Experiments on the Investigation of the Breakdown of NaCl Crystals

N. M. MELANKHOLIN AND V. R. REGEL'
Institute of Crystallography, Academy of Sciences, USSR
(Submitted to JETP editor June 14, 1954)
J. Exper. Theoret. Phys. USSR 29, 817-821 (December, 1955)

The development of cracks during the stretching of specimens of NaCl monocrystals was studied by the method of cinematography in polarized light. It was shown that the cracks spread intermittently. The development of the cracks is prevented by the shearing bands which they have to cross. It is assumed that the propagation of the cracks is prevented by the regions of the shearing bands in which compression stresses are present. Regions of shearing bands having stretching stresses apparently do not prevent their development.

A CONSIDERABLE number of investigations have been devoted to the study of the processes of shear origination and the breakdown of NaCl monocrystals. Of these investigations, the studies of Stepanov and his co-workers[1-7] are of great importance. In these studies the process of the artificial origination of shear and that of the appearance of cracks was investigated. However, the main conclusions regarding the relationships of the appearance and of the growth of cracks in crystals were arrived at by Stepanov on the basis of indirect experiments[8-10], and the data of direct observations are cited by him only in one investigation[4], and these are limited to a small number of experiments. On the basis of his investigations, Stepanov arrives at definite conclusions regarding the relationship between the appearance of cracks and shear-origination, stating that the formation of shear facilitates the origination of cracks. The effect of plastic deformation on the development of an already initiated origin of a crack hardly received any attention in these investigations. In the present investigation we are continuing the studies of Stepanov on the phenomena of the breakdown of NaCl monocrystals by direct observations of the origination and growth of cracks. Observations of shear-origination were also carried out simultaneously. The results of the latter observations will be published in another paper.

The growth of cracks was observed on specimens subjected to a gradually increasing stretching tension. The direct observation of the stretched specimens under a microscope in polarized light using crossed polaroids was later replaced by microcinematography under the same conditions. The specimens studied were of rectangular cross section having an area of 15-30 mm^2 and a length of 40-50 mm, and were spalled along the cleavage planes from natural crystals of rock salt of the Artemov region.

All the specimens studied were subjected to an initial anneal, which was carried out in accordance with the following schedule: first the specimens were heated gradually for two hours in a muffle furnace up to a temperature of 600° C. They were maintained at this temperature for 2-2½ hours, then gradually cooled for 4-5 hours, down to a temperature of 200° C. After this the furnace was disconnected. As a result of such an anneal, the primary shear bands (present in the nonannealed specimens and clearly visible with crossed polaroids) disappeared or became hardly noticeable. The annealed specimens were washed with water so as to remove the surface defects as much as possible.

Copper foil terminals were glued with BF-2 glue to the ends of the specimens. These terminals were used to secure the specimens to the ends of a self-centered suspension device. As shown by our experiment, such a simple device of attaching the terminals was found to be very reliable and

---

1 A. V. Stepanov, J. Exper. Theoret. Phys. USSR 17, 606 (1947).

2 A. V. Stepanov, J. Exper. Theoret. Phys. USSR 18, 741 (1948).

3 A. V. Stepanov and E. A. Mil'kamanovich, J. Exper. Theorèt. Phys. USSR 18, 773 (1948).

4 A. V. Stepanov, J. Exper. Theoret. Phys. USSR 18, 776 (1948).

5 A. V. Stepanov and E. A. Mil'kamanovich, J. Exper. Theoret. Phys. USSR 21, 401 (1951).

6 A. V. Stepanov and E. A. Mil'kamanovich, J. Exper. Theoret. Phys. USSR 21, 409 (1951).

7 A. V. Stepanov, Izv. Akad. Nauk SSSR, Ser. Fiz. 17, 342 (1953).

8 A. V. Stepanov, J. Exper. Theoret. Phys. USSR 7, 1168 (1937).

9 A. V. Stepanov, J. Exper. Theoret. Phys. USSR 7, 663 (1937)

10 A. V. Stepanov, Izv. Akad. Nauk SSSR, Otd. Matem. estest. nauk 6, 797 (1937).

can be recommended for use not only with NaCl crystals, but also with crystals of other substances.

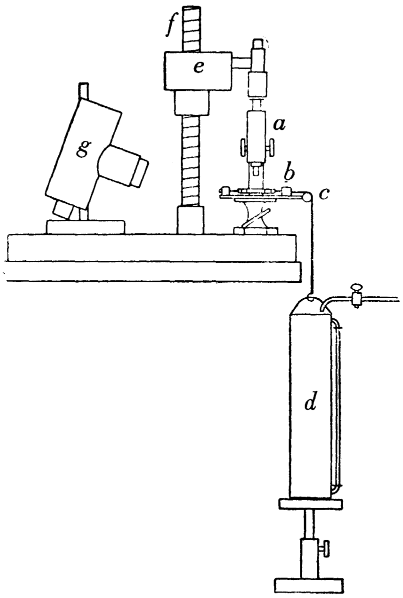

FIG. 1. Diagram of the apparatus for the microcinematography of the process of shear origination and breakdown in monocrystals.

A special apparatus was set up for the microfilming of the growth of cracks (Fig. 1). A heavy brass frame *b*, having two adapters (stationary and movable), was fastened to the table of the microscope. The terminals, glued to the specimens under investigation, were attached to this frame, and the movable adapter was connected to a copper truss spanned over the clock *c*. A high cylindrical vessel *d*, filled with water slowly flowing out through a syphon, was suspended from a hook which was attached to the end of the truss. A communicating vertical tube was attached to the vessel, making it possible to take readings of the quantity of water poured into the vessel by the scale marked on it. A 16 mm cine-camera *e*, which was attached to the vertical column *f*, was installed under the microscope. A projection lamp *g* of 300 W with a condenser was used as the light source.

Owing to the comparative slowness of the processes under study, the photographig was slow. For the automatic control of the photographing, a so-called *Zeitraffer** was developed--it is an instrument which makes it possible to regulate and maintain a constant frequency during photographing. The *Zeitraffer* made it possible, when necessary, to change over quickly from one frequency of photographing to another, and it also ensured the automatic switching on of the projector a definite time before the photographing of each frame, and switching off after the picture was taken, when the time interval between frames was fairly long.

In the majority of experiments the photography was carried out within the time intervals of 8 seconds. The rate of loading was of the order of 5 gm/mm^2 per minute, varying (within small limits) from experiment to experiment, and even during the experiment. In several of the experiments, the further loading was stopped after attaining a definite tension and continuous photographing was carried out at constant tension. In these cases the time intervals between the frames was increased to 15 minutes and longer. Noting down from time to time the value of the load and the corresponding number of the frame during the experiment, it was possible later, when analyzing the film obtained, to establish which of the processes originate or develop for the particular loads.

In order to facilitate the origination of cracks, scratches perpendicular to the length of the ribs of the specimen were made (prior to the experiment) on two adjacent faces of each specimen with the aid of a Martens sclerometer. Later the scratches were made not with a sclerometer, which produces a plastic line with smooth straight edges, but with a thin sharp needle producing a frangible line with broken edges. Such a line gave rise to more microdefects from which the breakdown of the specimen could originate.

The specimen was fastened into adapters in a position such that one of its side faces was perpendicular to the optical axis of the microscope. The crossed polaroids (one of which was fastened on the table and the other on the object of the microscope) were installed in a manner such that the vibrations which pass through them were parallel, and perpendicular to the axis of the specimen. With such a position of the polaroids, the axis of the optical indicatrix, produced by the stretching, was parallel to the direction of the vibrations in one of the polaroids and, therefore, the stretched specimen remained opaque in the crossed polaroids. On the dark background of the specimen, the end of each crack is always clearly visible owing to the presence around it of overstrain, which produces local translucencies in the form of two or four light beams issuing from the end of the crack (Fig. 2*a*).

In addition to the cracks, there are also clearly visible on the dark background of the specimen the

* The apparatus was developed and constructed at our request by V. G. Govorkov to whom the authors express their gratitude.

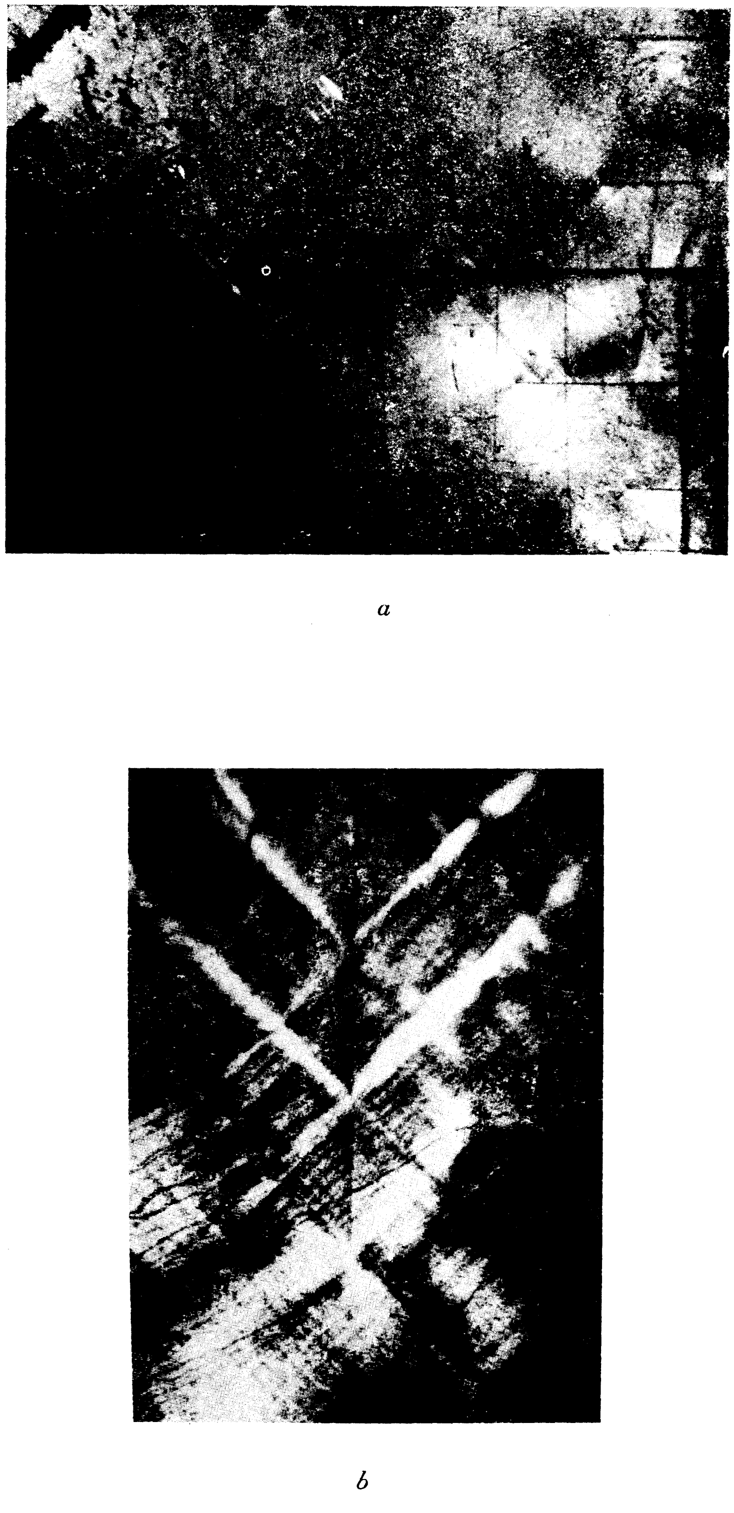

FIG. 2. Microphotographs of cracks in NaCl crystals (crossed polaroids): *a* – cracks originated during the stretching of the specimen; *b* – a crack spreading along the cleavage plane during the spalling of the specimen. Magnification × 22.

light shearing bands produced along the planes (110), in which the stresses are no longer orientated along the axis of the specimen, but approximately along the shears. The most intense shear bands emanate from the trace of the scratch. It should be noted that the observations of the shear bands in two directions, perpendicular to two adjacent faces of the specimen, show that frequent, intensely developed shear bands can be observed at sufficient tension only in one of these directions, while in the other one can see only relatively rare single traces of slipping.

Observation of the growth of cracks and the microcinematography of this process were hindered by the condition that the growth of cracks, as a rule, does not start from the scratch. Therefore, even in the cases when the scratch was made with a needle instead of the sclerometer, it was not possible to predict where the origins of the cracks will appear, and which of these will prove to be the most dangerous and will lead to the breakdown of the specimen. Only in the last experiments we found a method of initiating origins of cracks artificially by pressing lightly with a sharp razor blade on the rib of the specimen. When this is done, a small crack is produced at the rib itself, which, at sufficient load, begins to grow, extending into the interior of the specimen and finally leading to its breakdown. However, this method was only slightly used in our experiments, since by the time we discovered it, all the main experiments on the study of the development of cracks, designated for this investigation, had already been carried out.

The breakdown of the specimens occurred mainly at tensions of ~ 200 gm/mm^2 (varying within the limits 175 to 285 gm/mm^2). The growth of the crack, leading to the breakdown of the specimen, always begins at the rib, apparently from some microdefect which was not removed by the washing of the specimen. The cracks begin to grow noticeably at tensions of 140 to 185 gm/mm^2. In some of the experiments, the specimens studied were maintained for 24 hours and longer under a constant load. Moreover, no further growth was observed in the cracks which were sufficiently well developed.

As was stated, under our experimental conditions the breakdown almost never occurred along the scratches made on the surface of the specimen. Of the 24 specimens studied at a gradually increasing load, only three broke down along the scratches, and two of these did not break down along the scratches from which the most intensely developed shears issued. Only one of the specimens broke down along the scratch with intense shears, similar to the case described by Stepanov[4]; moreover, this breakdown occurred at the instant when the apparatus received a strong knock. All our experiments showed that the crack, having originated at the rib of the specimen, does not spread uniformly in all the directions, but at first always proceeds as a narrow band along that face of the specimen which is located in the zone of intense shears. The growth of the crack, moreover, proceeds intermittently and is visible to the eye when the specimen is observed under the microscope. Wherever there is a halt in the growth of the crack a pair of crossed shears is produced along both planes of sliding at the end of the crack. These shears form a row of small squares by crossing each other. The common diagonal of the squares is the crack (Fig. 2 *a*); in some cases the squares are almost of the same dimensions, in others they gradually decrease.

A similar picture is observed also in the cases when the crack results from causes other than of stretching the specimen. In Fig. 2 *b*, for example, one of the specimens is shown which has a crack produced along the cleavage plane during a longitudinal spalling of the specimen (by knocking along the scalpel); the crack in this case did not reach the end of the specimen.

Such an intermittent development of the crack may be observed when we analyze the surface of rupture of the specimen, which has a definite relief showing the history of the process of propagation of the crack during breakdown. The subsequent positions of the edge of the crack are visible on the surface of rupture in the form of a number of concentric contours. These contours are crossed by a system of lines indicating the directions of the propagation of cracks in all the sections of the surface.

The picture observed on the surface of rupture makes it possible to subdivide it into two parts. In Fig. 3, the first section of the surface of rupture is shown on a slightly enlarged scale (this occupies a relatively small portion of the surface of rupture). The growth of the crack in this stage of the breakdown proceeded mainly along the edge of the specimen in a direction shown by the arrow *A* in Fig. 3. The development of the crack in a direction perpendicular to the arrow proceeded much slower.

In Fig. 3 *b*, a general picture of the surface of rupture is given, from which it is evident that, after overcoming a certain "barrier", the crack begins to propagate only in the direction perpendicular to the initial direction (shown by arrow *B*);

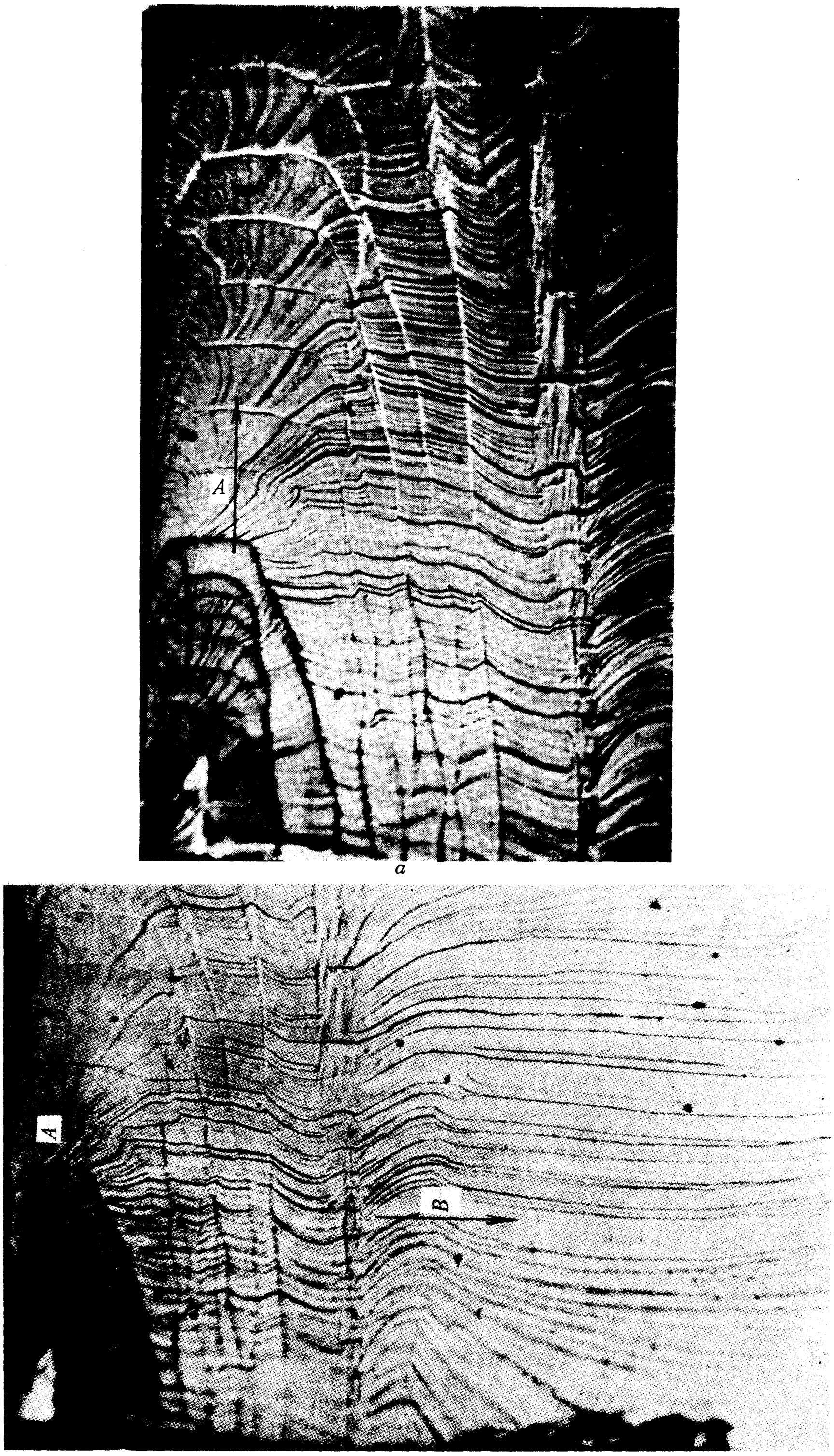

FIG. 3. Microphotograph of the surface of rupture of the NaCl crystal. The relief of the surface shows that the crack, having started from the angle of the specimen, had spread mainly along one side of the specimen (*A*), and only after having traversed the whole of the specimen, it began to grow in a perpendicular direction (*B*). *a* – magnification × 30; *b* – magnification × 20.

in this second section of the surface of rupture we no longer observe transverse contours, indicating the existence of halts in the development of the crack.

Frequently, during the loading, a row of cracks is produced simultaneously on the specimens. These cracks can be observed through a microscope, as well as by the naked eye in reflected light. In the latter case the cracks are clearly visible as light bands proceeding, as a rule, either along one of the faces, or along the two opposite faces of the specimen (Fig. 4). The plane of the cracks is perpendicular to the ribs of the specimen. Generally, as soon as one of such cracks approaches the opposite edge of the specimen, its total breakdown occurs in a very brief interval of time. During this time the crack no longer develops in a direction perpendicular to its initial direction of propagation.

FIG. 4. A view of the cracks in reflected light. It can be observed that the cracks develop in the form of bands only along the two opposite faces.

It is of interest to note that in the microphotographs of the cracks during the initial stage of their development it is clearly evident that the width of the crack increases noticeably from its end towards the beginning (Fig. 5). Therefore, the crack does not have an elliptical cross section, as was assumed by some investigators (such an assumption was introduced, for example, by Griffiths for the simplification of the mathematical calculations), but it has a wedge-like form (as was assumed by Frenkel'[11]).

[11] Ia. I. Frenkel', Zh. Tekhn. Fiz. 22, 1857 (1952).

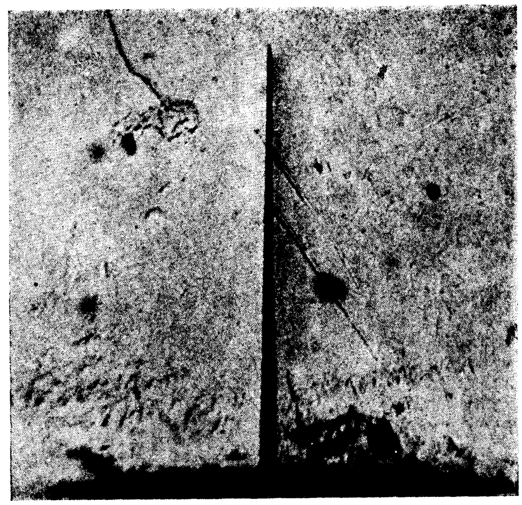

FIG. 5. Microphotograph of a crack developing from the angle of the specimen. The wedge-like appearance of the crack can be observed. Magnification × 30.

Thus, from the observations described above, it follows that during the initial stage of growth, the crack spreads easily in a direction parallel to the planes of strongly developed shear, but it crosses them with difficulty. In some of the specimens one can see clearly that the further growth of the crack is hindered by the intense shear against which the end of the crack thrusts (Fig. 6). Also, in agreement with this is the fact noted above, that the cracks do not originate from the scratches accompanied by intensely developed shears, and originate rarely from the scratches with weak shears, irrespective of the fact that the scratch is undoubtedly accompanied by microdefects, which facilitate the origination of cracks. The growth of a crack may begin from the scratch only in the case when the microdefects necessary for the origin of the crack, with the exception of the microdefects produced by the deposition of the scratch, are removed from the surface of the specimen by careful washing. Since the scratch itself cannot prevent the development of a crack, it is possible to assume that its growth is prevented by the intensely developed shears, always proceeding from the scratch.

One may suppose that the growth of cracks is prevented, on the one hand, by a dislocation in the homogeneity of the crystalline lattice and, on the other hand, by the presence of compression stresses in the vicinity of the sliding surfaces. The regions of the shear bands having stretching stresses apparently should not prevent the de-

FIG. 6. Microphotograph of a crack stopped at the line of shear (crossed polaroids).

velopment of cracks.

From the above one can assume that the plastic deformation of the NaCl crystal leads to its localized strengthening, which prevents the development of cracks. If this assumption is true, then one may consider that the process of shear origination doesnot prepare the breakdown of the NaCl crystal and does not facilitate it, as it was supposed earlier, but, on the contrary, hinders it. Plastic deformation, apparently, only accompanies the breakdown of the crystal, having been produced by the same causes, that is, by the action of tensions and by the presence of microdefects. Of course, the processes of shear origination always precede the breakdown of the crystal, but this is dependent only on the fact that these proceed at lower tensions than the breakdown; otherwise, they could not take place at all.

In the light of the above, one can explain the fact of the strengthening of the NaCl crystals, which have undergone plastic deformation when stretched in water, expressly as the result of the hindrance of the growth of cracks by the shears. This excludes the second of the two explanations of this fact proposed by Klassen-Nekhliudova[12], which is based completely on the ideas of Stepanov.

The investigation carried out shows that direct observation of the origination and growth of cracks in crystals can provide additional new material, substantiating or disproving the conclusions drawn indirectly from the experiments on the determination of the strength of crystals. Further work in this direction and, in particular, the obtaining of quantitative data on the kinetics of the growth of cracks in crystals, are highly desirable. The conclusions of Stepanov regarding the effect of plastic deformation on the appearance of origins of breakdown (but not on the growth of cracks) should also be substantiated by direct experiments.

In conclusion, the authors express their gratitude to Professor Klassen-Nekhliudova for valuable suggestions and discussion of this work.

[12] M. V. Klassen-Nekhliudova, J. Exper. Theoret. Phys. USSR **6**, 584 (1936).

Translated by E. Rabkin
276