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SSC-133

EFFECT OF SUBSTRUCTURE ON CLEAVAGE IN IRON CRYSTALS

by

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January 29, 1962

Dear Sir:

As a part of its research effort in the field of brittle fracture, the Ship Structure Committee is sponsoring a study at Massachusetts Institute of Technology of the influence of metallurgical structure on the fracture behavior of ship steel. Herewith is the Fifth Progress Report, SSC-133, of this project entitled Effect of Substructure on Cleavage in Iron Crystals by W. F. Flanagan, B. L. Averbach, and Morris Cohen.

This project is being conducted under the advisory guidance of the Committee on Ship Steel of the National Academy of Sciences-National Research Council.

This report is being distributed to individuals and groups associated with or interested in the work of the Ship Structure Committee. Comments concerning this report are solicited.

Sincerely yours,

J. A. Alger, Jr.
Rear Admiral, U. S. Coast Guard
Chairman, Ship Structure Committee

Serial No. SSC-133

Fifth Progress Report
of
Project SR-136

to the

SHIP STRUCTURE COMMITTEE

on

EFFECT OF SUBSTRUCTURE ON CLEAVAGE IN IRON CRYSTALS

by

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Washington, D. C.
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January 29, 1962

ABSTRACT

The influence of substructure on the cleavage transition temperature in iron single crystals has been investigated. Substructure was introduced by prestraining up to 10 per cent and annealing, with the dislocation density increasing correspondingly from an initial value of about 10^7 to about 10^{10} cm^{-2}. The yield properties of the crystals with substructure were somewhat higher while the brittle transition temperature was raised about 40 C. Twinning preceded cleavage in these tests, and all the cleavage microcracks observed were associated with twins. The microcracks were located either along the twin/matrix interfaces or within the twins, but not in the matrix itself nor at intersecting twins. Cleavage appears to be initiated by the action of twinning, rather than by the role of twins as barriers to slip.

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INTRODUCTION

Allen et al[1] have indicated that the brittle-ductile transition temperature in iron single crystals is a function of the orientation of the tensile axis and that the transition temperature is raised as the tensile axis approaches the cube pole. Biggs and Pratt[2] have shown that decarburization lowers the transition temperature of iron single crystals; they have also demonstrated that prestraining at room temperature lowers the transition temperature, apparently by inhibiting mechanical twinning. Twin interfaces have been considered as barriers for dislocation pile-ups[3,4] and consequently twinning has been postulated as an important prerequisite for the cleavage transition in iron crystals. Metallographic evidence for cracks associated with intersecting twins in bcc single crystals has been found by Cahn[5] in molybdenum and by Hull[6] in iron -3 per cent silicon. On the other hand, Low[7] has shown that microcracks in iron -3 per cent silicon may originate at the twin/matrix interface.

Substructure is known to have an effect on the transition temperature in polycrystals[8,9] but there has been little corresponding work on single crystals. This research was undertaken to investigate the role of substructure in the cleavage behavior of iron crystals. It is shown that substructure does influence the cleavage transition temperature, but it is also evident that mechanical twinning plays an important part in the cleavage of iron single crystals at low temperatures.

EXPERIMENTAL PROCEDURE

Iron single-crystal tensile specimens were prepared by a strain, gradient-anneal method. One-half inch diameter bars of vacuum melted iron (0.024 C, 0.004 N, 0.0023 O, 0.001/0.005 Mn, 0.008 Si, 0.001/0.003 Al, 0.006 Ni, 0.001/0.004 Mo, 0.003/0.006 Co, 0.001/0.003 Cu, 0.006 Sn) were rolled to 1/16-in. strips. These strips were then annealed at 800—870 C for 1 hour in hydrogen and slowly cooled to room temperature. The critical strain for subsequent crystal growth was achieved by passing a Lüders band along the entire length of the strip, from which flat tensile specimens with a gage section about 1/4-in. wide were then machined. Surface defects were removed by electropolishing[10] in 5 per cent by volume of perchloric acid (70—72 per cent) in glacial acetic acid at 15—20 C. The specimens were then drawn through a temperature gradient (peak temperature ~850 C) at 1/2 cm/hour in an atmosphere of tank hydrogen. The carbon content dropped to about 0.002 per cent during this gradient anneal.

Johnson[11] has shown that discontinuous yielding enhances crystal growth in iron, but that the magnitude of the strain is relatively unimportant. He found that the boundary movement at the temperature of crystal growth may be limited by regions of austenite which are dispersed through the ferrite and which must be removed by decarburization before growth can proceed. This growth inhibition may suppress extraneous recrystallization and thus provide some advantage for using ferrite saturated with carbon in order to attain single-crystal growth. The critical strains varied from 2.5 to 5 per cent and the temperature at the growth interface was about 790 C.

The substructure was introduced by deforming crystals 3 and 10 per cent, and then annealing them (together with the unstrained ones) for 1 hour at 200 C and 3 hours at 650 C, followed by furnace cooling. Two methods were employed to detect the substructure. Specimens were electropolished in the aforementioned perchloric acid-acetic acid electrolyte at a current density of about 1 amp/cm^2 and 40 volts,[10] and were then etched by lowering the voltage to about 1/4 volt. Some samples were etched by immersion for 1 minute in 1 per cent nital. These etching techniques developed etch pits on faces close to the (100) or (110) orientation; the pits were counted to give etch-pit densities. Polygonized structures defined by networks of dislocations were not generally observed,

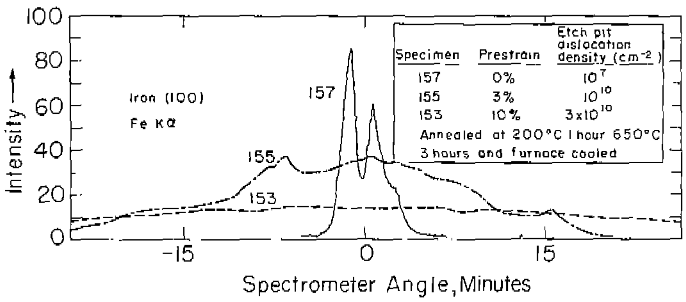

Fig. 1. Double crystal rocking curves of iron single crystals with varying prestrain followed by annealing.

and so the substructure was expressed in terms of dislocation density.

Double-crystal rocking curves were also obtained using FeKα radiation and two crystals oriented so that their (100) faces were parallel. A relatively perfect crystal with a rocking-curve width at half-maximum of less than 1 minute of arc was selected as the first crystal, and the second crystal was the specimen whose substructure was to be investigated. The presence of dislocations broadens the half-widths of the rocking curve. The dislocation densities were determined in a manner similar to that of Gay, Hirsch and Kelley.[12] This method was refined[13] to take into account the line-broadening effects of lattice bending, local strain and particle size, as well as of misorientations, but the results were essentially the same.

The x-ray and etch-pit methods indicated dislocation densities of the same order of magnitude. Typical rocking curves are plotted in Fig. 1 and the corresponding etch-pit densities are listed. The rocking curve for the undeformed crystal shows a well resolved small-angle boundary with a tilt of about 1.5 minutes, but other regions in the same crystal did not necessarily contain such boundaries. The rocking curves of the prestrained and annealed crystals disclosed more indications of small-angle boundaries but generally these could not be resolved. For purposes of comparison, the dislocation densities of the unstrained crystals were about 10^7, for crystals prestrained 3 per cent (and annealed) about 10^{10} and for crystals prestrained 10 per cent (and annealed) about 3×10^{10} (see Fig. 1).

The specimens were tested in a motorized vertical Hounsfield tensometer at a rate of 0.0035 in./min. Strains were measured by the cross-head movement and also by means of electrical-resistance strain gages mounted on the specimen.

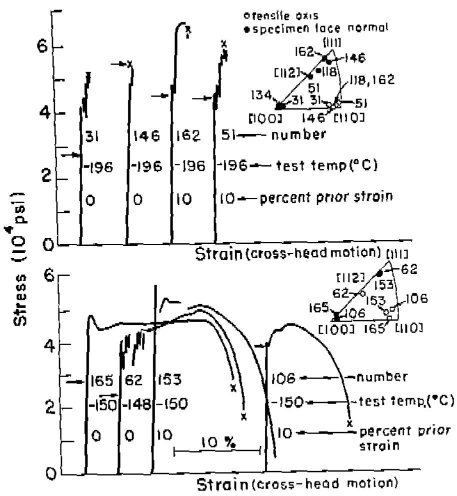

Fig. 2. Tensile curves for iron single crystals. Prestrained crystals were subsequently annealed (see text). Arrows indicate proportional limits determined by strain gages.

Tests were conducted between -140 and 170 C in a cryostat which was arranged with an outer chamber containing liquid nitrogen and an inner chamber containing heating elements to provide temperature control over a small range. A temperature controller was used to regulate the liquid nitrogen flow. Thermocouples were attached to each end of the gage section, and tests were not made until the temperature had been constant for 20 minutes.

RESULTS

Typical engineering stress-strain curves for a number of crystal orientations and test temperatures are shown in Fig. 2. The tensile axis in most of the crystals tested was close to the [110] direction, and the specimen-face normals tended to cluster near the [100] direction or between the [112] and [111] directions. Thus, there was little opportunity to explore systematically the effects of tensile direction.

Proportional limits, as determined by electrical-resistance strain gages to a sensitivity of 10^{-5}, are shown by horizontal arrows on the curves. In some instances, slip initiated outside the strain-gage area, and hence the apparent absence of plastic flow prior to fracture in these curves does not mean that it did not occur elsewhere. The discontinuous breaks in the stress-strain curves are the result of mechanical twinning. Twins were observed in all crystals which failed by cleavage.

The ductility-transition behavior is shown in Fig. 3. It is evident that the transition in these crystals is rather well defined, occurring at about -190 C in the virgin crystals and at about -150 C in the prestrained and annealed crystals. The transition temperature for the prestrained and annealed state was more sharply defined than for the unstrained state. This difference probably resulted from the likelihood that any inadvertent damage in handling would have a greater effect on the virgin crystals than on the prestrained and annealed ones. No systematic trend with respect to crystal orientation was found in these studies.

Three slip systems with equivalent critical resolved shear stresses[1] were assumed to operate in iron. The proportional limit, and upper and lower yield stresses were resolved along the shear direction on the pertinent plane and the resulting values are presented in Figs. 4—6. The introduction of substructure raises the proportional limit and yield strength somewhat, although there is considerable scatter. Gross yielding often started with twin formation, as indicated in these figures. The lower yield strengths of these crystals lie considerably below the values observed for polycrystalline specimens of the same material (Fig. 6).

Fracture stresses were found to be too erratic to warrant reporting; there were large uncertainties in the breaking load and final cross section.

DISCUSSION

Since a wide range of tensile-axis orientations was not available here, the effect of crystal orientation on the transition temperature could not be studied in detail. Allen et al[1] showed that the brittle-ductile behavior of iron crystals depends on orientation at -196 C, but the interdependence of orientation and temperature was not obtained. In the present work, the tensile axis varied within approximately 15 degrees about the cube pole for crystals with each type of substructure, and yet there was no apparent effect on the transition temperature. Also, the ratio of the normal (with respect to the cleavage plane) to the applied stress was calculated for specimens that underwent reduction in area before cleavage, and it was found that the reduction in area did not vary

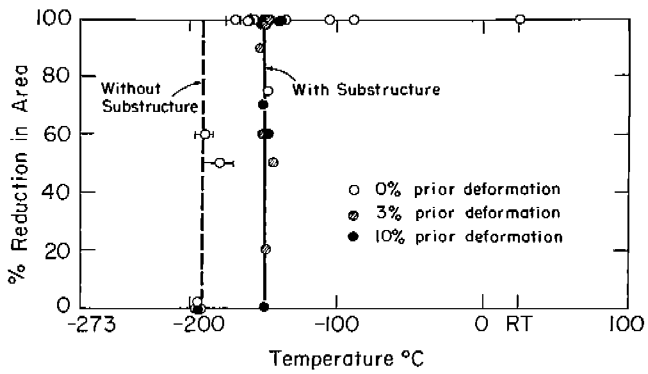

Fig. 3. Reduction in area of iron single crystals. Prestrained crystals were subsequently annealed (see text).

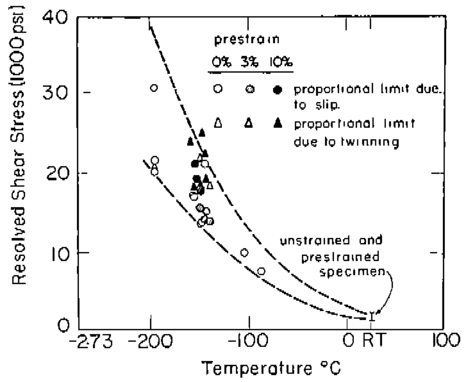

Fig. 4. Proportional limit of iron single crystals. Prestrained crystals were subsequently annealed (see text).

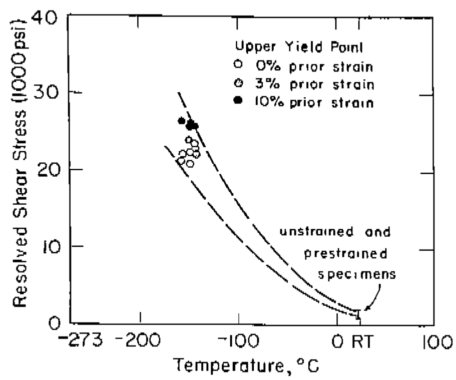

Fig. 5. Upper yield point of iron single crystals. Prestrained crystals were subsequently annealed (see text).

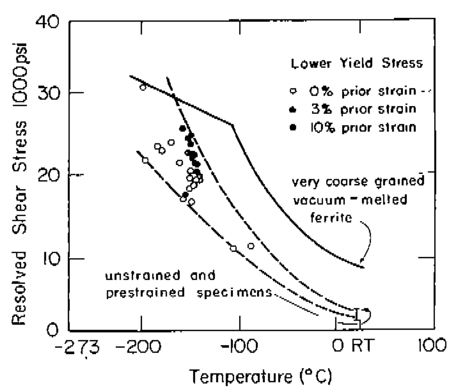

Fig. 6. Lower yield stress of iron single crystals. Prestrained crystals were subsequently annealed (see text).

in any regular manner with this ratio. This would seem to suggest that the transition was not primarily dependent on the normal stress (or orientation) within the present limited testing range.

It is interesting to note that the transition temperatures shown by Biggs and Pratt[2] for crystals with the same carbon content are very close (-183 C) to those observed here. They found that carbon raises the transition temperature and manganese lowers it; however, this effect of composition appears to be smaller than the effect of substructure found in the present work.

Biggs and Pratt[2] observed a <u>decrease</u> in the transition temperature of specimens which had been prestrained (but not annealed) prior to testing. The present investigation shows that annealing following such a prestrain <u>raises</u> the transition compared to the original state. This difference undoubtedly arises from the influence of prestrain on twinning; prestrain tends to suppress mechanical twinning whereas subsequent annealing tends to restore the ability of the crystal to undergo twinning.

Biggs and Pratt[2] obtained evidence that a critical stress is necessary for cleavage in iron in that the fracture stress was constant when resolved on the plane of maximum shear. It was concluded that yielding is a prerequisite of fracture. Consequently, the increase in transition temperature caused by substructure found in the present investigation may arise from the increase in flow stress. Certain inconsistencies appear, however: (1) the lower yield is significantly higher for crystals prestrained 10 per cent than for crystals prestrained 3 per cent, but the transition temperature is not correspondingly affected, and (2) the scatter in flow stress is not reflected in the transition-temperature data.

In addition, no correlation was found with respect to the crystal dimensions* if the latter were assumed to correspond to an effective slip

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*The crystal dimension in the direction of slip could vary through wide limits from specimen to specimen, even in cases where the tensile-axis orientations were similar, because of the rectangular cross section of the crystals.

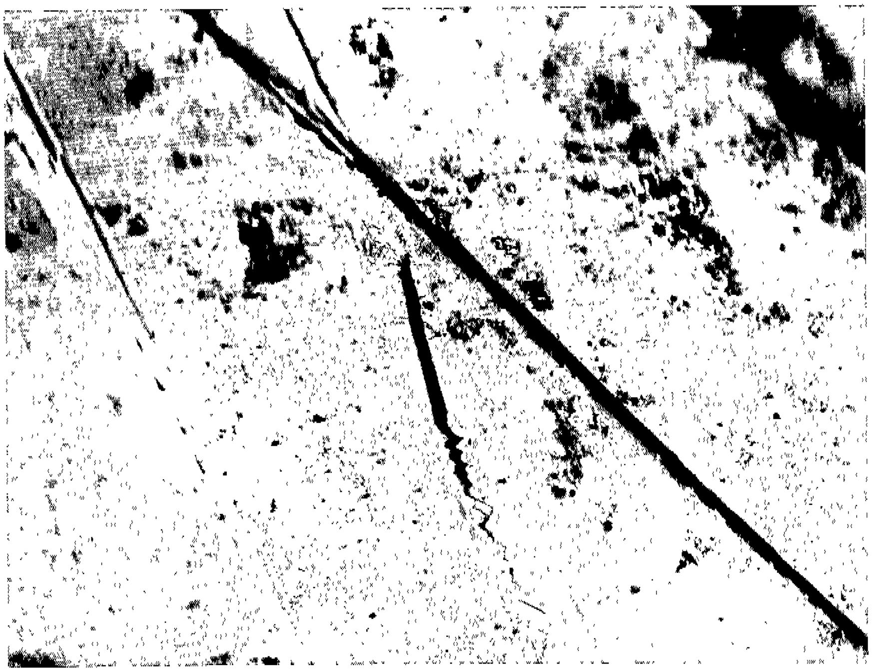

Fig. 7. Microcracks in deformation twin of iron crystal. Tensile axis is horizontal. The long almost-vertical crack is along a direction parallel to a (100) trace of the matrix. The zig-zag extension consists of (112) -type facets corresponding to a jagged twin/matrix interface. The specimen-surface orientation is near the (112) plane. As deformed, 500X.

length;[14] evidently, dislocation barriers other than the surface must be active in initiating cleavage. On the other hand, twin formation was observed in all crystals which failed by cleavage. Primary twins, with few exceptions, propagated across the entire crystal. These were generally bounded by a coherent interface on one side of the twin and by a jagged interface on the other. Zig-zag microcracks were often detected along the jagged twin/matrix interfaces and in some instances these microcracks were connected with larger cracks within the twins. Figure 7 shows such a crack. It is significant that the main cleavage facet within the twin was found to lie along a (100) or cleavage plane of the matrix. Evidently, such cracks were formed during the twin formation and were probably associated with the high local strains resulting from the twin generation. Cracks at twin intersections were not observed in these specimens even after many twins had formed. Cleavage cracks were not found outside of twins or twin/matrix interfaces unless the cracks were secondary to the final cleavage failure.

Some crystals cleaved after a ductile crack had started. In these cases, the cleavage crack originated well away from the ductile crack and propagated through the necked region without any apparent interference.

Flow and Twinning

Despite the scatter of results, there is a small increase in the proportional limit and the upper and lower yield strengths with increasing dislocation density when the latter is increased by orders of magnitude. The scatter in these properties could not be correlated with variations in orientation or carbon content.

If the Cottrell-Bilby model for twinning in bcc metals[15] is taken to be operative, and if it is assumed that the critical step in the twinning process is the dissociation of an $\frac{a}{2}[111]$ dislocation into partials, the stress should be resolved onto the dissociation plane. Such analysis of the data gave no support that this is the critical step. If the partials were already present, the critical step should be the activation of the rotating partial in the twinning plane. However, by resolving the twinning stresses onto the twinning plane in the direction of shear, the scatter was not reduced.

Because no evidence for a critical-stress law was found in these measurements, it was suspected that (in view of the rectangular cross section of the specimen) there might be a geometric effect caused by the crystal dimension in the direction of the slip or twinning. This dimension could vary markedly from specimen-to-specimen, even within the limited range of orientations encountered here. Such an effect might be analogous to the grain-size dependence of the stress for slip or twinning in polycrystalline ferrite.[9] However, no correction could be invoked that would eliminate the scatter or point toward a critical-stress law. Conceivably, the geometry of rectangular specimens leads to complications that obscure the expected orientation dependence.

Cleavage Mechanisms

No metallographic evidence was obtained to establish the mechanism whereby cleavage cracks nucleated. Microcracks were not found in untwinned regions of the crystals, unless they were connected with the final failure, thus suggesting that initiation is the critical step for cleavage in these single crystals. This is even more strongly indicated by the cases in which cleavage cracks propagated through necked regions where the energy for crack propagation should be relatively high.

The cleavage-fracture stresses, where they could be measured, fell within the scatter band for the initiation of slip, as would be expected from current theories. The Cottrell model of intersecting slip[4] was applied to crystal No. 162, in which only slight deformation preceded fracture at -196 C. Taking the crystal dimension in the direction of the operating shear as a measure of the slip distance, the surface energy associated with crack initiation was calculated to be 13—20,000 ergs/cm^2, which is about the value determined for the initiation of microcracks in polycrystalline iron.[9]

The fact that the transition temperature increases with the fineness of the substructure (i.e. increasing dislocation density) but decreases with grain refinement[9] points up the different ways in which substructure and regular grain boundaries influence cleavage fracture. Both substructure and grain boundaries increase the flow stress, but they behave dissimilarly relative to cleavage propagation. Whereas grain boundaries act as barriers to crack propagation, causing an increase in the effective surface energy to $\sim 10^5$ ergs/cm^2 the substructure introduced by prestraining and annealing appears to offer comparatively little resistance to the propagation of cleavage cracks.[9] This explains why no microcracks were found to stop within the matrix of the single crystals, initiation being the critical step for complete cleavage.

The microcracks observed in the single crystals are invariably associated with twins, and lie either at the twin/matrix interface or inside the twin along $\{100\}$ traces of the matrix. The latter indicates that the twins and cracks may grow simultaneously. Possibly, such microcracks act to relieve the twinning stress. No evidence was found to show that cleavage is caused by twins as barriers to slip. Under the conditions studied here, a cleavage crack that enters the untwinned matrix can propagate through the crystal to ultimate fracture before the stresses are removed.

Barriers to slip in the usual sense seem to be missing in single crystals. The surface cannot be expected to withstand the dislocation pile-ups as grain boundaries do. This is confirmed by the insensitivity of flow stress to the crystal dimension in the shear direction, and suggests that the "burst" of dislocations postulated by Cottrell[4] may be supplied by the high transient stresses associated with twinning.

The decrease in transition temperature with prestraining (but without subsequent annealing) found by Biggs and Pratt[3] can be, as they proposed, an effect of twin inhibition. If their specimens had been annealed after the prestraining, the ability to undergo twinning would have been restored and, because of the increased flow stress attributed to substructure, the transition temperature would probably have been raised, as found here.

CONCLUSIONS

1. Substructure raises the brittle-ductile transition temperature in iron crystals by about 40 C for an increase in dislocation density from 10^7 to 10^{10} cm^{-2}.

2. Cleavage in iron crystals studied here appears to be initiated by the high local stresses accompanying twinning, rather than by the role of twins as barriers to slip.

3. Substructure raises the flow stress of iron crystals, but does not seem to increase the resistance to cleavage-crack propagation.

4. Orientation, at least in the limited range investigated, has no appreciable effect on the brittle-ductile transition temperature in iron.

5. Critical stress laws for slip, twinning, or cleavage were not found in these experiments, perhaps owing to the rectangular section of the strip specimens.

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