DEFORMATION OF SINGLE CALCITE CRYSTALS UNDER HIGH CONFINING PRESSURES*

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Abstract

To supplement the work of investigation of the characteristics of deformation of the calcite aggregates, limestone and marble, some experiments of the deformation of single crystals of calcite under conditions of high confining pressure have been performed. The change in properties with pressure is remarkably different from those observed in the aggregates which suggests the possibility that a considerable portion of the deformation of the aggregates takes place by inter-crystalline deformation. The mechanism of deformation of single crystals does not change with pressure alone, and $10\bar{1}2$ twinning is the dominant means of deformation. It is found that twinning occurs on those twin planes where the deforming force resolved on the plane and in the direction and sense of twinning is a maximum.

Introduction

In order to understand the mechanism of deformation of crystalline aggregates it is necessary first to know the mechanism of the component crystals. A few experiments have been made on the deformation of calcite single crystals under conditions of high confining pressures in

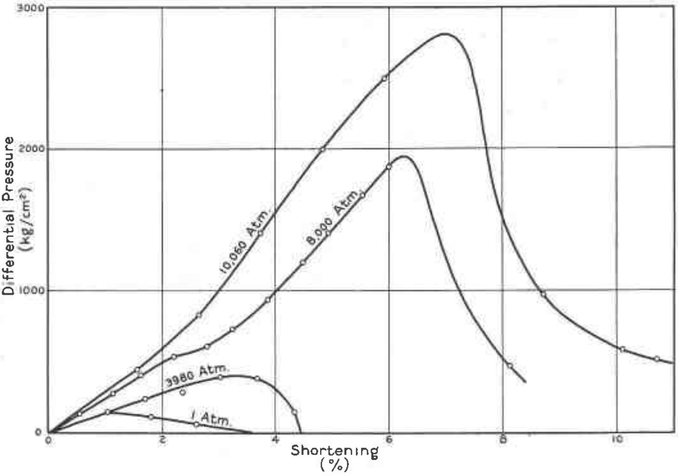

Fig. 1. Stress-strain curves for single crystals of calcite deformed under confining pressures to 1 to 10,000 atmospheres, showing the work hardening of crystals deformed under high pressure.

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the laboratory and the results are presented in this paper to supplement the data already published on limestone and marble.[1]

TECHNIQUE

These experiments were performed in a high pressure cylinder of the type described in an earlier paper[2] designed to permit observation of the behavior of specimens when surrounded by a confining pressure exerted by a liquid, and then deformed by a differential force applied longitudinally to the specimen. The strain (shortening of the specimen) is measured directly by an extensometer, and the differential pressure is obtained by measuring the force on the piston in contact with the specimen and subtracting from that the components due to the hydrostatic pressure and the friction of the packing.

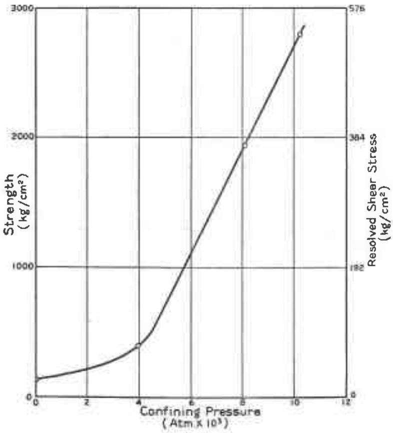

FIG. 2. The increase of strength with confining pressure for calcite single crystals.

The crystals from which these specimens were cut were clear Iceland spar, free from cleavage flaws and also free from any twinning that could be detected visibly. The cleavage fragments were in part obtained from Ward's Natural Science Establishment, and in part were donated by the Harvard Mineralogical Museum.

All the specimens dealt with in this paper were either cylinders or prisms oriented with their long axis parallel to the intersection of two cleavage faces, this direction being selected for ease of preparation. The cylinders were about 1 cm. in diameter and 2 cm. long.

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[1] Griggs, David, Deformation of rocks under high confining pressures: *Jour. Geol.*, vol. **44**, pp. 541–577, 1936.

[2] *Ibid.*, p. 545.

No jacket was applied to the specimen. The liquid (kerosene) was in direct contact with the crystal surface.

All the experiments were carried out at room temperature, and at a constant rate of deformation (.005 cm./min. approximately) so that the effects of time and temperature were not measured, but eliminated.

CHARACTERISTICS OF DEFORMATION

The change in the mechanical properties is shown in the accompanying graphs, Figs. 1–3. Figure 1 shows the stress-strain relations as a function of confining pressure. It is interesting that although the plasticity of the crystals is increased by the confining pressure, this effect is not so great as the increase in strength. Thus, for example, as can be seen from Figs. 2 and 3, the ductility is increased 5.83 times by a confining pressure of 10,000 atmospheres, while the ultimate strength is increased 20.30 times by the same confining pressure.

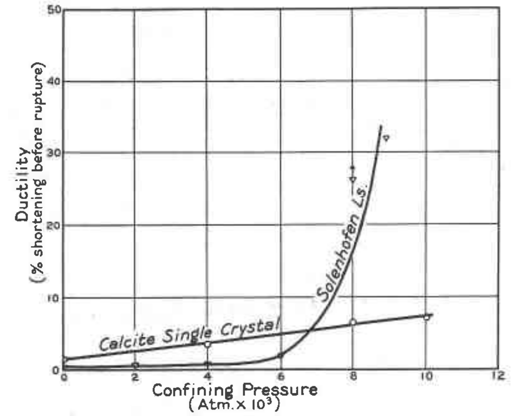

FIG. 3. Comparison of the change in ductility with the pressure in the case of calcite single crystals and in the case of Solenhofen limestone.

Curiously enough, just the opposite is true of the Solenhofen limestone, and the figures will be given here for comparison. Ductility is increased 46.0 times by a confining pressure of 8910 atmospheres, while the strength is increased only 4.7 times.

The break in the 8000 atmosphere stress-strain curve is not completely understood. Conceivably it might be due to something giving way within the apparatus, but there was no other indication of this. If it is a true curve of the crystal's behavior, then we have a close approximation to the behavior of many metals at the yield point. This would not be surprising, because calcite aggregates have been shown to have many properties in common with metal aggregates, but it is very interesting if it occurs thus just as a transient phenomenon during the change in properties as confining pressure is increased.

The writer was surprised to find that the ductility of single crystals of calcite at high pressure was so much lower than that of the aggregates. Marble has been deformed 24% without rupture at 10,000 atmospheres and Solenhofen limestone has been deformed more than 30% at the same pressure without rupture, but as shown, the single crystals of calcite ruptured after only 7% deformation at 10,060 atmospheres.

MECHANISM OF DEFORMATION

In a single crystal it is relatively easy to determine the mechanism of deformation. The first thing which is noticed is that the specimen which was originally a cylinder has been changed so that its section is an ellipse. Figure 4 shows half of this elliptical section in the cylinder deformed at 10,060 atm. In this case the ratio of the long to the short axes was 1.18.

When the thin-sections of Figs. 4 and 5 are set up on the universal stage and the angles between the planes of deformation and the optic axes of the crystals measured, it is found that all the noticeable deformation took place on twin planes of the $10\bar{1}2$ type.

The actual crystal strength may be determined by resolving the compressive force on the twinning plane and in the direction of the twinning, by the formula:

$$\text{Resolved shear stress} = \mathrm{P} \sin \alpha \cos \alpha \cos \phi$$

where P = compressive pressure, α = angle between the direction of compression and the normal to the twin plane, ϕ = angle between the projection of the direction of compression and the direction of twinning.

In the case studied, this resolved shear stress is equal to .192 of the compressive pressure on the operative twin plane, and the values of crystal strength are shown in Fig. 2.

It has been shown in the case of metal crystals that this resolved shear stress is the critical factor governing translation and twinning.[3] Thus, twinning occurs on those twin planes on which the resolved shear stress is a maximum. In the present experiments there was not sufficient variety of orientation to give much evidence for this. However, investigation of the twinning in marble has shown that this "Law of Maximum Resolved Shear Stress" governs calcite twinning.

A caution to anyone calculating this stress is that it must be not only in the right direction, but in the right *sense* to produce twinning. This

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[3] Gough, H. J., Crystalline structure in relation to failure of Metals: Edgar Marburg Lecture, *Am. Soc. Testing Materials,* vol. **33,** Pt. II, p. 22, 1933.

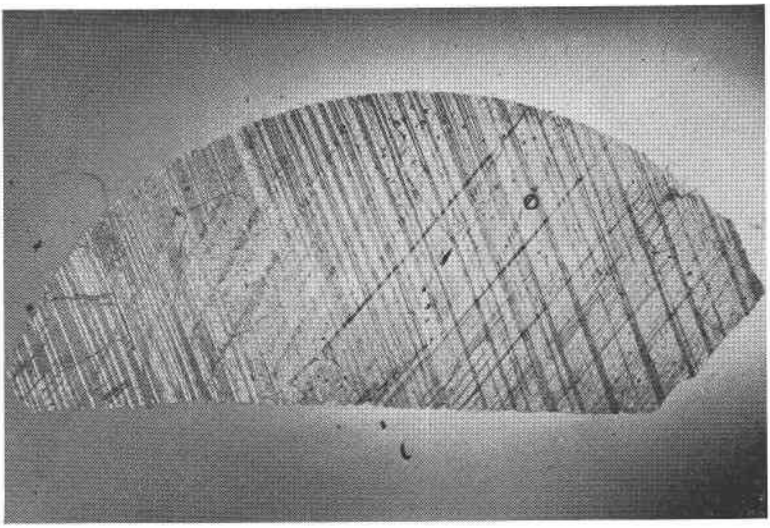

FIG. 4. Photograph of thin-section of single calcite crystal normal to the cylinder axis after deformation at 10,000 atmospheres, showing three sets of twins and cleavage. Magnification 8.5. Nicols uncrossed.

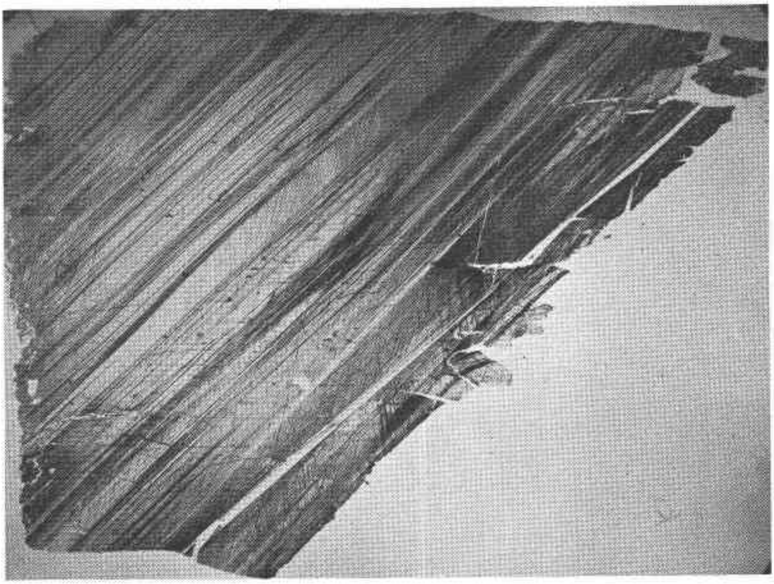

FIG. 5. Photograph of thin-section of calcite crystal parallel to the cylinder axis after deformation at 10,060 atmospheres, showing offsets by the dominant twinning, also other twins and cleavage. Magnification same as above, nicols uncrossed axis of compression is horizontal.

is, of course, obvious from geometrical considerations of the twinning deformation. It is not a necessary condition governing slip, however, since slip may occur in both senses in a given direction in general.

SIGNIFICANCE OF THE RESULTS

Perhaps the most significant observation in this investigation is the low ductility of the single crystals as compared to aggregates of the same crystals. This might immediately lead one to suspect that deformation in aggregates takes place partly through inter-crystalline deformation. It is difficult to determine the relative amounts of intra- and inter-crystalline motion, and at present, this question must be left unanswered, but it is hoped that petrofabric study of deformed marble which Mrs. E. B. Knopf is undertaking in cooperation with the writer may give the complete answer.

This study has shown at least some of the rules of intra-crystalline deformation, and has indicated that at high pressure, the $10\bar{1}2$ twinning is still the most important mechanism of deformation. No pure translation (slip) was noticed, and if present at all, was a very minor factor.

It is possible that the conditions of molecular stability may be altered materially by increased temperature and that under conditions similar to those existing in metamorphism, other mechanisms of crystal deformation become operative. The writer is undertaking a series of high temperature experiments which may give some light on this problem. It is not thought probable that long continued stresses such as might exist in geologic deformations would induce any different reaction, so the effect of time alone is discounted as affecting the *nature* of the deformation. It would certainly affect the *amount* of deformation, and hence the ductility. It is hoped that this factor may be investigated also in the near future.

It is a pleasure to acknowledge the invaluable help of Professor P. W. Bridgman in all the high pressure work. This investigation was made possible by a joint grant from the Committee on Geophysical Research and the Society of Fellows of Harvard University. The writer is also indebted to the Harvard Mineralogy Department for many of the specimens of calcite, which are difficult to obtain.