BI-7 TWINNING, SLIP AND BRITTLE FRACTURE IN VERY LOW CARBON IRON

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ABSTRACT

Tensile tests of iron single crystals and of polycrystalline iron specimens with various grain sizes were carried out at low temperatures down to -196°C. Microcracks and fracture sources were examined metallographically. The carbon content was so low that no carbides were visible in the specimens. The cleavage fractures of iron single crystals occurred from twin-twin intersections or from twin-included grain intersections. Twins induced fracture in polycrystalline iron when the grain size was very coarse or when the grain boundary was weak. Slip also induced cleavage or boundary fracture in polycrystalline iron over a wide range of grain size.

INTRODUCTION

The relation between the low temperature plastic deformation and the brittle fracture or semi-brittle fracture has been a subject of many discussions. (1) And yet, reports were rather scarce on iron to show experimentally under what conditions and how the slip or twinning is related to the brittle fracture. The purpose of this presentation is to give some answers to these questions on carbide-free iron.

The presentation is based on two recent experimental works: one on iron single crystals (2) and the other on polycrystalline iron. (3) They both consisted of low temperature tensile tests and metallographical observations. In either experiment, the carbon content was so low that no carbides were visible under microscope. The results of the above two works are consistent and complementary with each other and thus this unified presentation was considered to be useful. More details will be given elsewhere. (2,3)

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2. FRACTURE OF SINGLE CRYSTALS

2.1 Experimental procedure

Sheet specimens of iron single crystals of various impurity levels (P, F and C specimens) were made by strain annealing method in temperature gradient furnaces. P specimens were made of puron iron obtained from Westinghouse Electric Company and contained a fairly high amount of oxygen. F specimens were made of ferrovac E iron and had a good purity. C specimens were made of electrolytic iron and then slightly carburized. Their impurity contents are given in Table 1. As will be described later, many single crystals fractured from included grains. And the carbon and oxygen contents were expected to affect the fracture tendency of their boundaries. And thus above specimens with different carbon and oxygen contents were tested.

Tensile tests of those single crystals were carried out at low temperatures down to -196°C. The gauge part size of the tensile specimens was $25 \times 5 \times 1.2$ mm^3 in P specimens and $20 \times 6 \times 1.0$ mm^3 in C specimens. The F specimens were somewhat smaller. The strain rate was 0.015 per minute in P and F specimens and 0.2 per minute in C specimens.

It is important to note that these specimens included a number of small isolated grains mostly of a diameter of 50-200 microns embedded in the single crystal matrix. Such included grains were reported by several workers (4,5) and appear to be practically unavoidable in iron single crystals.

After the tensile tests, microcracks were carefully examined under microscope. Also, the cleavage fracture source was identified by tracing back the markings on the cleavage face of each specimen.

2.2 Results on P and F specimens

The tensile directions of P specimens tested at various temperatures are given in Fig. 1.

At -196°C and -178°C, all the P specimens fractured by cleavage. And the plastic deformation prior to the fracture was either mainly due to twinning up to several percent or twinning plus slip. Occasionally, specimens fractured without noticeable plastic deformation on the recorded stress-strain curves. Only in specimens with tensile axis away from [001] , slip deformation was appreciable.

At -160°C and -140°C, twinning and cleavage fracture were observed in most of the specimens. An appreciable amounts of macroscopic slip occurred in all the specimens before the fracture.

At -110°C, neither twinning nor cleavage fracture occurred. The specimens had the tensile directions under which twinning and cleavage fracture were predominant at lower temperatures. And the specimen fractured with nearly 100% reduction in area of cross section.

Microcracks, in particular those far from the cleavage face were rarely reported in iron single crystals. In P specimens, however, a variety of microcracks were observed at twin-twin intersections (A-type), at twin-included grain intersections (B-type) and at other sites (C-type). These were scattered over the whole gauge part of specimens. But no microcracks were found in specimens which did not twin or in regions where no twins were observed.

A-type cracks formed where twins were stopped by other twins on various sites as shown in Figs. 2, 3 and 4. B-type cracks formed where twins were stopped by included grains as shown in Figs. 5(a) and (b). C-type cracks (Figs. 6, 7 and 8) were observed around twins, in twins, on twin interfaces and so on. Incidentally, cleavage cracks were often stopped by other twins as observed in Fig. 3 and Fig. 5.

The cleavage initiation source, which was identified on the cleavage face, was either a twin-twin intersection or an included grain. And thus A- and B-type cracks are considered to have been the most important in the cleavage fracture. In Fig. 9(a) showing a cleavage face, cleavage markings are observed to radiate from a thick black line running from upper left to lower right. That the black line is actually a joint trace of two twins intersecting on the cleavage face is clear from Figs. 9(b) and (c) which show the specimen surface close to the cleavage face. In Fig. 10 which shows another cleavage face, cleavage markings are observed to radiate from an included grain in the centre of the picture. Twin traces are also observed in the vicinity. Specimens fractured more frequently from included grains than from twin-twin intersections.

Qualitatively similar fracture behaviors and microcracks were observed in F specimens in which the oxygen content was much lower than in P specimens.

2.3 Results on C specimens

All the five of tested C specimens were cut out from one carburized single crystal sheet and had the same tensile axis nearly of <110> . Out of five, four specimens fractured by cleavage in a rather brittle manner during twinning. The other one slipped much after twinning and fractured with almost 100% reduction of area. The representative stress-strain curves are shown in Fig. 11. Some number of small microcracks were observed in twins or on twin boundaries. But non of them were grain boundary cracks or cleavage cracks. The cleavage fracture source was, nevertheless, the included grain again in every C specimen fractured by cleavage.

2.4 Fracture mechanism of iron single crystals

The formation of A and B type microcracks which were the most important microcracks in the fracture can most reasonably be understood as follows. When a massive twin is stopped by another twin or by an included grain, a large concentrated stress appears at the head of the stopped twin. (6) This concentrated stress causes a crack, when the stress release by slip does not effectively occur, directly on $\{100\}$ plane or on a weak interface such as a twin boundary or a grain boundary. Interface cracks sometimes switch to cleavage cracks.

No much discussion will be given here on C group cracks which were not important in the fracture event. However, it will be noted that cracks as shown in Figs. 6 and 7 can possibly be understood as cracks which formed on the boundaries of small barrier twins at twin-twin intersections. That is, they may substantially be A type cracks. Here, the small barrier twins could not be observed presumably because they disappeared by detwinning leaving only cracks. In fact, the size, shape and orientation of those cracks were such that are expected to be according to this idea.

The cleavage fracture in C specimens occurred only during twinning but never during slip. And the fracture sources were again observed to be included grains. Thus, the fracture initiation mechanism in C specimens is considered to be essentially the same with that in P and F specimens: twin-included grain intersection.

As was described already, no B-type cracks and no cleavage cracks were observed in C specimens. This was so, although many twin-included grain intersections were observed. And in such a sense, the fracture initiation was more difficult but the fracture propagation was easier in C specimens than in P and F specimens.

The observed statistical frequency of the crack or fracture initiation at twin-grain intersections was several percent of the total number of twin-included grain intersections, in P and F specimens at -196°C. Whereas it must have been less than one percent in C specimens. This difference can be interpreted as due to the effect of carbon to strengthen the grain boundary in iron. (7) The frequency of the crack formation at twin-twin intersections was less than one percent of the total number of twin-twin intersections in any specimen.

3. FRACTURE OF POLYCRYSTALLINE IRON

3.1 Experimental procedure

Polycrystalline iron specimens with gauge part size of $20\times6\times1\mathrm{mm}^3$ were vacuum annealed at various temperatures and then furnace-cooled to control the grain size. The heattreatment, impurity content and the resulted grain size are given in Table 2.

These specimens were extended in liquid nitrogen either directly or after an application of prestrain at room temperature to suppress twinning during the subsequent low temperature tests. The strain rate was 5mm per minute.

During the heattreatment for the grain size control, the carbon content

changed slightly as shown in Table 2. In order to check if such changes were trivial or significant for the fracture behavior, a number of specimens were slightly decarburized in wet hydrogen and then homogenized in vacuum. From the tests of these specimens, it was found that an amount of carbon as little as 0.0011% - 0.0016% was enough to strengthen the grain boundary in this iron. And such slight variations of impurity contents in the range as shown in Table 2 were proved not to be substantial in their fracture behaviors.

3.2 Results

Stress-strain curves of some specimens tested in liquid nitrogen with or without room temperature prestrain are shown in Fig. 12 (a) and (b). Only in prestrained D(1200μ) specimens, occasionally shear type fracture occurred with almost 100% reduction in area. Otherwise, the fracture was predominantly cleavage type throughout the whole range of grain size, regardless of the amount of prestrain. The area of grain boundary fracture was only 5% or so of the whole fracture face. Only little area of fibrous fracture was observed. Substantially no microcracks were observed in any specimen, except for a few of D specimens. And thus, the fracture was rather initiation-controlled in this polycrystalline iron.

In D(1200μ), I(643μ) and M(388μ) specimens, the stress-strain curve was not reproducible but fairly different from specimen to specimen. However, the general trend as follows was observed. D(1200μ) specimens, unless prestrain was applied, fractured by cleavage mostly after a small smount of elongation due to twinning. If prestrain was applied, the elongation at the low temperature increased markedly, as shown in Fig. 12. Thus, most of the D specimens are considered to have fractured by a twin-induced mechanism, if not prestrained. The mechanism of this twin-induced fracture initiation is believed to have been the same as that occurred in single crystals. Slip does not appear to have had an effective tendency to induce fracture in D specimens, consistently with the result obtained on single crystals.

Unprestrained I(643μ) and M(388μ) specimens sometimes fractured by cleavage during twinning as unprestrained D(1200μ) specimens did. Differently from D specimens, however, elongation of unprestrained I and M specimens fairly often stretched beyond the twinning elongation into the region of slip deformation. And thus, it can be said that the tendency of fracture initiation by twins decreases pretty sharply with decreasing grain size. This trend becomes clearer when specimens of finer grain size are considered later.

If the specimens were prestrained, the elongation of I(643μ) and M(388μ) specimens was not larger but smaller than that of prestrained D(1200μ) specimens. This is rather an unusual dependence of elongation upon grain size and may somehow be related to the situation that the grain diameter of D specimens is comparable with the specimen thickness. In the range of finer grain size, the dependence of elongation upon grain size was usual as will be observed on Fig. 12.

In the range of grain size from 227μ to 50μ in diameter, the stress-strain curves were reproducible. And the stress-strain curves of unprestrained and prestrained specimens were pretty well overlapped as shown in Fig. 12, so far as the amount of prestrain was not very large, say less than 10%. In other words, the equation of state was valid in an approximate sense in this iron. Moreover, prestrain did not substantially change the elongation before the fracture, though twinning was suppressed. Thus, in this range of grain size, no twins but slip is believed to have induced the fracture. The tendency of fracture initiation by slip decreased with decreasing grain size in such a sense that the fracture occurred at a higher stress and after a larger amount of elongation.

It will be noted that many twins were observed in all the unprestrained specimens, even though they were fine in fine grains. The twinning occurred together with the Luder's band propagation in A(50 μ) specimens. In all the other specimens with coarser grain size, it preceded the yielding by slip.

In D(1200 μ), I(643 μ) and M(388 μ) specimens of coarser grain size, it was often possible to locate the position of primary source of cleavage fracture. The source was on a grain boundary in major cases but occasionally in a grain. In particular, the fracture initiation inside of a grain was rare in prestrained specimens.

3.3 Fracture mechanism and the grain size

It was concluded from the experimental results that, twins initiate the fracture so long as the grain diameter is more than several hundred microns. However, they do not initiate fracture in the specimens of finer grain size, unless second phases such as carbides exist.

From the mechanism described in 2.4, three points are considered to be important to determine the tendency for twins to cause cracks. (1) Magnitude of stress concentration caused by twins. (2) Easiness of stress release by slip or by other twins. (3) Existence of weak plane, weak interface or weak second phase. As for the grain size, it limits the size of twins and thus limits the magnitude of the stress concentration. This would be the most important reason of sharp grain size dependence of the fracture initiation tendency of twins.

As was already described, slip did not have much tendency to cause cleavage fracture in single crystals and in polycrystalline specimens with the grain diameter of 1.2mm, the order of the specimen thickness. However, the tendency of slip-induced fracture was pretty large in specimens with grain diameters of 643-388 microns. It decreased again with further decrease of the grain diameter.

The mechanism by which slip initiates the semi-brittle fracture is not clear. And thus the reason why slip had little tendency to induce fracture in single crystals and in extremely coarse grain size is not clear either. It may be related to the level of flow stress or to the easiness of stress release depending on the ratio of the grain size to the specimen size. Also, it is probable that a point where three grain boundaries meet together

is important in the slip-induced fracture initiation. Such points do not exist in single crystals, even though they include isolated grains.

So far, the specimens in which the fracture was predominantly cleavage type were described. However, after some treatments, such as an annealing in wet hydrogen not followed by vacuum homogenization, intercrystalline fracture occurred after rather little amount of elongation. Examples of stress-strain curves of such specimens are shown in Fig. 13. These specimens were, after the annealing for grain size control, annealed in wet hydrogen for two days at 700°C and furnace cooled. They were quite brittle at liquid nitrogen temperature. However, their fracture stress was close to the twinning stress of the specimens which had the same grain size but which were not annealed in wet hydrogen, unless prestrain was applied. And the prestrain increased the fracture stress. Apparently, in such specimens with weak grain boundary, either twins or slip can induce fracture very easily, even if the grain size is pretty fine. And yet, it seems that plastic deformation was necessary for the fracture initiation.

4. CONCLUSIONS

Concerning the relation between the low temperature deformation and brittle or semi-brittle fracture initiation, the followings were concluded under the present experimental conditions.

(1) Twins can initiate fracture when they are stopped by other twins or by grain boundaries. The mechanism works effectively in single crystals and in polycrystalline iron with very coarse grain size. If the grain boundaries are weak, it works in a range of much finer grain size.

(2) Slip can initiate cleavage and boundary fracture. The tendency of fracture initiation by slip increases with increasing grain size generally. However, if the grain size becomes extremely coarse and in single crystals, the tendency decreases again. The tendency also depends sharply on the grain boundary strength.

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Table 1. Impurities in iron single crystals

Specimen	C	N	O	S	P	Si	Mn
P	.002	.001	.06	.002	<.001	<.001	.005
F	<.001	.001	.003	.006	.003	.006	.001
C	.010	.001	.03	.004	.001	<.001	.001

In weight percent

Table 2. Polycrystalline iron specimens

Composition as received: weight percent

C	Total N	O	Si	Mn	P	S	Sol.Al	Insol.Al	Mo	Cu	Cr	Ni
.0047	.0065	.0042	.16	.087	.002	.007	.004	.028	.016	.016	.001	.006

Specimens after vacuum annealings:

Specimen	Vacuum annealing (1-5x10^{-5} Torr)	Grain diameter (microns)	C	N	O
			(weight percent)		
A	855°Cx0.5hr	50	.0026	.0069	.0040
J	700°Cx0.5hr	78	.0055		.0032
K	950°Cx0.5hr	122			
C	835°Cx60hr + 955°Cx16.5hr	135	.0026	.0077	.0033
E	835°Cx60hr + 955°Cx33hr	172	.0039	.0074	.0039
F	as C above + 1045°Cx4hr	181			
H	1045°Cx4hr + 1115°Cx4hr	227	.0031	.0056	.0038
M	1160°Cx4hr	388			
I	1180°Cx4hr	643	.0025		.0035
D	1215°Cx4hr	1200	.0016	.0019	.0055

Partially decarburized specimens, A', E', H' and D', contained 0.0011 - 0.0016%C and 0.0038 - 0.0053%O.

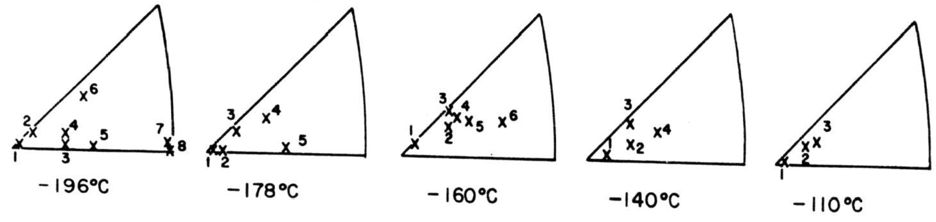


Fig. 1 Direction of the tensile axis of puron specimens tested at various temperatures. The specimen number is given.

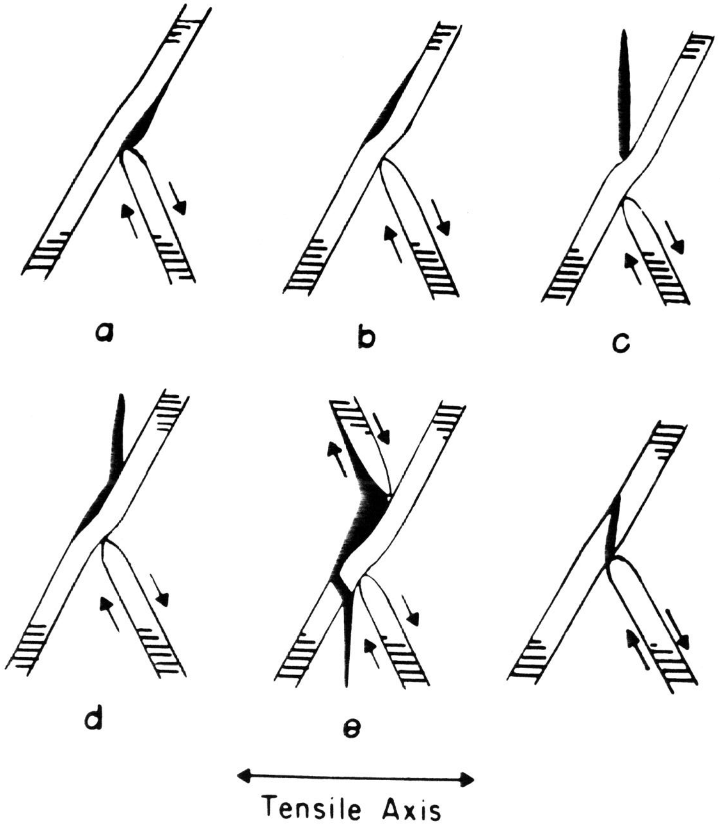
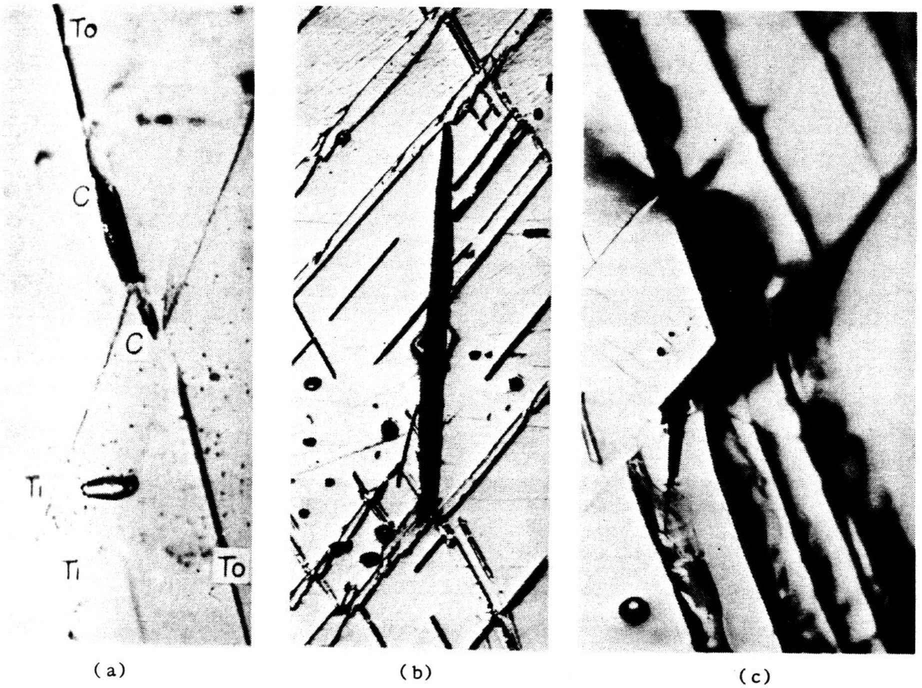
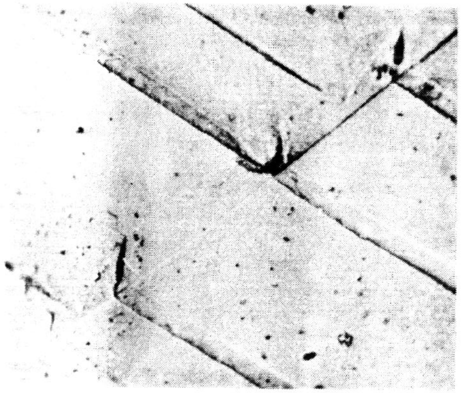


Fig. 2 Various microcracks (black regions) observed at intersections of twins (partly hatched regions), schematically shown. Direction of twinning shear is given by arrows.



(a) (b) (c)



(d)

Fig. 3 Cracks at twin intersections. (a) Inter face cracks, C, on a twin, T_0, caused by twins, T_1. Original surface. P4, -196°C. x900. (b) Cleavage crack. Polished surface. P4, -196°C. x230. (c) Crack partly on a twin boundary and partly of cleavage type. Original surface. P4, -196°C. x450. (d) Cracks on { 110 } planes in the barrier twin. Original surface. P7, -196°C. x460.

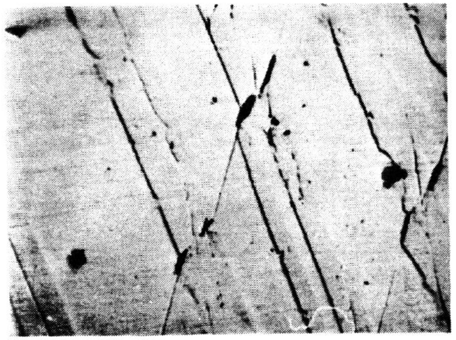


Fig. 4 Cracks on twin boundaries at twin-twin intersections. P4, -196°C. 460X.

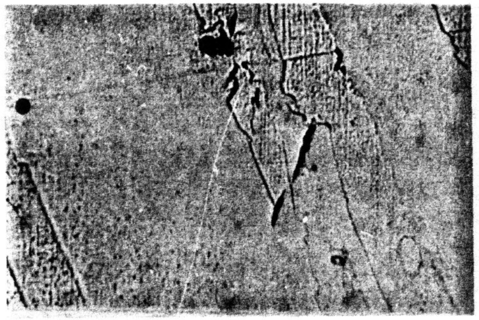
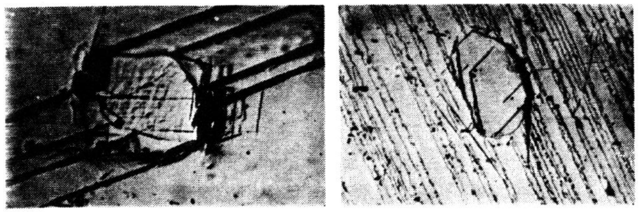
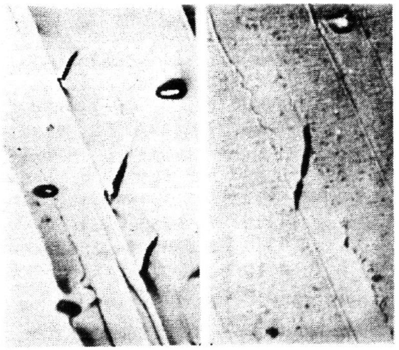


Fig. 6 Three cracks around twins. Polished. P4, -196°C, ×500.



(a) (b)

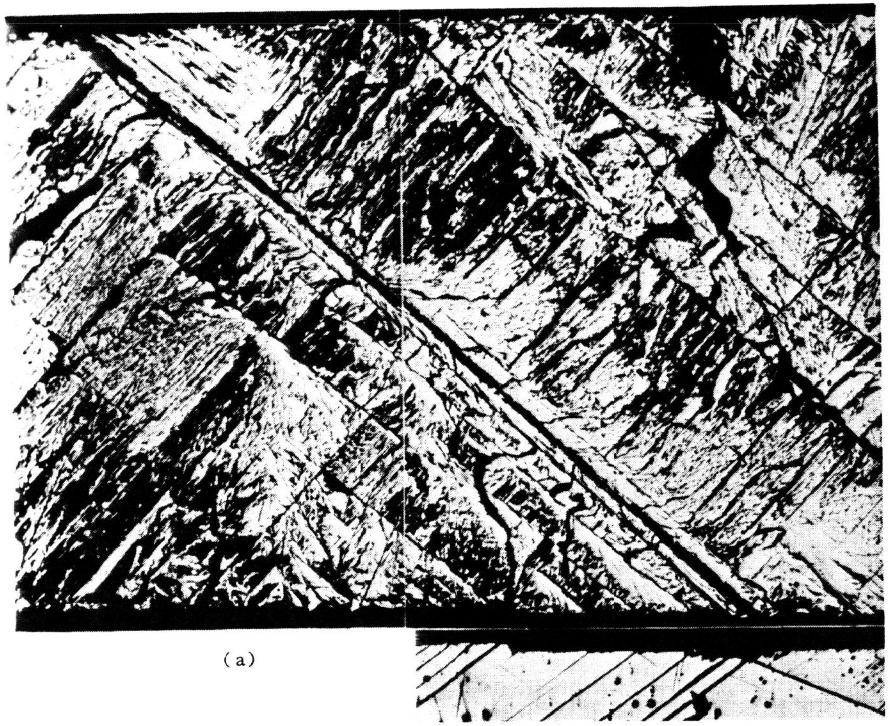
Fig. 5 Cracks at included grains. (a) Boundary cracks associated with twins. Original surface. P8, -196°C. 160X. (b) Boundary crack which switched tangentially to a cleavage crack. Polished. P4, -196°C. 125X.



(a) (b)

Fig. 7 Cracks in twins. P4, -196°C. 370X. (a) Original surface. (b) Polished.

Fig. 8 Cracks associated with a twin. Original surface. P4, -160°C. 370X.



(a)

(b)

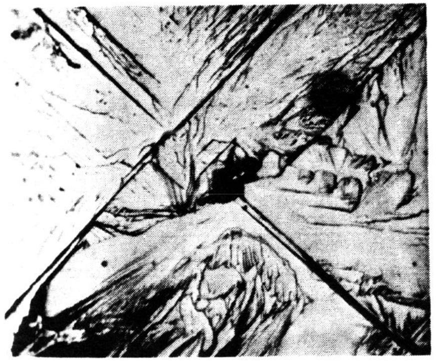
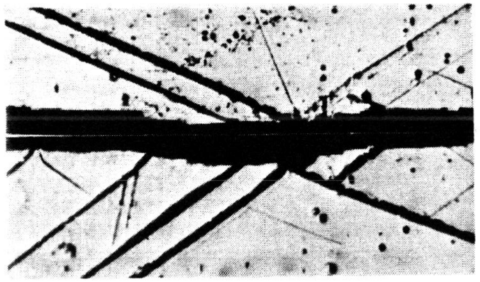


Fig. 10 Cleavage face around an included grain where the fracture started. 120X.



(c)

Fig. 9 Cleavage fracture from a twin-twin intersection. (a) Cleavage face. P5, -196°C. 86X. Thick black line from upper left to lower right is the trace of two intersecting twins. (b) Specimen surface close to the cleavage face. 85X. Two twins correspond to the trace observed on the cleavage face. (c) As in (b). 170X.

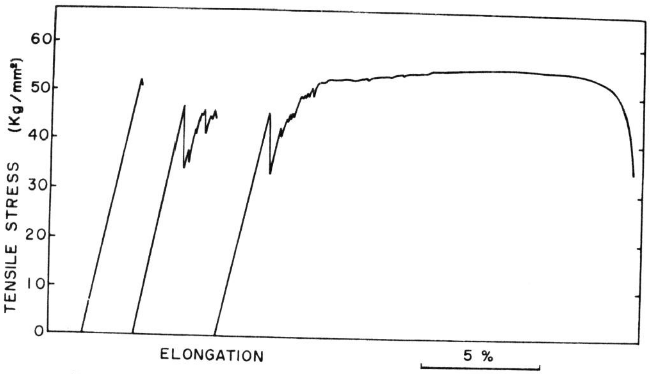


Fig. 11 Stress-strain curves of three 0.010% carbon iron single crystals of the same tensile axis close to <110> tested at -196°C.

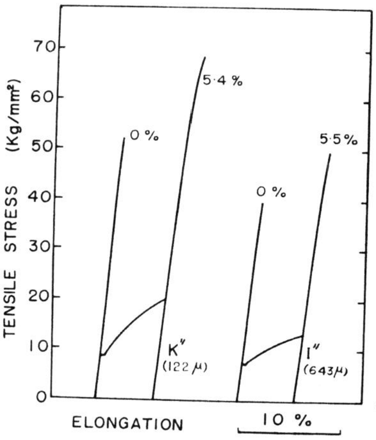


Fig. 13

Stress-strain curves of wet hydrogen annealed specimens tested at -196°C. The amounts of the prestrain are given.

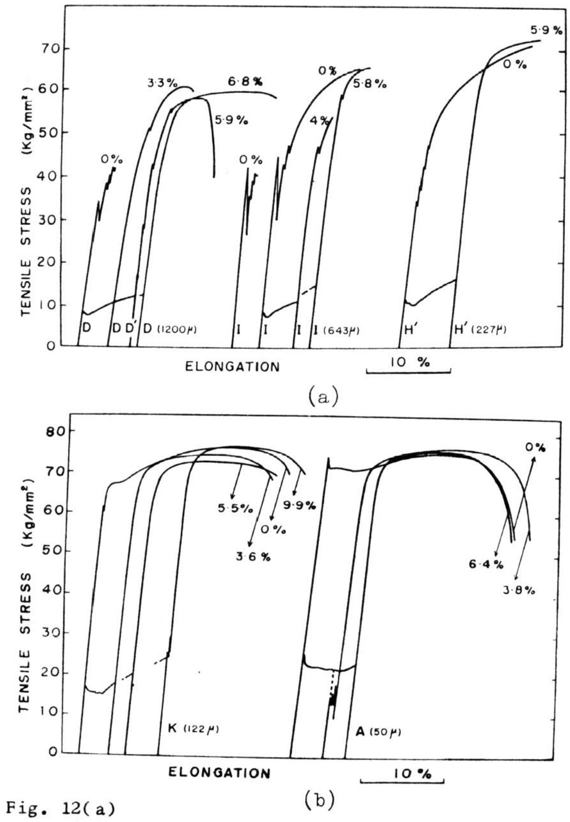


Fig. 12(a) and (b)

Stress-strain curves of Polycrystalline iron specimens tested at -196°C after various amounts of prestrain. The grain diameters and the amounts of prestrain are given.