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Dislocation distribution in ruby single crystals under loading at high temperatures

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Evolution of dislocation structure in ruby single crystals under four-point bending at $T = 1490°C$ has been studied using the etch pit method. The small-angle substructure boundaries have been observed, the dislocation types have been identified and their densities have been measured. The screw and edge dislocation density has been shown to be distributed symmetrically about the sample neutral axis [1100]] and about the [0001] one. The starting shear stress has been first measured and found to be 200 MPa in the prismatic slip system. This value is associated with the friction stress of the crystal lattice, that is, with the Peierls-Nabarro stress value in the $(1\bar{1}00)$ prism plane along the [1120] direction. The starting stress value is to be known to treat thermomechanically high-perfection ruby single crystals and to solve the problem of long-term laser stability.

Методом ямок травления исследовалась эволюция дислокационной структуры в монокристаллах рубина при 4-х точечном изгибе при температуре $T = 1490°C$. Выявлены субструктурные малоугловые границы, идентифицированы виды дислокаций и измерены их плотности. Показано, что плотность винтовых и краевых дислокаций распределяется симметрично относительно нейтральной оси образца $[1\bar{1}00]$ и относительно оси [0001]. Впервые измерена величина сдвигового стартового напряжения, которая в системе призматического скольжения оказалась равной 200 МПа. Эта величина связывается с напряжением трения кристаллической решетки, то есть с величиной напряжения Пайерлса-Набарро в плоскости призмы $(1\bar{1}00)$ в направлении $[11\bar{2}0]$. Отмечается, что величина стартового напряжения необходима для использования термомеханической обработки высокосовершенных монокристаллов рубина и решения проблемы долговременной работоспособности лазеров.

Ruby single crystals are used widely in the laser production [1, 2]. Since the ruby laser long-term operating stability depends heavily on the structure perfection of the ruby crystals [3, 4], a need arises in the production of high-perfection ruby single crystals by using the optimum temperature and strain conditions in their thermal and mechanical treatment. The preliminary studies aimed at the selection of optimum temperature and strain conditions of thermal and mechanical treatment carried out using the modeling zinc single crystals (published in part in [5]) have shown a substantial improvement in the crystal structure perfection due to thermomechanical treatment. Thus, the mean dislocation density was reduced by three decimal orders as compared to the untreated single crystals. The small-angle substructure boundaries become destroyed and leave the crystal due to thermomechanical treatment. These experi-

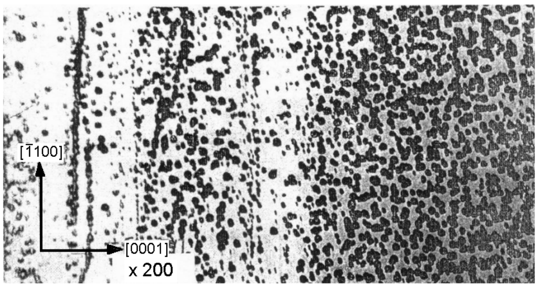

Fig. 1. Etch pattern of the sample (11$\bar{2}$0) plane after loading, ×200.

ments made it possible to establish the necessity and sufficiency of the selected temperature and force conditions of the thermomechanical treatment. To solve the long-term operating stability problem of the laser, the high-perfection ruby single crystals are required. To obtain such crystals, the evolution of the crystal structure state at high temperatures under loading should be studied. It is just the purpose of this work.

The laser ruby single crystals of high optical homogeneity were grown from the (Al_2O_3 + 3 % CrO_3) raw material using the modified Verneuil technique by controlling technologic regimes during growing [6]. The samples were cut out using a diamond saw. Those were shaped as rectangular parallelepipeds of 90×15×2 mm^3 size with faces corresponding to the basis plane (0001) and to the prism ones with longitudinal axis directed along [1$\bar{1}$00]. The samples were loaded by four-point bending about the [11$\bar{2}$0] axis at the temperature of 1490°C. The load application points were positioned on the upper and lower (0001) planes of the sample. The structure state was studied using the selective chemical etching of the basis and prism planes as described in [7]. It is worth to note that the sample surface should be prepared thoroughly prior to etching. This can be provided by the surface grinding and polishing using special laps.

The structure state of the initial (unloaded) ruby samples is characterized by the average density of prismatic dislocations $\bar{\rho}_p = 5\cdot10^5$ cm^{-2} and that of basis ones $\bar{\rho}_b = 10^6$ cm^{-2}. The small-angle twisting boundaries are formed with the linear density of screw dislocations $\bar{\rho}_s = 3\cdot10^3$ cm^{-1} and the slope ones with linear density of edge dislocations $\bar{\rho}_s = 1.5\cdot10^3$ cm^{-1}. The dislocation net of twisting boundaries is formed due to the reaction

$$\frac{1}{3}[2\bar{1}\,\bar{1}0] + \frac{1}{3}[\bar{1}2\,\bar{1}0] + \frac{1}{3}[\bar{1}\,\bar{1}20] = 0. \quad (1)$$

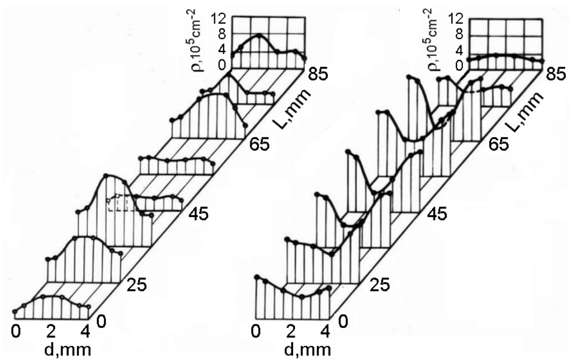

Fig. 2. Distribution of screw and edge dislocations along the sample neutral axis [1$\bar{1}$00] and along the [0001] axis.

This reaction is energy-favorable.

The ternary dislocation nodes hinder the displacement of the screw basis dislocation net and retard the dislocation rearrangement during the sample loading. Fig. 1 presents the etch pattern of the prism (11$\bar{2}$0) plane of a ruby sample after the loading is applied. A substantial redistribution of the dislocation structure is observed as compared to the above-mentioned initial structure state. The maximum dislocation density is seen to be near the upper and lower (0001) planes of the sample symmetrically about the neutral [1$\bar{1}$00] axis. It is to note that, at the selected crystallographic orientation and the loading geometry, there are essentially no shear stresses in the easy basis slip system (0001)[11$\bar{2}$0] while in the prismatic slip system (1100)[1120], substantial components of shear stresses act, thus forming the sample structure state.

Consideration of the etching pattern crystal geometry on the (1$\bar{1}$00) plane makes it possible to identify the dislocation type, to measure the density $\bar{\rho}$ values for each type and the distribution character with respect to the sample neutral axis. In Fig. 2, presented are the edge and screw dislocation densities along the longitudinal axis as functions of the coordinate perpendicular to that axis. For the dependences obtained, that coordinate is directed along [0001]. The $\rho(L)$ plots demonstrate the symmetric distribution of dislocation density with respect to the sample neutral axis. The dislocation density decrease along the [0001] direction characterizes the decreasing shear stresses from the maxima at the upper (stretched) (0001) and the lower (compressed) (0001) sample surfaces down to a minimum ion the sample middle. The results obtained allow us to measure the fundamental characteristic of dislocations, that is, the starting stress for their motion. In ideal crystals, this stress answers to the crystal lattice resistance, that is, to the Peierls-Nabarro stress, while in real crystals, it can be the value of local fields of internal long-range stresses resulting from the dislocation accumulations. In our case, the dislocations in the $(10\bar{1}0)[\bar{1}2\bar{1}0]$ slip system move in the variable field of shear stresses from the sample upper and lower surfaces towards its neutral axis and stop near it, since the stress applied is equal to the internal one, that is, the starting one. To estimate the normal stresses at the dislocation stopping point, let the following expression be used [8]:

$$\sigma_y = \frac{6 \cdot P \cdot K}{a \cdot h^3} \cdot y \approx 11.0 \ \ MPa, \tag{2}$$

where $P = 5$ kg is the load applied; $K = 15$ mm, the distance between the loading points at the (0001) sample planes; $a = 15$ mm, the sample width; $h = 2$ mm, the sample height; $y = 0.3$ mm, the distance from the sample neutral axis to the dislocation stopping point.

Taking into account the sample crystal geometry, the starting shear stress is evaluated as

$$\tau_c = \sigma_y \cdot \cos\psi \cdot \sin\varphi \approx 3.5 \ MPa, \tag{3}$$

where ψ is the angle between the shearing direction $[11\bar{2}0]$ and the sample neutral axis; φ, that between the prism slip plane $(1\bar{1}00)$ and the sample neutral axis.

It is seen from the etch pattern (Fig. 1) that in the dislocation stopping point near the sample neutral axis $[1\bar{1}00]$, there are no sources of internal long-range stresses, as the dislocation accumulations or small-angle subboundaries. Therefore, the measured value of the starting shear stress can be related to the friction stress of the crystal lattice, that is, to the Peierls-Nabarro stress in the prism plane $(1\bar{1}00)$ and the $[11\bar{2}0]$ direction. A comparison of the τ_s value with the critical shear stress in the prismatic slip system determined macroscopically using strain curves [9] shows that the latter exceeds τ_s by several times. Note that the starting shear stress measurements are believed to be an effective experimental method to determine the crystal lattice resistance against the dislocation motion in defect-free single crystals. Thus, the starting shear stress measurement and determination of dislocation distribution under loading of ruby single crystals at high temperatures make it possible to determine the optimum parameter values of the crystal thermomechanical treatment in order to improve its structure perfection and to solve the problem of the laser long-term operating stability.

To conclude, the structure state evolution in ruby single crystals under four-point loading (pure bending) at $T = 1490°C$ has been studied. The regularities of dislocation distribution with respect to the sample neutral axis have been determined and the symmetric arrangement along [0001] has been established. The starting shear stress for the dislocation motion in the prismatic slip system $(1\bar{1}00)[11\bar{2}0]$ has been first measured. The selection criteria of optimum parameter values for the crystal thermomechanical treatment in order to improve its structure perfection and to provide the laser long-term operating stability have been considered.

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Розподіл дислокацій у монокристалах рубіну при високотемпературному навантаженні

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Методом ямок травлення досліджено еволюцію дислокаційної структури у монокристалах рубіну при чотирьохточковому вигині при температурі T = 1490°C. Виявлено субструктурні малокутові границі, ідентифіковано види дислокацій та виміряно їх густини. Показано, що густина гвинтових та крайових дислокацій розподіляється симетрично відносно нейтральної осі зразка [$1\bar{1}00$] та відносно осі [0001]. Вперше виміряно величину стартової напруги зсуву, котра у системі призматичного ковзання виявилася рівною 200 МПа. Ця величина пов'язується з напругою тертя кристалічної гратки, тобто, з величиною напруження Пайєрлса-Набарро у площині призми ($1\bar{1}00$) у напрямі [$11\bar{2}0$]. Величина стартової напруги є необхідною для застосування термомеханічної обробки високодосконалих монокристалів рубіну та для вирішення проблеми довготривалої працездатності лазерів.