

# THE INFLUENCE OF PLASTIC DEFORMATION ON CRACK PROPAGATION IN SODIUM CHLORIDE

M. Breucker and R. Steinbrech

Lehrstuhl fuer Werkstoffwissenschaften A, Abteilung Chemietechnik  
Universitaet Dortmund, Federal Republic of Germany

## ABSTRACT

The influence of the plastic predeformation on crack propagation in sodium chloride single crystals was investigated at room temperature, using a double torsion arrangement. The crack resistance force  $R$  was determined as the elastic energy release per fracture area increase.  $R$ -values decreased with predeformation and X-ray-irradiation from 11 N/m for untreated specimens to 2.6 N/m for irradiated crystals.

In addition  $\ln v$ - $R$ -curves have been calculated, showing a linear correlation with higher slopes for embrittled crystals.

## KEYWORDS

Sodium chloride single crystals; double torsion tests; fracture; crack resistance force; predeformation; irradiation; kinetics of crack growth.

## 1. INTRODUCTION

The surface energy of ionic crystals is usually measured by the experimental techniques of fracture mechanics. For example Gilman (1960) and Wiederhorn and co-workers (1970) determined the effective surface energy of the cleavage plane of various ionic crystals with Double-Cantilever-Beam - (DCB) - specimens. To perform such experiments, crystals are normally embrittled by radiation, doping or low temperatures. The specimens fail catastrophically within the quasielastic region.

In contrast, the fracture behaviour of soft, untreated specimens is characterized by considerable plastic deformation superimposing crack growth. Very often the cleavage crack propagates discontinuously in steps, without catastrophic failure. The evaluation of fracture mechanical parameters for soft crystals has been usually restricted to the first crack step.

Both fracture load  $P$  and theoretically determined change of compliance with cracklength  $dC/da$  enter into the calculation of the crack resistance force  $R$  according to

$$R = \frac{P^2}{2B} \left( \frac{dC}{da} \right) \quad (1)$$

$B$ : specimen thickness

The basic relation (1) sometimes resulted in experimental surface energies even smaller than the theoretical value with a considerable scatter. Wiederhorn and co-workers (1970) interpreted this effect as plastic deformation assisted crack growth.

It is the purpose of this paper to propose a more reliable and reproducible technique of evaluation, in order to determine the influence of plastic deformation on the crack resistance force. A method for the determination of  $R$  will be outlined taking into account many individual crack steps. Some first results will be presented.

## 2. EXPERIMENTAL PROCEDURE

For the investigations double torsion - (DT) - specimens of NaCl-single crystals have been used (Fig. 1). This specimen configuration is well established for brittle materials (Evans, 1973). The specimens were loaded symmetrically to a previously inserted cleavage notch. The specimen surfaces correspond to  $\{100\}$  planes. Crack propagation followed the  $\{100\}$  cleavage plane.

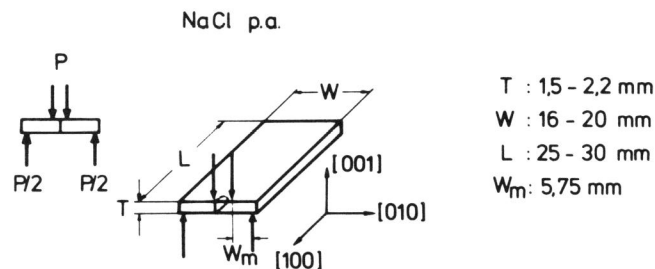


Fig. 1: DT-specimen geometry

All investigations were performed in air at room temperature. The substructure of the crystals prior to testing was varied by predeformation and X-ray-irradiation. Four types of specimens were investigated:

- untreated as grown crystals (named soft in the following)
- predeformed crystals (The specimens were cleaved from a larger sample, which was predeformed in compression to 1% shear strain. The predeformation occurred on slip systems which remained stress free during the DT-test.)
- irradiated crystals (Specimens were irradiated either at doses of 2 kR (called irradiation I) or at doses of 450 kR (called irradiation II). With the higher dose two radiation patterns were chosen: homogenous irradiation of the whole specimen and homogenous irradiation of only half the specimen.)

To explain the experimental situation, Fig. 2 shows a typical load-deflection curve.

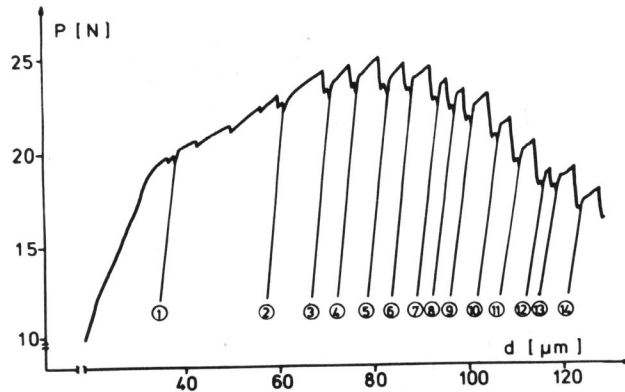


Fig. 2: Typical load - (P) - deflection - (d) - curve of a soft crystal including compliance measurement after load drops

At first the load deflection curve increases quasi linearly until the onset of plastic deformation. Within the plastic region many sharp load drops can be observed. The load reaches a maximum after some plastic deformation and then falls off again, contrary to constant load curves as expected for brittle materials. Thus a conventional method of R determination must be ambiguous because of the uncertainty of which load to take.

Therefore a compliance procedure was applied:

After strong load drops, a load cycle was performed to measure the actual compliance of the specimen. The decay of load with time for large load drops was stored with high resolution in a transient time converter and plotted afterwards. The deformation cycles were repeated until the specimen fractured completely. Then the fracture surface was inspected.

### 3. RESULTS

The load drops can be correlated to discrete steps in crack propagation. Figure 3a shows the fracture surface of a soft crystal. The curved lines mark the positions of the crack tip after discrete steps. The curvature of the lines indicates, that the crack moved from left to right. The area between any two of the crack arrest lines corresponds to the increase in fractured area during a single load drop. The crack does not extend over the whole specimen thickness. There always remains an uncracked region within the compression zone during the test. Strongly irradiated crystals show a similar behaviour, but less and greater crack steps are observed as shown in Fig. 3b. It should be mentioned, that this specimen fractured completely with the next crack step.

The fracture surfaces have been etched in order to reveal the dislocations. It is apparent that the dislocation density of the soft crystal is several times greater than in the strongly irradiated specimen. For the latter only grown-in dislocations can be observed within the crack step area. The crack tip is however surrounded by a higher dislocation density. The dislocations in front of the crack tip are introduced during further plastic bulk deformation before the crack advances a further step. Thus obstacles to crack propagation are created, which depend on the special type of DT-deformation. The fracture surface of the irradiated crystal shows the typical etch pit pattern for coarse slip.

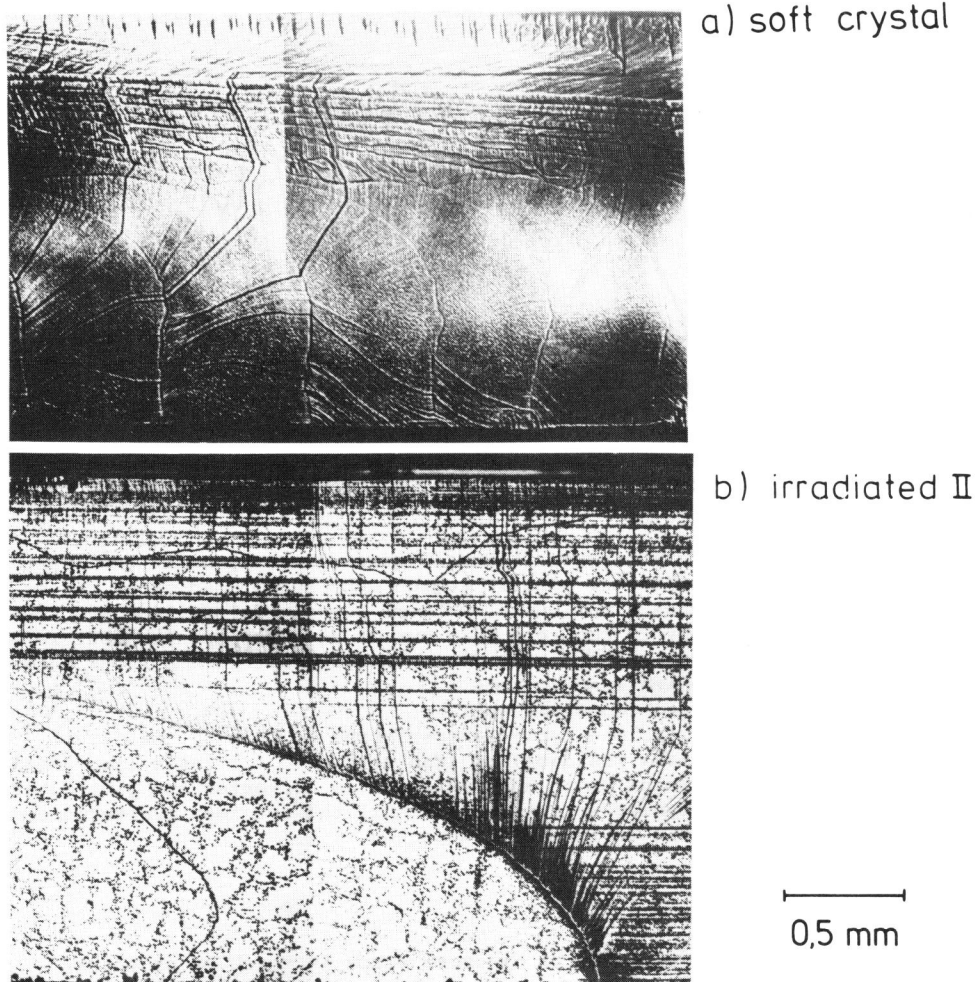


Fig. 3: Micrographs of etched fracture surfaces. Crack propagation from left to right as indicated by the curvature of the crack arrest lines

### 3.1 Crack Resistance Force

Schematically, typical fracture surfaces of soft and strongly irradiated specimens are outlined in Fig. 4. The upper fracture surface refers to the specimen of Fig. 2. After some smaller steps, the crack front reaches the typical shape, commonly observed in DT-specimens (Trantina, 1977; Virkar and Gordon, 1975). The first small steps are not taken into account for the R determination. Because the cracks do not extend over the whole specimen thickness, and because the crack tip is strongly curved, the evaluation of R was based on crack areas rather than on crack lengths.

The decay of load for a single load drop is shown in Fig. 5. As can be seen there is a sudden sharp load drop which is followed by a slight increase in load until the crosshead is stopped. The time constant of the subsequent plastic relaxation is large compared with the less than 1 second for the load drop. From this it can be concluded, that the amount of plastic relaxation accompanying the sudden load drops is rather small. The load drop thus is uniquely correlated with the increase of fracture surface.

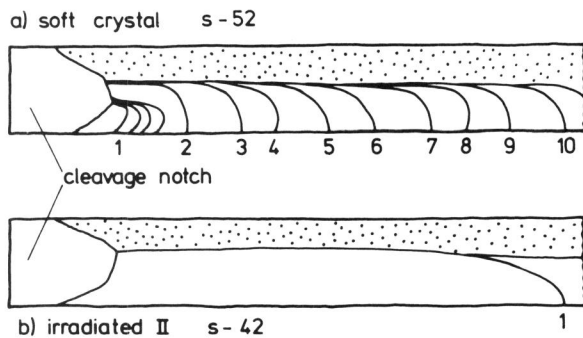


Fig. 4: Schematic view of fracture surfaces

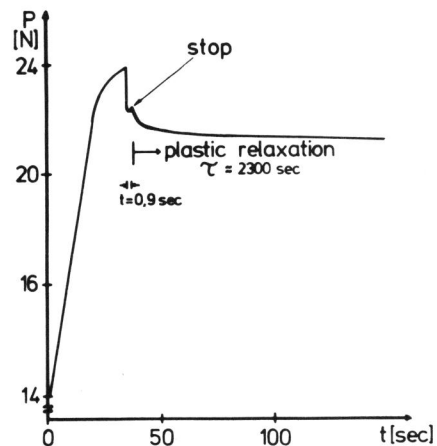


Fig. 5: Load-time curve of a single load drop with succeeding plastic relaxation

The upper part of Fig. 6 is a plot of the absolute value of the load  $P$  versus the increase in cracked area  $\Delta F$ . Obviously, for a wide range of loads,  $\Delta F$  is independent of  $P$ . However the graph of the magnitude of the load drop  $\Delta P$  versus  $\Delta F$  shows a good linear relation going through the origin. The numbers represent concurring load drops. Since the load decay during plastic relaxation depends on the absolute value of  $P$ , it is considered a reasonable assumption that plastic relaxation does not contribute much to the load drops. To generalize the results, a  $\Delta P$ - $\Delta F$  plot for a number of specimens is given in Fig. 7.

Again there is a linear dependence between  $\Delta P$  and  $\Delta F$ , while  $\Delta F$  is independent of the load  $P$ , which varies by almost an order of magnitude.

Based on this observation, single load drops of the load deflection curve have been used to determine the crack resistance force  $R$ . The method, based on an elastic analysis, is outlined in Fig. 8, where a section of the load deflection curve in Fig. 2 is enlarged.

The actual compliance  $C$  after each step has been measured, e.g. (12). From this the compliance immediately before the crack step has been constructed in the following way:

First the compliance line (12) is displaced parallelly to intersect the minimum load after the load drop. Then the point of zero load is connected with the maximum load at the beginning of the load drop. The resulting shaded area is to a good approximation a triangle and determines the amount of energy released during a crack step  $\Delta U$ . Taking this construction,  $\Delta C/\Delta F$  is greater than the theoretical value by factors up to 3.

The compliance of the preceding step (11) has not been used, because there is plastic deformation between subsequent crack steps. It has been observed, that the compliance of unnotched specimens decreases with plastic deformation.

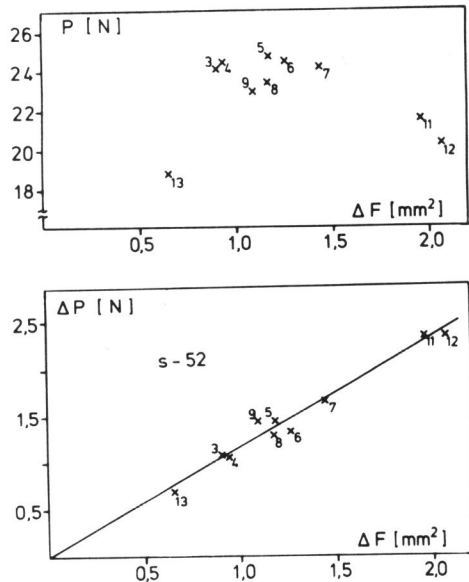


Fig. 6: Load P vs. increase in fracture area  $\Delta F$  in comparison to load decrease  $\Delta P$  vs. increase in fracture area  $\Delta F$

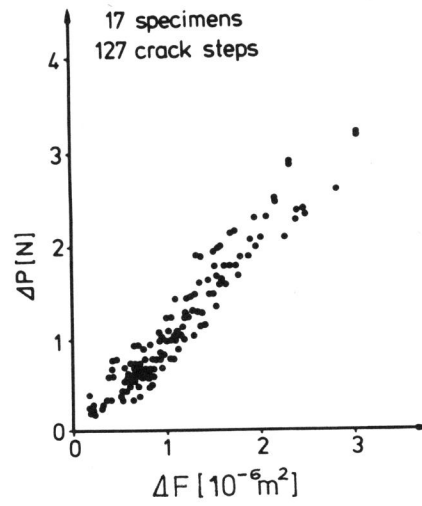


Fig. 7: Load decrease  $\Delta P$  vs. fracture area increase  $\Delta F$  for several specimens

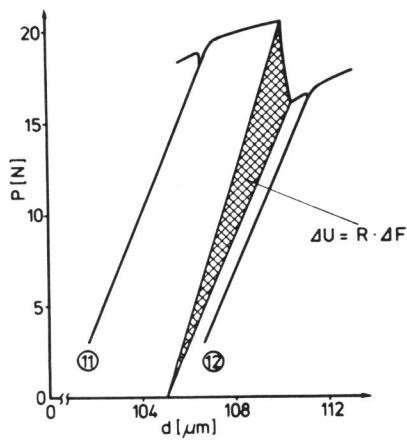


Fig. 8: Determination of the elastic energy release during a single load drop

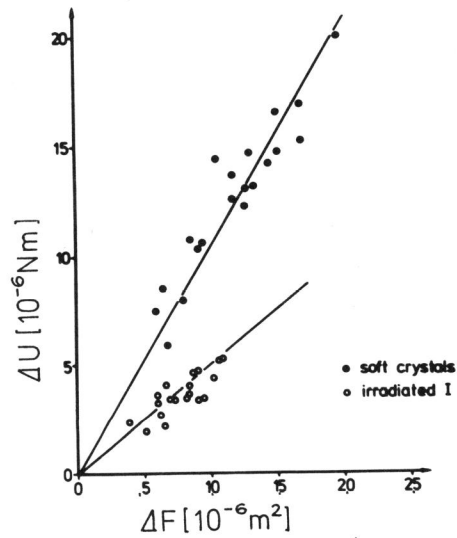


Fig. 9: Energy release  $\Delta U$  vs. fracture area increase  $\Delta F$  to determine the crack resistance force

A graph with the  $\Delta U$  values evaluated in the described way, plotted against the associated increase in cracked area  $\Delta F$  for some load drops of several soft and slightly irradiated specimens respectively is shown in Fig. 9. The scatter from the straight line is within the limits of experimental error, and was determined by errors in determining  $\Delta U$ .

With  $\Delta U / \Delta F = R$ , the linear correlation in the  $\Delta U$ - $\Delta F$  plot indicates, that  $R$  is constant within a single specimen as well as for a variety of specimens of the same obstacle structure. Similar plots have been made for different types of specimens too. The results are summarized in Table 1.

Tab. 1: Crack resistance forces for different specimen types

crystal property	Number of crack steps considered	R [N/m]
soft crystals	60	$11 \pm 2$
predeformed	5	$\approx 6$
irradiated I	25	$5 \pm 1.3$
irradiated II	2	$\approx 2.8$

theoretical value  $2\gamma = 0.2 - 0.4$  N/m

For soft, untreated specimens we find  $R$  values of 11 N/m, which exceed twice the theoretical surface energy  $2\gamma$  by a factor of 30. This can be explained by the interaction of the crack with the great number of dislocations produced during DT-testing. If one prevents the formation of such a high dislocation density,  $R$  decreases.

The predeformed specimens have  $R$  values of 6 N/m. During predeformation, slip occurred on glide systems oblique to those being active during DT-testing. Dislocations introduced by predeformation thus prevent the development of a dislocation structure similar to that of soft specimens.

The effect of irradiation points in the same direction. We find  $R$  values of 5 N/m for irradiation I-specimens and of 2.8 N/m for irradiation II-specimens. The  $R$  value for irradiation II-specimens is based on relatively few measurements only. However it is supported by the observation, that in partially irradiated specimens, the crack propagated stepwise in the untreated region, but failure was always catastrophic as soon as the crack reached the irradiated part. Our results can be compared with data published by Wiederhorn and co-workers (1970). The  $R$  values they obtained, were based on fracture mechanical formulae. Their  $R$  values are lower by factors of between 5 and 10 and show considerably more scatter than ours.

Two effects can explain the discrepancies:

First, DCB specimens develop a different dislocation structure. Second, both critical load and the theoretical  $dC/da$  enter into the calculation according to Eq. (1).  $dC/da$  values determined according to the previously introduced triangle construction, are considerably higher than the theoretical ones. This yields higher R values. At the beginning of deformation, the load deflection curve is strongly influenced by the geometry of the cleavage notch. The final crack shape is developed only after the first few small steps, as has been shown in Fig. 4a. In particular, the load values in the first region underly strong scatter. We think that it is more reliable to use the more direct method of calculation proposed here, as it makes use of the true energy release and the true increase in fractured area.

### 3.2 Kinetics of Crack Growth

In addition to the R value determination, an attempt has been made to study the kinetics of crack growth. This has been possible by high resolution in time of the individual load drops. The method used is shown in Fig. 10.

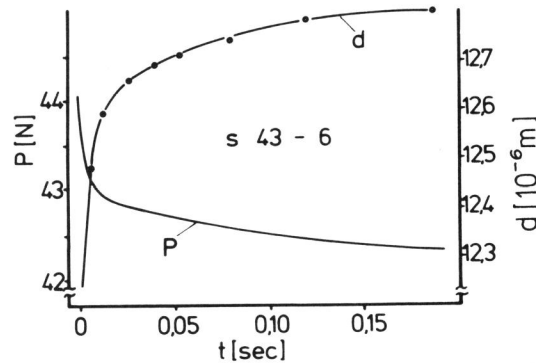


Fig. 10: Load P vs. time t during load drop and calculated deflection d vs. time

A single load drop-time curve as recorded with the transient time converter is plotted. It was not possible to measure the deflection d with time with the same accuracy. It was thus calculated assuming the same time dependence as for the load:

$$d(t) = d(0) - \frac{\Delta d}{\Delta P} (P(0) - P(t)) \quad (2)$$

The knowledge of deflection and load at any time during load drops, enables the computation of incremental fracture area increases:

$$\delta F = \frac{\delta C}{\Delta C / \Delta F} \quad (3)$$

The increase in cracked area with time is then only a function of the load change within a load drop.



The crack velocity can thus be computed from the load change:

$$\frac{\delta F}{\delta t} = \frac{1}{\Delta C / \Delta F} \cdot \frac{1}{\delta t} \left( \frac{\delta d}{P} - \frac{d}{P^2} \delta P \right) \quad (4)$$

Figure 11 shows logarithmic plots of the crack velocity  $v$  versus the crack resistance force  $R$  for four specimens of the same type. The scatter is relatively small and the dependence between  $\ln v$  and  $R$  appears to be linear. It can be seen on the interrupted x-axis that there is a certain amount of scatter in the  $R$  values from specimen to specimen of the same type.

Analogous graphs for irradiated specimens show qualitatively the same behaviour. The slope is higher for the more brittle crystals. To understand the information given by the slope, further investigations regarding the microstructural analysis of the kinetics of crack growth are in progress.

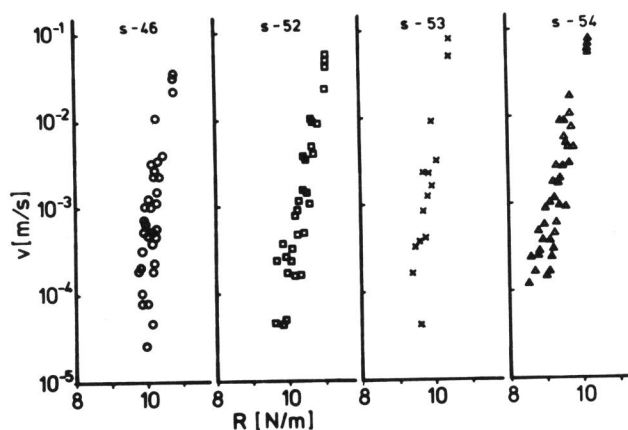


Fig. 11: Kinetics of crack growth for several soft specimens

#### 4. SUMMARY

The method presented here enables to obtain fracture mechanical data for semi-brittle NaCl crystals with satisfactory reproducibility. The scatter is much lower than encountered with other techniques. The trend towards more embrittlement for irradiated or predeformed crystals can be rationalized in terms of different dislocation densities as obstacles to the propagating crack.

Thus it might become possible in future, to detect local changes in the obstacle substructure from the analysis of the large number of crack steps in a single specimen by using our method of evaluation.

#### ACKNOWLEDGEMENT

The authors wish to thank Prof. Dr. W. Schaarwächter and R. Knehans for helpful discussions. The NaCl single crystals have been provided by Dr. W. Skrotzki (SFB 126 Goettingen Clausthal).

## REFERENCES

- Evans, A. G., (1973), A Simple Method for Evaluating Slow Crack Growth in Brittle Materials. Int. J. Fracture 9, 267-275
- Gilman, J. J., (1960), J. Appl. Phys. 31, 2208-18
- Trantina, G. G., (1977), Stress Analysis of the Double Torsion Specimen J. Am. Cer. Soc., July-Aug. 77, 338-341
- Virkar, A. V., Gordon, R. S., (1975), Crack Front Profiles in Double Torsion Specimens. J. Am. Cer. Soc., 58, 536 ff
- Wiederhorn, S. M., Moses, R. L., Bean, B. L., (1970), Plastic Deformation and the Fracture Surface Energy of Sodium Chloride. J. Am. Cer. Soc. 53, 18-23