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Fracture of $Gd_2\ (MoO_4)_3$ single crystals

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Fracture behaviour of $Gd_2\ (MoO_4)_3$ single crystal, which is improper ferroelectric–ferroelastic, has been examined by means of point loading. There are three fracture planes in gadolinium molybdat (GMO): {100} – main cleavage plane, {210} and {110} – secondary cleavage ones. It is shown that cracks have a tendency to transit from secondary planes up to cubic plane and vice versa. This would be considered as the main cause of river pattern appearance on fracture surfaces of samples. Mechanical twinning and crack growth are independent channels for relaxation of elastic energy in GMO, which do not connected between themselves, so excluding of twinning leads to increase of crack length. © *1999 Kluwer Academic Publishers*

1. Introduction

It is well known fact that plasticity prevents to growth of brittle cracks in crystals. Indeed, in thin metallic foil, dislocation emission leads to crack tip blunting and transition from sharp V-shape crack (brittle crack) to zigzag crack (ductile crack) [1]. Plastic deformation ahead notch in bulk samples of cubic metals does not allow it to advance as brittle crack, too [2]. On the other hand, slip and twinning near crack edges only accompany crack growth in brittle metals [3]. No deformation tracks have been observed near cracks in majority of non-metallic crystals, inasmuch as they are not deformed ever at temperatures higher then their temperatures of brittle-to-ductile transition [4]. However, it does not mean that deformation processes could not repress or brake growth of cracks in these materials. Fracture behaviour of non-metallic crystals, which are plastically deformed, is examined in this work. Gadolinium molybdat has been chosen as model material, because mechanical stress induces mechanical twins in it* [5].

2. Experimental

Crystal of $Gd_2\ (MoO_4)_3$ (GMO) represents a transparent substance, which is improper ferroelectric–ferroelastic possesses cubic-type lattice [6]. Mechanical stresses could induce an appearance of ferroelectric domains in the material. In polarised light domain structure looks like alternation of dark and bright strips, whose width is about some hundreds micrometers and length is compared with crystal size. Axis of domains is directed along ⟨110⟩ crystallographic direction. The mechanism of ferroelectric domain forming is similar to mechanical twinning [5]. It should be noted that GMO crystals cleave in ⟨110⟩ direction on {100} plane. Samples for tests have been cut from large crystals grown by Czochralski method. The plane of their working surfaces is normal to polar axis. They have a shape of parallelepiped with grains lie on cubic plane and edges are parallel to ⟨110⟩. Size of them is about $5 \times 2 \times 1$ mm. Before the test single crystals were mechanically polished. Diamond pastes have been used as abrasive. One set of samples is in multidomain state (MDS), while another set contains crystals in single domain state (SDS).

Fracture behaviour and deformation processes in GMO crystals are studied by means of point loading [7]. Light microscope equipped by Vickers microhardness device has been used in this work. Design of the facility allows rotating the diamond pyramid around its axis on 360°. The initial position of pyramid and crystal is when diagonals of indent are parallel to ⟨110⟩ direction. The angle between diagonals and ⟨110⟩ in contra-clock-watch direction will be denoted as angle of pyramid turn. The following angles are considered: 0°, 5°, 10°, 15°, 20°, 30°, 45°, 60° and 90°. Twelve indents (four ones in row and three ones in column) are inflicted on samples through equal distance between neighbours (3, 4 or 5 diagonals of indent) for every angle of turn. All indents are documented with a help of optical microscope. Distributions of both cracks and deformation tracks around indents are studied on photographs. At the first step, an angle between crack direction and axis ⟨110⟩ has been measured, after that crack plane is defined from crystallographic table [8]. Fracture surfaces of samples are examined on light microscope. Experimental data are represented in the table.

3. Results

Measurements have shown that microhardness of crystals in MDS continues to be a constant for all angle of pyramid turn ($\sim 3000 \pm 200$ MPa). Distributions of cracks and twins around indents do not depend on the

* Of course, its plasticity (<1%) is considerably lower in compare with metals.

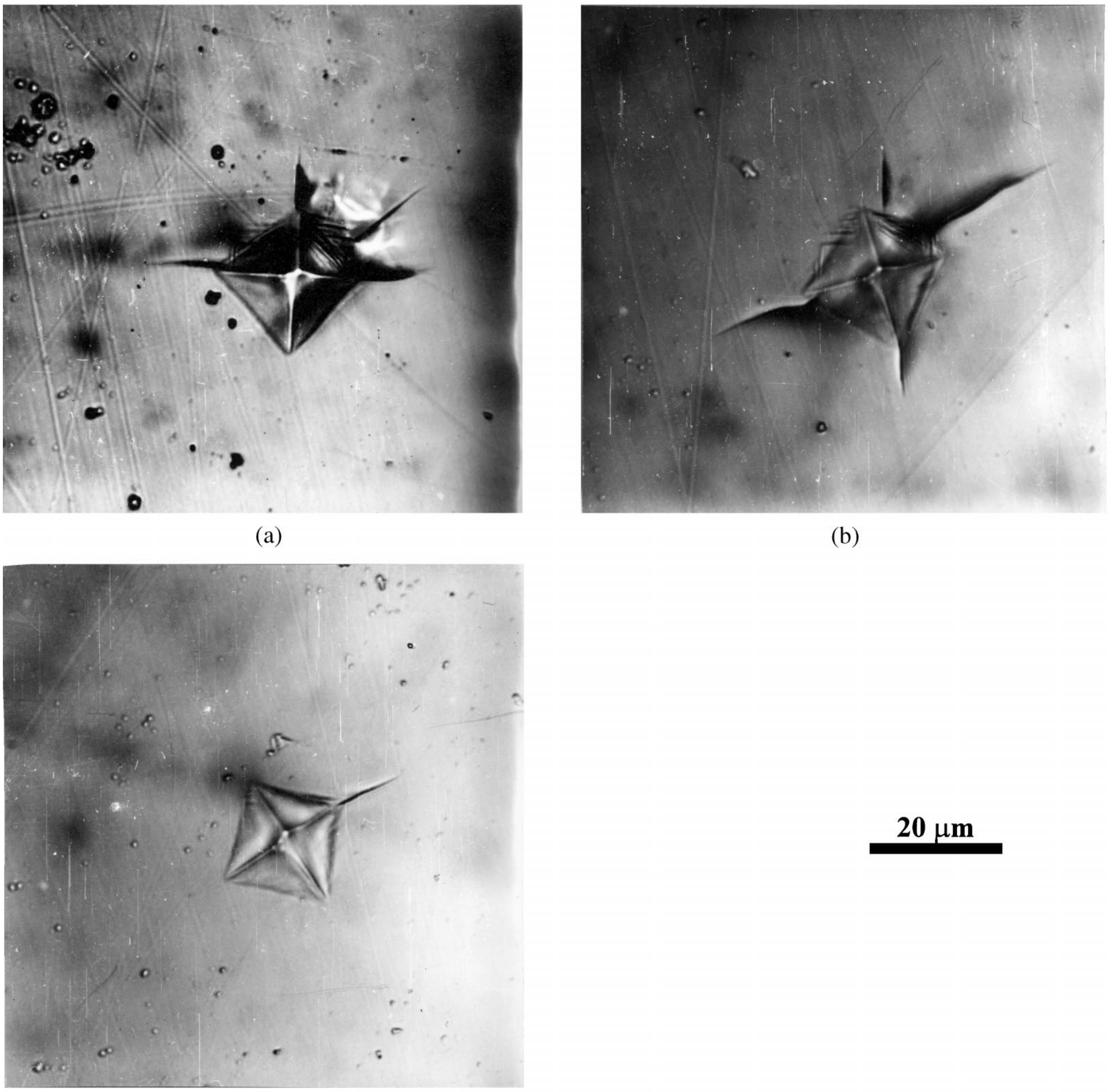

Figure 1 Microphotographs of indents in GMO single crystal at MDS: (a) angle of pyramid turn 0°; (b) 15°; (c) 30°.

distance between them. At initial position of pyramid (angle of 0°) cracks and deformation tracks are observed near indents (Fig. 1a). As a rule, cracks lie in ⟨110⟩ direction on cubic plane, but some of them could advance on {210} or {110} planes. Their average length is about 10 μm for all cleavage planes. Majority of cracks has V-shape with a sharp tip. There are few transitions of individual cracks from {100} onto {210}, when resulted crack possesses broken profile. Deformation tracks near indents look like twin lamellas having size of 6–8 μm in length and 1 μm in width. Their axis coincides with ⟨110⟩ direction, but they always direct normally to the nearest neighbour crack.

At turn of 15° fracture behaviour of GMO does not change. The main plane of crack growth continues to be {100}, although the number of cracks advanced in cubic plane is less than for previous case. Secondary cleavage planes are activated, too. The only thing that can change is the shape of cracks, which becomes undulating (Fig. 1b). Profile of twin lamellas is also changing, but this circumstance does not influence on their quantity and size. Drastic lowering of twin number up to zero takes place at turn of 30° and 60° (Fig. 1c). Simultaneously, the quantity of cracks in cubic plane decreases from few dozens up to 3–5 pieces. Majority of cracks begins to advance in the secondary cleavage plane {210}. It should be noted that average crack length does depend on neither cleavage plane nor angle of pyramid turn.

Crystallographic attestation has shown that macroscopic fracture plane is parallel to {100}, although microscopic relief is clearly visible here. There are two kinds of relief in every MDS crystals. The first type would be estimated as a brittle intercrystalline fracture (BIF) (Fig. 2a). It is well known that dangerous crack, whose growth leads to failure of sample, could advance along ferroelectric domain boundary [5]. The second type is a brittle transcrystalline fracture (BTF), so river patterns are observed on fracture surface (Fig. 2b). Ratio between the areas with BIF and BTF for GMO crystals is approximately 1 : 5.

In contrast to described above, microhardness of GMO crystal in SDS begins to decrease at turns on more than 20°: this value changes from 3000 MPa at

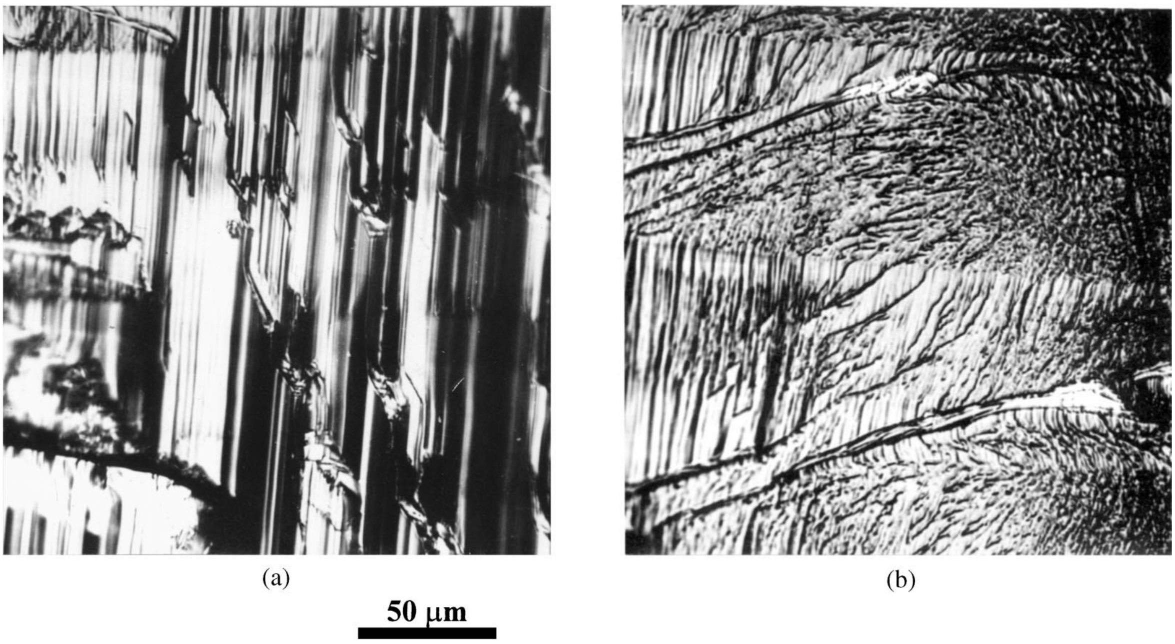

Figure 2 Fracture surface of GMO single crystal in MSD: (a) area of brittle intercrystalline fracture (BIF); (b) area of brittle transcrystalline fracture (BTF).

0–20° up to 2600 MPa at 45°. Simultaneously, number of twin lamellas near indents falls from 262 pieces at 0° up to 15 ones at 45°. Total quantity of cracks is also lowering from 70 pieces at 0–10° up to 50 pieces at 20–60°. For angle of turn 0°, average length of cracks in cubic plane is more than crack length in secondary cleavage planes (see Fig. 3a). Their number is order to five dozens, while number of cracks in {210} is twenty. In opposite to MDS, cracks do not advance in {110}. Also, there are equal number of transitions of cracks {210} → {100} and back. More then 250 lamellas are observed near dozen indents. For previous case, their number was about one hundred. No differences are revealed in shape and size of twins in compare with MDS.

Turn of pyramid at 5° does not lead to changing of fracture behaviour of GMO crystal (Fig. 3b). Crack length in {210} grows up in two times (Fig. 3c) and number of twin decreases in two times at turn of 20°. In so doing, no transitions of crack {100} → {210} are observed anymore. Numbers of cracks in {100} and {210} stand equal to twenty pieces at 30° and 60°, while quantity of twins is lowering in two times. Average length of cracks becomes 20 μm, including cracks in {110} (Fig. 3d). At angle of 45°, majority of cracks appears on {110} plane. They are long cracks with length of 26 μm (Fig. 3e). Also, new transition of cracks from {110} to {210} is registered. Probabilities of cracking in cubic and {210} planes are equal, but cracks in {100} are longer. It would be noted that no twinning near indents is observed. BTF is the sole fracture mode for GMO crystals in SDS. In this case characteristics of cracks and twins do not depend on the distance between indents, too.

4. Discussion

Experiments have shown that GMO single crystal has three crystallographic planes where cracks could advance. Cubic plane is the main cleavage plane, since majority of detected cracks lies here. {210} and {110} are the secondary cleavage planes, which could be only activated at suitable orientations of diamond pyramid. Probability of the event that crack will grow on {210} is greater then probability of crack growth on {110}. Cracks have a tendency to transit from secondary cleavage planes to the cubic plane. It should be noted both crack lengths decrease in the limits of population standard deviation and twin lamellas stop to appear around indents for crystal in MDS at turn of 30° or 60°. Simultaneously, microhardness of sample grows up on 10% in compare with its average mean. For SDS number of twins around indents is lowering at angles greater then 15°, too. However, lengths of cracks on cubic plane increase at angles of 30° and 45° up to 25% and 50%, respectively, while cracks on secondary cleavage planes begin to arise at 20° up to 200%. Microhardness of crystals in SDS at 30°, 45° and 60° is less then its average mean on 15%. It would be concluded that strengthening of materials leads to decrease of crack lengths at big angles for MDS and at small angles for SDS.

Both macroscopic fracture plane and crack growth direction do change neither in the case of BIF nor in BTF. In general, the plane of mechanical twin or ferroelectric domain does not coincide with {100} for crystal having cubic lattice [8]. Therefore, area of BIF should appear on fracture surface of crystal in MDS when domains intersect the cubic plane (it is absent for crystal in SDS). Otherwise short river patterns are the main feature of fracture surfaces. Crack growth on the secondary cleavage plane would be considered as the main cause of river pattern creation. Indeed, crack could deviate from {100} on to {210} or {110}, but its growth on these planes is limited by few dozens of micrometers. After that crack returns on to cubic plane. As a result river pattern appears on fracture surface.

Small angles of turn are suitable orientations of diamond pyramid for inducing of mechanical twinning

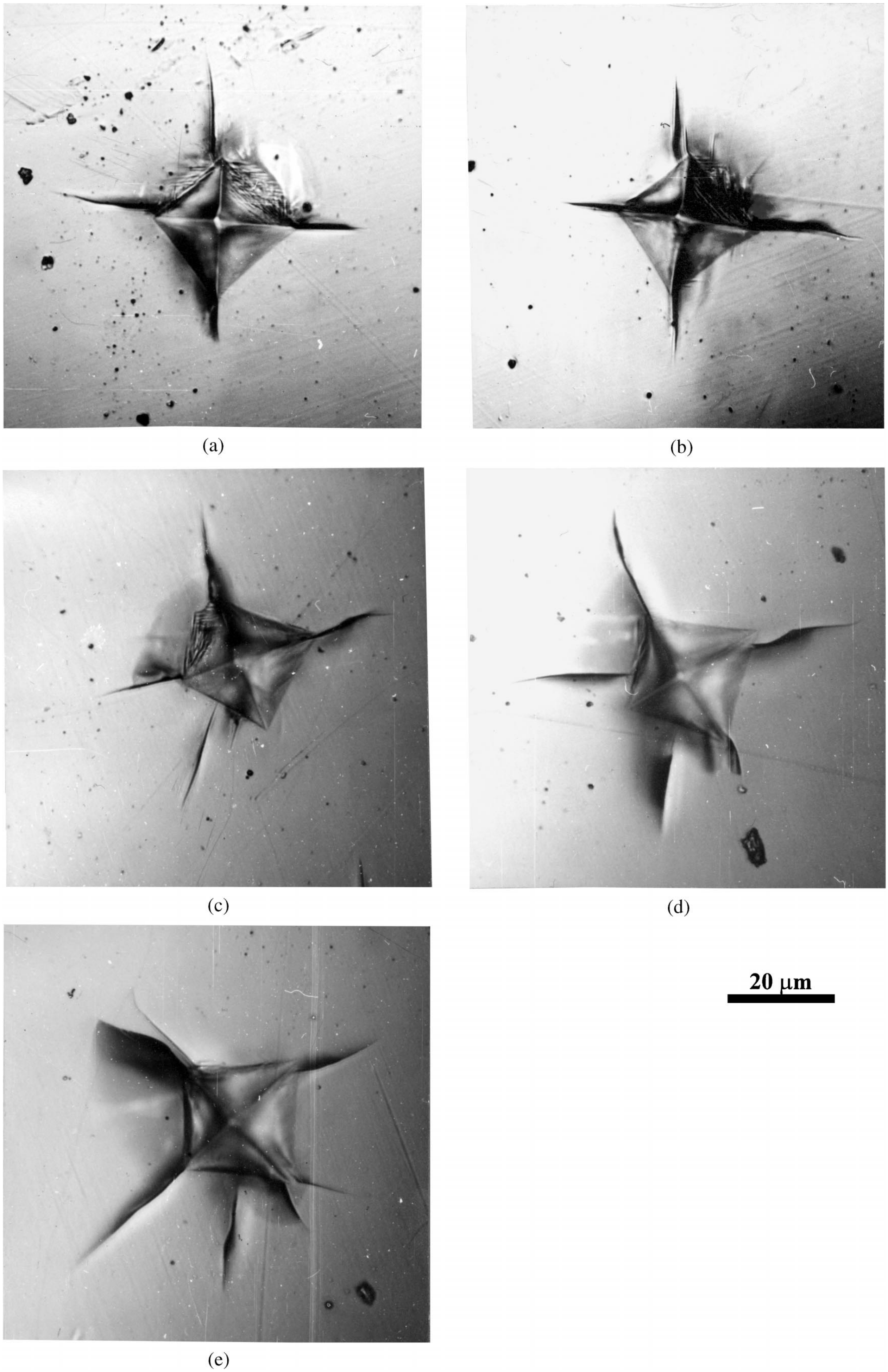

Figure 3 Microphotographs of indents in GMO single crystal at SDS: (a) angle of pyramid turn 0°; (b) 5°; (c) 20°; (d) 30°; (e) 45°.

TABLE I Storage of cracks and twins near indents in $Gd_2(MoO_4)_3$ single crystals

				Fracture plane						Transition of cracks from plane to plane					
				(100)		(210)		(110)		(210) → (100)		(110) → (210)		(100) → (210)	
Crystal	Angle of pyramid turn (°)	Micro-hardness (MPa)	Number of twins*	Number of cracks*	Length (μm)	Number of cracks*	Length (μm)	Number of cracks*	Length (μm)	Number of cracks*	Length (210) → Length (100) (μm)	Number of cracks*	Length (110) → Length (210) (μm)	Number of cracks*	Length (100) → Length (210) (μm)
Multi-	0	3000 ± 200	112	47	12 ± 5	6	11 ± 4	6	10 ± 6	—		—		3	9 ± 2 → 5 ± 2
domain	15	2900 ± 200	117	31	12 ± 3	6	14 ± 8	3	—	—		—		—	
state	30	3300 ± 200	—	3	—	15	9 ± 5	3	—	—		—		—	
	0	3100 ± 200	262	53	15 ± 6	20	9 ± 6	—	—	6	6 ± 4 → 11 ± 7	—		5	12 ± 6 → 9 ± 7
Single	5	3100 ± 200	239	52	16 ± 6	13	8 ± 3	—	—	3	5 ± 2 → 20 ± 4	—		4	12 ± 1 → 7 ± 1
domain	20	3000 ± 200	94	38	18 ± 7	14	22 ± 8	—	—	5	15 ± 3 → 17 ± 8	—		—	
state	30	2800 ± 200	42	23	20 ± 10	24	22 ± 10	4	—	3	15 ± 6 → 9 ± 1	—		—	
	45	2600 ± 200	15	15	26 ± 8	15	17 ± 10	27	26 ± 15	—		5	17 ± 6 → 8 ± 3	—	

$X \pm \sigma n$, where x is arithmetical mean; σ_n is population standard deviation; * is quantity per 12 indents.

around indents (see Table I), but the lengths of lamellas do not depend on magnitude of angle. No events have been observed when twins are leaving from crack edges or crack tips. This gives the base for conclusion that mechanical twinning does not accompany crack growth or cracks do not generate twins in the crystal, as it takes place in metallic materials [3, 9]. Consequently, mechanical twinning and cracking should be considered as independent and competing channels for relaxation of elastic energy in GMO. It means that removing one of them leads to the rising intensity of relaxation through another channel. Indeed, at big angles in SDS crack length increases in 50–100%, while quantity of twins falls up to 10–20 times. For MSD at big angles of pyramid turn, cracking on secondary cleavage plane {210} becomes the sole channel for relaxation of stresses, when mechanical twinning and cracks on cubic plane are absent.

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