NASA TECHNICAL REPORT

NASA TR R-186

NASA TR R-186

c. 1

ON THE MECHANISM OF DUCTILE BENDING IN IONIC CRYSTALS

by Charles E. May
Lewis Research Center
Cleveland, Ohio

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • DECEMBER 1963

ON THE MECHANISM OF DUCTILE BENDING

IN IONIC CRYSTALS

By Charles E. May

Lewis Research Center
Cleveland, Ohio

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

For sale by the Office of Technical Services, Department of Commerce,
Washington, D.C. 20230 -- Price $0.50

ON THE MECHANISM OF DUCTILE BENDING

IN IONIC CRYSTALS

SUMMARY

This report presents experimental evidence in support of a mechanism that explains the bending of face-centered cubic ionic single crystals such as sodium chloride. When a specimen is deformed in flexure, some new dislocations are generated while others are driven from the sample; some of the remaining dislocations align themselves along the {110} slip planes to produce "orientation" domain boundaries perpendicular to the slip planes. This type of process is generally referred to as mechanical polygonization. The existence of orientation domains was confirmed by an X-ray diffraction technique. The number of domains increases with increasing deflection, and there is slight evidence that the average angle between adjacent domains may also increase with increasing deflection. An approximate value for the average angle attained between adjacent domains is about $1/4^{\circ}$. Observation of curvature of the fracture faces also lends support to the correctness of the model.

A unique relaxation phenomenon appears to occur if a crystal is allowed to sit with a constant overall deflection; the curvature that is present apparently tends to become more uniform along the length of the specimen.

INTRODUCTION

For several years there has been great interest in the ductility of ionic single crystals such as sodium chloride (refs. 1 to 6). The determination of the degree of such ductility was most often carried out by means of flexure tests, either with three- or four-point loading. It has been known for a long time that, for face-centered cubic materials, such as sodium chloride and magnesium oxide, the {110} slip planes are the operative ones and that these are operative in the ⟨110⟩ direction. Models have been presented that illustrate the manner in which such slip and the accompanying redistribution of dislocations might be effective in producing a ductile bend in a single crystal (ref. 7).

This report presents a slight modification of such a model, includes experimental evidence to show that the model is correct, and illustrates certain generalities concerning the bending process. The evidence is based upon the dependence that the intensity of the X-ray reflection from the tension surface of sodium chloride has with respect to the angle that a bent single crystal makes with an incident X-ray beam. This dependence was determined as a function of the amount of bending. Furthermore, curvature of the fractures that occurred was observed, thus giving supporting evidence for the model presented.

PRESENTATION OF MODEL

Recent work (ref. 8) that involved the use of special Berg-Barrett X-ray diffraction techniques has produced photographs of bent lithium fluoride single crystals that reveal narrow domains (about 0.2 mm wide) of low dislocation density separated by still narrower regions of higher dislocation density. The domain boundaries are parallel to one of the {110} crystallographic planes. The existence of such domains suggests a reasonable model similar to that of Cahn (ref. 9, pp. 157-159) by which accumulation of dislocations could effectively produce a bend in an ionic crystal. A group of dislocations is considered to exist along the ($\bar{1}10$) plane (as shown schematically in fig. 1(a)). It should be noted that figure 1 defines the crystallographic plane notation used throughout this report. With the aid of figure 1(b), the effect of applying a bending force on such a crystal can be visualized. More dislocations are generated, and some are driven from the crystal through the upper and lower surfaces, as indicated in figure 1(b). Some of those remaining tend to line up, as indicated in figure 1(c). Such an array generates a series of domain boundaries perpendicular to the operative slip planes. The domains themselves are regions of low dislocation density, while the regions separating them are regions of relatively high dislocation density. Each domain is at a slight angle to its neighbor, and thus an effective bend is produced in the crystal. Furthermore, the (110) slip planes would also be expected to operate so that "orientation" domain boundaries would also be produced parallel to the ($\bar{1}10$) planes.

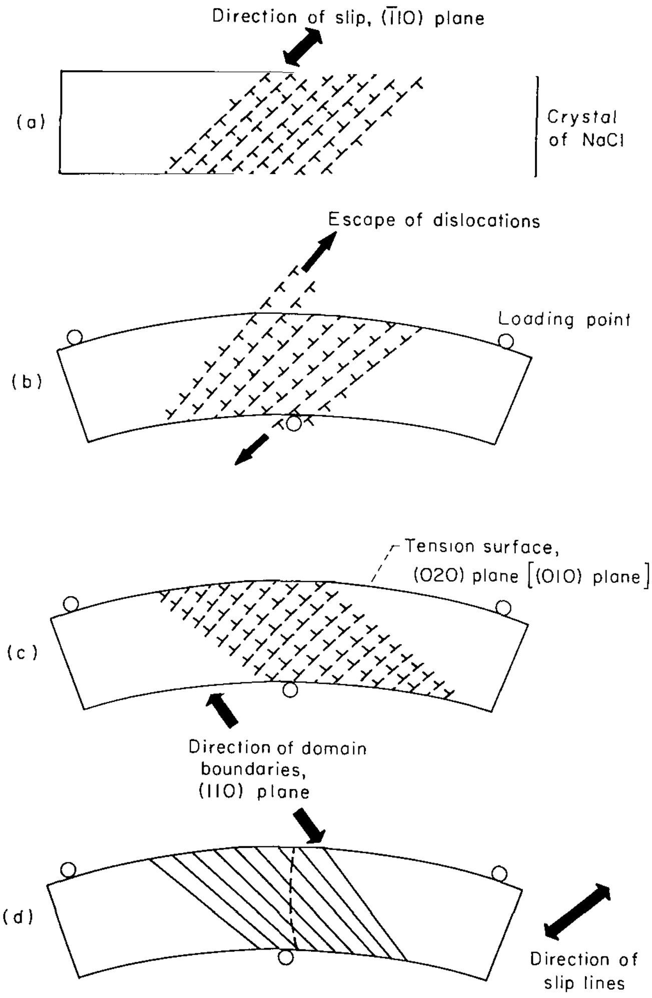

Figure 1. - Model of bending mechanism. Dashed line in (d) is expected direction of fracture with respect to domains, slip planes, and direction of bending; fracture along (100) plane.

If, however, only one slip system would be operative, as in the case of potassium chloride (ref. 10), a unique phenomenon should be observable, as illustrated in figure 1(d). A fracture crack along the (100) plane should have a curvature, since such a crack would penetrate many domains with each successive domain making a larger angle with respect to the first domain than the preceding ones would be making.

EXPERIMENTAL

The validity of the model might be experimentally confirmed in at least three ways: (1) by etch-pit studies of dislocations revealing the domain boundaries, (2) by X-ray diffraction techniques that could reveal the existence of slight angles between the domains but not the existence of a single slip system, and (3) by observation of curvature for a fracture parallel to the (100) planes, as indicated in figure 1(d) and noted before.

Preliminary observations of etch pits on sodium chloride crystals, which were severely strained in flexure, were made but failed to provide evidence for the existence of domain boundaries. Any alignment of dislocations was masked by the high density of dislocations.

The X-ray diffraction technique used required the apparatus shown schematically in figure 2. It consists primarily of a Norelco X-ray diffractometer equipped with a copper target and a nickel filter. However, the normal specimen holder is replaced by a specially constructed three-point flexure loading device. The device is designed to hold a $1\frac{1}{2}$- by $\frac{1}{4}$- by $\frac{1}{8}$-inch single crystal so that the central portion of the tension surface of the crystal (1.13 cm along the face as calculated from the geometry) is hit by the incident X-ray beam. The Geiger counter of the diffractometer is fixed at 31.69° for 2θ, so that the portion of the beam diffracted from the (020) planes of sodium chloride by copper K_α radiation is directed into the Geiger tube. A 0.006-inch-width receiving slit is used together with a 1° divergence slit and a 1° antiscatter slit. The bending device is mounted on a shaft that can be rotated counterclockwise at a constant rate of 0.889° per minute. The alignment of the device and crystal are such that the bending direction is perpendicular to the axis of rotation. The distance between the upper loading points is approximately $1\frac{1}{4}$ inches, with the relative motion of the lowest point bisecting this distance.

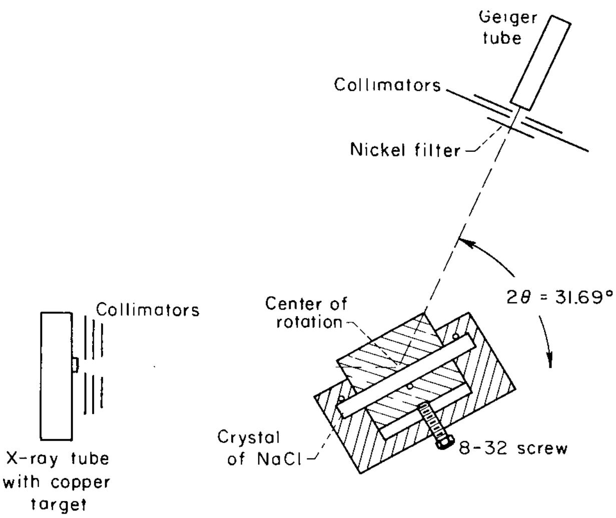

Figure 2. - Schematic of bending apparatus. Crystal bending device rate of 0.889° per minute where axis is same as that for Geiger tube.

A Harshaw melt-grown single crystal of sodium chloride was water polished and placed in position in the three-point loading device with a minimal deformation to the crystal. The angle that the top face of the crystal makes with the incident beam was set to a little less than θ where 2θ is 31.69°. The counterclockwise rotation of the specimen was begun, and the X-ray tube and recording equipment were started; the diffracted X-ray intensity was thus measured

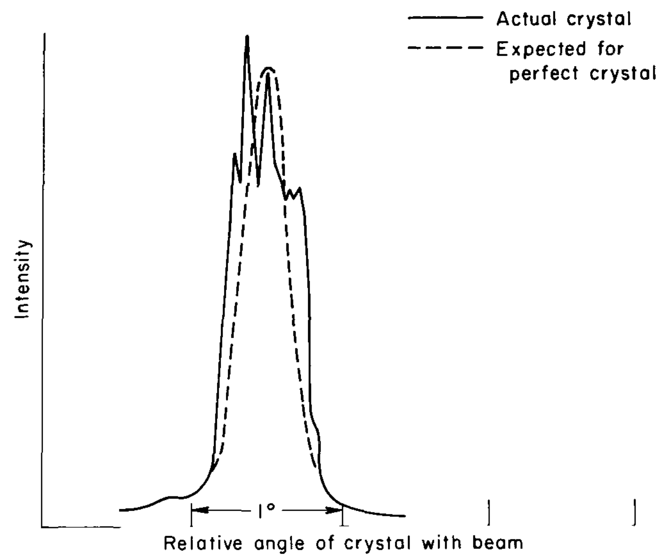

Figure 3. - Resolution of (020) line at zero deflection (specimen 6).

as a function of the angle of the crystal. The crystal was bent a known amount, and the diffracted intensity was again measured as a function of the angle. This procedure was continued until the crystal fractured. Occasionally, the scanning of the angle was repeated without additional deformation. In all, six crystals were tested. Figure 3 gives a scan of a typical crystal with essentially zero deformation, and figure 4 gives three consecutive scans of the angle for the deflections indicated (approx. 3/32 in.). Figure 5 gives the results of two consecutive scans with no deformation made between the scans; however, a period of 16 hours expired between scans.

The preliminary work was done by using film as the detector of the diffracted beam of incident "white" X-rays. These were essentially Laue patterns

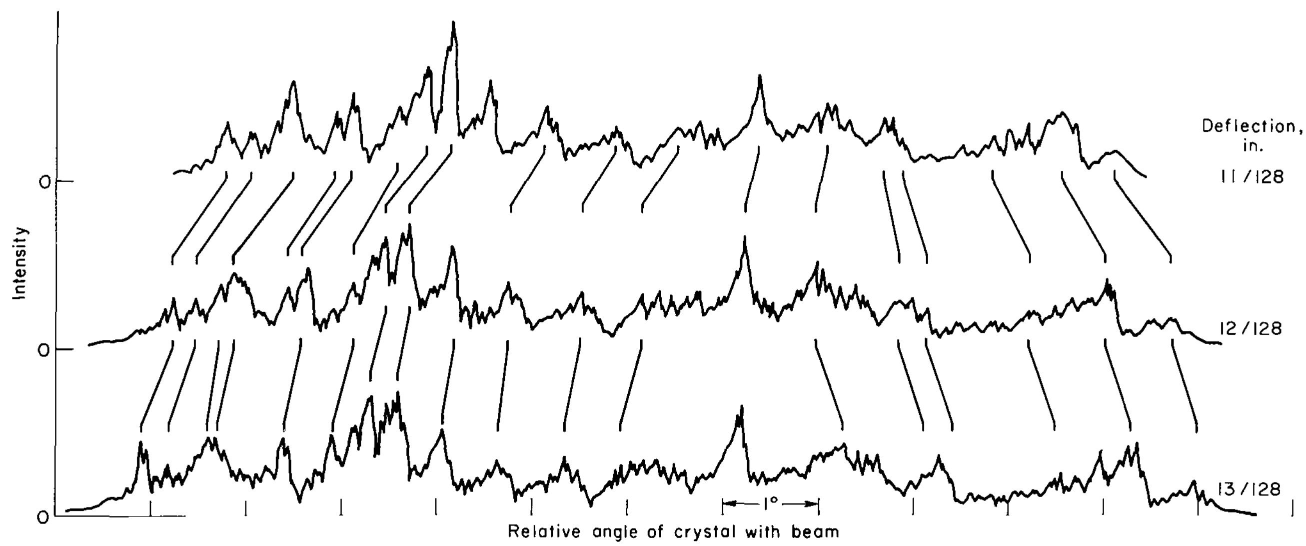

Figure 4. - Resolution of (020) line at deflections indicated (specimen 1).

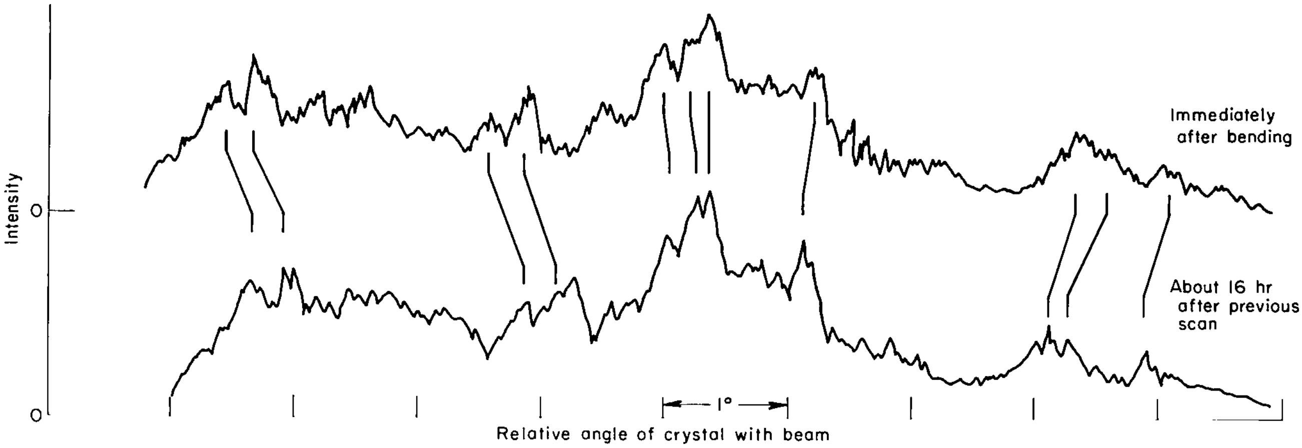

Figure 5. - Resolution of part of (020) line at 9/64-inch deflection (specimen 5).

(fig. 6) of bent single crystals. These experiments were followed by others that made use of a moving Geiger tube, a stationary crystal, and again "white" X-rays.

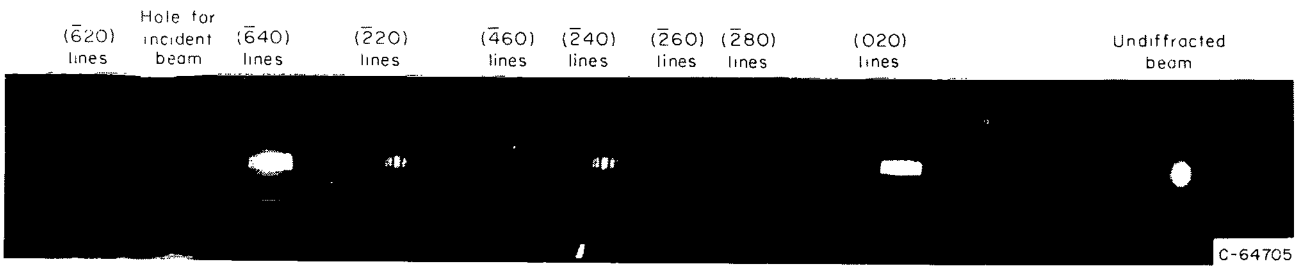

Figure 6. - Laue patterns of bent sodium chloride crystal.

Some of the data obtained from these experiments were informative and are discussed later. The final experimental setup, the one described above in detail, was found to be the most suitable for yielding the desired data.

After the X-ray diffraction studies were performed on the crystals, the fractured parts were inspected for any curvature and were examined under polarized light to detect the operative slip planes.

DISCUSSION

The method used was found to be capable of substantiating the proposed mechanism despite several minor drawbacks. The first of these concerns the simple rotation used. This type of rotation of the crystal does not produce the ideal relative position of a bent crystal with respect to the incident beam, since the surface planes of the crystal move out of the idealized position for diffraction. However, for small deflections in the crystal and thus small variances in the angle involved, the experimental setup proved to be quite satisfactory. The second drawback concerns the desirability of having a scan of the entire lengthwise direction of the specimen instead of only the middle third. This is prevented because of interference from the upper loading points as well as the narrowness of the beam required for good resolution. A third possible drawback concerns the fact that the actual length of the crystal scanned is slightly dependent on the amount of deflection. Experimentally, this dependence could not be detected.

The next aspect of the discussion concerns the expected results from the method. If a bent crystal possessed no alignment of dislocations, a gradual bending of the (020) crystallographic planes should exist and thus each diffraction pattern in figures 3, 4, and 5 should consist of a single broad peak; however, the presence of many distinct peaks in these figures indicates that the bending is not microscopically uniform but that the crystals possess more (020) planes at some angles than at others. From the experiment in which film is used as a detector, identical types of diffraction patterns can be seen to have resulted from all the observable sets of planes (fig. 6). This indicates that the observed diffraction is the result of domains that are at slight angles with respect to one another. If all the diffraction lines observable in figure 6 did not show the same resolution of fine structure, the resolution of the (020) line could be due merely to some type of distortion of the cubic lattice. One other comment should be made about figure 6. In the diffraction pattern for one particular plane, for example, the $(\bar{2}20)$, the lines are not perfectly parallel. This indicates that there may be some twist within a single domain along its extent across the top of the crystal. It is impossible for the simple scanning method used to detect such a twist.

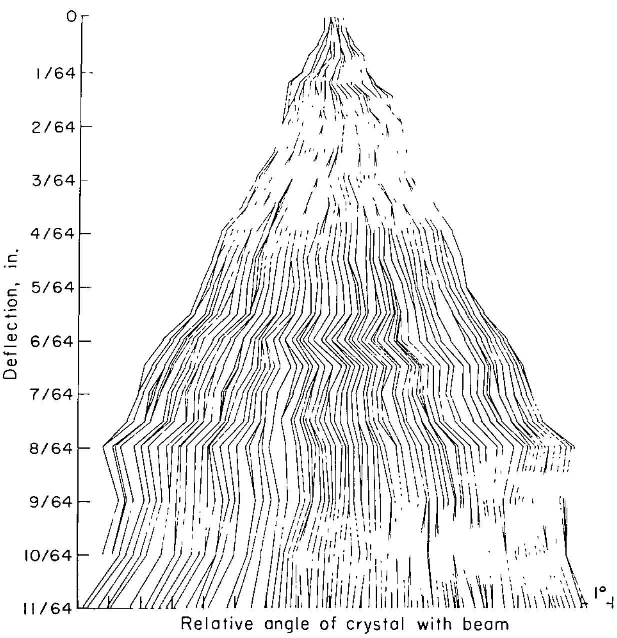

Figure 7. - Angular position of domains as a function of deflection (specimen 5).

By a comparison of the three curves in figure 4, retention of the majority of the diffraction peaks can be seen. Since each peak should correspond to a domain, it is possible to trace the development of such domains as a function of the amount of deflection (bend). Figure 7 is such a plot of the development of domains as well as the change of the angle between them as a function of deflection for one of the sodium chloride single crystals used. Each vertical dash represents a domain found at a particular degree of bending. The connecting lines indicate the reconstructed angular motion of these domains, while the crystal was being deformed in flexure. A small fraction of this exact reconstruction may be open to dispute, since occasionally it is difficult to trace a peak in one scan to the corresponding peak in the subsequent scan; however, this does not affect the generalities that can be drawn from figure 7 or the general model that is suggested by them. Similar plots resulted from the data of the five other specimens treated similarly as well as from the data obtained from the preliminary work in which the Laue technique was used with both film and Geiger tube detection. These plots indicate that as a crystal is deformed in flexure the number of distinct domains multiplies.

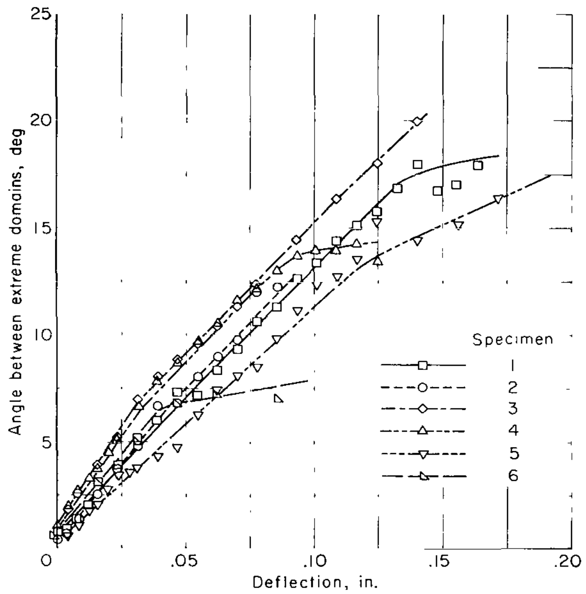

Figure 8. - Dependence of curvature on deflection.

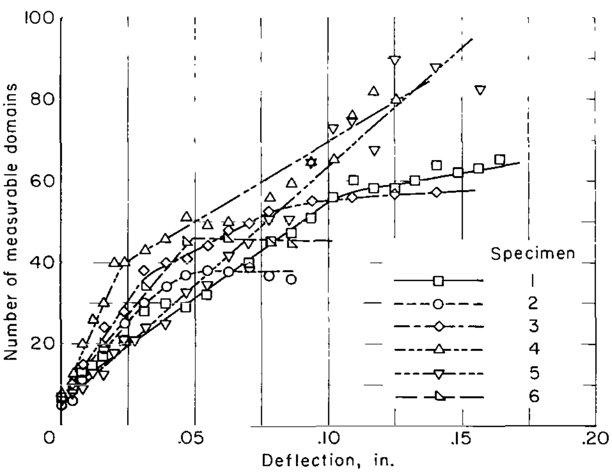

Figure 9. - Increase in number of domains with deflection.

To illustrate this and other points, three additional types of plots were made. First, the angle between the extreme domains was plotted as a function of deflection, as shown in figure 8. Next, the number of domains was counted for each deflection and plotted as a function of the deflection, as shown in figure 9. Finally, the average angle between domains was computed and plotted as a function of the deflection in figure 10.

For small deflections, it is certainly expected that the deflection should be roughly a linear function of the angle made between the two extreme domains detectable for a crystal. This is indeed shown experimentally in figure 8. The initial slope of the curve is about 131° per inch. Since the beam sees only about 0.346 of the crystal, the slope of a curve that gives the entire curvature (provided that it is uniform) would be 379° per inch. A value for this slope can also be readily calculated from the geometry of the bending jig if a small deflection is assumed. This value is found to be 366° per inch, which is in very good agreement with the experimental value. Figure 9 indicates that the number of domains, in general, increases with increasing deflection. The average width of a domain can be obtained by dividing the number of domains into the length of crystal struck by the X-ray beam. Thus, an order-of-magnitude value for the width of a domain would be

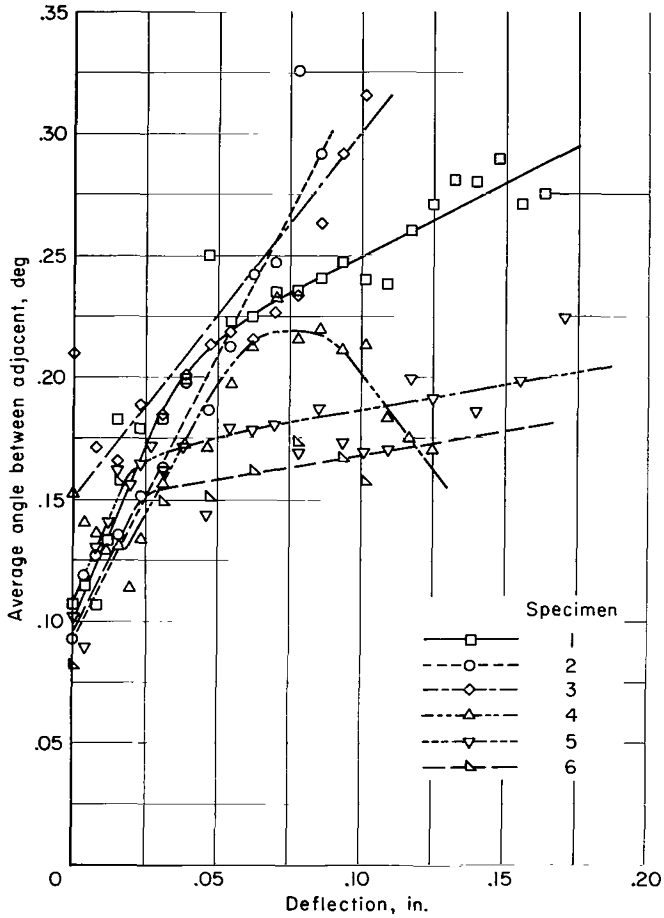

Figure 10. - Dependence of average angle between domains on deflection.

about 0.3 millimeter, which is in good agreement with earlier results (ref. 8). Figure 10, which gives the average angle between adjacent domains, shows some evidence that this value increases with increasing deflection. The order of magnitude for this value is about $1/4^{\circ}$.

Figure 5 also indicates that some type of "angular" relaxation occurred while the overall deflection was being held constant. Its magnitude is about 5 percent of the total deflection. The explanation of this phenomenon may be as follows. If the original deformation tended to be nonuniform along the length of the specimen, the angular deformation of the middle portion of the specimen would be greater than that which is caused by a uniform deformation. On standing, the deformation could redistribute, which would cause greater deformation in the outer portion of the crystal, but would allow the center portion, which was involved in the diffraction experiments, to recover as is experimentally observed.

Examination of the fractured specimens under polarized light revealed that, although both the (110) and $(\bar{1}10)$ planes were operative in a particular specimen, in any one particular region of the crystal only one set of planes was operative. Thus, curvature of the fractures should be observed. Indeed, in most of the specimens, even in the preliminary runs, fracture occurred with a slight curvature along the (100) plane. Such a curvature has been observed many times before in the fracture of sodium chloride specimens at Lewis. Whenever noted, the direction of the curvature with respect to the bend and slip planes was always the same as indicated in figure 1(d). To explain this direction of curvature, it is necessary for the domain boundaries to be perpendicular to the operative slip planes.

CONCLUDING REMARKS

In addition to generating clearer ideas concerning the bending mechanism, the present work indicates a vast amount of research that could be done. The present experiments could be repeated with the use of other surface treatments for the crystals to determine whether or not the mechanism of bending could be altered. Diffraction photographs similar to those in reference 8 should be taken to substantiate further that the domain boundaries are perpendicular to the slip planes. Any further investigation of the ductility of single crystals should be preceded by a determination of the nature of the orientation domains already present in the specimens as figure 3 would indicate.

Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio, November 6, 1963

REFERENCES

1. Gorum, A. E., Parker, E. R., and Pask, J. A.: Effect of Surface Conditions on Room-Temperature Ductility of Ionic Crystals. Jour. Am. Ceramic Soc., vol. 41, no. 5, May 1958, pp. 161-164.

2. Hulse, Charles O., and Pask, Joseph A.: Mechanical Properties of Magnesia Single Crystals in Compression. Jour. Am. Ceramic Soc., vol. 43, no. 7, July 1960, pp. 373-378.

3. Machlin, E. S., and Murray, G. T.: Effect of Atmosphere Conditions on the Brittleness of NaCl. Jour. Appl. Phys., vol. 30, no. 11, Nov. 1959, pp. 1731-1735.

4. Stearns, Carl A., Pack, Ann E., and Lad, Robert A.: Ductile Ceramics. I - Factors Affecting the Plasticity of Sodium Chloride, Lithium Fluoride, and Magnesium Oxide Single Crystals. NASA TN D-75, 1959.

5. Stearns, Carl A.: Effect of Aging Time After Cleavage on the Plasticity of MgO Single Crystals. Jour. Appl. Phys., vol. 31, no. 12, Dec. 1960, p. 2317.

6. Stokes, R. J., Johnston, T. L., and Li, C. H.: Effect of Surface Conditions on the Initiation of Plastic Flow in Magnesium Oxide. Third Tech. Rep., Honeywell Res. Center, Sept. 1958.

7. Cottrell, A. H.: Dislocations and Plastic Flow in Crystals. Clarendon Press (Oxford), 1958, pp. 181-184.

8. Yoshimatsu, Mitsurer: Observations on the Polygonization of Bent LiF Crystals by the Berg-Barrett Method. Jour. Phys. Soc., Japan, vol. 16, no. 11, Nov. 1961, pp. 2246-2253.

9. Cahn, R. W.: Internal Strains and Recrystallization. Prog. in Metal Phys., vol. II, B. Chalmers, ed., Butterworth's Sci. Pub., 1950, pp. 151-176.

10. Van Bueren, H. G.: Imperfections in Crystals. North Holland Pub. Co., 1960, pp. 570-572.