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and Inorganic Materials Research Division, Lawrence Radiation Laboratory,
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ABSTRACT

Stress-strain curves obtained in bending at temperatures up to 500°C are presented for large-grain specimens of polycrystalline lithium fluoride. The amount of plastic strain obtained before fracture is considerably greater at higher temperatures than at room temperature. The grain interactions and dislocation movements involved are discussed, with special reference to the effects of the {100} <110> slip system.

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INTRODUCTION

As a result of considerable research over the past few years, the mechanical behavior of single crystals of ionic solids, particularly those possessing the sodium chloride structure, is now fairly well understood. It has been found that specimens of high purity and high degree of mechanical perfection must be used to obtain useful results.

In trying to extend the study of ionic materials to polycrystalline specimens, it is found that conventional fabrication techniques introduce not only grain boundaries but also impurities and porosity in variable amounts. By the use of special techniques it is possible to obtain fully dense specimens of the lower melting point materials of the same purity as single crystals, but some limitation on the available grain size is unfortunately involved. However, it is felt that more useful information is to be obtained from specimens of this type than from the more conventionally fabricated materials.

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A recent study by Scott and Pask[1] on large-grained polycrystalline lithium fluoride specimens at room temperature showed that a small amount of plastic deformation always preceded fracture, and that some accommodation between deformation of adjoining grains was possible by means of fine slip at the grain boundaries. In one case the slip in the vicinity of the grain boundary was proved to be of the type <110> {100} instead of the usual <110> {110}, and this observation suggested that greater ductility might be achieved if movement on the {100} planes could be facilitated. W. G. Johnston[2] has observed that slip on the {100} planes in LiF at room temperature has a critical resolved shear stress approximately 15 times that on the {110} planes, but Gilman[3] has shown that at about 400°C the critical resolved shear stress on the {100} plane is comparable with that on the {110} plane.

An interesting contrast to these observations is provided by studies of bicrystals of MgO at room temperature by Stokes and Li.[4] They showed that fracture occurred almost as soon as slip started in one grain, because no accommodation at the grain boundary was possible and pile-up of dislocations nucleated a cleavage crack. At room temperature in MgO (with its high melting point of 2500°C compared to 842°C for LiF) no movement of dislocations on {100} planes was observed by Hulse, Copley, and Pask.[5]

Taylor[6] has pointed out that five independent slip modes must operate in each grain of a polycrystalline aggregate to maintain contact between grains when the aggregate is deformed. Although there are six independent modes in the <110> {110} system, there are certain orientations of stress (such as tension or compression along a <111> axis) for

which less than five modes have a non-zero resolved shear stress, and thus it would be expected that only limited ductility would be observed until further slip modes became available.

These observations suggested that the effect of temperature on the mechanical behavior of polycrystalline lithium fluoride would be particularly interesting, and this paper is a progress report on a study of this effect.

SPECIMENS

Powdered LiF of approximately 99.95% purity was further purified by a zone-refining technique (as used by Scott and Pask) until clear crystals were obtained. Analysis of the crystals showed that the only significant impurities were about 20 ppm of Mg and less than 10 ppm of Al. It has been experimentally demonstrated by Johnston[7] that Mg is the only impurity in this concentration range which significantly affects the mechanical properties of LiF, and that at a concentration of 75 ppm the yield stress at room temperature is affected by annealing, especially in the 200-100°C range. At a concentration of 3 ppm of Mg no annealing effects were observed.

Polycrystalline specimens were prepared by lowering a graphite crucible 3/8 in. by 2 in. by 2 in. containing molten purified LiF out of the hot zone of a vacuum furnace at a rate of 36 in. per hour. The resulting ingot was formed of crystals which nucleated at the bottom of the crucible and extended through the height of the ingot. A specimen 3/8 in. by 1/8 in. by 1-1/2 in. was cut off the bottom of the ingot so that grains traversed its thickness, the average grain diameter being about 1/8 in. After cutting, the specimen was held in a glass clamp and

rotated in hot (~100°C) phosphoric acid for several (~8) hours to polish off surface damage.

The specimens produced in this way were free from pores. No annealing was done after polishing because of the risk of increasing the grain size above its already large dimensions, but the technique of growth in which the specimen ingots were furnace-cooled ensured that the treatment over the critical annealing range was the same for all.

MEASUREMENTS

Stress-strain curves were obtained in four-point bending. This technique enables a test to be carried to failure without destroying the specimen completely, and allows simple specimen preparation. A disadvantage is that sweeping assumptions have to be made in order to extract values of stress and strain from the observations.

A stainless steel four-point bend rig was used in conjunction with a small Instron testing machine. The support span was 3/4 in. and the load span 1/4 in. The specimens had 12-16 grains within the support span. A small Kanthal-wound furnace surrounded the rig, and the temperature was measured by means of a chromel-alumel thermocouple placed near the specimen. Because of the sensitivity of LiF to thermal shock, heating and cooling rates of about 200°C per hour were used. The slowest strain rate available (0.04 cm/min = ~0.016 in./min) was used. Studies of single crystals have shown that the mechanical properties of LiF are insensitive to strain rate.

The data are expressed as outer fiber stress, which is calculated from an elastic approximation but is directly proportional to the load applied, vs the product of crosshead movement and specimen thickness.

This latter product is proportional to the outer fiber strain at low strains. The observations are thus presented as nearly as possible as obtained, and have only been adjusted to allow for the small differences in specimen dimensions. It has been assumed that the specimen bent into a circular arc, and strain values at fracture have been computed on this basis. Both the stress and strain values become increasingly unreliable as absolute values at high strains.

The maximum strain obtainable in a bend test is limited, because after bending through a certain amount the specimen tends to slip between the support points. With the specimens and bend rig used in this work, the maximum outer fiber strain which it was deemed prudent to use was in the range 6-7%.

RESULTS

Figure 1 shows the stress-strain curves obtained in this work, together with a room-temperature curve taken from Scott and Pask. It can be seen that up to 350°C the yield is followed by a range of high work-hardening before fracture sets in after a strain which tends to rise with temperature up to a maximum of about 3%. The 400°C and 500°C curves exhibit a sharp yield and lower work-hardening, and extend to over 6% strain. The 400°C specimen had one rather large grain occupying most of the load span, and most of the deformation took place in this grain. This condition probably accounts for the fact that the latter part of its stress-strain curve is nearly horizontal, and for the fact that the test had to be stopped before fracture occurred.

A test of reproducibility is provided by a second curve obtained at 300°C. If plotted, this curve would almost coincide with the one shown.

Fracture in this specimen occurred at about 5500 psi outer fiber stress and 3.2% outer fiber strain.

(As a comparison figure to bear in mind, the critical resolved shear stress at room temperature for the <110> {110} system was observed by Scott and Pask to be about 600 psi.)

Figure 2 shows the yield stresses as a function of temperature, together with a bar representing the range of values obtained at room temperature by Scott and Pask. This spread is such as to cast doubt on the validity of the slight rise of yield stress observed in the 300-400°C range, and it may be that the fall to about one-third of the room temperature value is followed by a range of approximately constant yield stress. A similar rise has been observed by May and Kronberg[8] in single crystals of MgO, and attributed to an impurity effect, but Phillips[9] observed a slight decrease in critical resolved shear stress in single crystals of LiF in the temperature range 300-500°C.

The fracture observed in the 100°C and 250°C specimens was of the type seen by Scott and Pask, i.e., principally of the cleavage type but initiated by a region of grain boundary fracture associated with a three-grain junction. In the 300°C and 350°C specimens the fracture lengths were about equally divided between grain boundary and cleavage, whereas the 500°C failure appeared to be completely of grain boundary type. In addition it was noted that the fracture showed more tendency to be associated with one of the load points as the temperature rose and the specimens became more plastic.

DISCUSSION

The change in the character of the yield, together with the increased ductility which occur between 350°C and 400°C, must almost certainly be attributed to the greater activity of the <110> {100} slip system in this temperature range. Gilman's[3] measurements, obtained in torsion tests, show that it is precisely in this temperature range that the critical resolved shear stress on this system falls by a factor of about five to approximate to that on the <110> {110} system. Observational evidence in favor of this explanation is that wavy slip traces were observed on the faces of the specimens tested at 400°C and 500°C. This waviness indicates that slip is no longer restricted to one set of parallel planes. It would thus seem that not until both systems of slip (making a total of 12 possible slip modes in the crystal) can act at about the same resolved shear stress, can the grains accommodate conveniently to the strains occurring in their neighbors in accordance with Taylor's condition.

The increasing tendency to fail at grain boundaries as the temperature is raised may be due to either or both of two causes: (1) decreased grain boundary strength, and (2) decreased ease of cleavage.

Although the possibility of the first of these cannot be entirely neglected, it is thought that the second is far more likely. The increased availability of slip systems means that stress concentrations at crack tips are more easily dissipated and crack propagation is made more difficult so that cleavage becomes less evident. As an experimental fact it is known that crystals of the NaCl structure cleave more readily at low temperatures. With the tendency to cleave thus reduced, failure occurs at regions of high stress concentration, and these are likely to be grain boundaries, where

adjustment of neighboring grains is unlikely to be perfect, and areas near load points where local stress concentrations must be considerable. The observed failure at grain boundaries can thus be explained without postulating that grain boundaries are inherently weak.

Work is continuing, and it is hoped to obtain more information about the processes taking place in the deformation and fracture of polycrystalline LiF. It is also hoped to extend the work to smaller-grained specimens.

ACKNOWLEDGMENTS

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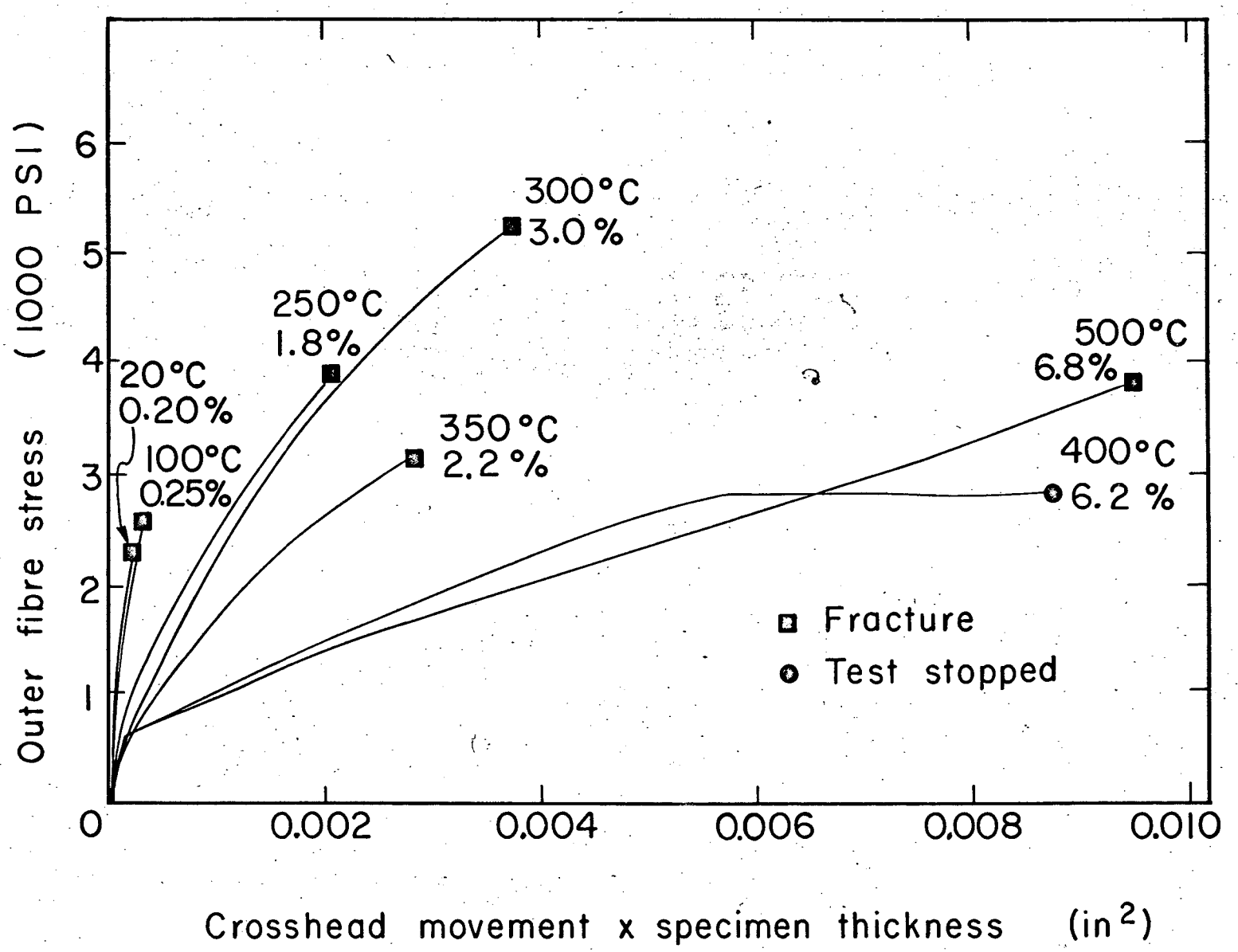
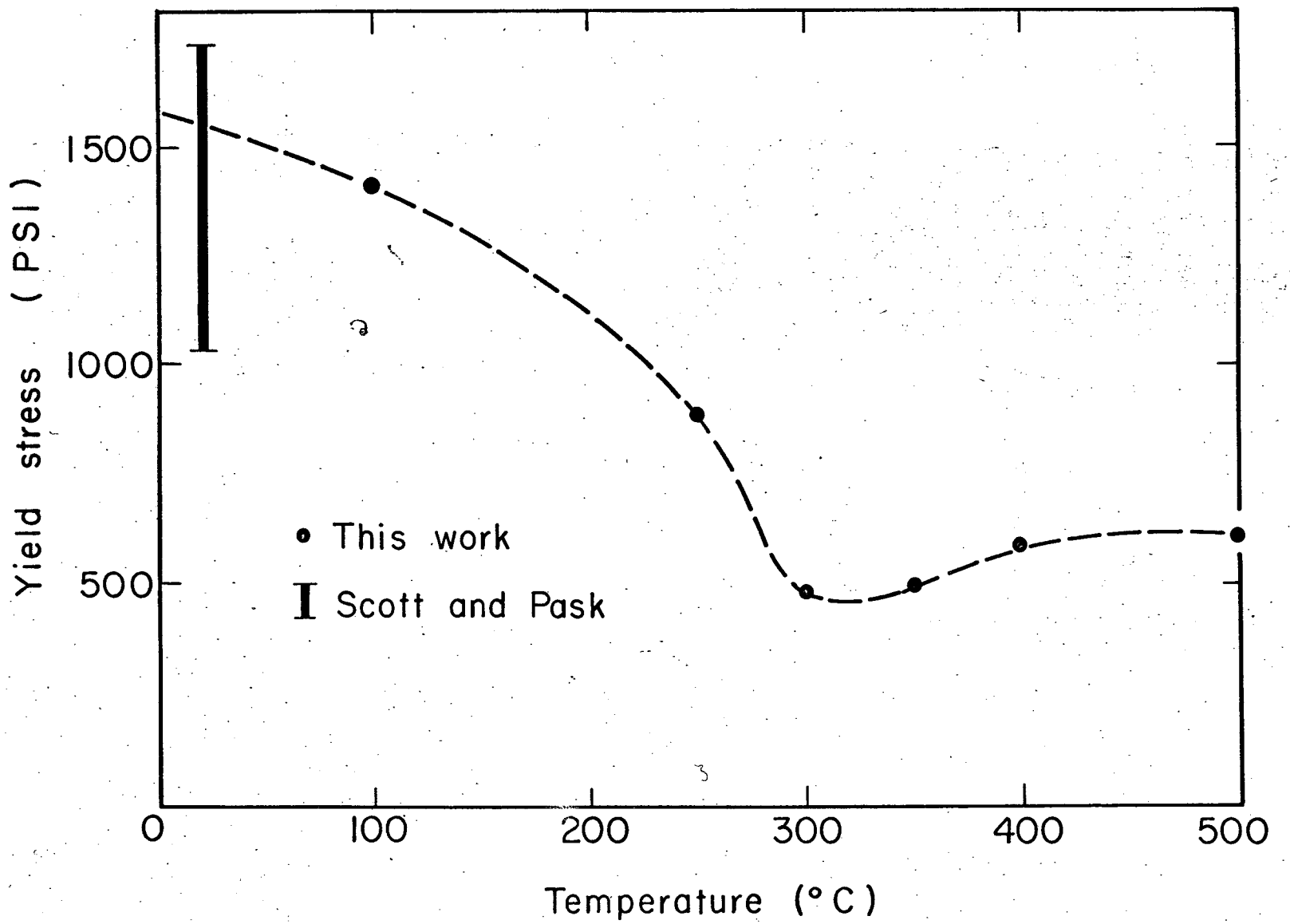

Outer fibre stress (1000 PSI)
6
5
4
3
2
1
0
0.002
0.004
0.006
0.008
0.010
Crosshead movement x specimen thickness (in²)
300°C
3.0 %
250°C
1.8%
20°C
0.20%
100°C
0.25%
350°C
2.2 %
500°C
6.8%
400°C
6.2 %
Fracture
Test stopped

MU-28188


Yield stress (PSI)
1500
1000
500
0
100
200
300
400
500
Temperature (°C)
This work
Scott and Pask

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