simplified. The results of recent investigations [6] testify that changes in deformation mechanism should be taken into account while analysing changes of DRX types. Unfortunately, the lack of experimental data does not allow to understand how the processes of high angle grain boundary formation and migration are connected with the character of slip in the material. So far the features of high angle grain boundary array when MDRX starts are not clear yet.

The purpose of the present investigation is twofold: (i) to study the microstructure evolution during hot plastic deformation in its interrelation with the character of crystallographic slip; (ii) to estimate the characteristics of grain boundaries at stages of substructure formation, RDRX and MDRX. Lithium fluoride single clystal has been chosen for investigations.

MATERIAL AND EXPERIMENTAL PROCEDURE

Samples 5x5x8 mm of LiF single crystals (less than 4 p.p.m divalent impurities) were cleaved along {100} faces. Prior to deformation all the samples were annealed at 1073 K for 4-5 hours with subsequent slow cooling in a furnace. Finally, the samples were mechanically and chemically polished. Compression tests were conducted on an universal testing machine "Schenck RMS-100M" at a rate of $\dot{\varepsilon}$ =10^{-3} s^{-1} , at a temperature of 1073 K with further cooling in air. For examination of microstructure the samples were etched in 30% solution of fluoboric acid. The deformation relief was studied by means of a scanning electron microscope JSM-840, the microstructure - by optical microscopes "Metaval" and "Neophot- 32".

RESULTS

Mechanical Testing. Five stages of deformation can be distinguished on σ-ε curve (Fig.1) These stages are different in their range and strain hardening rate (Tab.1).

Table 1.

Strain hardening rate θ (MPa) at different strain ranges.

	Strain range				
	≤ 0.05	0.05-0.11	0.11-0.24	0.24-0.31	≥ 0.31
θ	2.3	5.4	1.2	3.9	1.3

The maximum magnitude of the strain hardening rate is observed at stage II. The further deformation up to

Fig.1. Stress-strain curve of LiF: T=1073K, $\dot{\varepsilon}=10^{-3}$ s^{-1}.

$\varepsilon=0.6$, excluding a short region at stage IV, is characterised by small continuos increase in material hardening. At the same time, the rate of strain hardening at stage V is lower than the same value at stage I by nearly twice.
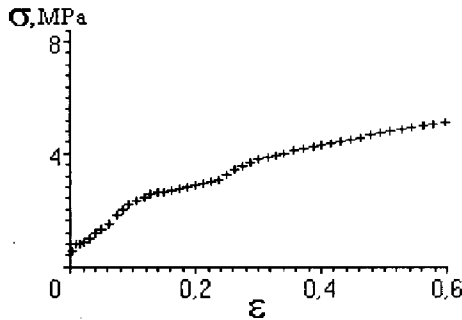
Microstructural changes. After ε =0.05, long range low angle grain boundaries are formed in single crystal of lithium fluoride along <110> direction (Fig.2a). These boundaries have been recognized after etching as long thick lines. They are non-uniformly arranged along the sample diagonal. Relatively short subboundaries locate along direction <$\bar{1}$10> at right angles to the long range boundaries. The original single crystal is subdivided into separate subgrains. The low angle grain boundaries in parallel with other crystallographic directions have been observed rarely. One should note that short low angle boundaries are resolved as strings of separate etch pits. Considerable dislocation density is observed inside subgrains.

An increase in strain up to $\varepsilon=0.11$ results in appearance of areas consisted of relatively fine equiaxed grains. Their size is approximately 30-40 μm. (Fig.2b, Tab.2). At present it is impossible to reveal a dominant orientation of their boundaries in relation to different crystallographic directions. Angles in triple junctions of these grains are close to 120°. Short boundaries with relatively low angles of misorientation are resolved near these boundaries (Fig.2c).

The other structural component is an area of coarse subgrains having mainly a right-angled shape and elongated along one of <110> directions. Angles in triple junctions are close to 90°. Dislocation walls subdivide them into small areas with a size close to that of fine subgrains.

At the strain interval $\varepsilon=0.11$-0.34 no qualitative changes of structure are observed. A volume of the first structural element increases while that of the second decreases. Thus, grains are of a circle shape or elongated along one of directions <110>, respectively. A mean size of crystallites decreases.

Table 2.

Mean size of structural elements and their size distribution (%) depending on strains; numerator - measurements along the axis of compression, denominator - tranverse to the axis of compression.

ε	Structural element size range (μm)								d
	≤ 16	16-22.4	22.4-32	32-44.8	44.8-64	64-89.6	89.6-128	128-201	μm
0.11	0.8/2.4	4.4/9.5	11.7/17.2	19.8/18.7	19.3/16.9	23.9/20	14.3/9	4.4/4.2	90.4/79.3
0.34	4.1/13.7	15.7/22.1	19.8/16.2	25.9/22.7	16.3/15.4	12.9/6.5	3.9/2.2	1.1/-	60.1/48.8
0.69	9.6/11.7	16.3/19.7	16.5/19.2	20.5/22.2	18.4/14.6	10.8/9.6	6/2.5	1.8/0.4	60.8/52.4

The number of crystallites with high angle grain boundaries increases during further deformation. After $\varepsilon=0.69$ a completely recrystallized structure has been formed. (Fig.2d). Individual dislocations and separate subgrains are revealed inside the crystallites. The average size of structural elements within the interval $\varepsilon=0.34$-0.69 is stable. (Tab.2). At the same time their morphology changes and elongation of grains in the direction of the material flow are observed.
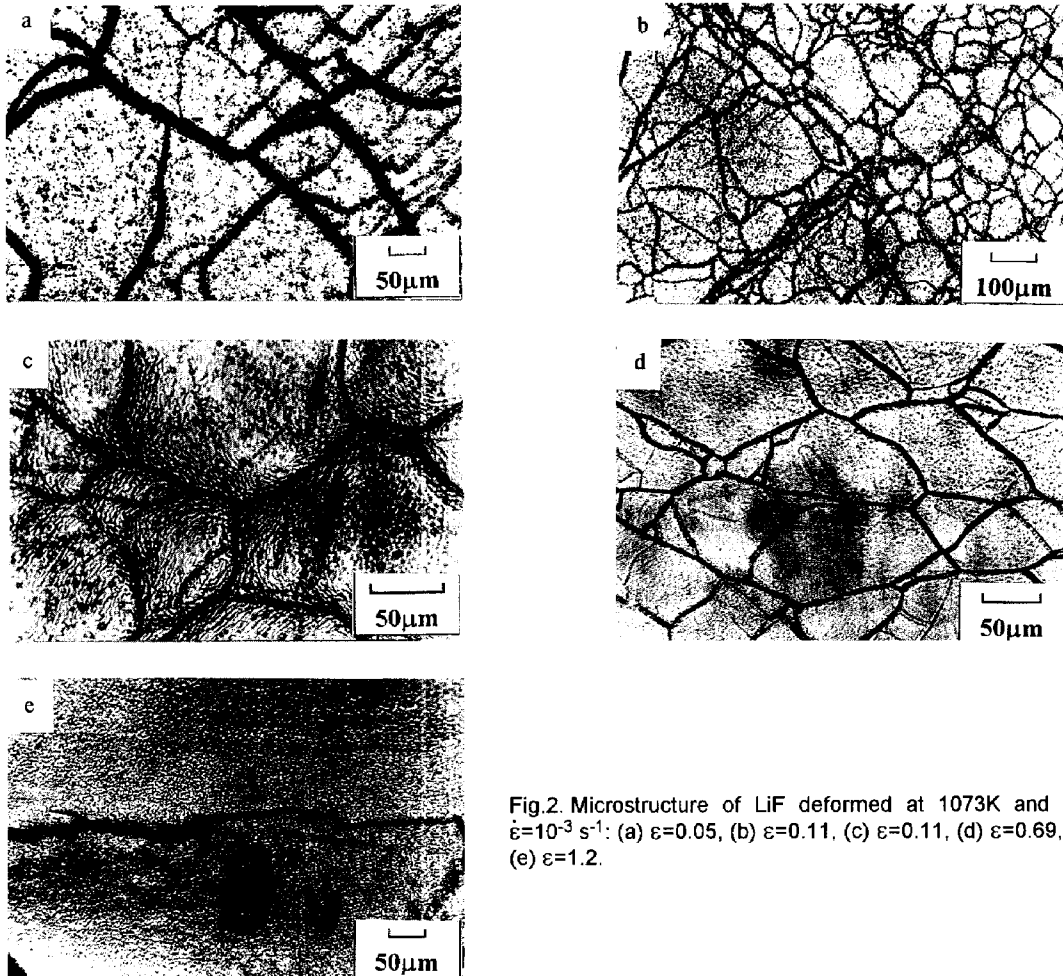
Fig.2. Microstructure of LiF deformed at 1073K and $\dot{\varepsilon}=10^{-3}$ s⁻¹: (a) ε=0.05, (b) ε=0.11, (c) ε=0.11, (d) ε=0.69, (e) ε=1.2.

After ε=1.2 the microstructure of LiF dramatically changes (Fig.2e). Within the body of the original single crystal only several grains are present. Their size is comparable with that of samples. There are ledges on boundaries of these grains. Local grain boundary bulging has been observed too. Relatively fine grains locate only in areas near the end of the sample and in some triple junctions. Bands consisting of a large number of etch pits are revealed inside coarse grains. This testifies that the grains were formed during deformation.

Deformation Relief. At the initial stage of deformation prevailing slip system <110>(110) acts. The fine features of this slip cluster in the bands. At the same time folds appear on the polished surface. Extensive cross-slip occurs near that at strain ε≥0.11 (Fig. 3a). As deformation proceeds, the number of slip bands and their width increases. Slip bands merge together. Simultaneously, the density of slip lines increases.

At ε=0.69 the slip develops along the system <110>(110). In the area with features of the primary slip system separate recrystallized grains mainly of a right-angled shape are observed. (Fig.3b) The presence of these grains causes a bend of the former deformation bands. A single slip occurs inside grains of an equiaxed shape.

DISCUSSION

The analysis of the results shows that in single crystals of LiF a type of microstructural evolution is mainly caused by the operation deformation mechanisms. The heterogeneous nature of slip is a reason of formation of a well-developed subgrain structure at the earliest stages of plastic flow [2]. Cooperative slip

causes the formation of twist subboundaries along slip lines. They are created by screw dislocations of like sign [2]. The appearance of transverse low angle grain boundaries may be attributed to alignment of edge components of these dislocations. However, it is more likely that these boundaries are formed by dislocations with other Burgers vector. The difference between the density of dislocations in longitudinal boundaries and that in transverse ones, corresponding to the difference in the number of slip lines formed at mutually perpendicular directions <110>, supports this conclusion.
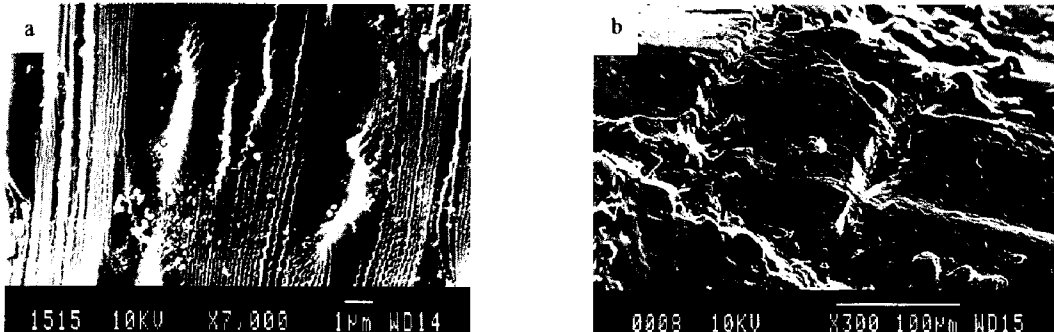

Fig.3. Deformation relief of LiF at 1073K and $\dot{\varepsilon}=10^{-3}$ s^{-1}: (a) $\varepsilon=0.11$, (b) $\varepsilon=0.69$.

The formation of coarse subgrains of a right-angled shape occurs at sites of deformation bands [2,7]. The increase in the width of slip bands and the density of slip lines leads to subdivision of coarse subgrains into fine ones. Migration of parallel subgrain boundaries leads to their recombination. If the boundaries contain a dislocation of like sign, the misorientation angle growth takes place. If they consist of dislocations of opposite signs, then they mutual annihilate [2]. Since the annihilation of boundary dislocations does not exceed 40% a steady growth of angles of misorientation is observed. This is the process of structural evolution from kink band-like arrangements of low angle boundaries to equiaxed subgrains. On the other hand, a formation of subboundaries from dislocations with other Burgers vectors is observed near such boundaries. The angle of misorientation of these latter subboundaries is less than 1° and due to migration they join boundaries with a higher misorientation [8]. Thus, the continuos growth of misorientation of separate low angle boundaries during deformation, that causes their transformation to boundaries of a common type during RDRX, occurs via the mechanism typical of materials with high SFE [8]. The growth of angles of misorientation increases the surface tension of boundaries. Consequently, crystallites acquire a more equilibrium shape with angles in triple junctions being close to 120°.

After $\varepsilon=0.69$ the final transformation of the subgranular structure to an equilibrium granular one takes place. Microscopic strain localization (at the slip line level) occurs in new grains. These two conditions are necessary for MDRX development and cause the existence of large critical strain for this type of DRX. Boundaries of some recrystallized grains with maximum misorientation and, consequently, maximum energy, evidently start extensive migration. To provide this it is also required that the direction of moving force P_p caused by the difference in the density of dislocations on both sides of the boundary should coincide with that of the moving force P_σ connected with the interrelation between dislocation pile-ups and this boundary [6]. The combination of all these conditions, as well as the specific features of the grain boundary structure in the ionic crystal causes an abnormally high rate of migration of some grain boundaries during hot plastic deformation.

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