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Abstract

Elementary processes in cleavage fracture of iron were investigated on single crystals of very pure, carburized and neutron irradiated iron. The yield stress of very pure iron was estimated to be smaller than 10 Kg/mm^2 even at 4.2°K , mechanism of initiation of crack associated with twin was suggested to be closely related to that of initial growth of twin, carbides precipitated inside and at boundary of grain were shown to decrease the stress of initiation of cracks and play an essential roles in the transition characteristics of low carbon steels, and neutron irradiation increased the rate of work-hardening together with the yield stress.

§ 1. Introduction

Iron is a material of much interest as it is a representative one of semi-brittle crystals, and has high sensitiveness for impurity contents and heat-treatments which involve many technical problems. So many experimental investigations have been accumulated for understanding of mechanisms in brittle fracture particularly through studies of elementary processes, but there still remain a lot of doubts yet to be clarified.

Opinions are divided on the magnitude of lattice resistance of dislocation, ⁽¹⁾⁽²⁾⁽³⁾⁽⁴⁾ which play very important role in the mechanism of cleavage. Roles of minor impurities of interstitial elements have not yet been separated from the characteristics of work-hardening and fracture. ⁽⁵⁾⁽⁴⁾⁽⁵⁾⁽⁶⁾ It is known that twinning is closely related to initiation of cleavage in pure iron, ⁽⁷⁾⁽⁸⁾⁽⁹⁾⁽¹⁰⁾ but its atomistic mechanism is not yet clear. The role of secondary phases is very important in the mechanism of low carbon steels. ⁽¹¹⁾⁽¹²⁾

On these themes some efforts were made in this paper. Since the range of those problems is very wide, we confined our studies mainly to single crystals, and particular efforts were made so as to separate the roles of impurities, particularly of interstitial elements. ⁽¹³⁾

§ 2. Specimens and test procedures

The two species of materials were used in our experiments. The one is the pure iron produced in our laboratory, firstly decarburizing carbonyl iron powder by annealing in wet hydrogen and then deoxidizing by melting it in pure hydrogen using pure alumina crucibles. ⁽¹³⁾ And the other is the zone melted iron produced by zone refining the sintered carbonyl iron rod by 2~9 passes in pure hydrogen and one pass in vacuum. Purities of these materials are listed in Table 1. Single crystals were produced by recrystallization method. The zone-melted iron was nitrized by about 0.005% before recrystallizing and denitrized after crystallization to single crystals. ⁽¹⁴⁾ Size of specimens were $1 \times 5 \text{ mm}$ cross section and $50 \sim 30 \text{ mm}$ in length. Specimens were further decarburized by annealing at 720°C in wet hydrogen for 24 hours to minimize carbon content. And some of them were then re-carburized and homogenized, and then some were water quenched, and the others cooled slowly in

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furnace. By these treatments carbon content of specimens were about 0.02%. And some of them were neutron irradiated by dose of $7 \times 10^{16} \text{ n/cm}^2$ ($> 1 \text{ MeV}$) in the reactor KUR in Kyoto University.

Tensile tests were performed using automatic control type testing machine TOM 5000, speed of cross head were fixed to 0.3 mm/min as a standard test, and test temperatures were 4.2, 65, 77, 90°K and higher. The strains were measured by means of wire strain gauge at temperatures higher than 65°K, and differential transformer at 4.2°K. Precisions for measurements of strains were 10^{-6} and 2×10^{-6} for the strain gauge and the differential transformer respectively.

§ 3. Results of experiments

1) Tensile tests of pure iron single crystals.

Since our specimens yet contain interstitial impurities of about < 10 ppm which suffice to lock all initial dislocations, we decided to measure yield and flow stresses at lower temperatures after pre-straining of 0.1% at room temperature.⁽⁵⁾ By this treatment we intended to produce fresh dislocations free from carbon locking. Fig. 1 shows an example of test at liquid nitrogen temperature. The yield stress is very low and followed by very rapid work-hardening and then work-softening at larger strains. When the pre-strain at room temperature was increased, the yield stresses at 77°K were unchanged, but the rate of work-hardening decreased rapidly and then saturated to nearly constant value, where the critical value of pre-strain was about 0.1%. Fig. 2 shows the comparison of the specimens of various orientations on tests at 77°K, where the abscissa is taken to be plastic strain. Fig. 3 shows the similar tests at liquid helium temperature. Then Fig. 4 shows the comparison of stress-strain curves of crystals of same orientation at various temperatures. As the temperature is lowered, the rate of hardening, flow stress and also the critical strain at which work softening commences increase. Fig. 5 shows some examples of the twinning and fracture characteristics of pre-strained single crystals. It is noted that twinning and cleavage fracture stresses are rather similar with those of as annealed specimens. The zone-melted iron single crystals showed essentially similar behaviors excepting that the flow stresses were a little lower than those of iron melted in crucible Fig. 6.

2) Observations of nucleus of crack of pure iron single crystals.

The type of nucleus of crack associated with crossed two twins were reported by Hull⁽⁷⁾ and Honda⁽⁹⁾ on simple tension tests of single crystals, and we also confirmed similar structures. But we shall report here another type of nucleus of crack which is frequently observed in notched single crystals. And it has interesting structure suggesting mechanism of initiation of cleavage.⁽⁹⁾⁽¹⁰⁾⁽¹³⁾

In our experiment, notches, which have 1 mm depth and radius of curvature 0.01 mm~5 mm, were sawed on one side of specimens. And some specimens were pre-strained by 1~10% at room temperature. Fig. 7a shows the orientation dependence of mode of fracture in tension test of notched specimens at the temperature of liquid oxygen. The abscissa is the angle between the direction [100] and axis of tension, and the ordinate is radius of curvature of notches. The open circle shows the type of origin of fracture in which cleavage started from a twin. While the half circle shows the type of origin in which cleavage is not associated with twin, and closed circle shows that of ductile fracture. And the number under the markings shows the number of specimens of the same orientation tested. While Fig. 7b shows the effects of pre-strain given at room temperature. In this case loads were applied by

shock bedding. Fig. 8 shows the dependence of fracture stress on radius of curvature. We may notice that the nominal fracture stresses were nearly equal to those of plain specimen and did not decrease as expected from the stress concentrations factors at the roots of notches.

Some examples of the structure of nucleus of crack observed on the surface of cleavage crack by optical and electron microscopes are shown in Figs. 11~16. And Fig. 10a shows the schematic sketch of the type of nucleus associated with a twin. In this type cleavage is started from a region of head of a twin which seems to be stopped at some distance apart from the root of notch, and one groove, or two in some cases, can be seen along the twin near the center of the head of twin, beside many striations crossing the twin Figs. 11 and 12. Figs. 13 and 14 show another examples where the twin is nearly parallel to the root of notch, and it is clear that many cracks are started independently from separate points at the boundary of twin.

Fig. 9 shows an example of the solid view of twin inside specimen, i.e. the surface of cleavage was removed successively by polishing, and the positions of twins were located. The curve *acb* is the notch, *cd* the surface of cleavage, while *ef* shows the front of twin associated with the origin of cleavage, and *gh* the front of twin nucleated at the tip of propagating crack. From these observations we determined that the twin associated with the origin of crack is one twin, and no crossing two twins, and the twin is stopped inside specimen near the root of notch. Topographical observations are summarized in the Figs. 10b, c and d. It can be seen that the distance between the root and the twin increases as the radius of curvature of notch increases.

Then Figs. 16a and b show the enlarged views of the same portion as Fig. 11 by electron microscope. It is presumed from careful examinations on the fine markings of striations that first cracks were initiated along the groove and that they were propagated in ductile manner inside twin, then transformed to cleavage cracks at some points on the boundary twin. An alternative explanation may be that cleavage cracks were first started on the boundary of twin followed by ductile fracture inside twin and lastly groove. The later process, however, fails us to explain all the evidences.

Origin of cleavage in pure iron is not always accompanied with twin, for instance, cleavages started sometimes at etch pits on the surface of plain specimens, or at the edge of ductile cavity produced near the root of notch when the test temperature is close to the transition temperature as shown by the half closed circles in Figs. 6 and 7 and illustrated by photographs Figs. 15a and b, or at the edge of cleavage crack once stopped.

3) Tests on carburized single crystals.

According to Allen's experiments⁽¹¹⁾ addition of small amount of carbon to pure iron caused in some cases very large increase of transition temperature in Charpy test, for instance, addition of 0.02% carbon increased the transition temperature by 150°C on furnace cooling, which covers the range of transition temperatures of engineering low carbon steels. To pursue the characteristic mechanisms of this effect, we determined to test on the effects of 0.02% carbon added to pure single crystals.

When the specimens were quenched in water, however, no appreciable effects were detected in fracture characteristics, though the flow stresses were increased by some amount. But when the specimens were cooled slowly in furnace, plate like carbides were formed, whose size was about 5 to 10 μ in length, and on simple tension tests the transition temperature was increased by small degree, and the transition became slower as shown by intermediate

reductions of area before onset of cleavage in Fig. 18, though the fracture stress was almost unchanged.

The discrepancies were, however, exaggerated appreciably on the tests of notched specimens. Single crystals of same orientation are compared in the Fig. 19. Pure iron shows lower transition temperature with sharp transition, while iron containing carbides has higher transition temperature with very slow transition. Compared with the case of simple tension test, fracture stress of notched specimens with large carbides is appreciably reduced in brittle range. Then by examinations of cleavage surfaces of iron containing carbide, it appeared that cleavage started from a carbide without accompanying twin in the transition range, which is shown by the markings X, and one example of nucleus of crack of this type is shown in Fig. 20, on the other hand, from a carbide met with a twin in the range of very low temperatures, this is shown by the marking Δ in Fig. 19.

4) Bi-crystals.

To obtain informations for the role of crystal boundary in the initiation and propagation of cleavage cracks, we investigated two groups of bi-crystals, i.e. these having boundaries nearly parallel and perpendicular to tensile axis. Tensile tests for pure iron bi-crystals are illustrated in Fig. 21a, where comparison of these with single crystals revealed the slower transition, which is illustrated by more or less amount of reduction of area before cleavage fracture in Fig. 21a, and also some micro-cracks which were found to have stopped inside crystals near the grain boundary. Then examining the origin of cracks, cleavages were frequently found to start at the boundary of twin stopped by the grain boundary as illustrated in Fig. 22. On the other hand, bi-crystals containing carbides, carburized by 0.02% carbon and furnace cooled, showed rather similar tensile characteristics as pure bi-crystals, Fig. 21b, but cracks were almost always initiated from carbides precipitated at crystal boundaries as illustrated by the photograph Fig. 23.

5) Effect of neutron irradiation.

As for the mechanism of irradiation hardening of pure iron, it is considered to be important to eliminate the effect of minor interstitial impurities though it was not yet examined in previous studies.⁽¹⁵⁾ Fig. 24 shows the comparisons of tensile tests for single crystals pre-strained by 0.1% at room temperature after irradiation of 7×10^{16} nvt for fast neutron with those unirradiated and having same orientations. It is noted that yield stress together with rate of hardening were increased by irradiation. The transition temperature of notched specimens, however, was not increased by the small amount of irradiation as shown in Fig. 25.

§ 4. Discussion

1) Concerning the yield and flow stress of pure iron, some divergence of opinions are pointed out. In fact, Petch⁽¹⁾ and Conrad⁽²⁾ had attributed the high yield and flow stress at low temperatures to large Peierls stress of dislocation caused by covalent character of interatomic forces characteristic in B.C.C. transition metals. Based on that idea Hahn⁽¹⁶⁾ explained the flow curves of iron using the Johnston-Gilman's theory.⁽¹⁷⁾ According to these theories lattice resistance of iron at 0°K was estimated to be about 50Kg/mm². On the other hand Kitajima⁽³⁾⁽¹³⁾ and Brown⁽⁴⁾ has asserted that the lattice resistance may not be so great but presumably smaller than some Kg/mm². Present measurements shows that it is smaller than 5 Kg/mm² at 4.2°K. The discrepancy may partly be attributed to the difference in precisions of

measurements of strains in these experiments. According to our results flow stresses show very rapid work-hardening in the range of small strains, which could be reasonably explained by the easiness of cross slip characteristic in B.C.C. metals. Measurements of activation energies at strains of 5×10^{-5} , 10^{-3} and 10^{-2} show the values of 0.15, 0.38, 0.57 eV respectively at 77°K. Fig. 4b, which seem to support the above deductions on the mechanism of work-hardening. Some criticisms for the proposed value of lattice resistance may hold that, the internal stresses caused by pre-straining at room temperature might lower the yield stresses and that movements of kinks contribute to small strains. On the former we may point out that the increases of lattice resistance at low temperatures may contribute additively to yield stresses irrespective of the internal stresses in our experiments, and on the latter, the contribution of movements of kinks may not exceed strain of about 10^{-5} , though for a final conclusion more careful experiments may be required on purity of specimen and analyses for very small strains. Stein and Low⁽⁸⁾ measured the yield stresses at 77°K on specimens which contained interstitial impurities as low as 5×10^{-5} ppm, but more precise measurements are considered to be necessary in the range of small strains, and on the effects of substitutional impurities and of many amount of jogs expected to be contained in the dislocations in annealed state.

2) According to our experiments the pre-straining of about 0.1% at room temperature, which masked the effect of small quantity of interstitial impurities on yield stress, however, did not have so much influence on the initiation stresses of twinning and also cleavage. This leads to the deductions that the initiation of twinning and cleavage may not depend essentially on the locking of dislocations by interstitial elements, but that rather on the absolute magnitudes of flow stresses which are insensitive for the history of small strains.

3) On the mechanism of initiation of cleavage associated with twin, the type of crossing two twins have been a common mechanism.⁽⁷⁾⁽⁸⁾ In our experiment,⁽⁹⁾⁽¹⁰⁾⁽¹³⁾ however, one twin caused many cleavage cracks in notched specimens. And the precise examinations for the structure of nucleus of crack, the initial start of crack was attributed to a groove structure appeared near the center of twin.

A possible explanation for this may be that, at first the origin of the groove inside the twin may be common with the midrib observed in twin as illustrated in Fig. 17, which seems to be closely related to the mechanism of growth of twin. The electron microscopic observations for midrib were reported by Nishiyama.⁽¹⁸⁾ And it is very probable that the midrib may be the trace of elastic twin which proceeded at high speed near sound wave in the initial stage of growth of twin.

Then we may suggest the mechanism whereby crack is initiated that, an elastic twin may propagate nucleating new twins by the distortion of lattice caused by very high stress at the edge of twin, then the same high stress may produce also cracks at the edge of twin which may be the cause of cracks first appeared along the groove. These cracks may then be propagated in ductile manner in company with further lateral growth of twin, in which climbing of twin dislocations may be the controlling mechanism, under high normal stress field near the notch, and transformed to cleavage cracks at some points on the boundary of twin, where Griffith's condition is satisfied. It can be seen in Fig. 16b that the fine striations crossing twin reveal the trace of ductile fracture and it's direction of propagation suggested above, and also the size of head region of twin is in accordance with the estimated

theoretical values.

The twin might be initiated at some place of maximum shear stress, and proceed toward the root of notch and be stopped nucleating cracks at the place where triaxiality is maximum consequently relaxation by strain is constrained. This mechanism may explain the topographical observations of the initiation of cleavage near root of notches Fig. 10.

The suggested mechanism of growth of cracks is considered not to contradict with the observations for those associated with the crossing two twins. And also the suggested mechanism of growth of twin may be substantiated by observations of the growth of twins nucleated at the edge of propagating cleavage cracks, because the climbing mechanism of twin dislocations may not suffice to explain so fast growth of twins as illustrated in Fig. 9.

4) The mechanism of initiation of cleavage is not confined to one type in the case of pure iron as explained previously, but various types of mechanisms have some common points. At first they are composed of the two stages, i.e. the stage of macroscopic stress concentrations at very sharp notches of about some tenths of micron produced by various causes, and that of nucleation of cleavage of atomistic size in the regions of stress concentration. In the later stage the model of coalescence of dislocations suggested by Cottrell⁽¹⁹⁾ may be a possible mechanism.

5) On the mechanism of cleavage originated from carbides, many factors may be important, i.e. stress concentration depending on the form of carbides, weakness at phase boundary, and also fracture of carbides.⁽¹²⁾ We have not definite conclusion for the relative prevalence on the later two factors, but we may point out that thin carbides are whisker like and can deform some percents elastically,⁽²⁰⁾ so that may provide firm barriers against piling up of dislocations. Apart from these detailed factors, our experiments showed that plate like carbides could initiate cleavage in association with slip at lower stresses and consequently at higher temperatures than the case of pure iron, and the mechanism was more insensitive for pre-straining.

These characteristics of carbides then may provide reasonable explanations for the large difference of brittle fractures between the two types of steels, i.e. furnace cooled low carbon steels and pure iron or water quenched steels. In fact, in pure iron the stress of initiation of cleavage is rather high so that the fracture stress is considered to depend on the critical condition at the earlier stage of propagation of cleavage inside the grain, and the initiation of cleavage is suppressed by pre-straining. On the other hand, in steels containing large carbides the initiation stress is low and not suppressed by pre-straining, so that the fracture stress is considered to be determined by the critical stress at the later stage of propagation of cleavage at the boundary of grains. Theoretical estimates⁽¹³⁾ for the fracture characteristics in these two types of steels dependent on the triaxiality of stress, temperature and pre-strain was shown to be able to explain reasonably the large differences, i.e. the lower transition temperature and sharp transition in pure iron in contrast to the higher transition temperature, slower transition and also higher sensitivity for notches in steels containing large carbides.

6) As for the effects of neutron irradiation on the stress-strain curve of pure iron, the lattice defects produced by irradiation may increase the lattice resistance of dislocation and decrease the mean free path of slip, consequently increase the yield stress and the rate of work-hardening.

5. Summary

Elementary processes in cleavage fracture of iron were investigated on very pure, carburized and neutron irradiated iron single crystals.

It was shown that the yield stresses of pure single crystals pre-strained by 0.1% at room temperature did not exceed 10 Kg/mm² even at 4.2°K, then the stress-strain curves showed very rapid work-hardening at earlier stage of straining and followed by work softening, while the twinning and cleavage stresses however were not so much influenced by the pre-straining.

Cleavage cracks were frequently found to be started from a twin not from a crossed two twins on single crystals which have various notches. On this type of nucleus of crack, cracks were found to be started initially along a groove, which was surmised to correspond to midrib in twin, and propagated in ductile manner inside twin, and then transformed to cleavage cracks at some points on the boundary of twin. As a possible mechanism of this type of initiation of crack, initial growth of cracks along the groove was attributed to nucleation of cracks at high stress field at the tip of propagating twin.

In contrast to pure iron, the iron carburized by 0.02% carbon and cooled in furnace showed higher transition temperature and slower transition. And the cracks were found to be started from carbides precipitated inside or at boundary of grain in company with slip, and the fracture stress was lower and more insensitive for pre-straining in the transition range. The characteristics was suggested to play important roles in explaining the fracture characteristics of low carbon steels.

Neutron irradiation increased the yield stress together with the rate of work-hardening.

Acknowledgements

The author's cordial thanks are due to the encouragements and valuable discussions of the late Professor Ishibashi in Kyushu University and the assistances in experimental works of Mr. S. Fukuda, K. Futagami, S. Nakazaki and Y. Nanamori, and also to the facilities in using the Reactor KUR offered by Reactor Research Institute of Kyoto University.

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Table 1

Pure iron melted in crucible	O 0.001%, N 0.0005%, C 0.001%, Si 0.01%, Al 0.001%.
Carbonyl iron powder, GA & F type HP	O 0.1 ~ 0.3%, N 0 ~ 0.05%, C 0.01% ~ 0.04%, Spectroscopically pure for metallic elements
Zone melted iron	Not determined, but recrystallization temperature was about 100°C lower than that of the pure iron melted in crucible, and $R_{300^{\circ}\text{K}}/R_{4.2^{\circ}\text{K}} \approx 200$

Cleavage Fracture of Iron Single Crystals

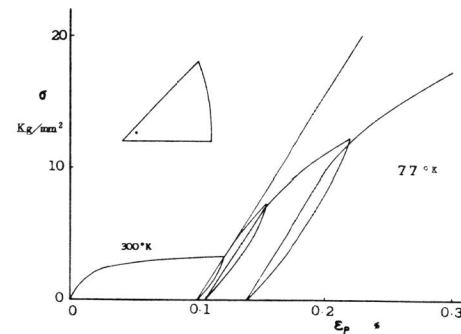


Fig. 1. Stress-strain curve at 77°K of single crystal strained by 0.1% strain at room temperature. Pure iron melted in crucible, same with other Figures excepting those noted.

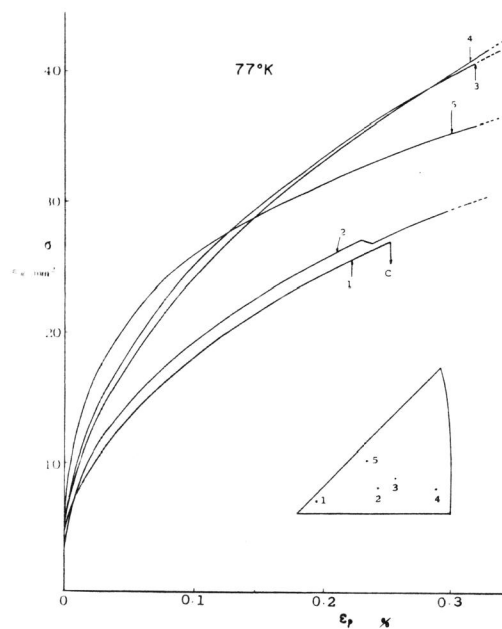


Fig. 2. Orientation dependence of stress-strain curves at 77°K of single crystals pre-strained by about 0.1% at room temperature. ϵ_p is plastic strain.

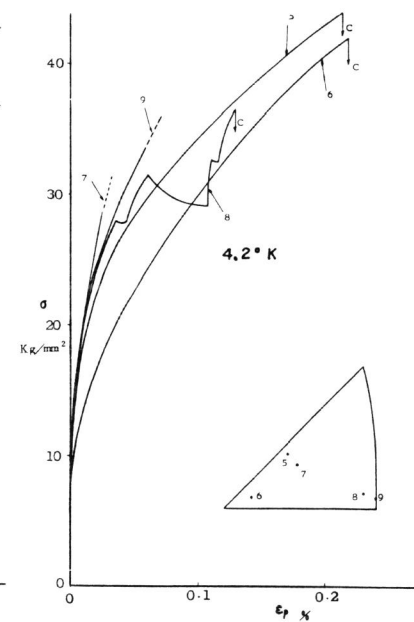


Fig. 3. Similar curves at 4.2°K to those of Fig. 2.

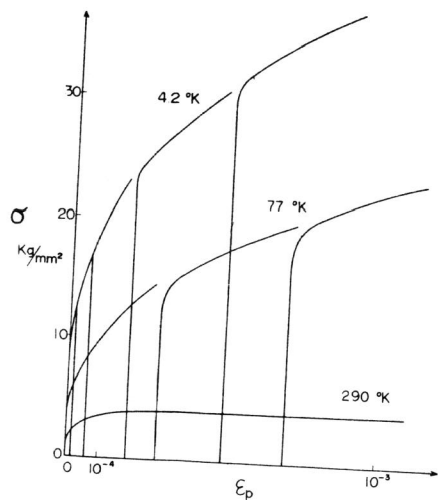


Fig. 4a. Temperature dependence of work-hardening curves of pre-strained single crystals which have same orientation. Pure iron zone-purified by 3 passes.

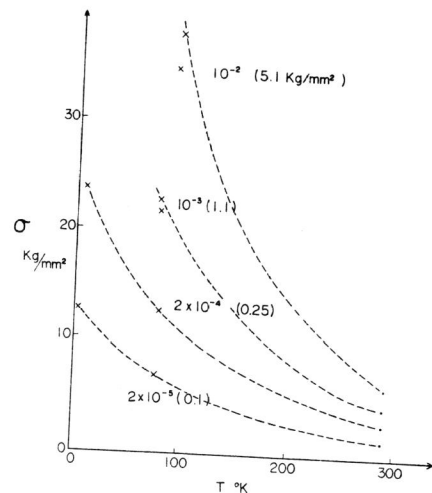


Fig. 4b. Dotted lines are flow stresses at equal plastic strains taken from Fig. 4a, in increments of flow stresses at 77 K with increasing of strain rate from 1.6×10^{-7} /sec to 1.6×10^{-5} /sec are shown in parentheses.

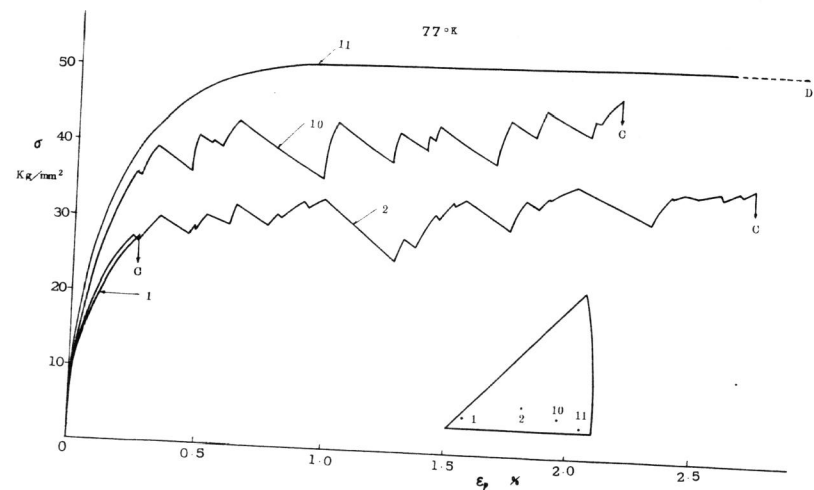


Fig. 5. Twinning and fracture characteristics of pre-strained single crystals.

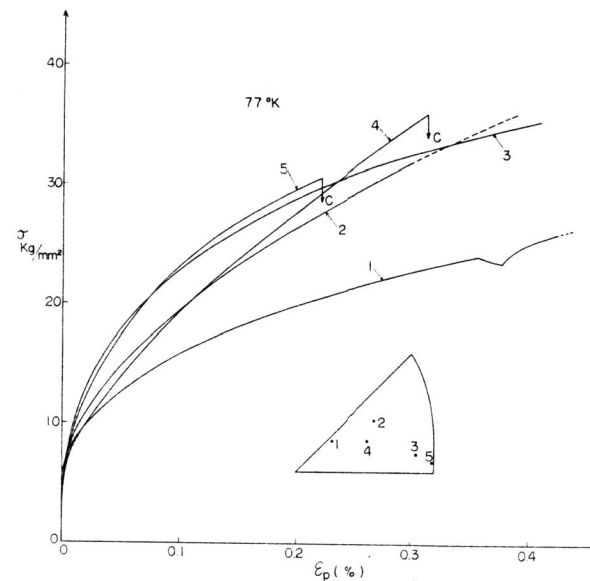


Fig. 6. Stress-strain curves at 77 K of single crystals pre-strained by 0.1%. Pure iron zone-purified by 10 passes.

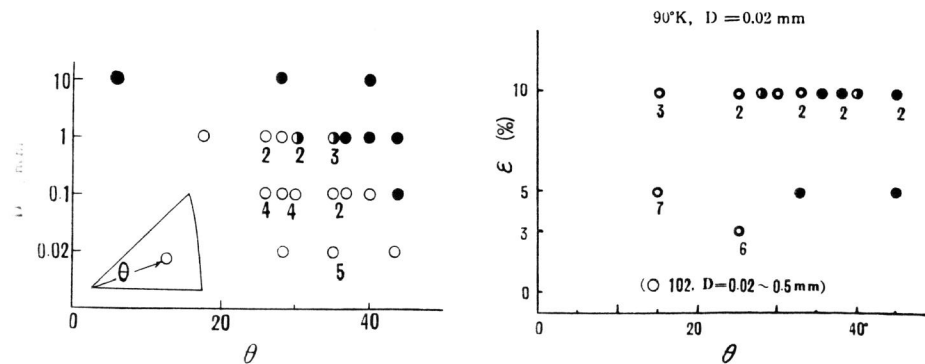


Fig. 7a. Orientation dependence of the mode of fracture of notched single crystals in static tension tests at 90 K. D is the radius of curvature at the root of notch, \bullet ; ductile fracture, \circ ; Cleavage fracture whose origin is accompanied with a twin, \odot ; Cleavage fracture whose origin is not accompanied with twin. The number under the markings is the number of specimens of same orientation tested.

Fig. 7b. Mode of fracture in shock bending tests of single crystals pre-strained at room temperature. ξ is magnitude of pre-strain. Markings are common with Fig. 6.

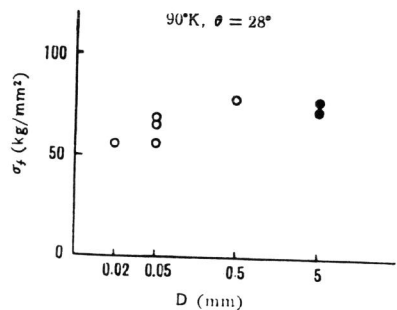


Fig. 8. Dependence of fracture stress on magnitude of radius of curvature. Markings are common with Fig. 6.

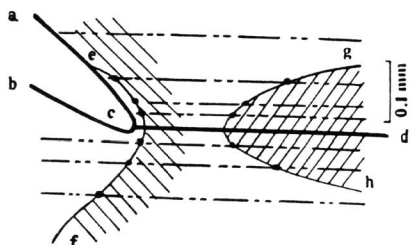
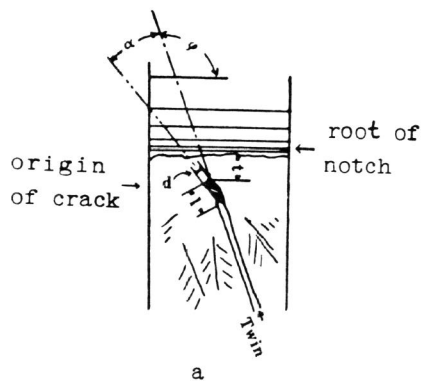
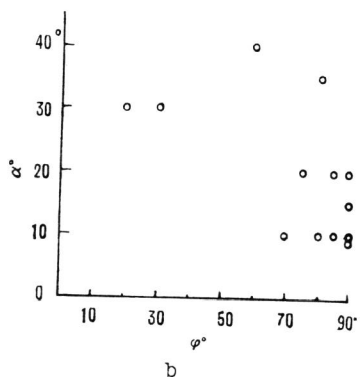


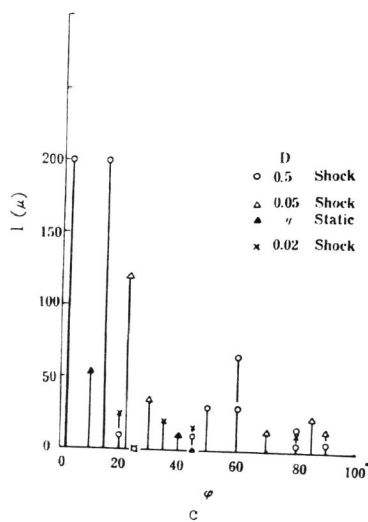
Fig. 9. Solid view of twins inside specimens.



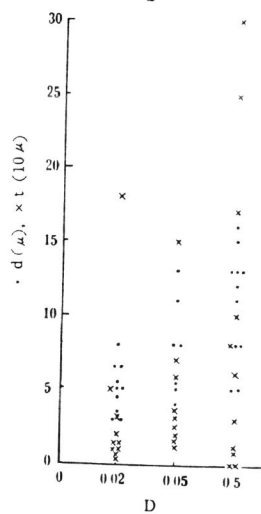
a



b

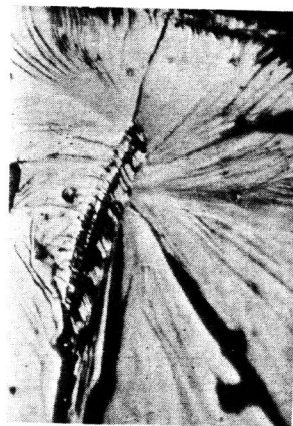


c



d

Fig. 10a, b, c, d. Topographical summaries for the origin of crack accompanied with a twin in notched specimens.



750x

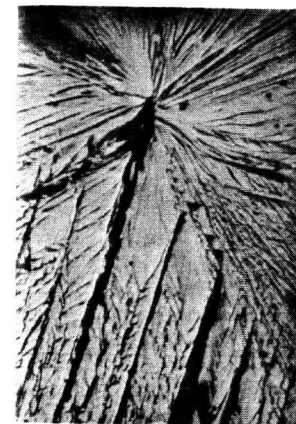


Fig. /2a

250x

Fig. 11. Some examples of origin of crack accompanied with a twin in notched specimens. $\phi \approx 90^\circ$

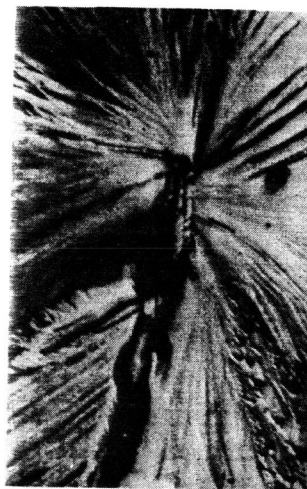


Fig. /2b

750x



Fig. /2c

750x

Fig. 12a, b, c. Some examples of origin of crack accompanied with a twin in notched specimens. $\phi \approx 90^\circ$



Fig. / 3a

250x

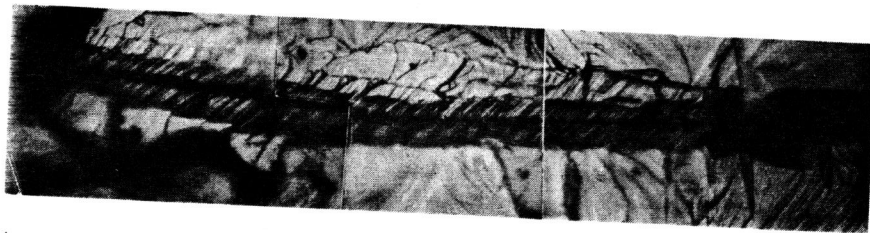


Fig. / 3b

550x



Fig. / 3c

550x

Fig. 13a, b, c. Some examples of origin of crack accompanied with a twin in notched specimens. $\varphi \approx 0^\circ$



650x

Fig. 14. Some examples of origin of crack accompanied with a twin in notched specimens. $\varphi \approx 0^\circ$

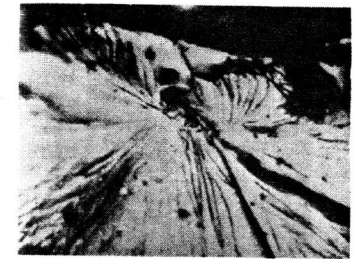


Fig. / 5a

750x

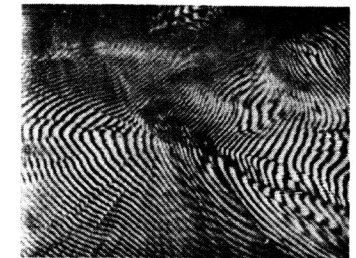


Fig. / 5b

750x

Fig. 15. Example of origin of crack not accompanied with twin and corresponding to the marking \bullet in Fig. 6 and 7.

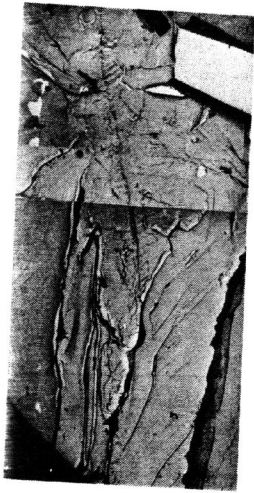
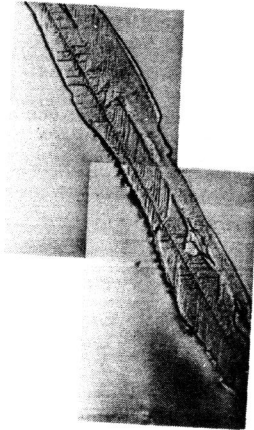


Fig. 16a 160x



200x

Fig. 17. Midrib pattern in a twin.



Fig. 16b 6500x

Fig. 16. Some examples of origin of crack accompanied with a twin in notched specimens. The same view of Fig. 11 enlarged by electron microscope.

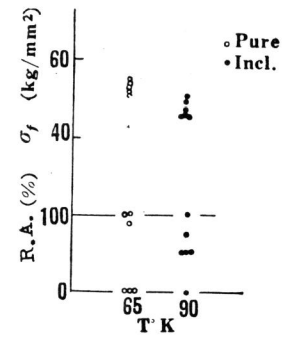


Fig. 18. Comparison of fracture characteristic between single crystals of pure iron and those containing carbides. Simple tension test, R.A.; reduction of area.

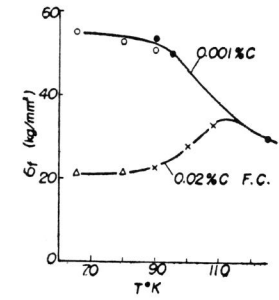
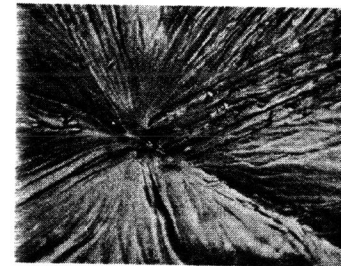


Fig. 19. Comparison of fracture characteristic between single crystals of pure iron and those containing carbides. Tension test of notched single crystals of same orientation. O, ●; common with Fig. 6, X; origin of crack started from a carbide without accompanying twin, Δ; origin of crack started from a carbide met with a twin.



650x

Fig. 20. Example of origin of crack started from a carbide as shown in Fig. 19.

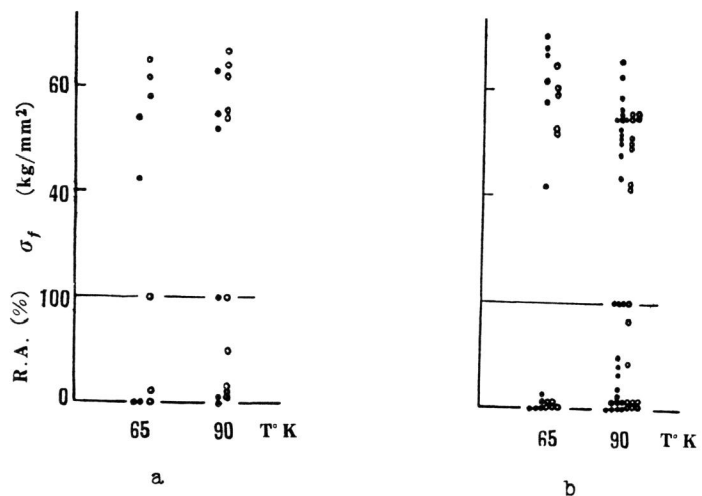
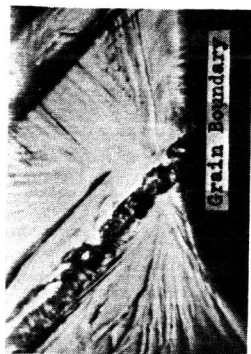
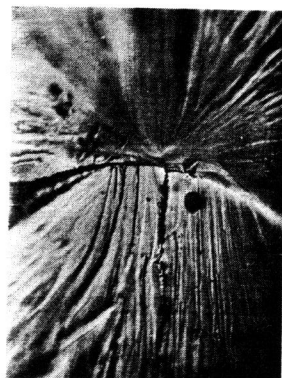


Fig. 21a, b. Simple tension tests of bi-crystals. Fig. 21a; pure iron, Fig. 21b; iron carburized by 0.02% carbon and furnace cooled. ○; boundary of crystals is nearly parallel to tension axis, ●; boundary of crystals is nearly perpendicular to tension axis.



650x

Fig. 22. Origin of crack in pure iron bi-crystal.



320x

Fig. 23. Origin of crack in bi-crystal of iron containing carbides.

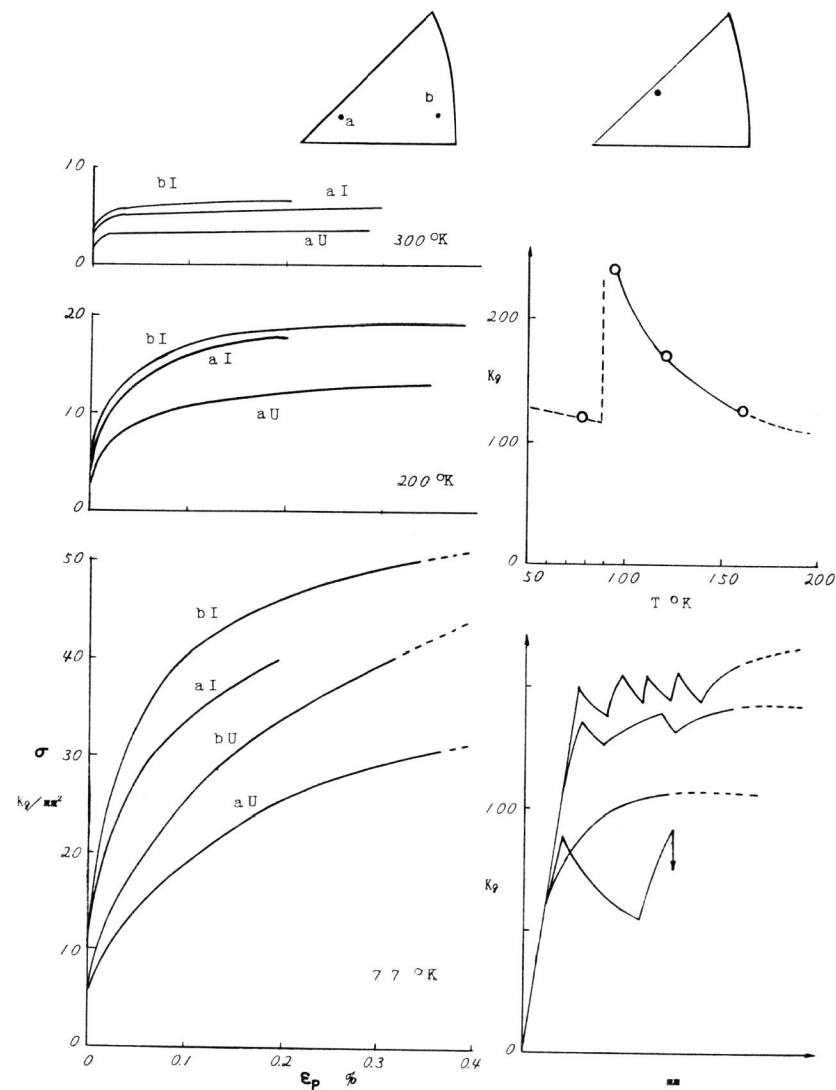


Fig. 24. Effects of neutron irradiation on stress-plastic strain curves of pure iron single crystals pre-strained by 0.1% at room temperature. Pure iron zone-purified by 2 passes.

Fig. 25. Fracture characteristics of neutron irradiated pure iron single crystals which have same orientation. Notched specimens.