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# TECHNICAL NOTE

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DUCTILE CERAMICS

I - FACTORS AFFECTING THE PLASTICITY OF SODIUM CHLORIDE  
LITHIUM FLUORIDE, AND MAGNESIUM OXIDE SINGLE CRYSTALS

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FLUORIDE, AND MAGNESIUM OXIDE SINGLE CRYSTALS

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SUMMARY

A study was made of the relative magnitude of the effects of various factors on the ductility of single crystals of sodium chloride (NaCl), lithium fluoride (LiF), and magnesium oxide (MgO). Specimen treatments included water-polishing, varying cleavage rate, annealing, quenching, X-irradiation, surface coating, aging, and combinations of some of these treatments. The mechanical behavior of the crystals was studied in flexure and in compression, the latter study being performed at both constant strain rate and constant load. Etch-pit studies were carried out to provide some pertinent information on the results of pretreatment on the dislocation concentration and distribution in the vicinity of the surface.

The load deformation curves for these ionic single crystals show an initial region of very low slope which proved to be due to anelastic deformation.

The extent of initial anelastic deformation is modified by specimen pretreatment in a way that suggests that this deformation is the result of expansion of cleaved-in dislocation loops, which can contract on the removal of the stress.

The effects of the various pretreatments on the load and deflection at fracture are in accord with the prediction one might make with regard to their effect on the nucleation of fatal surface cracks.

For NaCl, increases in ductility are always accompanied by increases in strength. The creep constants for NaCl are a function of treatments which affect the bulk structure but are not a function of treatments which only affect the surface.

## INTRODUCTION

Present-day demands for materials to be used at very high temperatures place a large share of the burden on ceramics. Although ceramics have been avoided where possible in the past because of their brittleness and poor resistance to thermal shock, proper design should overcome some of the problems associated with their use. However, the use of very brittle materials will always be the source of many difficulties, and any improvement in the plasticity of ceramics will be welcome.

Recently a great deal of attention has been turned toward the determination of the factors which are responsible for the low ductility of ceramic materials (refs. 1 to 6). The results of these investigations on single crystals indicate that the problem is a complex one. The ductility of materials such as sodium chloride (NaCl), lithium fluoride (LiF), and magnesium oxide (MgO) is affected by factors like purity, mode of cleavage, time after cleavage, and the atmosphere in which the specimens are stored. Marked changes in the ductility can be produced by specimen treatments as varied as X-irradiation and coating with a thin film of stearic acid.

The purpose of this report is to present the results of an investigation of the relative magnitude of the effects of the various factors on the plasticity of single crystals of NaCl, LiF, and MgO. The mechanical behavior at room temperature of the crystals was studied in flexure and in compression, the latter test being performed at both constant strain rate and constant load. The specimen treatments included water-polishing, varying cleavage rate, annealing, quenching, X-irradiation, surface coating, aging, and combinations of some of these treatments. The results obtained permitted some speculation concerning the relation between the state of the specimen and its behavior in terms of present concepts of the role played by crystal imperfections in deformation and fracture. Etch-pit studies using techniques of the type described in reference 7 were carried out to provide some pertinent information on the results of pretreatment on the dislocation concentration and distribution in the vicinity of the surface.

Some of the findings of this investigation have been applied to the preparation of polycrystalline NaCl specimens with improved ductility (ref. 8).

## EXPERIMENTAL PROCEDURE

## Specimens

The NaCl single crystals used in this study came from four groups of material purchased from the Harshaw Chemical Company. Each group was

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from a single melt. The specimens in group I were supplied as-cleaved to the approximate dimensions of 3 by 7 by 40 millimeters, and the specimens in group II were cleaved in our laboratory from large pieces of good cleavage material. These two groups of specimens were used exclusively for the flexure tests. The specimens in group III were supplied as-cleaved to the approximate dimensions of 6 by 6 by 20 millimeters, and these specimens were used for the compression tests, creep tests, and etch-pit studies. The specimens of group IV were cleaved at this laboratory from large pieces of good cleavage material and were used in the creep tests.

All the MgO single crystals used in this study were purchased from Semi-Elements, Incorporated, in the form of random pieces from a single melt. The specimens were cleaved to size in the laboratory.

The LiF specimens were cleaved to size from large pieces of purchased material.

#### Polishing

Two water-polishing procedures were used for the NaCl specimens. Specimens to be completely water-polished were immersed successively for 15 seconds in four 25-milliliter volumes of distilled water at room temperature. They were then rinsed with dry methanol and air-dried. The second procedure was used when only part of the specimen was to be water-polished. The face to be polished was passed successively over water-saturated cotton and methanol-saturated cotton and was finally air-dried.

The MgO specimens were polished by immersion in 85-percent phosphoric acid at 100° C for 30 minutes followed by a hot-water rinse and air-drying.

The LiF specimens were polished by two different methods. In the first method, the specimen was immersed in concentrated hydrofluoric acid for 1 minute, washed with water, and air-dried. In the second method, the specimen was immersed in concentrated hydrofluoric acid for 1 minute, washed in water, immersed in 14-molar ammonium hydroxide for 10 minutes, washed with water, and then air-dried.

#### Annealing and Quenching

All anneals, except those for the etch-pit studies, were performed by placing the specimens directly into a quartz tube supported in a tubular furnace maintained at the annealing temperature. In the case of the etch-pit studies the specimens were placed in the quartz tube at room temperature and then slowly heated to the annealing temperature.

The quartz tube was in both cases closed at one end and open to the air at the other end. The annealing temperature was controlled to  $\pm 10^{\circ}$  C.

The only difference between the annealing and quenching procedures was that annealed specimens were furnace-cooled, while quenched specimens were cooled to room temperature rapidly by immersing the hot specimen in a volume of carbon tetrachloride.

#### X-irradiation

Specimens to be irradiated were wrapped in aluminum foil and mounted on the end of the shaft of a 4-rpm clock motor. The specimens were rotated with their long dimension normal to the X-ray beam. The X-ray tube was a Machlett AEG-50 having a tungsten target and a beryllium window and was operated at 50 kilovolts and 30 milliamperes. For all specimens, the distance between the crystal and the window of the X-ray tube was 8 inches.

#### Miscellaneous Treatments

Some NaCl specimens were coated with silver in a vacuum evaporator. The silver was evaporated from a tungsten filament located 4 inches from the NaCl substrate. The computed thickness of the silver coating was 25 Å.

For the creep tests some specimens were cleaved in mineral oil and tested in the same or other media. The procedure was to place a large block of material in a vessel containing the mineral oil and cleave from it specimens whose faces were all new. The specimen was then placed in a cup in the mineral oil, and the cup containing mineral oil plus specimen was transferred to the creep apparatus, where the specimen was tested while still in the cup under oil. When testing was done in other media, simple transfer procedures were followed which ensured that the specimen was always confined to the liquid medium.

#### Flexure Tests

Specimens were tested in flexure in an Instron tensile testing machine equipped with a compression load cell. The bending jig is shown schematically in figure 1. The four-point loading method subjects an extended area of the specimen to a constant bending stress; this condition was deemed necessary to obtain representative data for specimens having a distribution of nucleation sites for destructive crack formation. The reactance and loading points, rigidly attached to the load cell and the movable crosshead, respectively, were 0.1035-inch blades

of Monel ground to a 0.0625-inch radius at the specimen contact end. The distance between centers of the loading points was 1.1770 inches, and the moment arm was 0.2690 inch long. The bending jig was aligned in the testing machine to within 0.0001 inch.

The Instron machine used is generally considered to be a "hard" machine. However, in these experiments, the load-cell deflection was great enough to be an important factor, and correction was always made for this factor. The load-cell deflection coefficient was experimentally determined to be  $1.00 \times 10^{-4}$  inch per pound of load for the load cell used.

All the bend tests were run in air at room temperature and at a constant bending rate. The data were obtained in the form of strip-chart traces.

Load-strain data were obtained in some experiments by use of wire resistance strain gages bonded with cement to the tension and/or compression faces of the specimens. The bond was cured for 50 hours at room temperature. Strains were measured with an SR-4 strain indicator.

#### Compression Tests

The compression tests were all run with the tensile testing machine equipped with a compression load cell. The tests were run in air at a constant compression rate. The deflection coefficient of the load cell used for these tests was experimentally determined to be  $2.0 \times 10^{-5}$  inch per pound of load.

#### Creep Tests

The compressive creep tests were run in the apparatus shown schematically in figure 2. The load was applied to the specimen in an instantaneous manner by means of the hydraulically operated loading platform. The creep of the specimen was measured by both a differential transformer and a dial indicator. The differential transformer was primarily used to measure the very rapid initial creep of the specimen. By connecting the differential transformer to a strip-chart recorder through a discriminator circuit, the actual creep curve could be automatically recorded. In most cases the initial instantaneous creep was so great that the accuracy of the recorded creep curve had to be limited to  $\pm 0.0005$  inch. To increase the accuracy of the experiment, a dial indicator with a sensitivity of  $\pm 0.000025$  was utilized in conjunction with the differential transformer.

The procedure was first to place a specimen in position under the small initial load of the measuring platform (1.97 lb), which represents

a stress of approximately 0.05 psi for the size specimen used. The dial indicator and differential transformer were then adjusted to read zero. The load required to produce the desired stress was placed on the loading platform, which was held in the raised position by air pressure. To start the test, the platform was lowered slowly, and at the instant the loading column made contact with the steel ball on the measuring platform, the full load was applied by rapidly exhausting the hydraulic cylinder. At this same instant the recorder and a timer were started. The creep was then measured as a function of time.

### Etch-Pit Studies on NaCl

The etchant used to produce dislocation etch pits was that mentioned in reference 9, that is, 3 grams of mercuric chloride per liter of absolute alcohol. Etching times of 30 seconds were used, and etching was always followed by a dry-acetone rinse.

## RESULTS

### Flexure Experiments

General characteristics of load-deflection curve. - The most striking feature exhibited by the specimens tested was the existence of an initial anelastic region having an extremely low slope. Figures 3 to 5 show the initial portions of the load-deflection curves for as-cleaved specimens of NaCl, LiF, and MgO. The Hooke's law line calculated from the elastic constant is included in each case for comparison.

In order to ensure that this behavior was characteristic of simple crystals of this type and not an artifact of the test procedure, flexure tests were run on polycrystalline nickel and copper. The resultant load-deflection curves (fig. 6) were linear, as were the load - tensile-strain curves obtained with the aid of strain gages mounted on the specimen tensile faces (fig. 7). The Young's moduli obtained were in good agreement with literature values.

Cyclic stressing showed that the initial deformation for these materials was anelastic. When a specimen was loaded into this anelastic range and unloaded, the deflection represented by the foot did not recover immediately, but rather recovery occurred over a period of several hours. Figure 8 illustrates a typical experiment with LiF. Photoelastic strain patterns observed through a polariscope also showed a slow recovery. The experiment could be repeated any number of times provided that enough time was allowed for recovery to take place.

The difference in behavior between the experimental specimens and that predicted from simple-beam theory is quite marked in these

experiments and definitely shows that simple-beam theory equations cannot be employed for these materials to convert load-deflection data to stress-strain data. In order to prove this point, load-strain data were obtained for NaCl with the aid of strain gages mounted on the tension and compression faces of the specimens. Preliminary experiments in which load-deflection and load-strain data were obtained simultaneously proved that the presence of the strain gage did not affect the deformation behavior. The log-log plots of the load-strain data in figures 9 and 10 show that the deformation in the outer fibers does not satisfy Hooke's law. The load - compressive-strain curve is linear and corresponds to a parabolic relation between load and compressive strain. The load - tensile-strain curve is nonlinear and does not lead to a simple relation between load and tensile strain.

Effect of specimen treatment. - The length of the initial foot of the load-deflection curve was found to depend in a marked way on the history of the specimen. In order that a quantitative comparison of the effect of various treatments might be made, the "foot length" was defined as the intercept on the deflection axis of the tangent drawn through the point of inflection. The point of inflection and the tangent were determined analytically. The foot-length variations with treatment for NaCl are listed as averages for five or more specimens in table I. It can be seen that large variations in the extent of anelastic deformation can be produced.

The effect of treatment on deflection and load at failure is indicated in tables II, III, and IV, and representative behavior is shown in figures 11 to 15 for all three materials. No foot is discernible on these curves because of the compressed deflection scale used to show the point of failure. The values of deflection at fracture and load at fracture for NaCl in table IV are averages for at least five specimens, and the indicated limits are computed probable errors. The data of table IV for NaCl are extensive enough to indicate that improvement in ductility is always accompanied by an increase in strength, the variation in ductility being more pronounced.

The variation of strain on the compression side of the specimen with load for NaCl can be described by the equation

$$P = n\epsilon^m$$

where  $P$  is the load in pounds,  $\epsilon$  is the compressive strain, which was obtained from the strain-gage measurements, and  $n$  and  $m$  are constants. The parameters  $n$  and  $m$  varied with the specimen pretreatment as shown in table V.

An effect of specimen size on ductility has been known to exist for MgO: thin, freshly cleaved specimens can be bent easily in the

hand, while thicker specimens are brittle. Figure 16 serves to illustrate this point as well as the effect of time after cleavage. Increasing the thickness from 0.07 to 0.12 centimeter reduces the deflection at fracture by two-thirds. MgO specimens as thick as the NaCl ones used in these studies (0.3 cm) exhibit no ductility whatever.

### Compression Experiments

Constant strain rate. - Stress-strain curves obtained for NaCl in compression showed the same characteristics as the curves obtained in flexure, namely an initial foot and the lack of a Hooke's law region. An example is shown in figure 17. Data obtained at 0.02 and 0.002 inch per minute are listed in table VI for specimens as-received, water-polished, quenched from 700° C into carbon tetrachloride, and annealed. The variation of the stress and strain at fracture is more pronounced at the lower strain rate, but in neither case are the effects as large as they are in flexure.

Constant load. - Room-temperature creep experiments at a stress of 1700 psi were conducted for all three materials. NaCl exhibited the logarithmic behavior (fig. 18) reported in reference 11, while LiF did not (fig. 19). MgO did not creep at this stress and temperature.

The creep constants  $a$  and  $b$  for the equation  $\epsilon = a \log t + b$ , where  $\epsilon$  is compressive strain and  $t$  the time, are listed for NaCl in table VII. Specimen treatments which primarily affected the bulk structure of the material, such as heavy irradiation with X-rays or quenching, produced measurable effects, while treatments which altered only the surface, for example, cleaving and testing in oil, were essentially ineffective.

### Etch-Pit Studies

The etch-pit technique of studying fresh dislocations, developed for LiF by the work of reference 7, was applied to NaCl single crystals. Large crystals (20 by 6 by 6 mm) were cleaved in half lengthwise and etched, and the freshly cleaved faces were examined microscopically. As can be seen in figure 20, the matching cleavage faces are essentially identical in dislocation etch-pit density, distribution, and arrangement. This result makes it possible to study the effect of various treatments on the fresh dislocation structure of a specimen. Crystals were cleaved in half, and one half was subjected to a specific treatment, while the matching half was not. Both halves were then etched and examined. The results of such investigations are shown in figures 21 to 23. Water-polishing and annealing both reduce the dislocation density, while quenching increases the density. It must be noted that this was a true annealing, that is, slow heating and slow cooling.

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Since fresh dislocations are produced in a specimen during cleavage, the cleavage process was examined in detail using the etch-pit technique. A lengthwise, bisecting cleavage crack was initiated in a crystal measuring 20 by 6 by 6 millimeters by striking a sharp blow on a chisel placed on an edge of the crystal. The partially cleaved crystal was immediately etched, and the faces normal to the plane of the crack were examined. Figure 24 shows a length of the crack as seen from both sides of the crystal. The side of the crystal containing the edge on which the chisel was placed shows regular arrays of dislocation etch pits and considerably less damage than the opposite side, which shows no regular arrays but a high density of etch pits ahead of and near the crack tip. It can also be seen from the figure that the damage extends to a considerable distance from the crack.

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An interesting result shown in figure 25 is that dislocations are still moving in a crystal for a considerable time after the applied stress is removed. This figure shows a portion of the face normal to the plane of the cleavage crack. The portion shown is far removed from the end of the crack (approximately 5 mm) and is on the side of the crystal containing the edge on which the chisel was placed. The flat-bottomed pits indicate that, during the 30 seconds of etching time, dislocations were still moving, and this was about 2 minutes after the stress applied during cleavage had been removed. It can be seen that the dislocations moved in a jogging manner with a velocity of approximately  $10^{-5}$  centimeter per second.

In figure 26 two etched cleavage faces are shown. For the crystal cleaved by successively propagating the crack by light taps on the chisel, a high density of etch pits can be seen, while for the crystal cleaved rapidly, that is, by propagating the crack with one sharp tap on the chisel, there are few regular arrays and a smaller density of dislocation etch pits.

## DISCUSSION

The experimental data reported herein indicate that the mechanical behavior of ionic crystals is dependent on a large number of variables. The data obtained with NaCl are extensive enough to permit some speculation on the mode by which anelastic motion of dislocations might occur and on the mode by which specimen treatment might affect the extent of anelastic deformation and the stress and deflection at fracture.

Recent work (refs. 4 and 7) on the nature of the dislocation loops introduced into a crystal during cleavage indicates that they might play an important role in the deformation. These dislocation loops differ from "grown-in" dislocations in that they can be caused to move readily on the application of low stress, probably because they are not encumbered

by a Cottrell atmosphere. The "cleaved-in" loops also differ in their distribution in the crystal from the grown-in ones in that they are far more numerous in the surface layers than in the interior.

These properties make it possible for the cleaved-in dislocations to produce two different effects on the deformation behavior. Their response to very low stresses could make them responsible for the initial anelastic deformation of small slope. Their large concentration at the surface would create a stress field which would inhibit the loss through the surface of dislocations moving along the slip planes in the later stages of deformation.

It is suggested that the application of low stress causes the cleaved-in loops to expand until they interact with the stress fields of dislocations which remain static under the low applied stress. The expansion of a particular loop would begin when the applied stress reached its activation stress and would cease when the opposing stress balanced the applied stress; for these low stresses, the latter point will be reached long before the two dislocations interact strongly enough to cause permanent locking. When the applied stress is removed, the loops will be free to contract under the influence of the back stress of static dislocations with the aid of thermal activation to overcome dissipative forces.

On the basis of this picture, the large decrease in foot length produced by water-polishing NaCl is due to the removal of the surface region containing a large number of cleaved-in dislocations. The increase in foot length for NaCl specimens quenched from 700° C to room temperature might be expected for two reasons: (1) the stresses introduced during quenching can create fresh dislocation loops having low activation stresses and (2) the high-temperature treatment boils off vacancies and impurity atoms from the grown-in dislocation and thus produces loops with lower activation stress.

In a well-annealed specimen, each dislocation has a Cottrell atmosphere, and thus a decrease in foot length would be expected to occur after annealing. The anneal used in the present study resulted in no significant change in foot length. However, the annealing procedure employed included a very rapid rise to the high temperature. Reference 3 reports that "up-quenching" gives rise to extensive slip. The additional dislocations thus produced would tend to increase the foot length, and it is impossible to predict the net result. The experimental results would indicate that the increase in dislocation concentration offset the effect of the decrease in loop length due to annealing.

The result of X-irradiation, namely a large increase in foot length, is in accord with the softening observed in the creep of NaCl after short irradiation (ref. 10). In that case it was proposed that the first effect

of irradiation by X-rays was to disperse vacancy clusters which were in the vicinity of dislocations. Such a dispersal would again create loops having lower activation stresses.

In addition to causing marked changes in the length of the initial portion of the load-deformation curve, the various methods of pretreating the specimens also affected the deflection and load at fracture. Since the failure of these crystals was observed to be ultimately caused by the nucleation of a crack on the tension face and its propagation through the crystal, it is of interest to examine the results in this light. The crack nucleus in all probability results from the interaction of a surface flaw with dislocations arriving at the surface. On this basis, it would be predicted that the pretreatment which would lead to the greatest ductility would be that which makes the surface most free of crack nucleation sites.

Electron diffraction studies of X-irradiated NaCl surfaces have illustrated the role of surface stresses in the disorientation of NaCl surfaces by X-irradiation (ref. 11). As-cleaved crystals were disrupted much more readily than were annealed or water-polished ones. A study of the growth on water-polished NaCl surfaces of nucleation sites for the crystallization of evaporated silver films (ref. 12) showed that surface irregularities grow by a thermally activated rate process. The application of stress to annealed water-polished crystals increased the rate of formation of the nucleation sites for crystallization of the silver. The observed improvements in deflection and load at fracture by treatments such as annealing and water treatment are in accord with the predictions that might be made with regard to their effect on crack nucleation.

Finally, it is interesting to consider the significance of the observation during the etching studies that the cleaved-in dislocations move for periods as long as several minutes after cleavage. This observation confirms the expectation that cleaved-in dislocations can move slowly to equilibrium positions as the result of the stress remaining after the applied stress is removed. It also suggests that a possible mode for the embrittling with time after cleavage of cleaved crystals may be the motion of these dislocations to the surface with the additional aid of the image force attraction and the resultant formation of crack nuclei. Dislocations with very low activation stresses would likewise be expected to contribute to the "instantaneous" deformation in creep. The data presently available indicate that a correlation may exist between the creep constant  $b$  and the foot length (the creep constant  $b$  being the strain at unit time). More extensive information is needed in order that this apparent correlation may be studied in more detail.

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## SUMMARY OF RESULTS

A study was made of the relative magnitude of the effects of various factors on the ductility of single crystals of NaCl, LiF, and MgO. Specimen treatments included water-polishing, varying cleavage rate, annealing, quenching, X-irradiation, surface coating, aging, and combinations of some of these treatments. The mechanical behavior of the crystals was studied in flexure and in compression, the latter study being performed at both constant strain rate and constant load. Etch-pit studies were carried out to provide some pertinent information on the results of pretreatment on the dislocation concentration and distribution in the vicinity of the surface. The results were as follows:

1. The load-deformation curves for these ionic single crystals show an initial region of very low slope, which proved to be due to anelastic deformation.

2. The extent of initial anelastic deformation is modified by specimen pretreatment in a way that suggests that this deformation is the result of expansion of cleaved-in dislocation loops which can contract on the removal of the stress.

3. The effects of the various pretreatments on the load and deflection at fracture are in accord with the prediction that might be made with regard to their effect on the nucleation of fatal surface cracks.

4. For NaCl, increases in ductility are always accompanied by increases in strength.

5. The creep constants for NaCl are a function of treatments which affect the bulk structure but are not a function of treatments which affect only the surface.

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TABLE I. - EFFECTS OF TREATMENTS ON LENGTH OF "FOOT"  
OF LOAD-DEFLECTION CURVE FOR SODIUM CHLORIDE  
SPECIMENS FROM GROUP I

[Tested at constant deflection rate of 0.002 in./min.]

Number of specimens	Treatment	Foot-length, in.
7	As-cleaved	$2.5 \pm 0.3 \times 10^{-4}$
5	Annealed 24 hr at 700° C and furnace-cooled	$2.4 \pm 0.3$
7	Water-polished	$0.6 \pm 0.1$
4	X-irradiated for 72 hr	$11.8 \pm 2.1$
5	Quenched after being held at 700° C for 24 hr to 25° C in $\text{CCl}_4$	$4.6 \pm 0.5$

TABLE II. - EFFECTS OF TREATMENTS ON DEFLECTION AND LOAD AT FAILURE  
AND ON LENGTH OF "FOOT" OF LOAD-DEFLECTION CURVE FOR  
LITHIUM FLUORIDE SPECIMENS

[Tested at constant deflection rate of 0.002 in./min.]

Specimen width, b, cm	Specimen thickness, d, cm	Treatment	Deflection at failure, in.	Load at failure, lb	Foot length, in.
0.6725 .7583 .7584	0.2002 .1768 .1976	As-cleaved	$142 \times 10^{-4}$ 200 119	1.90 1.93 2.13	$4.2 \times 10^{-4}$ 4.5 2.1
0.7275 .7552 .7551 .6685	0.2122 .1582 .1872 .2122	Etched 1 min in HF and then polished in $\text{NH}_4\text{OH}$	$370 \times 10^{-4}$ 399 214 380	2.86 1.55 2.07 2.35	$2.3 \times 10^{-4}$ 1.1 1.5 2.4
0.6725 .6465	0.2305 .2070	Etched 1 min in HF	$286 \times 10^{-4}$ 382	3.26 2.28	$0.3 \times 10^{-4}$ 1.0

TABLE III. - EFFECTS OF TREATMENTS ON DEFLECTION AND LOAD AT  
FAILURE AND ON LENGTH OF "FOOT" OF LOAD-DEFLECTION  
CURVE FOR MAGNESIUM OXIDE SPECIMENS

[Tested at constant deflection rate of 0.01 in./min.]

Specimen (a)	Specimen width, b, cm	Specimen thickness, d, cm	Treatment	Deflection at failure, in.	Load at failure, lb	Foot length, in.
1	0.6929	0.1206	As-cleaved, tested 30 sec after cleavage	131 $\times 10^{-4}$	22.10	8.5 $\times 10^{-4}$
2	.4008	.1174		66	10.98	6.3
3	.5070	.0805		40	4.58	8.8
4	.5194	.0721		412	6.54	8.5
5	.4714	.0620		110	3.78	8.7
1'	0.7061	0.1107	As-cleaved, tested 5 hr after cleavage	54 $\times 10^{-4}$	16.95	8.8 $\times 10^{-4}$
2'	.4539	.1275		19	6.82	5.5
3'	.5082	.1252		23	8.20	6.0
4'	.5159	.0864		166	8.58	5.5
5'	.4707	.0800		52	5.29	7.2
6	0.5789	0.0818	As-cleaved, tested 24 hr after cleavage	165 $\times 10^{-4}$	8.28	4.9 $\times 10^{-4}$
7	.6546	.0826		29	3.85	8.2
11	.6076	.0452		70	1.74	12.1
12	.6152	.0432		271	2.42	13.6
13	.6497	.0457		244	2.89	9.3
8	0.5850	0.0818	Polished 30 min in hot concentrated H <sub>3</sub> PO <sub>4</sub>	169 $\times 10^{-4}$	7.33	3.6 $\times 10^{-4}$
9	.5349	.0754		50	4.45	11.0
10	.5618	.0828		43	4.72	12.7
14	.5712	.0452		488	2.77	11.3
15	.5994	.0544		317	3.93	5.6
16	.5796	.0589		458	4.07	10.1

<sup>a</sup>Primed number denotes crystal cleaved from specimen with corresponding nonprimed number.

TABLE IV. - DEFORMATION AND LOAD AT FAILURE FOR SODIUM CHLORIDE  
SPECIMENS OF GROUP II SUBJECTED TO PRETREATMENT

[Tested at constant deflection rate of 0.002 in./min.]

Number of specimens	Treatment	Deflection at failure, in.	Load at failure, lb
11	As-cleaved	$47 \pm 5 \times 10^{-4}$	$2.1 \pm 0.2$
5	Annealed 24 hr at 700° C	$710 \pm 40$	$7.1 \pm 2.0$
5	Annealed 24 hr at 700° C and water-polished	$1450 \pm 160$	$14.5 \pm 3.9$
5	Heated 1 hr at 135° C	$38 \pm 5$	$2.2 \pm 0.3$
5	Heated 1 hr at 135° C and water-polished	$165 \pm 144$	$3.3 \pm 0.3$
10	Water-polished	$343 \pm 53$	$3.2 \pm 0.2$
6	Water-polished and heated 1 hr at 135° C	$367 \pm 98$	$4.8 \pm 0.5$
5	Water-polished and coated with silver	$176 \pm 11$	$2.4 \pm 0.5$
5	Coated with silver	$112 \pm 23$	$2.7 \pm 0.6$

TABLE V. - EFFECT OF TREATMENTS ON PARABOLIC EQUATION ( $P = \eta \epsilon^m$ )  
PARAMETERS FOR SODIUM CHLORIDE SPECIMENS

FROM GROUP I TESTED IN BENDING

[Constant deflection rate of 0.002 in./min.]

Number of specimens	Treatment	m	n
3	As-cleaved	$0.43 \pm 0.02$	$38 \pm 4$
3	Tension side water-polished	$.32 \pm 0.02$	$17 \pm 1$
3	Annealed 40 hr at 700° C	$.35 \pm 0.06$	$21 \pm 5$

TABLE VI. - EFFECTS OF TREATMENTS ON STRESS AND STRAIN

AT FAILURE FOR SODIUM CHLORIDE SPECIMEN

[Tested at constant rates of compression.]

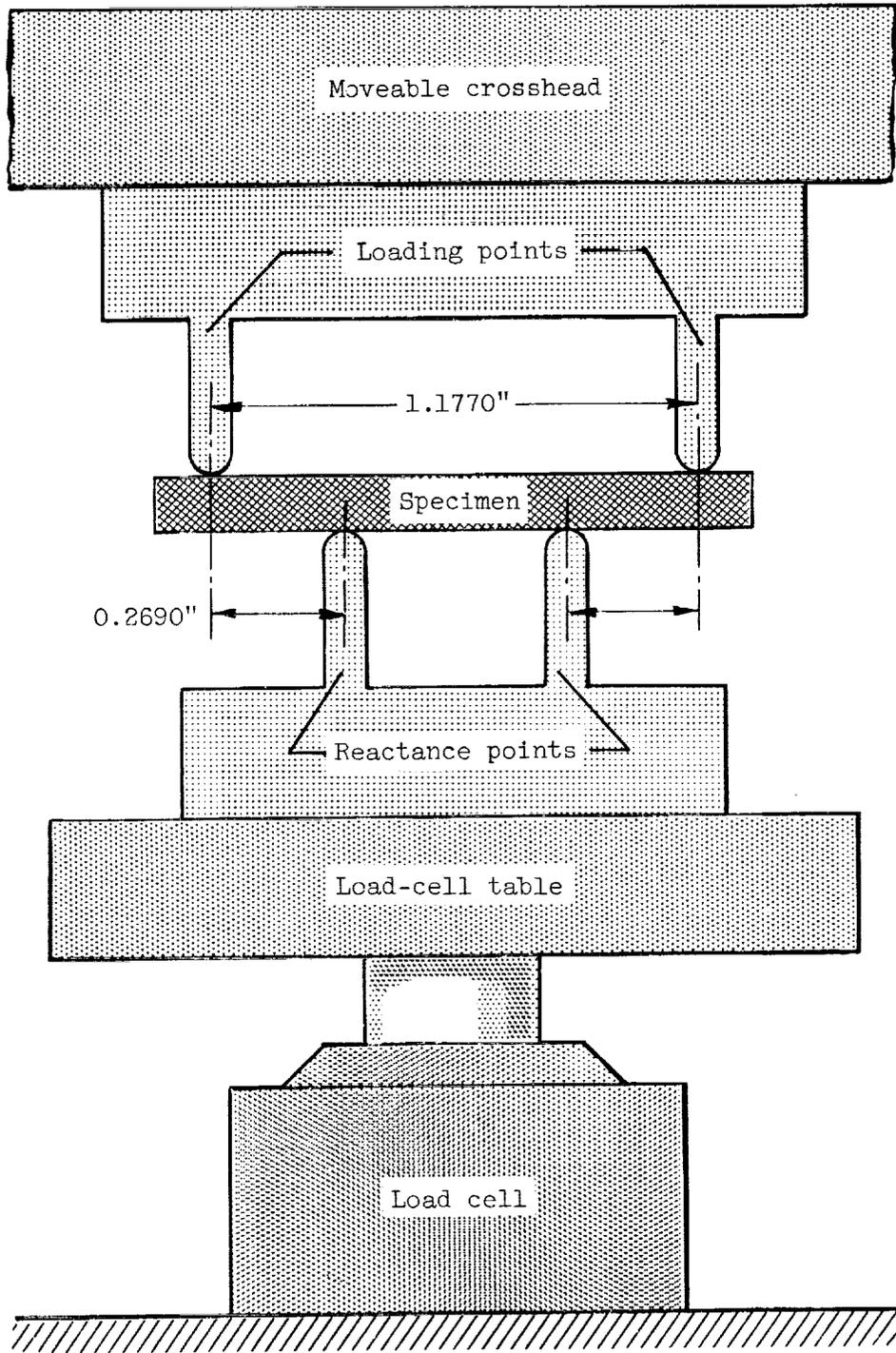
Compression rate, in./min	Treatment	Fracture stress, psi	Fracture strain, in./in.
0.02	As-cleaved	5204	$152.2 \times 10^{-3}$
	Water-polished	5933	163.3
	Quenched to 25° C in CCl <sub>4</sub> after being at 700° C for 4 hr	5100	134.8
	Annealed 4 hr at 700° C	5849	173.5
0.002	As-cleaved	4899	$152.2 \times 10^{-3}$
	Quenched to 25° C in CCl <sub>4</sub> after being at 700° C for 4 hr	7964	242.6
	Annealed 4 hr at 700° C	5893	199.8

TABLE VII. - EFFECTS OF TREATMENTS ON LOGARITHMIC CREEP CONSTANTS

(ε = a log t + b) FOR SODIUM CHLORIDE SPECIMENS

[Tested at constant stress of 1700 psi.]

Specimen group	Treatment	a	b
III	As-cleaved	$1.7 \pm 0.1 \times 10^{-4}$	$8.0 \pm 0.3 \times 10^{-2}$
	Water-polished	$2.1 \pm 0.1$	$6.2 \pm 0.3$
	Annealed 24 hr at 600° C	$2.5 \pm 0.3$	$6.8 \pm 1.0$
	Annealed 24 hr at 700° C	$2.8 \pm 0.2$	$6.8 \pm 0.3$
	X-irradiated 15.5 hr	$3.8 \pm 0.6$	$8.8 \pm 1.3$
	X-irradiated 40 hr	$4.4 \pm 0.9$	$11.2 \pm 0.2$
	X-irradiated 100 hr	$15.3 \pm 4.5$	$8.9 \pm 1.4$
	Annealed 24 hr at 600° C, water-polished and X-irradiated 15.5 hr	$7.1 \pm 3.2$	$7.6 \pm 0.8$
	Annealed 24 hr at 600° C, water-polished and X-irradiated 40 hr	$6.2 \pm 1.6$	$9.6 \pm 1.0$
	Annealed 4 hr at 700° C	$2.2 \pm 0.2$	$8.0 \pm 0.5$
	Annealed 4 hr at 700° C and quenched to 25° C in CCl <sub>4</sub>	$2.6 \pm 0.2$	$2.6 \pm 0.1$
IV	Cleaved in air and tested in air	$3.3 \pm 0.3 \times 10^{-4}$	$5.0 \pm 0.4 \times 10^{-2}$
	Cleaved in oil and tested in oil	$2.3 \pm 0.5$	$5.2 \pm 0.5$
	Cleaved in oil and tested in CCl <sub>4</sub>	$2.9 \pm 0.2$	$5.0 \pm 0.2$
	Cleaved in oil, washed with acetone, and tested in air	$2.7 \pm 0.3$	$4.2 \pm 0.1$
	Cleaved in oil, washed with ether, and tested in air	$2.7 \pm 0.3$	$4.1 \pm 0.1$



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Figure 1. - Bending jig with specimen in test position.

E-427

CP-3 back

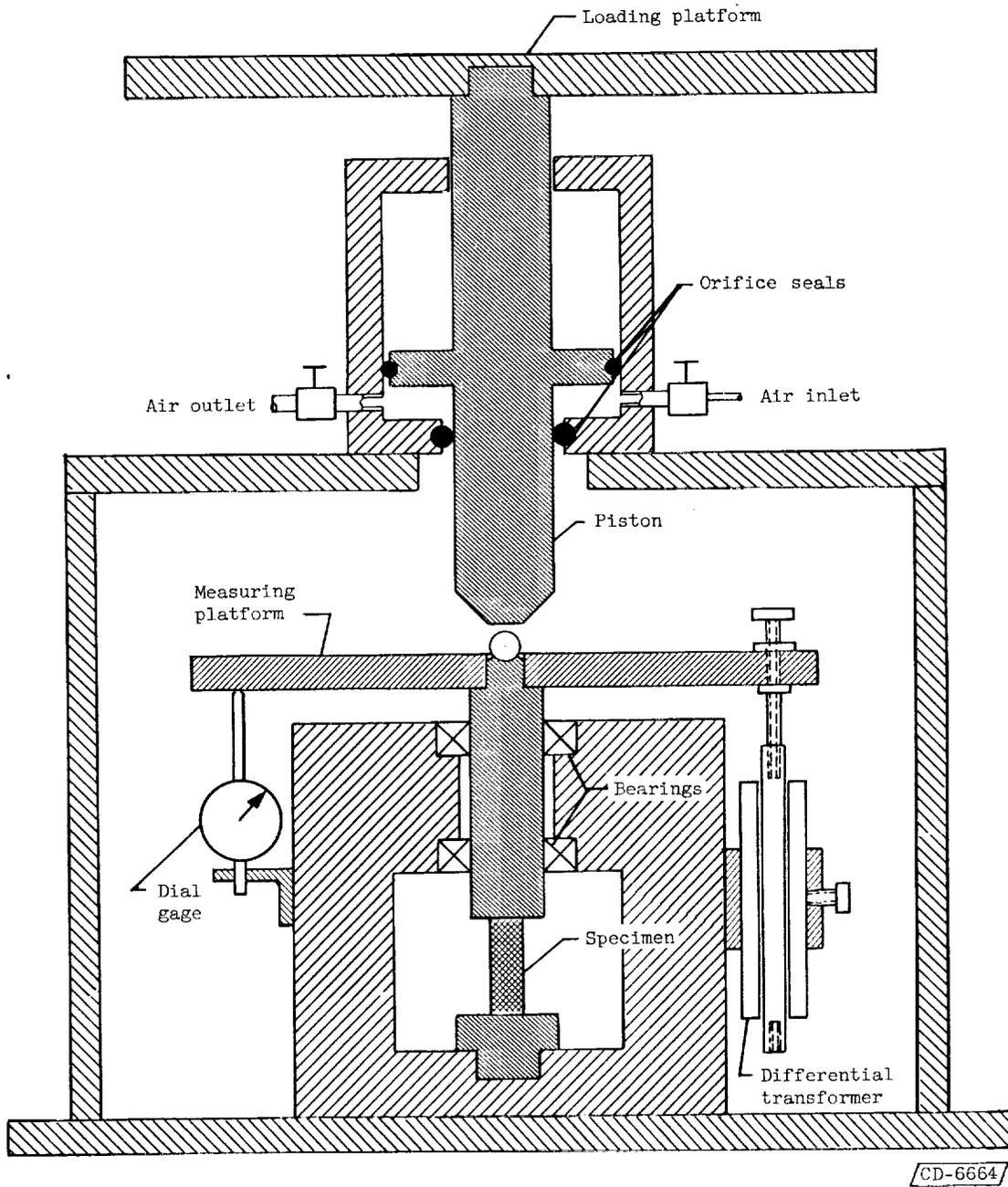


Figure 2. - Creep apparatus with specimen in test position.

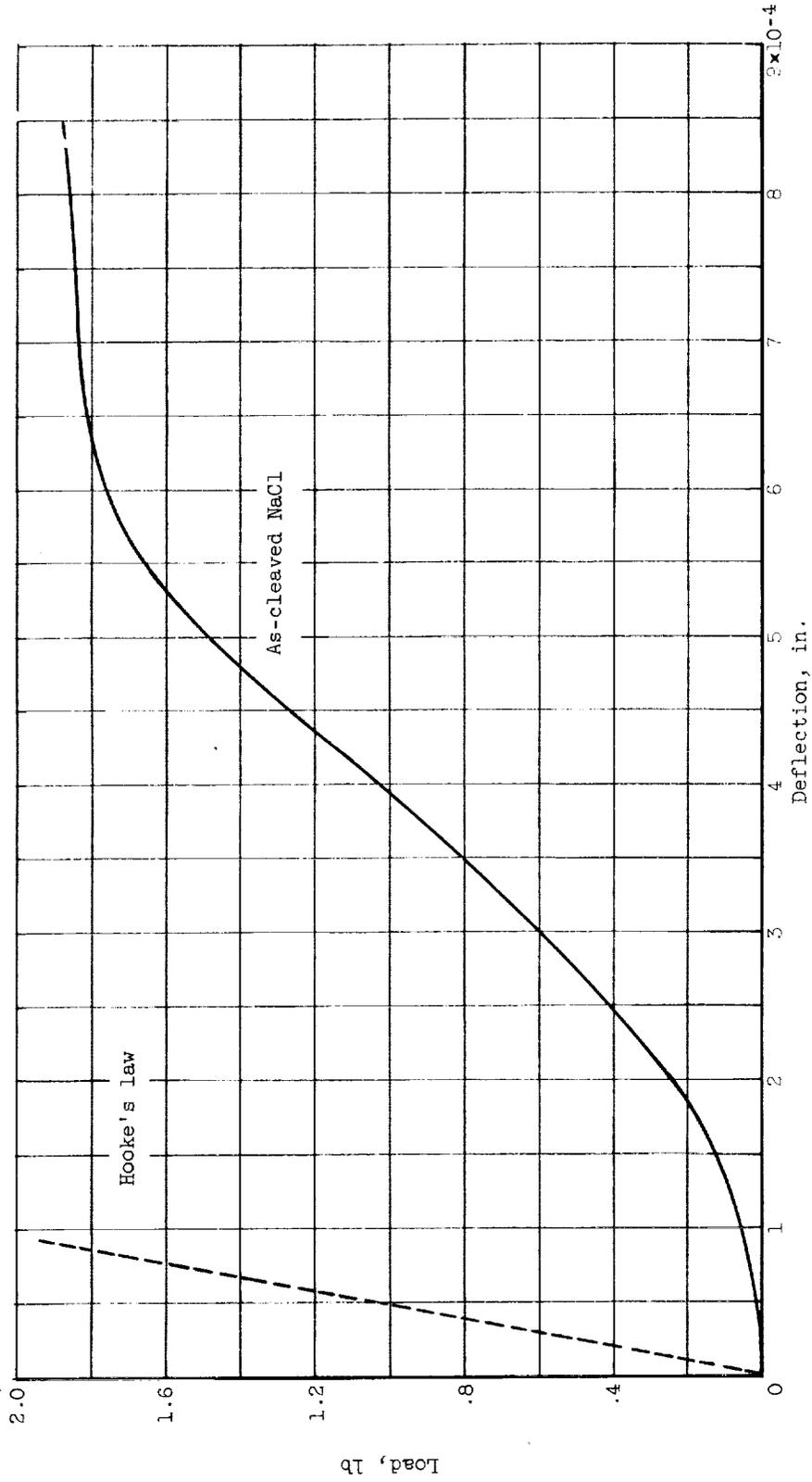


Figure 3. - Initial portion of load-deflection curve for as-cleaved sodium chloride single crystal showing low-slope anelastic region. Crystal width, 0.7750 centimeter; thickness, 0.3110 centimeter.

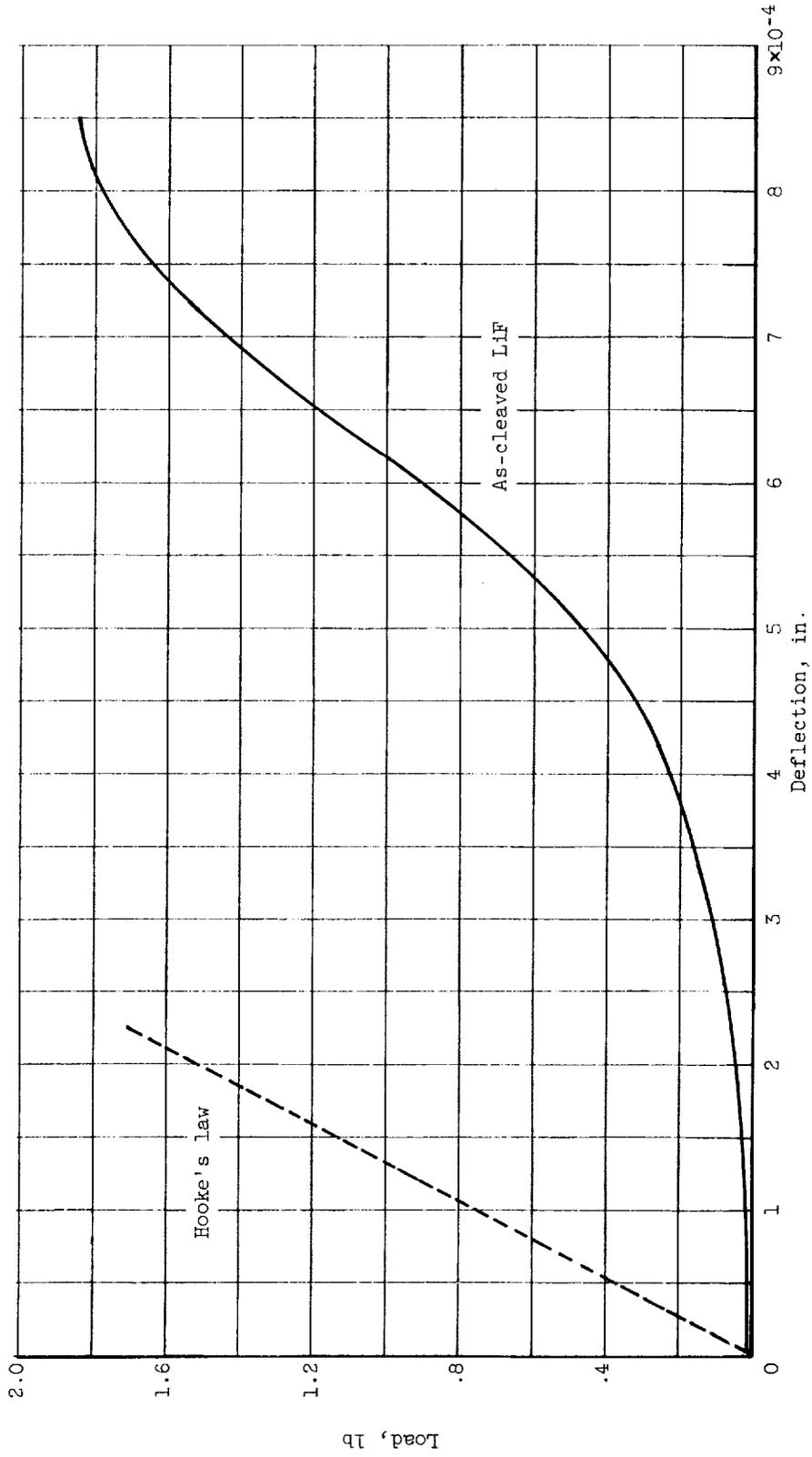


Figure 4. - Initial portion of load-deflection curve for as-cleaved lithium fluoride single crystal showing low-slope anelastic region. Crystal width, 0.6725 centimeter; thickness, 0.2002 centimeter.

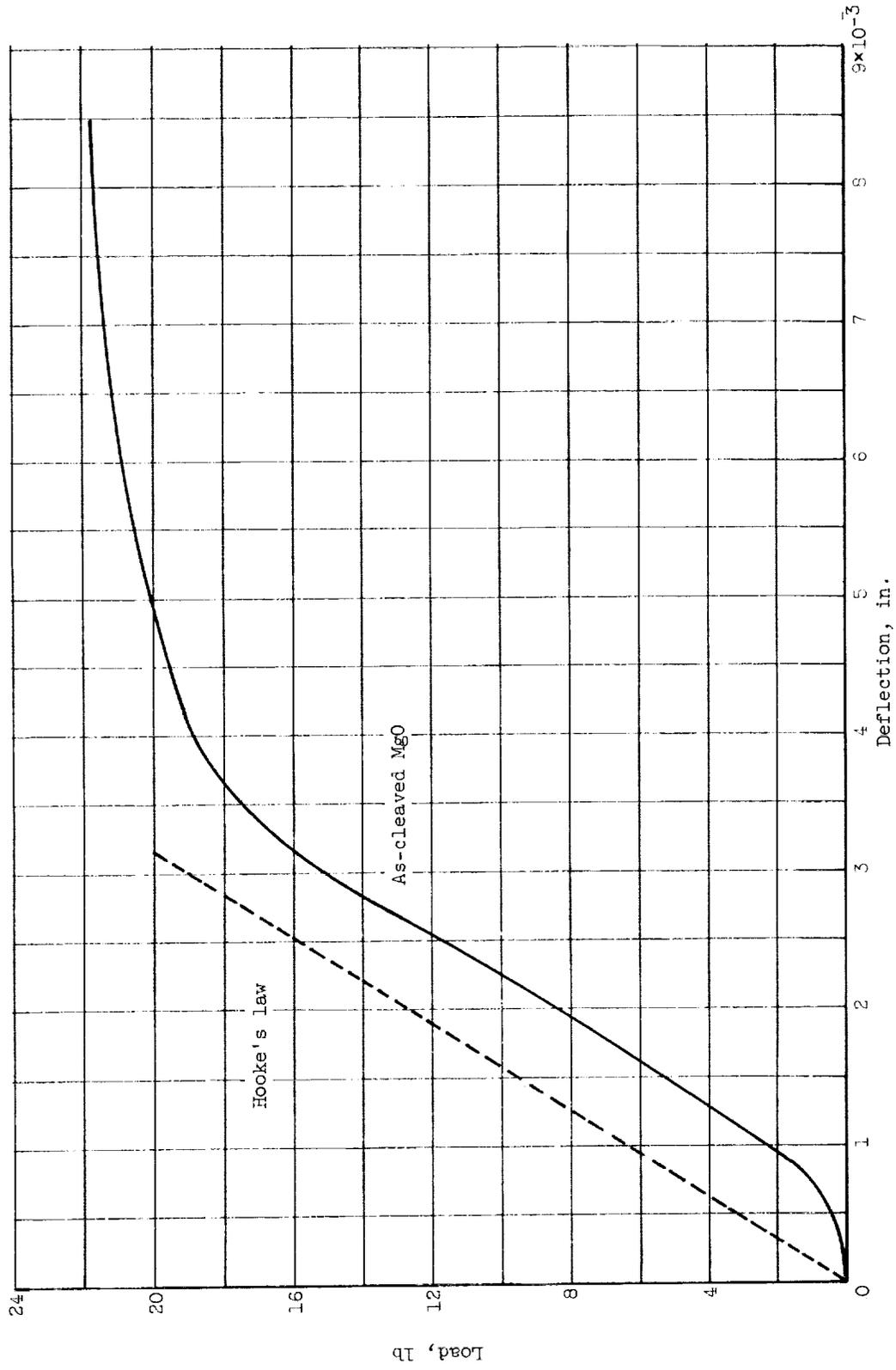


Figure 5. - Initial portion of load-deflection curve for as-cleaved magnesium oxide single crystal showing low-slope anelastic region. Crystal width, 0.6929 centimeter; thickness, 0.1207 centimeter.

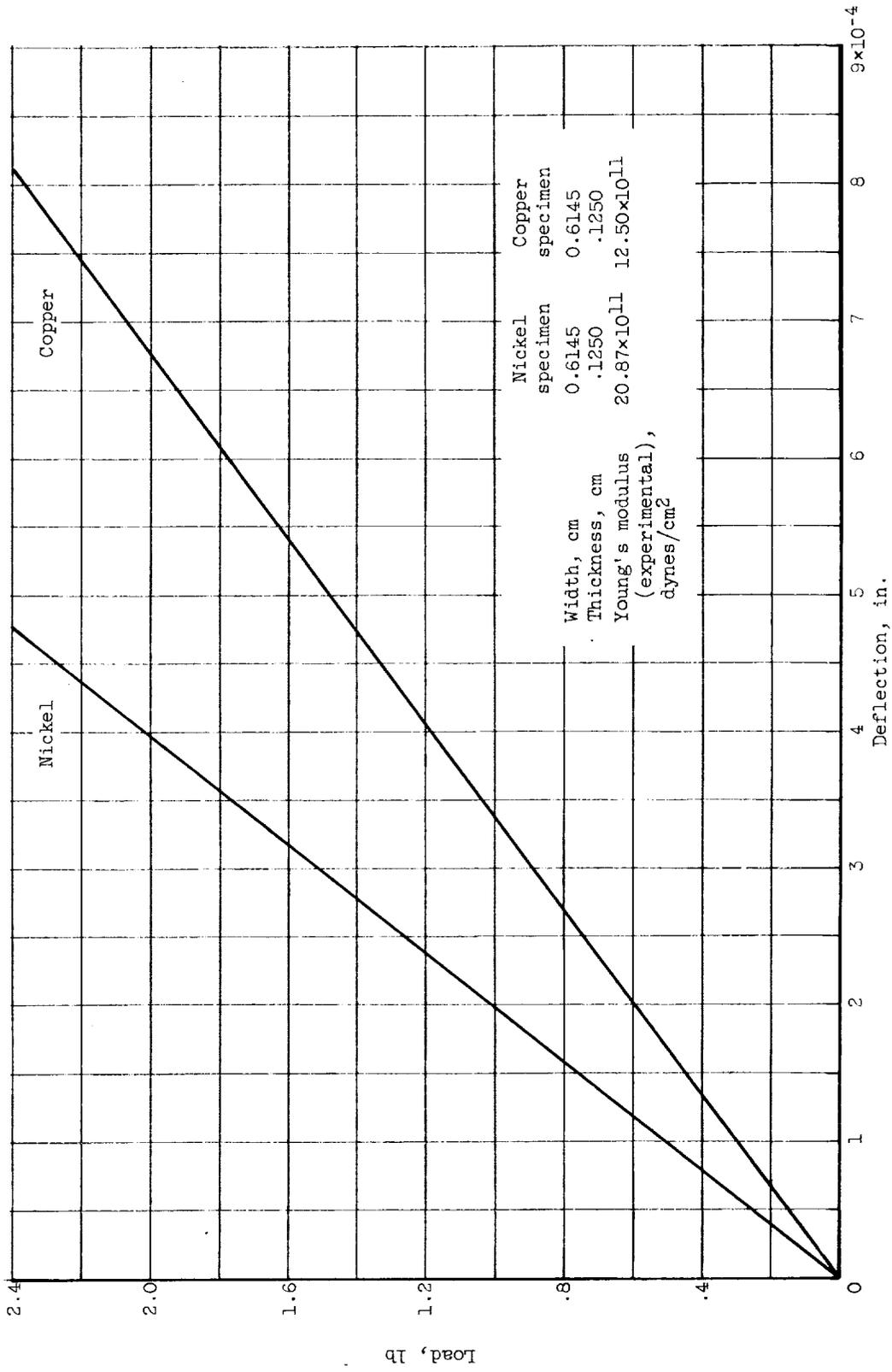


Figure 6. - Load-deflection curves for polycrystalline nickel and copper specimens showing straight-line Hooke's law dependence.

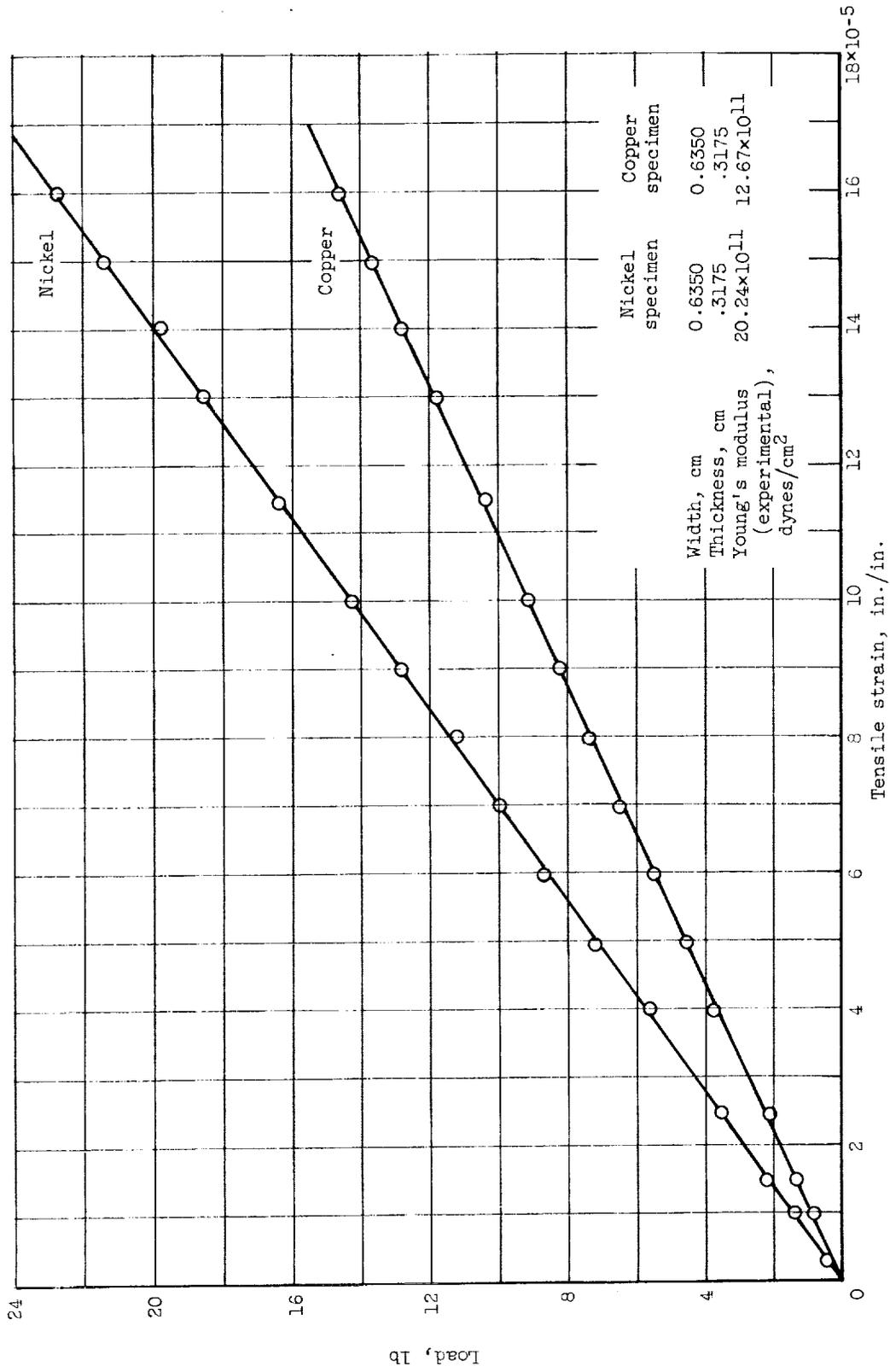


Figure 7. - Load-strain curves for polycrystalline nickel and copper specimens showing straight-line Hooke's law dependence.

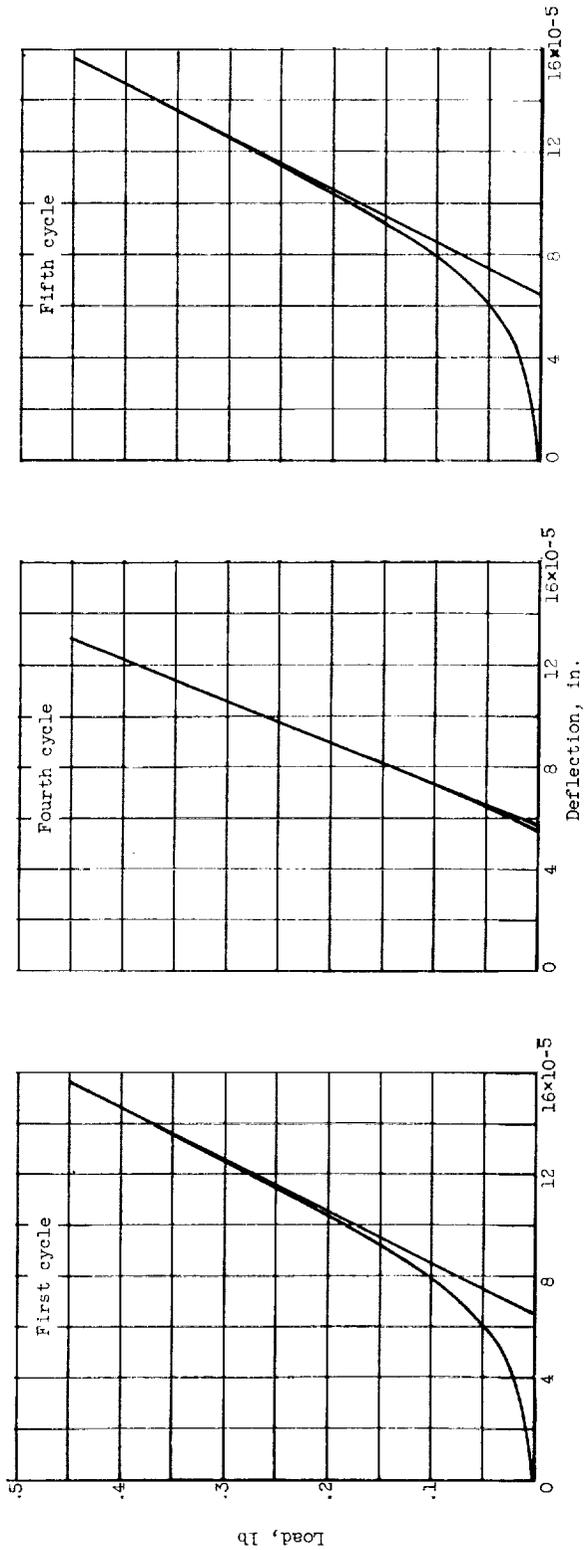


Figure 8. - Cyclic loading and unloading of lithium fluoride single crystal showing anelastic behavior. First through fourth cycles were run consecutively; fifth was run 24 hours after fourth.

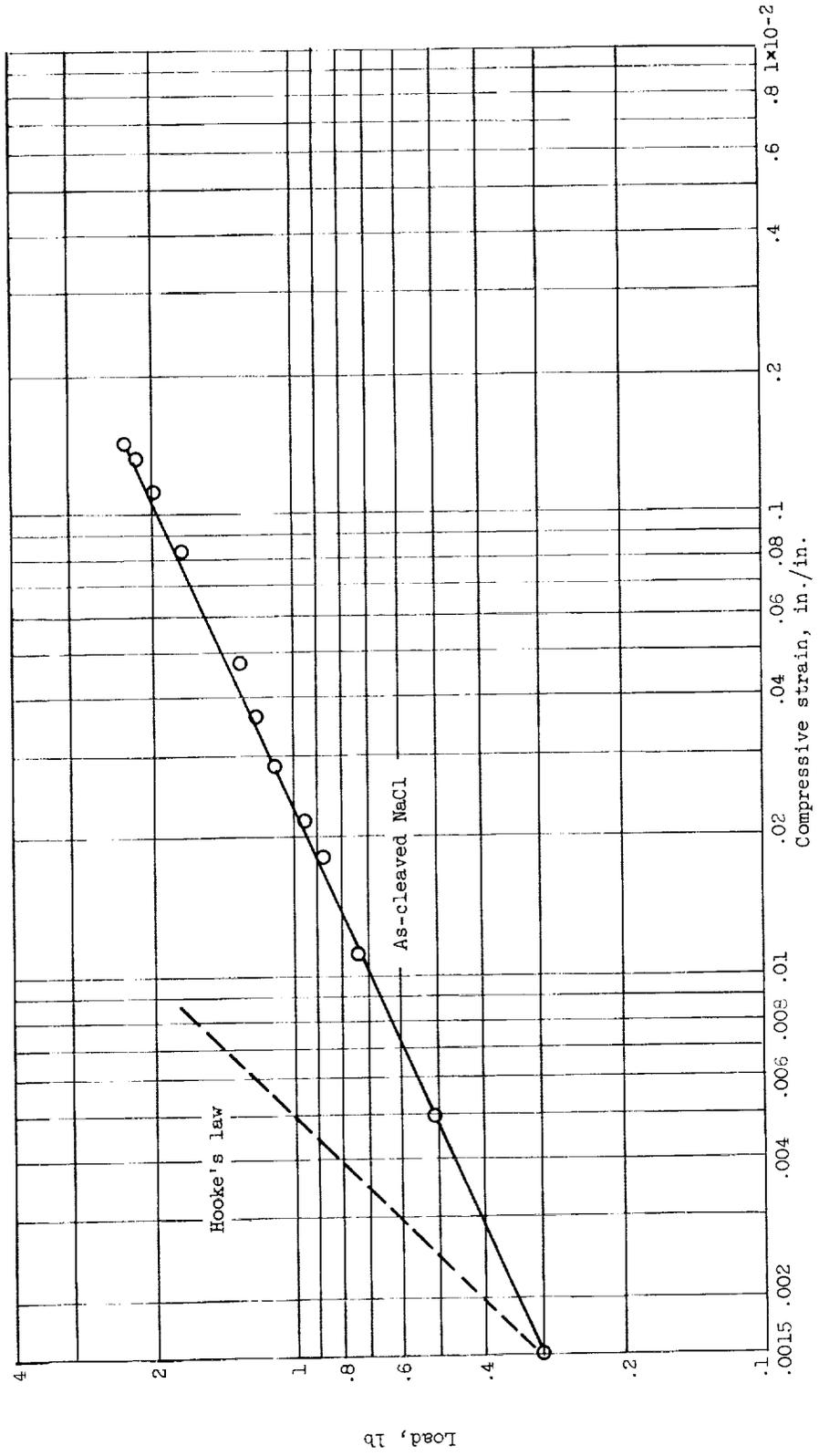


Figure 9. - Load - Compressive-strain curve for sodium chloride single crystal showing deviation from Hooke's law. Crystal width, 0.7324 centimeter; thickness, 0.3045 centimeter.

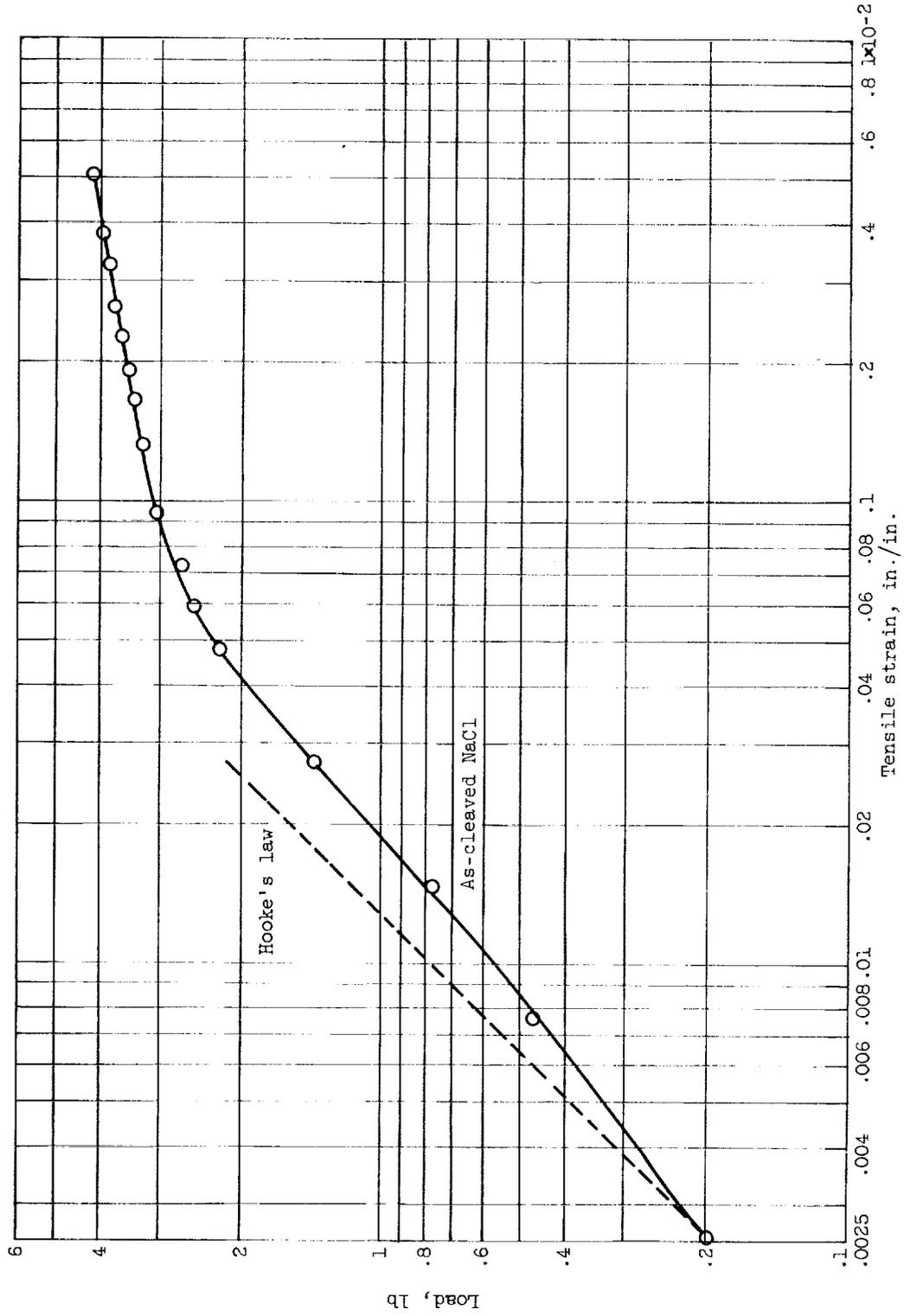


Figure 10. - Load - tensile-strain curve for sodium chloride single crystal showing lack of straight-line dependence and deviation from Hooke's law. Crystal width, 0.7324 centimeter; thickness, 0.3045 centimeter.

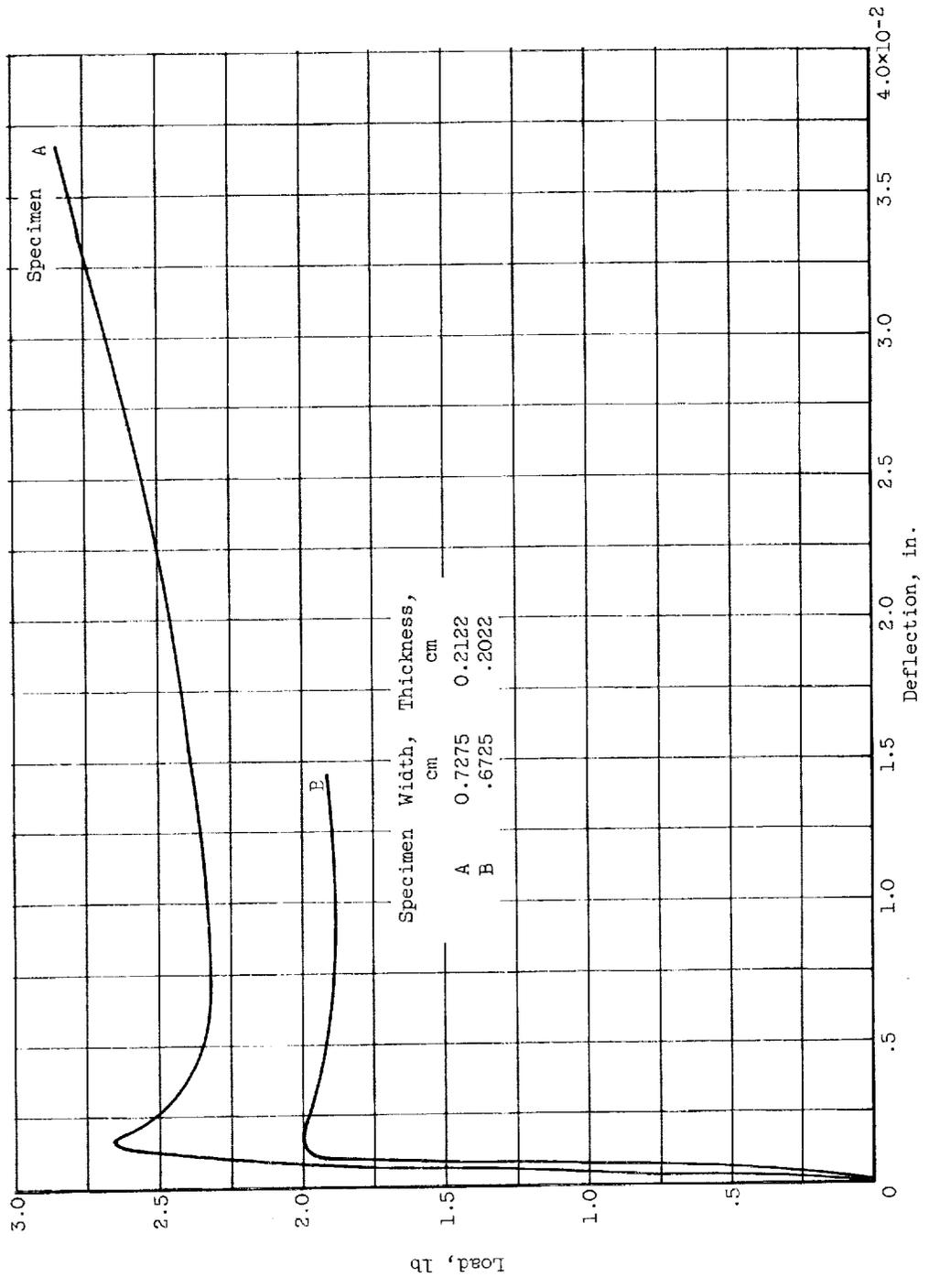


Figure 11. - Effect of chemical polishing on load-deflection curves for lithium fluoride single crystals. Specimen A, etched with concentrated hydrofluoric acid and polished in ammonium hydroxide; B, as-cleaved.

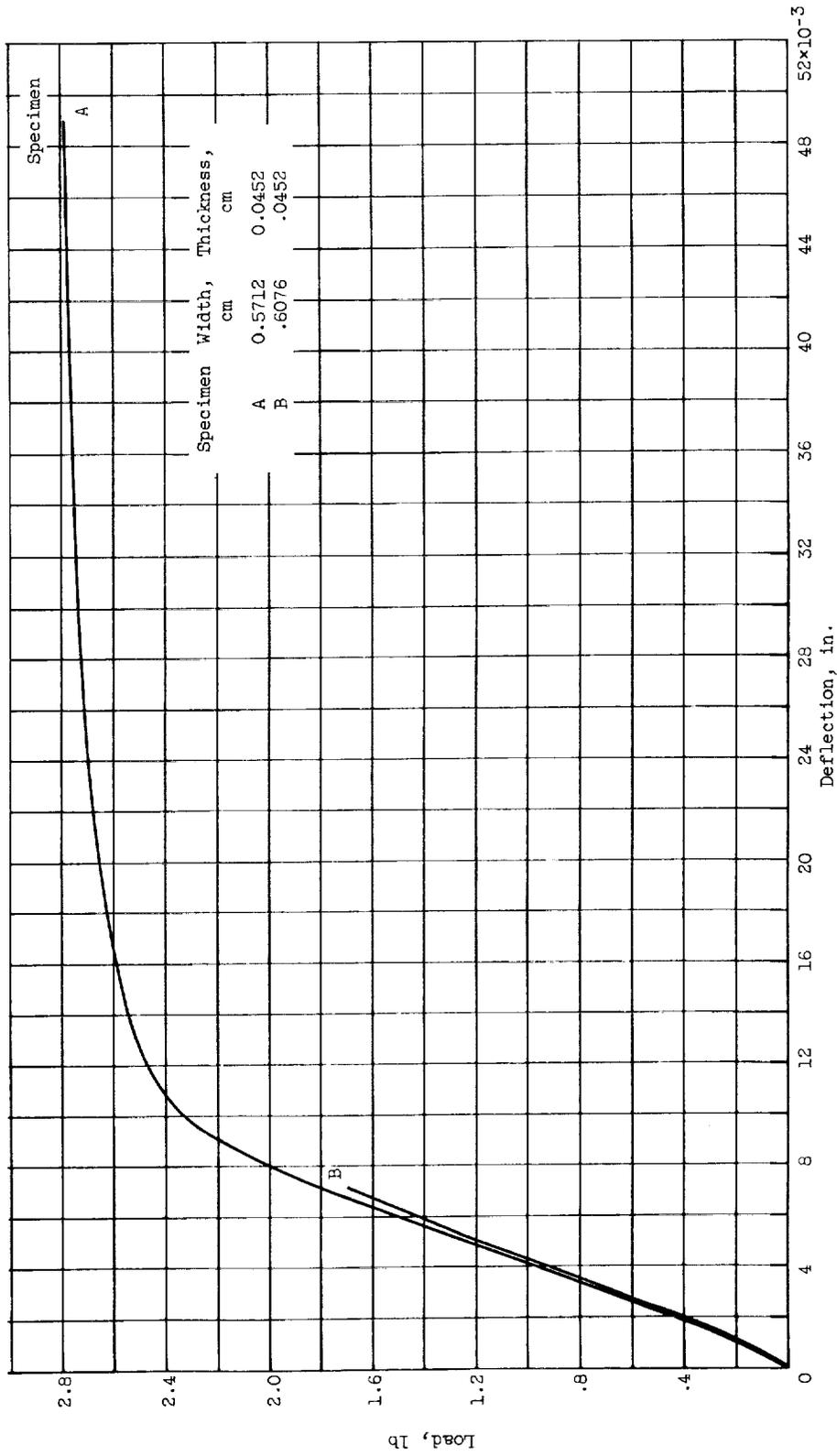


Figure 12. - Effect of chemical polishing on load-deflection curves for magnesium oxide single crystals. Specimen A, polished with concentrated phosphoric acid; B, as-cleaved.

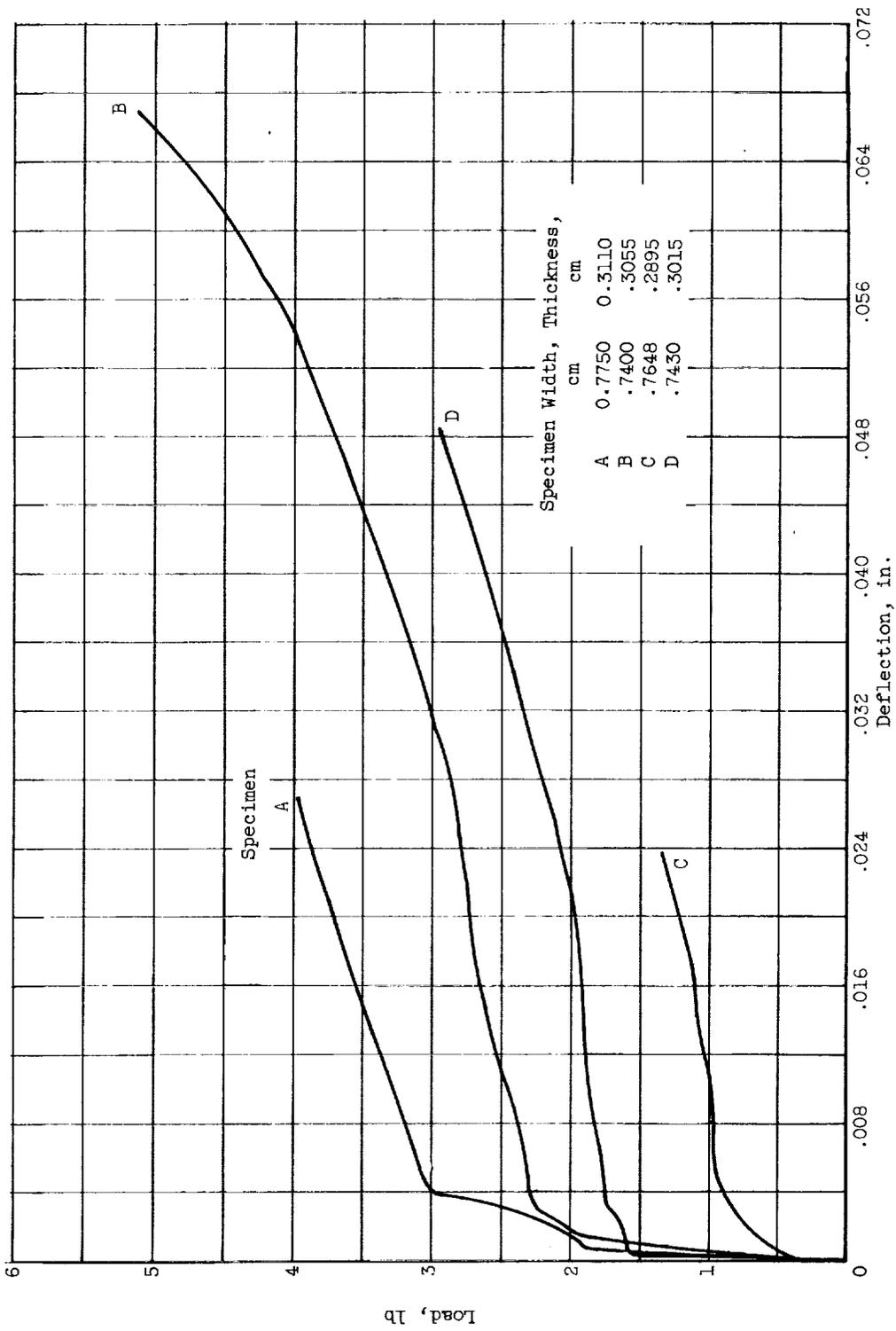


Figure 13. - Effect of partial and complete polishing on load-deflection curves for sodium chloride single crystals. Specimen A, as-cleaved; B, tension side water-polished; C, compression side water-polished; D, both sides water-polished.

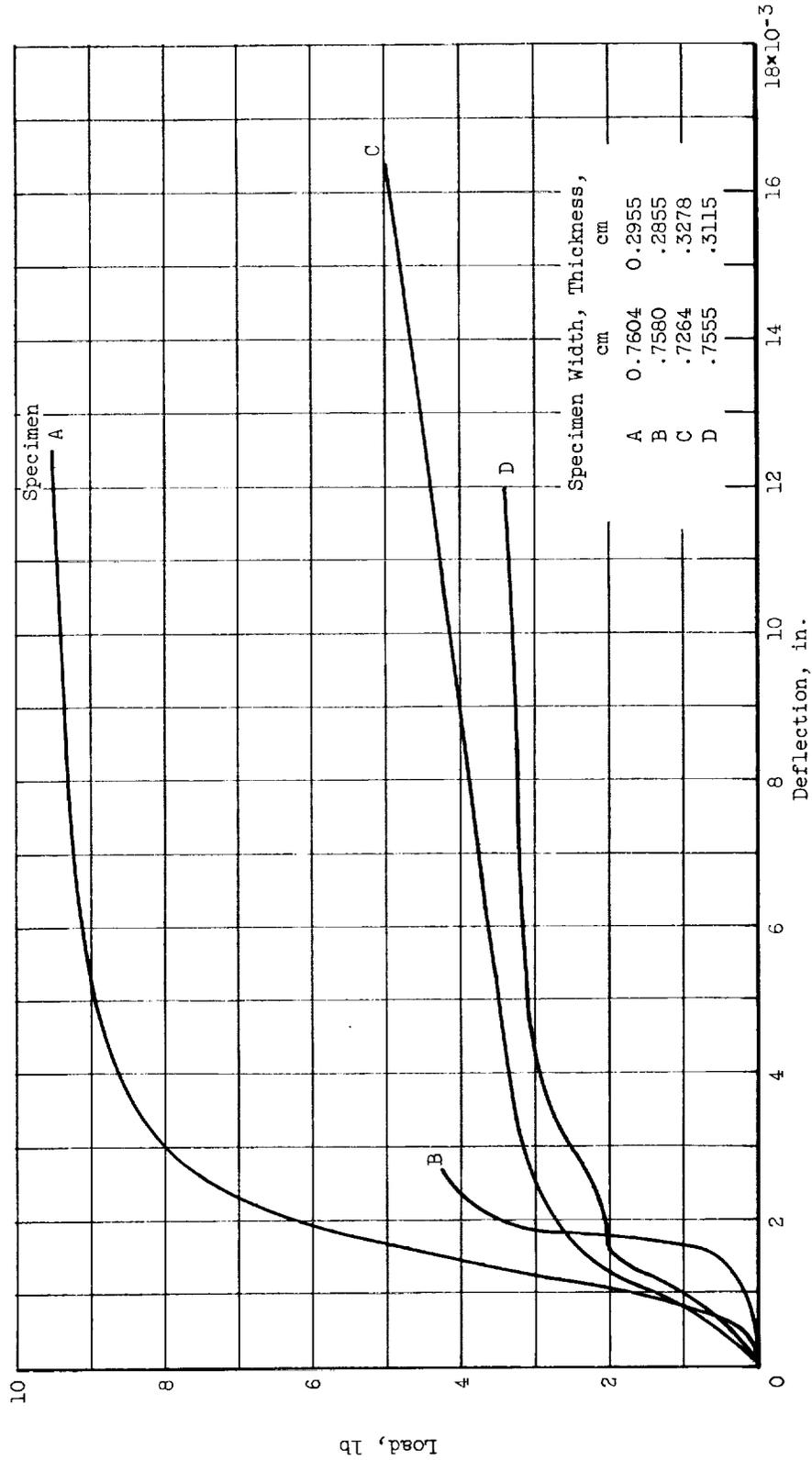


Figure 14. - Effect of bulk treatments on load-deflection curves for sodium chloride single crystals. Specimen A, quenched from 750° C; B, X-irradiated 72 hours; C, annealed 40 hours at 750° C; D, as-cleaved.

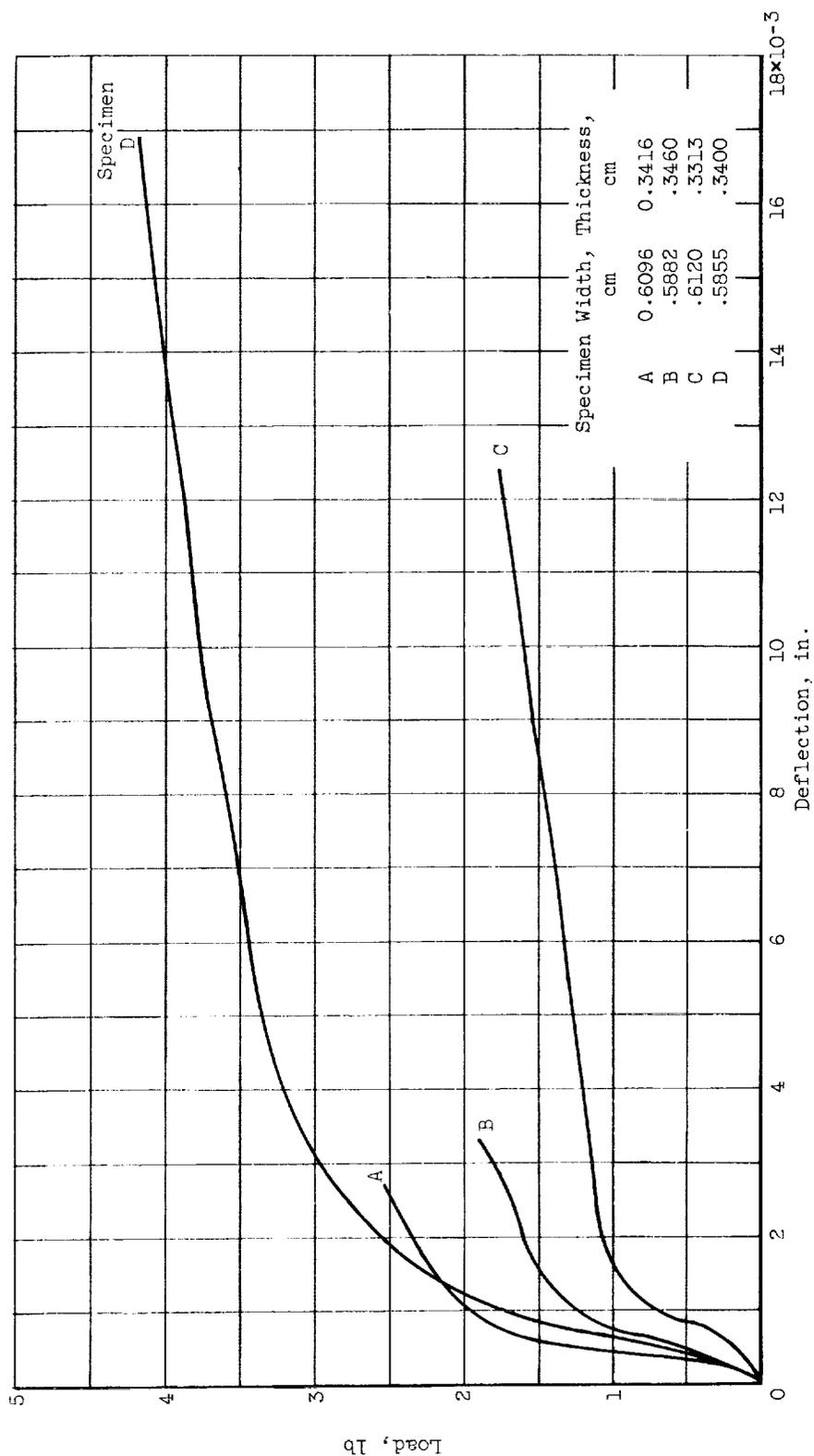


Figure 15. - Effects of various treatments on load-deflection curves for sodium chloride single crystals. Specimen A, as-cleaved; B, heated 1 hour at 135° C; C, coated with silver; D, heated 1 hour at 135° C and then water-polished.

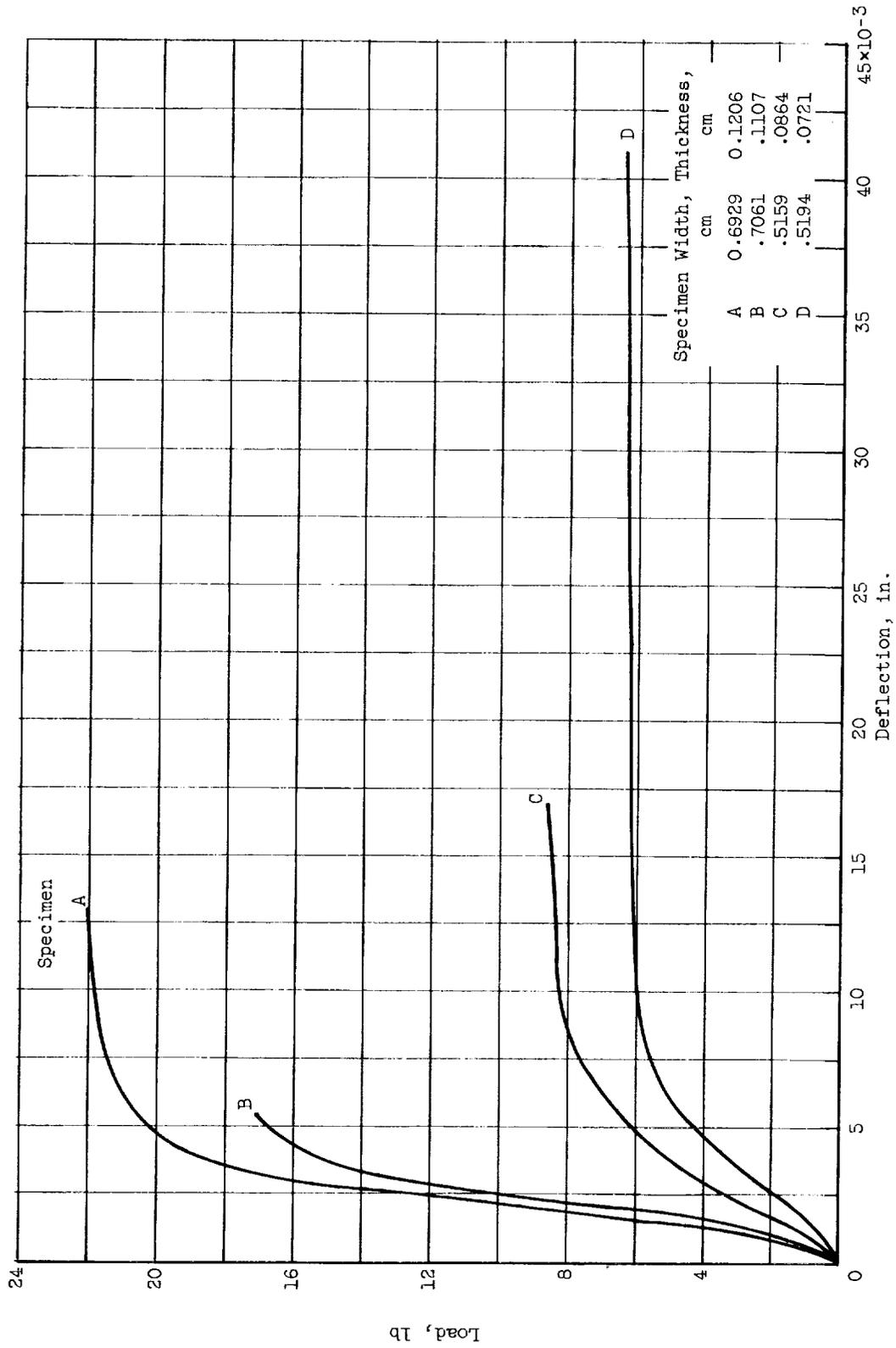


Figure 16. - Load-deflection curves for as-cleaved magnesium oxide single crystals showing time and size effects. Specimens A and D tested 30 seconds after cleavage; B and C tested 5 hours after cleavage.

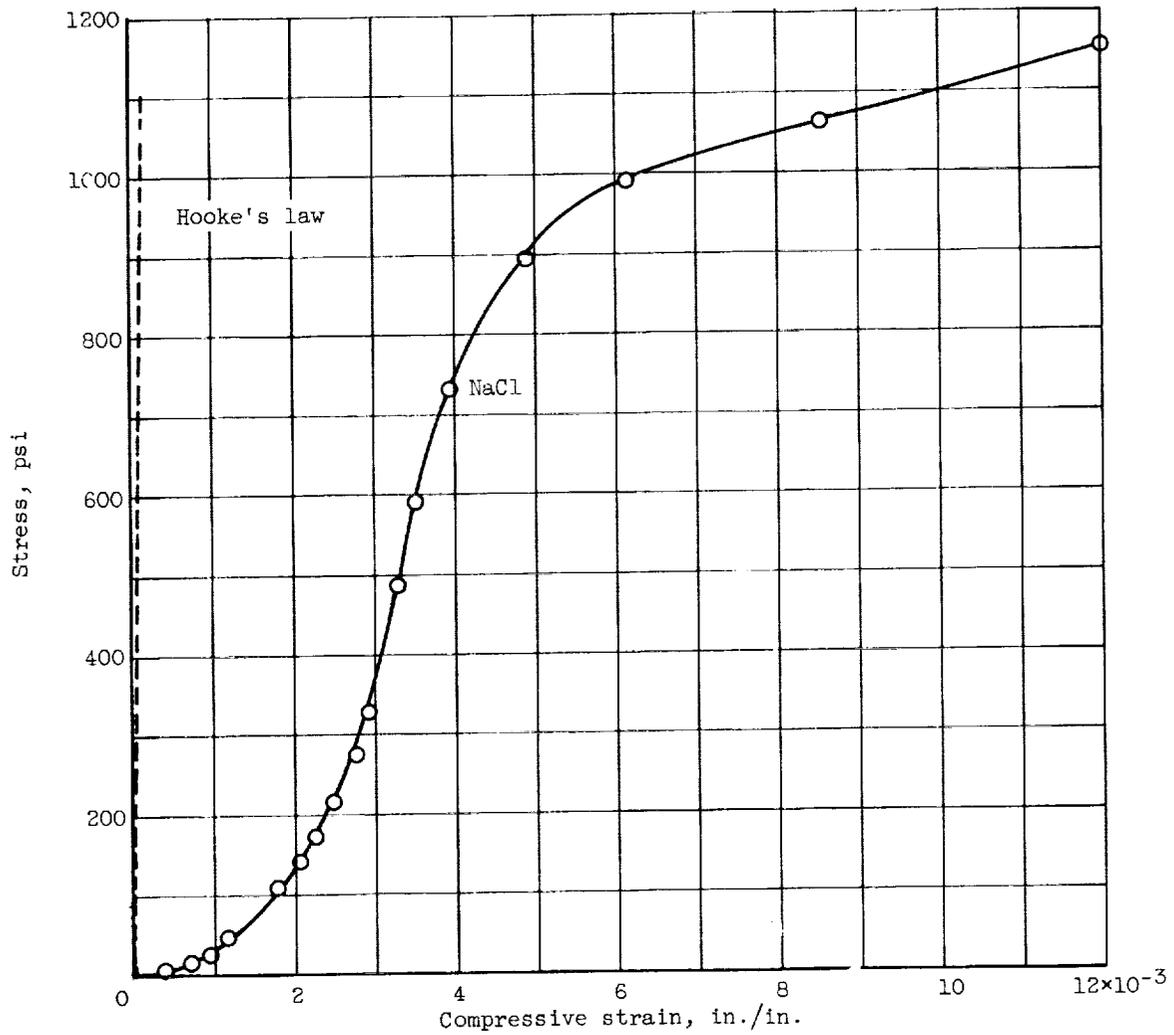


Figure 17. - Initial portion of stress-strain curve for sodium chloride single crystal tested in compression at constant compression rate of 0.002 inch per minute. Specimen quenched from 750° C to 25° C in carbon tetrachloride.

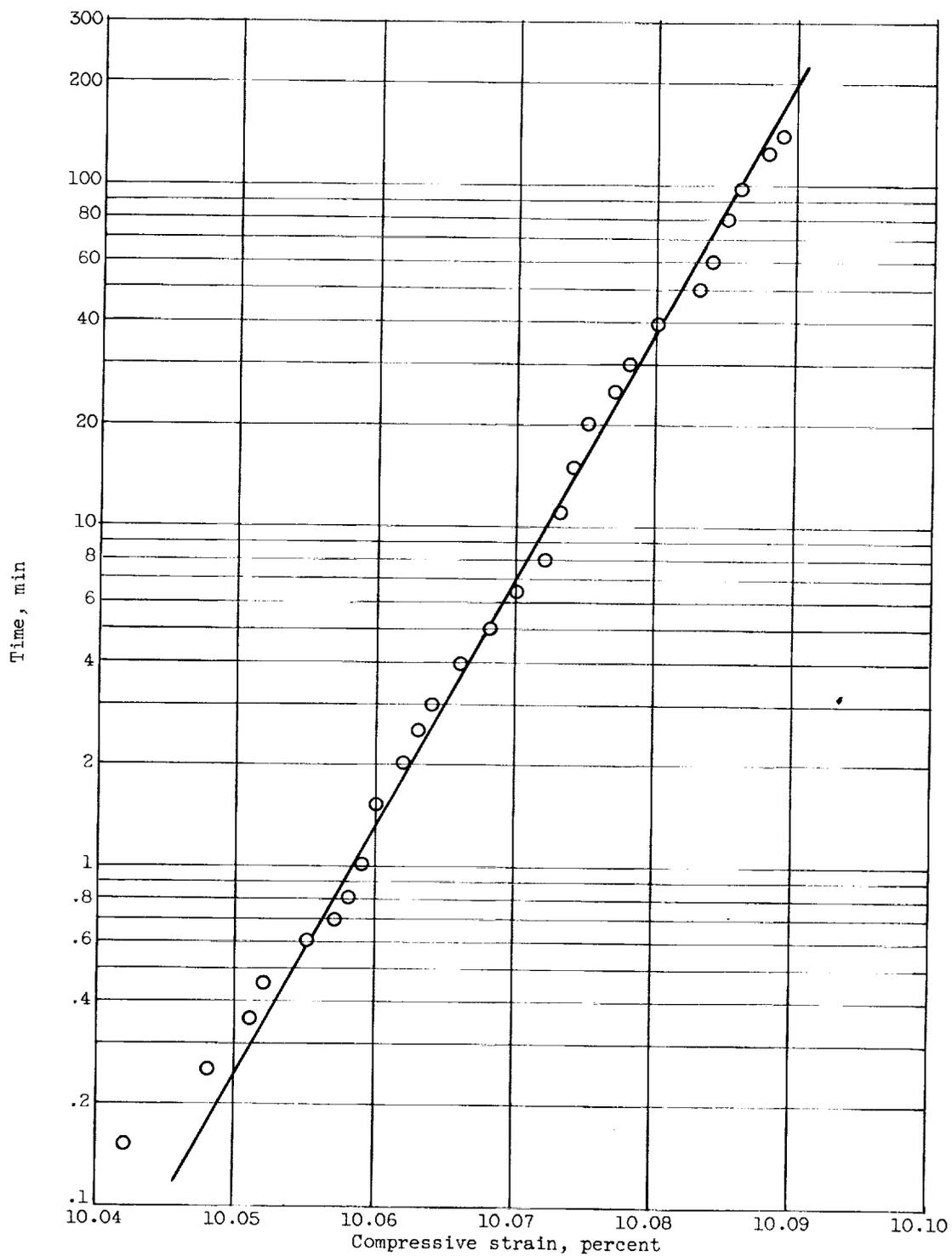
E-427  
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Figure 18. - Time plotted against percent strain for as-cleaved sodium chloride single crystal showing straight-line dependence and fit of creep curve to equation  $\epsilon = a \log t + b$ .

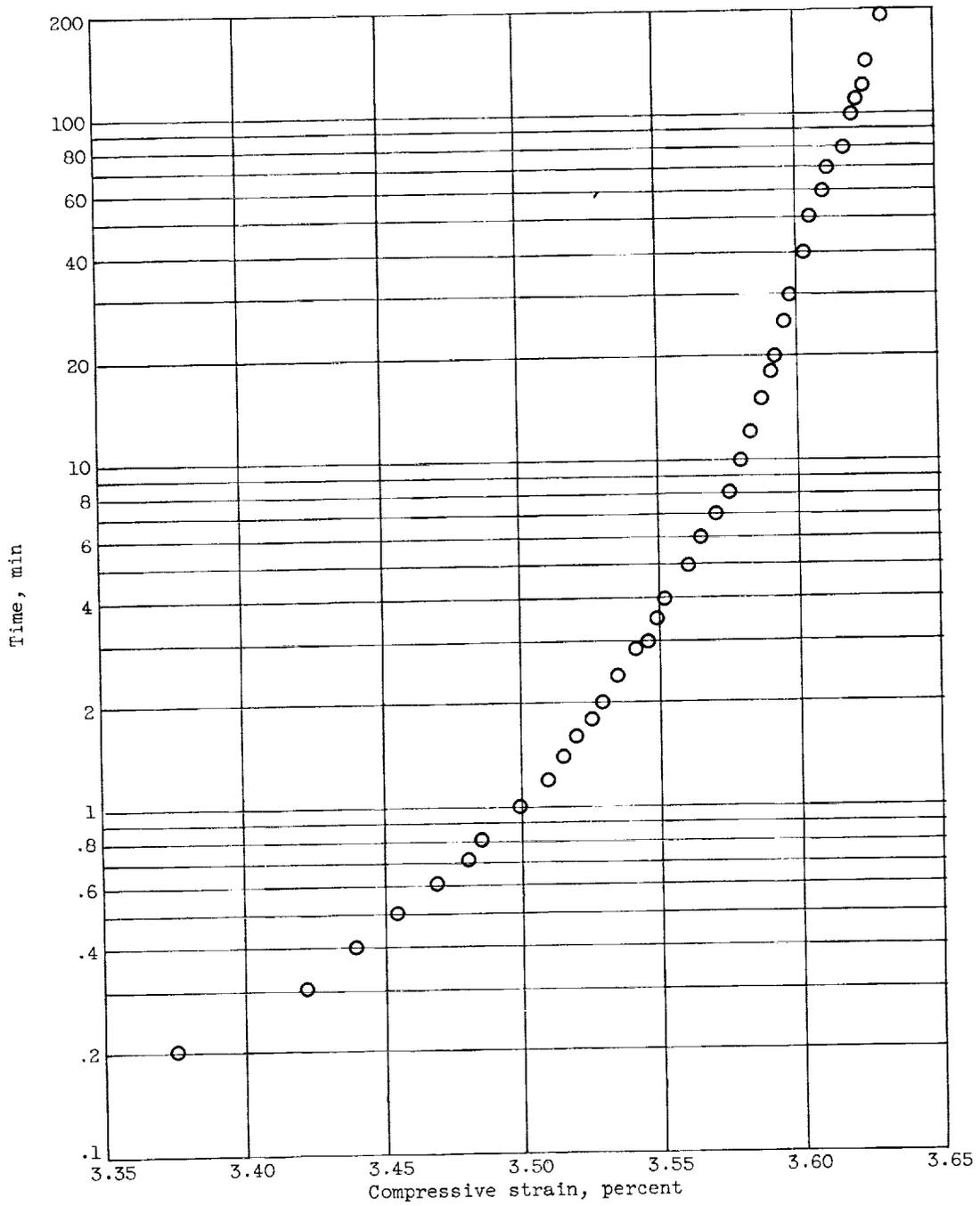


Figure 19. - Time plotted against percent strain for as-cleaved lithium fluoride single crystal showing lack of straight-line dependence.

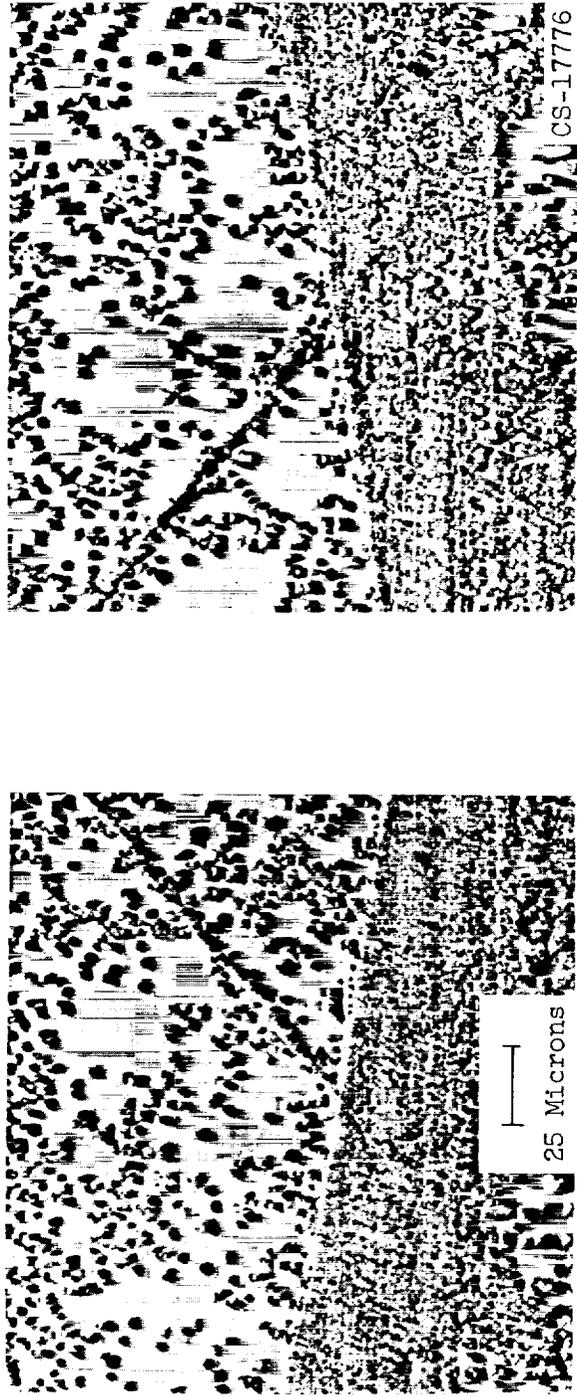


Figure 20. - Matching cleavage faces of sodium chloride single crystal showing correspondence of dislocation etch pits. Faces etched 30 seconds.



(a) As-cleaved; etched 30 seconds.

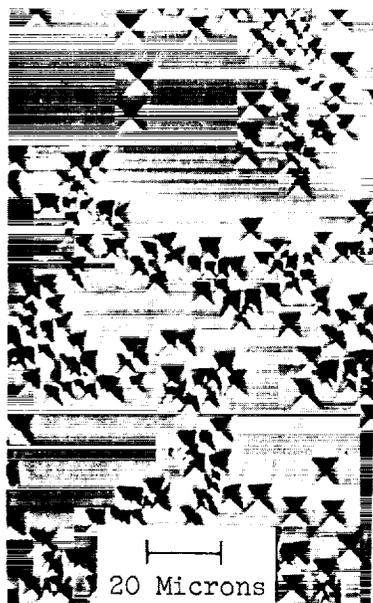


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(b) Water-polished slightly; etched 30 seconds.

Figure 21. - Effect of water-polishing on dislocation-etch-pit density.

E-427



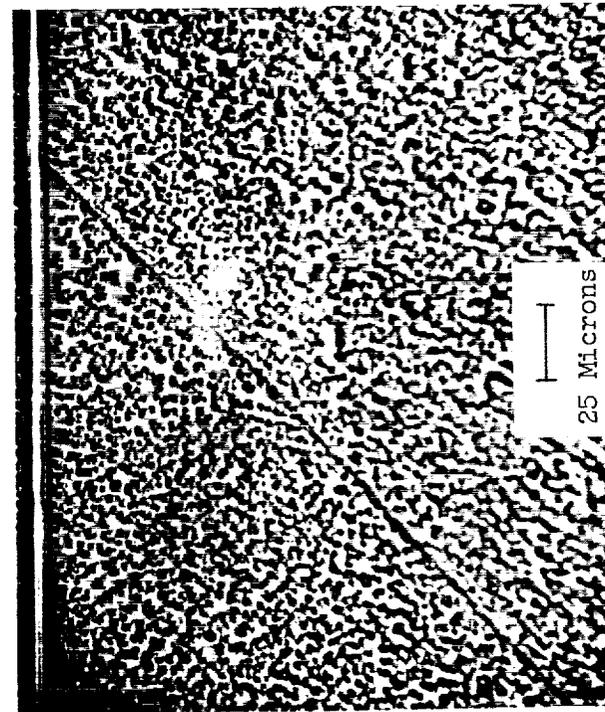
(a) As-cleaved; etched  
30 seconds.

CS-1777

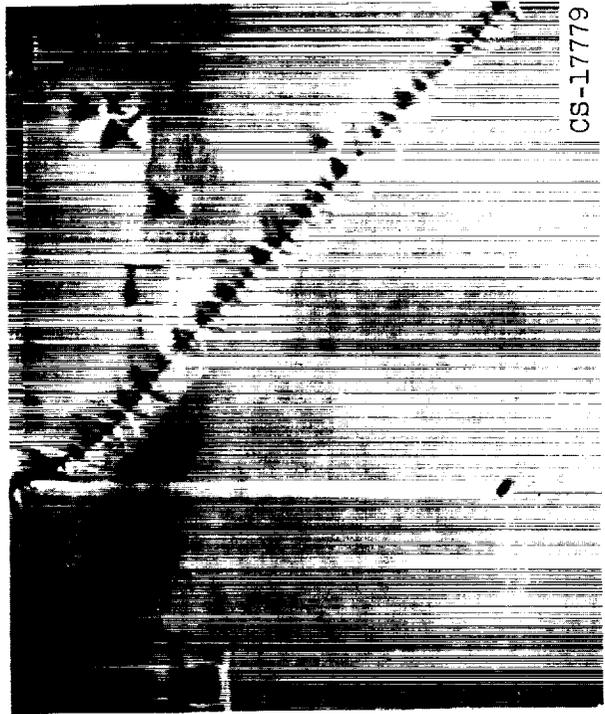


(b) Quenched from 700°  
to 25° C in carbon  
tetrachloride; etched  
30 seconds.

Figure 22. - Effect of quenching on dislocation-etch-pit density.

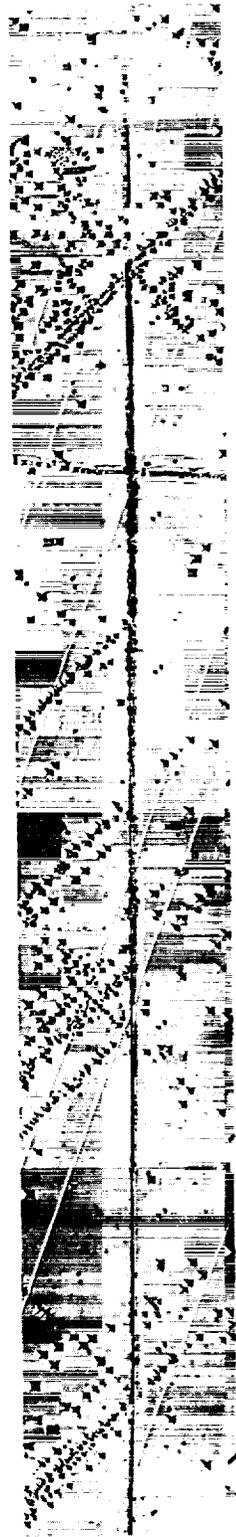


(a) As-cleaved; etched  
30 seconds.

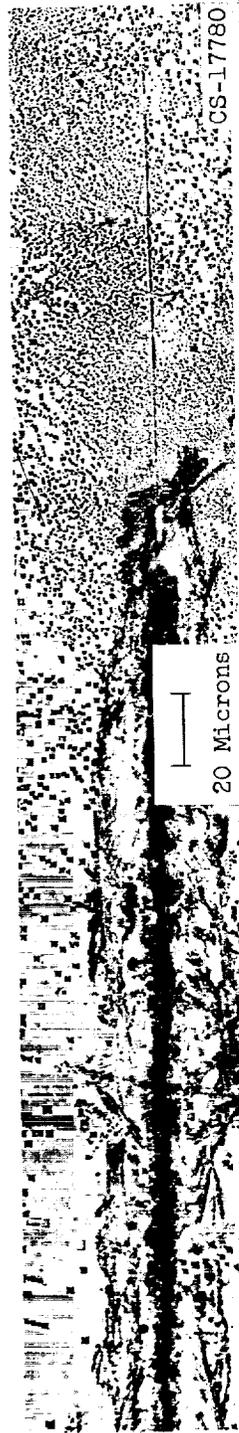


(b) Annealed 12 hours at  
700° C; etched 30 seconds.

Figure 23. - Effect of annealing on dislocation-etch-pit density and distribution.



(a) Normal to side of crack initiation.

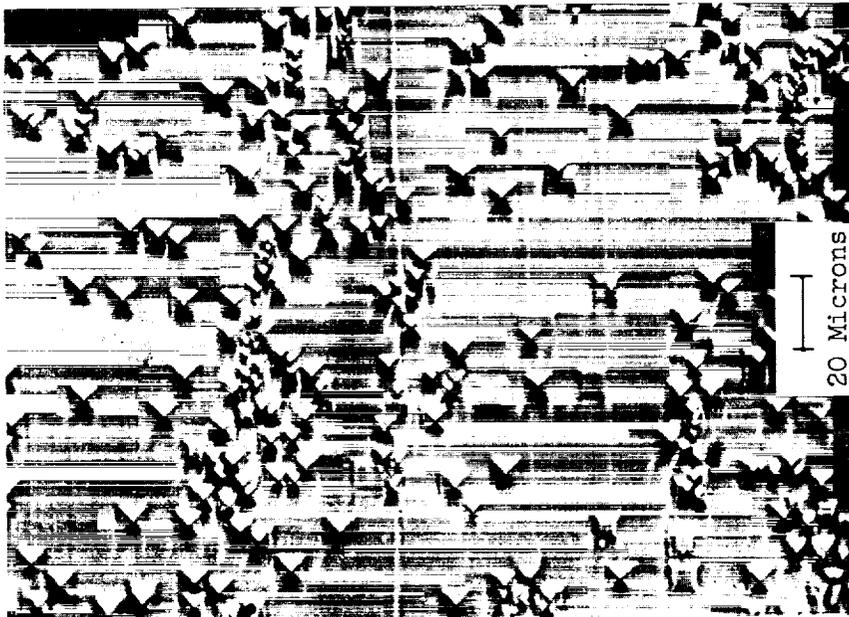


(b) Normal to side opposite crack initiation.

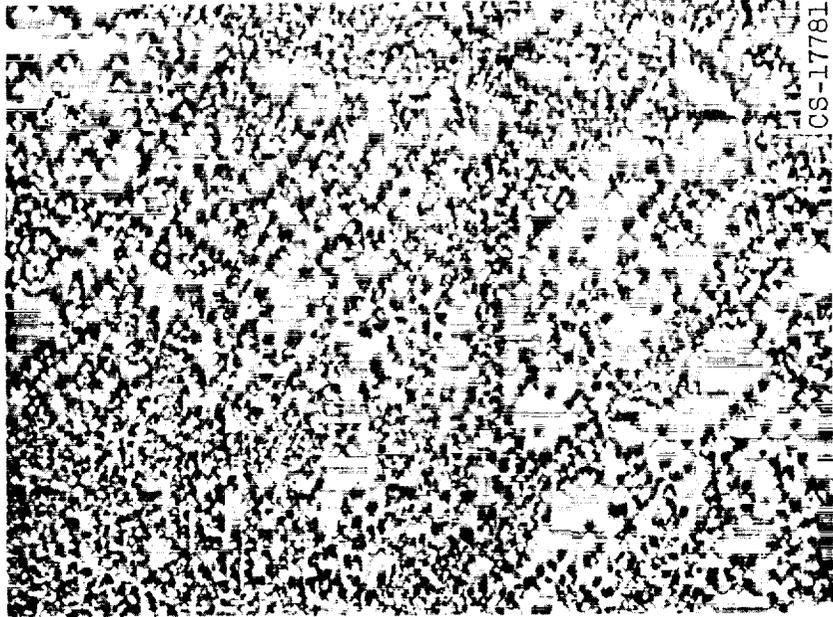
Figure 24. - Cleavage crack in sodium chloride single crystal showing dislocation arrays and damage produced by cleavage.



Figure 25. - Cleavage face of sodium chloride single crystal showing flat-bottomed pits which indicate movement of dislocations.



(a) Cleavage face of rapidly cleaved crystal; etched 30 seconds.



(b) Cleavage face of slowly cleaved crystal; etched 30 seconds.

Figure 26. - Effect of cleavage rate on dislocation-etch-pit density and distribution in sodium chloride single crystal.

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