Effects of Small Amount of Impurities on the Yield Stress of Iron Single Crystals*

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Synopsis

Iron single crystals with the $[110]$ extension axis containing small amount of carbon (about 20 ppm and less than 1 ppm) and nickel (0.05 to 0.005%) were extended in a range of temperature between 200° and -195°C. The effect of impurities on the lower yield stress was recognized only on the athermal component. Decarburization and addition of a small amount of nickel suppressed the appearance of twinning deformation, and in some cases twinning deformation was completely suppressed at -195°C. Large glide bands which appear in as-grown crystals at the beginning of deformation were not observed on decarburized crystals; this fact means the increase in the number of mobile dislocation initially present or dislocation sources in decarburized crystals. It was concluded that a small amount of impurities gave large effects to the upper yield stress and its related phenomena, but they gave very small effects to the lower yield stress.

I. Introduction

An important problem in the theory of crystal plasticity is the explanation of temperature and strain rate dependence of yield stress and flow stress of an ideally pure crystal. The strong temperature dependence of yield stress of iron or other bcc metals is considered to be the intrinsic property of crystal or the Peierls stress for the motion of dislocation.^{1),2)} This explanation, however, has not been well accepted because there have been some objections from the fact that the impurities with tetragonal distortion have a large contribution to the temperature dependence of yield stress in iron.³⁾

The effect of small amount of impurities on the yield stress of iron has been investigated mainly on interstitial impurities such as carbon^{4),5)} and nitrogen atoms,^{6),7)} but the conclusions obtained from these experiments are conflicting. It is expected that tensile characteristics of iron would be also affected by small amount, about 0.01%, of metallic impurities. Then, investigations of the effects of substitutional metallic impurities on the mechanical properties of iron will be important to get an insight into the properties of ideally pure iron. In the present report, iron single crystals of an identical crystal orientation which contained 0.05 to 0.005% of nickel were prepared, and the yield point behaviour was observed after various heat treatments in a range of temperature between -196° and 200°C.

II. Experimental Procedure

Electrolytic iron with weight of 50 kg was melted in a vacuum induction furnace, deoxidized by carbon in an argon atmosphere and casted in vacuum. Such ingots contained about 0.01% carbon and about 0.002% oxygen. It was hot-forged to a plate of 25 mm in thickness. This plate could be rolled at room

temperature free from cracking in a rolling mill. Subsequent processes of cold-rolling, annealing and growth of large single crystals of 1 mm in thickness were the same as the previous method.⁸⁾ Crystals which contained small amount of nickel were prepared from 7-kg ingots in which 0.02 and 0.05% of nickel were added respectively. These ingots were hot-forged and cold-rolled to sheets of 1 mm thick by the same procedure as the pure iron. The process for the growth of large crystals from these sheets was slightly different from that of pure iron, because the cold-rolled sheets which contained 0.05 and 0.02% nickel exhibited secondary recrystallization in annealing at above 800°C for 3 hr. Accordingly, the cold-rolled sheets were firstly annealed at 800° or 780°C for about 15 min in dry-hydrogen and cooled in the furnace. Critical strain was applied at about 70°C in order to reduce the Lüders strain less than 3%. The other processes were the same as the previous method.⁸⁾

Chemical analysis of an as-grown crystal of pure iron showed the following impurities in wt%:

C: 0.002	Si: 0.001	Mn: 0.002	P: 0.001
S: 0.002	Ni: 0.005	Co: 0.010	Cu: 0.001
Al: 0.001			

Content of nickel in the irons in which 0.02 and 0.05% of nickel were added was 0.019 and 0.043% respectively in chemical analysis. Thus the three kinds of specimens are called 0.005%Ni-Fe (or pure iron), 0.02%Ni-Fe, and 0.05%Ni-Fe crystals.

Dimensions of test specimens used in this experiment are shown in Fig. 1. All the specimens had the (100) plane on the sheet surface and the $[011]$ direction in the extension axis. After mechanical shaping and polishing, surface layers of the specimens were removed about 50 microns by chemical polishing.

In order to change the amount and the distribution of carbon atoms, following four heat treatments were selected.

(1) Annealing at 850°C for 4 hr in vacuum of 2×10^{-5} mmHg followed by furnace cool. This process will be called as-grown.

(2) Annealing at 200°C for 3 hr and water quenching after the process (1).

(3) Annealing at 700°C for 48 hr in wet-hydrogen saturated with water vapour at room temperature and 700°C for 18 hr in dry-hydrogen followed by furnace cool.

(4) Annealing at 850°C for 11 days in hydrogen purified by circulating through a ZrH_2 furnace⁴⁾ after

* Received February 28, 1968.

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Fig. 1. Test specimen and grips

the process (3).

Tensile tests were made in an Instron type testing machine. Some of the specimens were extended under a constant load in a lever type creep testing machine. Tests were carried out in a temperature controlled silicon-oil bath above room temperature and iso-pentane or liquid nitrogen bath below room temperature.

III. Experimental Results

1. Load-Elongation Curve

The load-elongation curves of pure irons extended at 200°C are shown in Fig. 2. The strain rate was $4 \times 10^{-3} \text{ sec}^{-1}$. As-grown crystal showed larger work-hardening rate and larger flow stress than the decarburized crystals. The load-elongation curve of as-grown crystal was not smooth in the later stage because of the dynamical strain aging of solute carbon atoms. Workhardening rate of the decarburized crystal was smaller than that of the as-grown crystal in the initial stage, and it became much smaller after an elongation more than 10%. There was no difference between the decarburized crystal with heat treatment in wet-hydrogen and that in ZrH_2 -purified hydrogen. The solubility of cementite in iron has been reported to be 0.0001% at 200°C.⁹⁾ Then, it may be considered that the as-grown crystals contained effective carbon atoms more than 0.0001% and the decarburized crystals much less than 0.0001%. The crystals of 0.05%Ni-Fe showed the same load-elongation curve as pure iron (Fig. 2) at 200°C.

The load-elongation curves of iron single crystals which contained different amounts of nickel after various heat treatments were measured at room temperature and are shown in Fig. 3. The upper yield stresses and the stress drops at yield points were the largest in as-grown crystals for all nickel contents. Upper yield point could not be eliminated from the load-elongation

Fig. 2. Load-elongation curves of pure irons containing different amounts of carbon impurity at 200°C

Fig. 3. Load-elongation curves of pure irons, 0.02%Ni-Fe, and 0.05%Ni-Fe crystals extended at 25°C after three kinds of heat treatments indicated

curves after decarburization or quenching into water from 200°C. In pure iron, the lower yield stresses were substantially constant in three kinds of heat treatments. The effect of heat treatment on the lower yield stress and the initial work-hardening rate were the largest in 0.05%Ni-Fe crystals. The large effect of heat treatment on the 0.05%Ni-Fe crystals suggests that the

Fig. 4. Load-elongation curves of single crystals of pure iron and 0.05%Ni-Fe extended at 114°K after three kinds of heat treatments indicated

initial work-hardening rate is affected by a small amount of metallic impurities and heat treatments. Such an effect was the most prominent at room temperature, and it was not observed at -78°C .

At lower temperature than -100°C , twinning deformation could be observed in as-grown crystals of pure iron, in agreement with previous reports.^{10,11} Fig. 4 shows the load-elongation curves obtained at 114°K. As-grown crystals of pure iron were deformed always by twinning at 114°K, but decarburized crystals were deformed sometimes without twinning. Twinning deformation was not observed in 0.05%Ni-Fe crystals after all heat treatments as shown in Fig. 4. The crystals which were quenched from 200°C had large upper yield stress but did not show twinning deformation.

Load-elongation curves of crystals extended at 78°K at crosshead speeds of 5 mm/min and 0.05 mm/min are shown in Fig. 5. At higher strain rate, 5 mm/min, the crystal which had no twin was only 0.05%Ni-Fe crystals quenched from 200°C. At low strain rate, 0.05 mm/min, decarburized specimens deformed without twinning, and as-grown crystal of 0.05%Ni-Fe showed twinning deformation after slight yield drop. The upper yield stress or twinning stress of 0.05%Ni-Fe crystals was higher than that of pure iron for each heat treatment. These results show that addition of a small amount of nickel suppresses the appearance of twinning deformation in iron single crystals. Similar effects were observed on the crystals after decarburization and quenching from 200°C. Twinning deformation was very sensitive to heat treatment.

2. Yield Stress

Lower Yield Stress

Lower yield stress was defined in the present investigation as the minimum true stress in the initial part of true stress-strain curve calculated from observed load-elongation curve. The variation of lower yield

Fig. 5. Load-elongation curves of single crystals of pure iron and 0.05%Ni-Fe after three kinds of heat treatments indicated. The specimens were extended at 78°K under a crosshead speed of (a) 5 mm/min and (b) 0.05 mm/min.

stress in wet-hydrogen treated pure iron with temperature at various crosshead speeds is shown in Fig. 6. This datum has been analysed in another paper.¹² In the present paper, the curve of the strain rate of $4 \times 10^{-3} \text{ sec}^{-1}$ was used as the reference curve for comparison of the temperature dependence of lower yield stress of various crystals.

Lower yield stresses of as-grown and decarburized crystals of pure iron and 0.05%Ni-Fe crystals were plotted in Fig. 7 as a function of temperature. The solid line is the reference curve obtained from Fig. 6. Lower yield stresses of ZrH₂ treated pure iron and 0.05%Ni-Fe crystals were same as those of wet-hydrogen treated pure iron (reference curve) within the experimental accuracy. Lower yield stresses of as-grown crystals of pure iron were slightly larger than those of the wet-hydrogen treated pure iron. The as-grown crystals of 0.05%Ni-Fe showed larger lower

yield stresses than those of the wet-hydrogen treated pure iron by about 2 kg/mm² in the whole range of temperature. The lower yield stresses of the crystals which were quenched from 200°C were substantially same as those of as-grown crystals even at low temperature. An example can be seen on the curves of 0.05%Ni-Fe crystals in Fig. 4. The flow stress of pure iron after twinning deformation, as shown in Fig. 5,

Fig. 6. Temperature dependence of lower yield stress in wet-hydrogen treated iron single crystals extended in a range of crosshead speed of 500 to 0.005 mm/min¹²⁾

Fig. 7. Temperature dependence of lower yield stress of single crystals of pure iron and 0.05%Ni-Fe in as-grown and decarburized conditions

was essentially same in the quenched and decarburized crystals in (a) and the quenched and as-grown crystals in (b). Accordingly, it is concluded that the effect of impurities on the yield stress of iron single crystals appears only on the athermal part of the yield stress.

Upper Yield Stress

Upper yield stresses of all crystals extended at a crosshead speed of 5 mm/min were plotted in Fig. 8. The dashed line is probably the highest limit of the upper yield stresses in all crystals. Reduction of upper yield stress by decarburization or quenching from 200°C was prominent at about room temperature. Upper yield stress of decarburized crystals disappeared at above 100°C. At low temperature about 200°K, the variation of upper yield stress by heat treatment was less prominent, presumably because of large scattering of observed upper yield stresses. At 114° and 78°K, upper yield stresses or twinning stresses were large in 0.05%Ni-Fe crystals in comparison with pure iron, and also in the quenched crystals both of pure iron and 0.05%Ni-Fe crystals. Because of the scattering data, more quantitative comparison of upper yield stress in Fig. 8 could not be made at low temperatures.

3. Yield Delay Time

Delay time phenomenon for yielding in a constant-load extension test has essentially the same meaning as the upper yield phenomenon. The creep tests were made with a great care for the alignment of specimen and grips with the use of a guide pipe. Typical creep curves of as-grown and decarburized pure irons are shown in Fig. 9. Both crystals were extended under the same stress of 30 kg/mm² at 153°K. The delay

Fig. 8. Plot of upper yield stresses and twinning stresses of single crystals of pure iron and 0.05%Ni-Fe in as-grown, quenched-from-200°C, and decarburized states

time was defined by the time at elongation of 0.1%. The observed delay times were plotted in Fig. 10. In these tests the load applied to a specimen was so selected as to have a delay time of the order of 10 to 1000 sec. At 195° and 153°K, the delay times of decarburized crystals were about ten times smaller than those of as-grown crystals. At 114°K, the as-grown crystals did not show yielding after several thousand seconds under a stress of 40 kg/mm², then the crystals were extended at a stress of 46 kg/mm². At 78°K, all as-grown crystals were deformed by twinning and no yield delay time was observed. Then, only the data of decarburized crystals are shown in Fig. 10.

4. Slip Line Observation

When an as-grown crystal is extended by small

Fig. 9. Creep curves of as-grown and decarburized crystals of pure iron at 153°K under a stress of 30 kg/mm²

Fig. 10. Comparison of the time at 0.1% elongation under constant load tests between as-grown and decarburized crystals of pure iron, 0.02%Ni-Fe and 0.05%Ni-Fe

amount at slightly below room temperature, many glide bands were observed on the crystal surface. Such glide bands have been observed extensively by Takeuchi.¹³⁾ The observation of the surface structure of crystals after extension was made on the crystals which were extended under constant load in a previous section. An example of a glide band pattern which is similar to the pictures in the previous paper¹³⁾ is shown in Photo. 1. Such a glide band pattern could not be observed on a decarburized specimen, in which slip lines or any surface unevenness could not be detected on both macroscopic and microscopic observations at 195°K. A macrophotograph of an as-grown crystal extended at 153°K by 0.5% under 30 kg/mm² is shown in Photo. 2. This photograph shows that plastic deformation in the specimen was strongly localized. Such a phenomenon is similar to nucleation and growth of a Lüders band in polycrystals. The decarburized crystal extended under the same condition as the crystal shown in Photo. 2 is shown in Photo. 3. Although the surface gradually curved was seen on the crystal surface, no slip lines were detected even on the severely deformed region. Photo. 4 is another photograph of the decarburized pure iron which was ex-

Photo. 1. Macrophotograph of an as-grown single crystal of pure iron extended under a stress of 18 kg/mm² by 0.26% in 246 sec at 195°K (×8) (4/7)

Photo. 2. Macrophotograph of an as-grown single crystal of pure iron extended under a stress of 30 kg/mm² by 0.53% in 442 sec at 153°K (×8) (4/7)

Photo. 3. Macrophotograph of a decarburized single crystal of pure iron extended under a stress of 30 kg/mm² by 0.54% in 161 sec at 153°K (×8) (4/7)

Photo. 4. Macrograph of a decarburized single crystal of pure iron extended under a stress of 40 kg/mm² by 0.52% in 57 sec at 114°K ($\times 8$) ($\frac{4}{7}$)

Photo. 5. Macrograph of a decarburized crystal of 0.05% Ni-Fe extended under a stress of 62 kg/mm² by about 2% in 391 sec at 78°K ($\times 8$) ($\frac{4}{7}$)

Photo. 6. Slip lines observed on the crystal shown in Photo. 5. ($\times 70$) ($\frac{5}{8}$)

tended at 114°K by 0.5% under 40 kg/mm². The inhomogeneity of deformation in large scale was more pronounced than that in Photo. 3, and no fine structure such as slip lines was detected in the deformed region. Glide band patterns and the macroscopic pictures of the 0.05%Ni-Fe crystals were same as those of pure iron in both as-grown and decarburized conditions. Photo. 5 is a picture of 0.05%Ni-Fe crystals extended at 78°K by about 2% under a stress of 62 kg/mm². At this temperature, slip bands which cross the whole section of specimen similar to Photo. 2 were not detected. Similar pictures were also observed on decarburized pure iron extended at 78°K.

Slip planes which are determined from slip line on as-grown crystals in the pictures in Photos. 1 and 2 are nearly {112} planes. Photo. 6 is a microphotograph of the crystal shown in Photo. 5. The slip plane which was determined from the slip traces on Photo. 6 was nearly {110} planes at 78°K. Similar transition of the slip plane from {112} to {110} planes at low tem-

Fig. 11. Schematic drawing of four stress-strain curves and illustration of yield stresses

perature has been observed on 3%Si-Fe crystals by Taoka et al.¹⁴⁾

IV. Discussion

1. Yield Stress

Yield stress is a quantity which has a close relation to the dynamical properties of dislocation in crystals. There are several experimental methods by which the yield stress is defined. It is necessary, here, to clarify the meaning of these yield stresses. Typical stress-strain (or load-elongation) curves around yield point are shown schematically in Fig. 11. *A* is a stress-strain curve after a prestraining at high temperature, *B* the curve which exhibits no upper yield point, *C* the curve in which stress decreases gradually after upper yield point, and *D* the curve in which stress decreases abruptly after upper yield point. Four kinds of yield stresses have been defined on these stress-strain curves: proportional limit yield stress *P*, extrapolated yield stress *E*, upper yield stress *U*, and lower yield stress *L*.

Brown et al.^{15),16)} have observed the proportional limit yield stress with the strain sensitivity of 10^{-6} in the experiment of the type *A*, and have shown that the yield stress of high purity iron was independent of temperature. The yield stress obtained from such an experiment is very small comparing with the other yield stress defined in Fig. 11. The motion of dislocations corresponding to this type of microyield stress has been said to be the different kind of motion from the so-called gross yielding.¹⁶⁾ Wynblatt and Dorn¹⁷⁾ have discussed the temperature dependence of yield stress of 2%Mn-Fe polycrystals equivalent to the extrapolated yield stress obtained from the experiment of the type *A* in Fig. 11. This type of determination of yield stress has an advantage of eliminating inhomogeneous deformation or twinning deformation at low temperature, but has a shortage that the stress is not a real yield stress but a yield stress of crystals which were work-hardened by prestraining.

Stress-strain curves of the type *B* are seen in pure fcc metals. In case of iron single crystals, this type of stress-strain curves has been presented even at low temperature.^{4),5)} It is necessary to recognize whether the stress-strain curve of the type *B* is the true characteristics of the specimen or not. For this purpose,

it is important to recognize that a crystal which shows a stress-strain curve of the type *C* or *D* under an appropriate testing condition may exhibit a curve of the type *B* under inappropriate testing conditions. As an example, a load-elongation curve of an iron single crystal is shown in Fig. 12. The specimen had a shape of rectangular rod of $2 \times 3 \times 38 \text{ mm}^3$ without reduced section in the middle part of the specimen, and the specimen contained the same impurities and was subjected to the same heat treatment as the present wet-hydrogen treated crystals. A sharp upper yield point was expected to appear at -120°C in this specimen, but it disappeared completely in Fig. 12.

Fig. 12. A load-elongation curve of a specimen which had no reduced section in the middle part of the specimen extended at 153°K . The specimen size was $2 \times 3 \times 38 \text{ mm}^3$.

Stress-strain curve of the type *C* can be observed on a crystals which shows a rapid increase of the density of mobile dislocations at yield point. Stress-strain curve of the type *D* is an extreme case of the type *C*. In this case, the number of mobile dislocations initially present is zero and the upper yield stress is determined by the nucleation stress of glide band which appears firstly in the specimen. A typical example of this type of stress-strain curve has been observed on MgO crystals.¹⁸⁾

An important relation in the dynamical properties of dislocation is the dislocation velocity as a function of stress and temperature. Johnston's phenomenological theory¹⁹⁾ of yield point has revealed the relation between macroscopic yield behaviour and dislocation velocity and its density in various crystals. It has also been shown experimentally that this theory can be applied to crystals which have yield point of the type *C* in Fig. 11.

In the present investigation, the iron single crystals had a gradual yield point around room temperature and sharp yield point at low temperature, and emphases were placed on the upper yield stress and the lower yield stress of the $[110]$ type iron single crystals. The former has a close relation with the sources of dislocations or glide bands in crystals, and the latter has a relation with dynamical properties of dislocations in

motion.

2. Upper Yield Stress

The effect of impurities on upper yield stress is discussed together with twinning stress. The appearance of twinning deformation in iron was very sensitive to very small amount of impurities such as carbon and nickel. Twinning deformation appeared below 153°K in as-grown crystals of pure iron. Similar results have been reported in previous papers.^{10),11)} The as-grown $0.05\%\text{Ni-Fe}$ crystals, however, did not show twinning deformation down to 114°K . This result is consistent with the fact that the twinning deformation is easier in purer iron. Macroscopic pictures of glide band of as-grown crystals at low temperature as shown in Photos. 1 and 2 were same in the pure iron and $0.05\%\text{Ni-Fe}$ crystals. Then a possible explanation is that the growth processes of glide bands in which work-hardening plays an important role are different in pure iron from less pure iron containing small amount of nickel. The suppression of twinning deformation at low temperature was the most prominent in the $0.05\%\text{Ni-Fe}$ crystals quenched from 200°C . The surface observation of the $0.05\%\text{Ni-Fe}$ crystals after deformation at 78°K showed that the large glide bands, which seemed to appear at the abrupt yield drop, had fine structures within them. This observation also suggests that the growth of glide band is affected more strongly when a small amount of solute carbon and nickel atoms coexist in iron crystals.

Fig. 3 showed that upper yield stress was decreased by decarburization at room temperature. Such tendency could be observed above -35°C on load-elongation curves. The delay times for yielding under creep tests shown in Fig. 10 were shorter in decarburized crystals than in as-grown crystals. This observation has the same meaning as small upper yield stress in decarburized crystals. In the present experiments the delay time was defined by the time at 0.1% elongation. The delay time, t_d , can be expressed by^{19),20)}

$$t_d = \frac{1}{b\alpha v} \ln \frac{n_d}{n_o}$$

where b is the Burgers vector, v the dislocation velocity, α the proportional constant between dislocation density and plastic strain ($n = \alpha \epsilon$), and n_d and n_o the dislocation density at the delay time and at the start of deformation, respectively. This equation shows that the decrease of t_d by $1/10$ corresponds to the 10^4 times increase of the mobile dislocation density initially present, n_o . The disappearance of glide bands on the decarburized crystals can be explained by the increase of this dislocation density.

The fact that the transition temperature for the appearance of twinning deformation was decreased by decarburization has a close relation to the disappearance of glide bands. The nucleation of twin has been considered to be assisted by the stress concentration which appear at the tips of growing glide bands under applied stress.^{10),11)} Then the observations on the decarburized crystals are consistent with this interpretation.

3. Lower Yield Stress

The result in Fig. 7 showed that the temperature dependence of lower yield stress did not change within the range of impurities in the present investigation. The effect of impurity appeared only on the athermal component of yield stress of the order of 2 kg/mm² at most. Similar result has been reported on 2% Mn-Fe polycrystals.¹⁷⁾ The lower yield stress of crystals which contained about 1 ppm of solute carbon atoms was equal to or slightly less than that of as-grown crystals. This result indicates that lower yield stress is insensitive to a small amount of solute carbon atoms of the order of 1 ppm. The effective solute carbon atoms which were contained in as-grown crystals and decarburized crystals are considered to be less than 1 ppm. Then, the effect of solute carbon atoms on the dynamic motion of dislocations in these crystals is considered to be negligible. The fact that the lower yield stress of as-grown crystals is slightly larger (1 to 2 kg/mm²) than that of decarburized crystals must be explained as secondary effects, such as the number of glide band sources and the inhomogeneity of strain distribution around the yield point.

Solution hardening in iron single crystals by various elements such as Al, Be, Ge, Mo, Si, Ti, and V has been investigated by S. Takeuchi et al.²¹⁾ Their result has shown that solution hardening rates of the above elements in iron were in the range of 1 to 10 kg/mm²/at%. The total amount of substitutional impurities in the present iron is considered to be less than 0.05%. Then, if the average solution hardening rate of impurity atoms is assumed to be 4 kg/mm²/%, the solution hardening by impurity atoms in the present iron is less than 0.2 kg/mm², which is the same order of magnitude as the scattering of the observed lower yield stresses in the present experiment. Therefore, it is concluded that the solid line in Fig. 7 and the temperature and strain rate dependence of lower yield stress in Fig. 6 can be regarded as the yield stress of ideally pure iron single crystals, if the uncertainty of the order of 1 kg/mm² or less is allowed.

V. Summary

(1) The yield point behaviour of iron single crystals with the [110] extension axis containing small amount of carbon (about 20 ppm and less than 1 ppm) and nickel (0.05 to 0.005%) was extended in a range of temperature between 473° and 78°K.

(2) Addition of small amount of nickel of the order of 0.02 to 0.05% suppressed the appearance of twinning deformation at low temperature.

(3) Decarburization of crystals suppressed the appearance of twinning deformation especially in pure

iron, and in some case twins did not appear even at 78°K.

(4) Large glide bands which appeared in as-grown crystals at the beginning of deformation slightly below room temperature were not observed on the decarburized crystals.

(5) Upper yield stress and nucleation and growth processes of deformed regions in iron single crystals were influenced strongly by small amount of impurities and the differences in heat treatments.

(6) The effect of impurities and heat-treatments on the lower yield stress was very small, and it was observed only on the athermal component of the lower yield stress.

Acknowledgements

The writer expresses his thanks to Mr. A. Yoshikawa for the use of the furnaces for decarburization of iron single crystals by wet-hydrogen and ZrH₂-purified dry-hydrogen. His thanks are also due to Messrs. R. Honda, A. Yoshikawa, S. Takeuchi, and S. Ikeda for helpful discussion.

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