FOREWORD

This report was prepared by Manufacturing Laboratories, Inc., Cambridge Massachusetts, on Air Force Contract No. AF33(616)-6348, under Project No. 7021, "Solid State Research and Properties of Matter", Task No. 73653 "Mechanisms of Flow and Fracture of Metallic and Nonmetallic Crystalline Substances".

The efforts were accomplished under the cognizance of the Advanced Metallurgical Studies Branch of the Materials Central, Directorate of Advanced Systems Technology, Wright Air Development Division, with the technical work directed by Capt. Richard Gerhardt as Project Engineer.

This report covers work from 1 February 1960 to 31 January 1961.

Dr. Paul J. Fopiano was principal investigator of the body of this report, and Dr. S. Andrew Kulin served as supervisor on the program.

ABSTRACT

The material in this report is primarily devoted to work carried out during the period 1 February 1960 to 31 January 1961. However, it also, concerns the results of the period 1 March 1959 to 31 January 1960. An important part of the initial contract period was the development of suitable techniques and apparatus for the strain-anneal growth of iron single crystals. The factors affecting this growth, however, have been of continuing interest throughout the entire period. The metallography and etch pitting of annealed and strained single crystal specimens have been developed and shown to give consistent results. While x-ray and light micrographs of extra-large grained specimens showed qualitative agreement, the technique was not felt to be sufficiently quantitative for our purposes. The double crystal spectrometer is felt, however, to be of most interest in this program. Furthermore, the desirability of being able to follow the effect of strain on the rocking curve of one particular area of an iron single crystal has led to the construction of a tensile apparatus capable of being mounted directly on the double crystal spectrometer. The methods and results of employing this technique are discussed in considerable detail. The analysis of these results based on a dislocation model is presented.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER

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I. INTRODUCTION

The investigation of the role of dislocations in iron single crystals can be divided into two major groups: the growth of single crystals and the observations of substructure. The growth of single crystals involves the selection of material, the preliminary treatment of the material (including recrystallization and critical straining) and, finally, the gradient growth of single crystals. The factors affecting the strain-anneal growth of single crystals (rather than merely a presentation of optimum conditions) will be of primary interest in the report.

Metallographic as well as x-ray methods for the observation of substructure have been employed during the course of this investigation. Previous summary reports (Nos. 3 and 6) have dealt with the characterization of substructure of the as-grown single crystal by several techniques and the nature of the results obtainable. These results suggest that quantitative results can best be obtained by the use of double crystal spectrometry. The effect of strain on the rocking curve will be of primary interest in this report where the strain is introduced in the specimen in situ on the double crystal spectrometer. A description both of techniques and results will be discussed. In conclusion, an interpretation of the observations based on dislocation theory will be presented.

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II. GROWTH OF SINGLE CRYSTALS

Three compositions of iron have been investigated with an effort to understanding the factors involved in the strain anneal growth of single crystals. These compositions are vacuum melted iron, Armco iron, and silicon iron. The results of this work will be described below.

A. General Description

In review, the strain anneal growth of iron single crystals in general requires four basic steps:

(1) Cold work

(2) Recrystallization anneal

(3) Critical strain

(4) Crystal growth

Within the framework of prior history, composition and these four steps are a large number of variables. The results of a large number of runs in the case of vacuum melted iron has permitted a reduction of the possible variables to the following:

(1) Carbon content

(2) Prior cold work

(3) Recrystallization temperature

(4) Amount and type of critical strain

(5) Peak gradient temperature for crystal growth

The variables have been shown (Progress Reports Numbers 3 and 6) to critically affect the success of the strain anneal procedure. Other possible variables have been shown to have a less critical effect. The work reported here has been concerned with increasing the yield of single crystals as well as shedding light on the factors involved in the strain anneal growth of single crystals. The work has been also extended to include Armco iron and silicon iron.

Work on the growth of iron single crystals from vacuum melted iron has been shifted to another composition Cr-3 (Crucible Ferrovac E). The development of a generalized technique for determining the optimum recrystallization temperature and the peak gradient temperature and the addition of what is called a polygonization anneal after critical straining have been the major developments during this period. Other variables have also been more closely defined.

B. Recrystallization and Peak Gradient Temperature

Inasmuch as the temperature at which the cold worked specimens are treated is critical to the strain-anneal growth of iron single crystals, two concurrent heat treatments are proposed to facilitate the selection of both the recrystallization and peak gradient temperatures. These consist of two anneals in a stationary gradient furnace. The first such anneal consists of placing a cold worked specimen in the gradient (the peak temperature of which is well into the austenite region) for a period of one hour. It is known from previous experience that recrystallization temperatures in the range 700 to 850°C were most favorable for the strain anneal growth of single crystals. From the appearance of the specimen, a zone extending from 880°C down to 760°C is clearly different from the recrystallized zones both at higher and lower temperatures. The higher temperature is associated with a coarsening temperature while the lower temperature is not well understood except that its boundary is less sharp than the higher temperature limit. Recrystallization temperatures in the range 760-875°C proved to be most favorable for subsequent crystal growth of Cr-3.

The second stationary gradient anneal which is preceded by critical straining (Lüder's or a 4 to 5 percent homogeneous strain) consists of a grain growth anneal as opposed to the previous recrystallization anneal. The purpose of this anneal is to select the optimum peak gradient temperature. A region of exaggerated grain growth is readily observed corresponding to a range of temperatures favorable for growth. Here again, previous experience is helpful in evaluating the significance of the observed grain structure, since the observed range of temperatures corresponds to those found favorable for grain growth.

It is expected that these two gradient anneals can be applied, at least in part, to a variety of materials. For iron, such an investigation may take the form of varying the carbon content over a range from 0.005 to 0.023 pct. C. The above technique when applied to silicon iron indicated that only the second anneal produced useful results. A peak gradient temperature in the range 1050 - 1200°C is observed to yield favorable results.

C. Effect of Prior Cold Rolling

The effect of the degree of cold rolling has been investigated in order to determine if the reduction in area or the gage section controls the final result. Stock initially 3/4 and 1/2 inches in diameter was rolled to the thicknesses tabulated in Table 1. The reductions favorable for the growth of single crystals are marked with an asterisk. From these observations, about a 75% reduction in area and also a gage section of the order of 1/16 inch or less is required as initial conditions for the subsequent strain anneal growth of vacuum melted iron single crystals.

Increased prior-cold work (up to 95 percent), cross-rolling, and a sharper gradient have all tended to improve the yield of vacuum melted iron single crystals. The first two modifications are known to increase the degree of preferred orientation in iron. It is assumed, therefore, that by sharpening the texture, there exists less chance for the subsequent growth of minor textural elements.

Table 1

Effect of Prior Cold Rolling on Growth of Single Crystals

Initial Diameter	Final Gage Thickness	% Reduction in Area
3/4	.121	71.8
3/4	.090	79.4
3/4	.075	87.5
3/4	.059	85.3*
3/4	.025	93.5*
1/2	.061	75.2*
1/2	.180	91.7*

*Refer to those final gage sections favorable for crystal growth.

The result of the addition of a water jacket has been to steepen the leading edge of the gradient. Again, it is felt that an improvement in the yield of large single crystals is realized but is difficult to substantiate.

D. Effect of Carbon Content

The effect of carbon on the growth of single crystals has been investigated in vacuum-melted and Armco irons. In vacuum melted iron, the specimens were given a decarburization treatment during the recrystallization treatment (prior to critical straining) or during the subsequent polygonization treatment (after critical straining). In the former, subsequent gradient growth was far less effective in producing exaggerated grain growth than the latter. The interpretation may be related to the French investigation in which they indicate the impossibility of growing iron single crystals from vacuum-melted iron. Their explanation suggested that the substructure produced by the critical straining of such iron was too stable to permit exaggerated grain growth. They were successful in obtaining single crystals only when the crystals have been recarburized prior to the strain anneal treatments. Our interpretation, however, suggests that the removal of carbon increases the difficulties in introducing a critical strain, rather than in any inherent stability of the substructure. This point of view is supported by the fact that the removal of carbon after the critical straining assists rather than inhibits the subsequent growth of single crystals.

The techniques employed for the growth of single crystals of vacuum melted iron have been applied to the growth of single crystals of Armco iron. It was found, however, that the decarburizing treatments described above were necessary to obtain exaggerated grain growth. It was observed that, for decarburizing treatments (during polygonization anneal) which were of insufficient duration, a gradient in exaggerated growth paralleling the apparent carbon gradient was found. As yet, no extremely large (greater than one cm.) grains have been produced in Armco iron.

E. Effect of Atmosphere

The effect of atmosphere has been investigated in considerable detail. Critically strained and polygonized specimens were gradient grown in helium, argon, wet hydrogen, dry hydrogen, forming gas, and argon 15 percent hydrogen. In the first two atmospheres, considerable development of surface parasitic growth occurred. In the latter three atmospheres, insignificant surface parasites formed. The effect of wet hydrogen on parasite formation lies between these extremes. The reason for the parasite formation is associated with the presence of free oxygen which is known to have a strong effect on the surface energy of the exposed grain. That the atmosphere is not of primary importance in the growth of our relatively thick (0.045 in.) strip stock is shown by the ability to expose a single crystal by extensive chemical thinning even in cases of specimens which had developed a severe parasitic structure.

F. Polygonization Anneal

An additional heat treatment following critical straining has been introduced in the program for the strain anneal growth of vacuum melted iron single crystals.

The temperature range for this anneal has been found to be 1200-1400°F. Its addition was suggested by the work of Dunn wherein he subjected critically strained silicon iron to a low temperature anneal prior to gradient annealing at a higher temperature. It is referred to as a polygonization anneal in this investigation on the basis of the classical definition of the term. The role played by this anneal in the growth of large single crystals, however, is to produce selected growth of some grains so that they exceed some critical size.

The result of this polygonization anneal (either carburizing or decarburizing) has been to increase the probability for growth of iron single crystals. Strains of 2-3 percent in excess of the critical have yielded good single crystals when subjected to the polygonization anneal, whereas such strains without the anneal invariably produce a fine grained structure. While the polygonizing anneal is not a rigid requirement for the growth of single crystals, it is felt that its application makes the growth of single crystals more probable by making the critical strain requirement less rigid.

G. Relationship Between Some of the Variables

In vacuum melted iron, a definite correlation between the recrystallization temperature, the yield point stress, and the yield point (Lüder's) strain has been observed. The results are tabulated in Table 2. A relatively sharp break in Lüder's strain is observed to occur for recrystallization temperatures at and above 780°C. This break corresponds to the strains found to be most advantageous in the subsequent growth of single crystals.

H. The Growth of Silicon Iron Single Crystals

The growth of silicon iron single crystals has been attempted for three basic compositions, commercial 4.25 silicon iron, high purity 3 percent silicon iron, and doped (manganese and sulfur) high purity 3 percent silicon iron. All were melted in an atmosphere of helium in a molybdenum wound pot furnace. Because of the excessively large grain size obtained, it was necessary to hot forge and hot roll all stock in order to prevent severe cracking. Moderate success in the growth of single crystals was observed only in the case of the doped material.

The two stage method described by Dunn and Nonkin [5] was the starting point in the growing of large crystals. The results (only on the doped material) established that:

(1) The two stage reduction method yields in all cases extremely large grained material.

(2) Peak growing temperatures between 1030 and 1200°C are effective in obtaining exaggerated grain growth.

(3) Straining is not only unnecessary but is detrimental to the growth of large grained crystals by the two stage method.

(4) Three hours at the peak temperature is sufficient to produce the required growth.

Table 2

Effect of the Recrystallization Temperature on the Yield Point Elongation in Vacuum Melted Iron

Recrystallization Temperature (°C)	Yield Point (lbs.)	Elongation (%)
610	880	14.8
665	890	14.0
720	780	10.0
720	770	8.9
725	620	9.1
780	380	5.1
830	270	2.5
830	290	3.0
830	250	4.0
835	-	2.1

Attempts to increase the final gage section above 0.015 inch by the application of the strain anneal techniques employed in the case of vacuum melted iron have led to, in general, poor results. This is apparently related to the extremely inhomogeneous nature of the tensile straining process whereby the material is observed to yield by sections. In other words, while a sharp yield point is observed, the Lüders bands do not propagate, but, rather, different regions of the specimen yield discontinuously. It is felt that overstraining occurs in sections that first yielded and, therefore, prevented exaggerated grain growth. The addition of more carbon by employing Armco iron in place of the vacuum melted iron in the doped high purity 3 percent silicon iron did not produce any observable change in the Lüder band propagation.

III. TENSILE STRAINING OF IRON SINGLE CRYSTALS

A. Equipment

Apparatus has been constructed whereby a specimen can be strained on the diffractometer. The requirement that the same area be irradiated was originally satisfied by machining a left and right handed thread on opposite ends of a shaft. It was thereby possible to strain the sample by turning the shaft and because the threads were of equal pitch, an area (A) on the sample would increase by a small increment (dA), but the centerline would remain constant. For small strains (up to 3 pct.), the irradiated area would remain essentially constant throughout the straining period.

The design of this apparatus proved to have several weaknesses. The first was the necessity for removing the apparatus from the diffractometer in order to strain the sample, since the forces necessary to turn the shaft are large. The application of such force onto an essentially delicate instrument is not desirable. Secondly, the weight of the tension apparatus because of the threaded shaft and the necessity for rigidity tended to weight excessively the double goniometer on which the tensile jig was mounted. The possibility for disalignment was therefore always present. Finally, continual removal of the tensile assembly and double goniometer from the diffractometer for the introduction of small increments of strain is felt to complicate the experimental procedures unnecessarily and perhaps prohibitively.

An improvement of this apparatus has therefore been made in which a hydraulic system has replaced the mechanical one described above. The advantages of this are many and will be enumerated:

(1) It is possible to load and unload the specimen as many times as felt necessary without increasing the risk of misalignment. The latter, "unloading", was particularly difficult in the mechanical device because of the uncertainty of knowing when zero load was reached. The possibility of compressive bending was therefore always present.

(2) The necessity of continually removing the tensile apparatus from the diffractometer in order to increase the strain is eliminated.

(3) A reduction in the weight of the apparatus has been accomplished, and

(4) The ability to measure directly the applied load (from a pressure gage in the hydraulic system) enables one to not only conveniently determine the "no-load" condition, but, with the introduction of a strain gage on the reverse side of the specimen, to carry out a complete stress-strain curve.

Because of the small volume of hydraulic fluid, the tensile apparatus is very sensitive to drops in load. It was necessary to increase this volume because of the excessive sensitivity of the original design.

B. Experimental Procedure

A tensile specimen of the form shown in Fig. 1 is mounted on the tensile apparatus which is in the position of the second crystal in the double crystal spectrometer. Rocking curves before and after each increment of plastic strain are taken. Exposures of the diffracted beam are taken at all relative maxima in the rocking curve. In general, an effort is made to search the sample so that a single maxima is obtained.

The ability of the hydraulic straining apparatus to maintain the same area of the tensile specimen in the x-ray beam has been demonstrated several times. The form of the observed rocking curve remained identifiable throughout the entire initial stages of deformation. Only after severe straining is another rocking curve obtained, indicating some lattice rotation within the specimen area.

All specimens which were deformed during this report period were vacuum melted iron single crystals with (211) face normals and [110] tensile direction. Elastic and plastic strains have been considered with emphasis, of course, on the plastic deformation. All specimens have been deformed in place as the second crystal of the double crystal spectrometer.

The introduction of plastic strain is carried out by a load-unload procedure. The stress is increased in steps of 1 to 2 ksi and is measured indirectly by the measurement of pressure within the hydraulic system. The strain is measured by S R-4 gages which have been mounted on the reverse side of the strip specimens. The strain for each stress-level appears to be sigmoidal in time which means that the strain rate at each stress level passes through a maximum value. Except in the early stages of plastic deformation, equilibrium strain conditions are impractical to attain because of the long times involved. Quasi-equilibrium has been assumed to be reached when the strain rate at each stress level approaches some low value. The lower yield range is found to be very sensitive to the value chosen for the final rate. The lower yield increases for lower final strain rates. The magnitude of the strain is also higher for the lower strain rate. Figs. 2 and 3 show the effect of this final strain rate on the lower yield range as well as the magnitude of the plastic strain for low and high final strain rates respectively.

C. Experimental Results

1. Elastic Strains

From the elastic constants and suitable orthorgonal transformations of axes, the strain conditions for two single crystal configurations have been calculated. For the (100) [110] condition with the applied stress along the [110] axis, we obtain

$$\sigma_{[011]} = 51.2 \times 10^{6} \epsilon_{[100]}$$

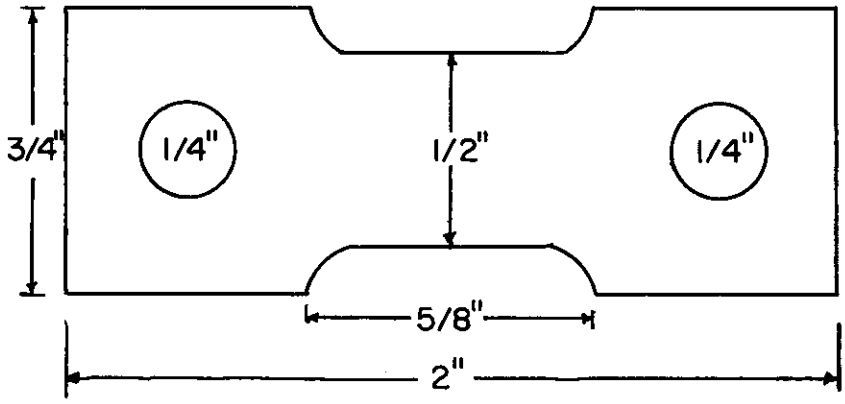

FIG. 1 TENSILE SPECIMEN

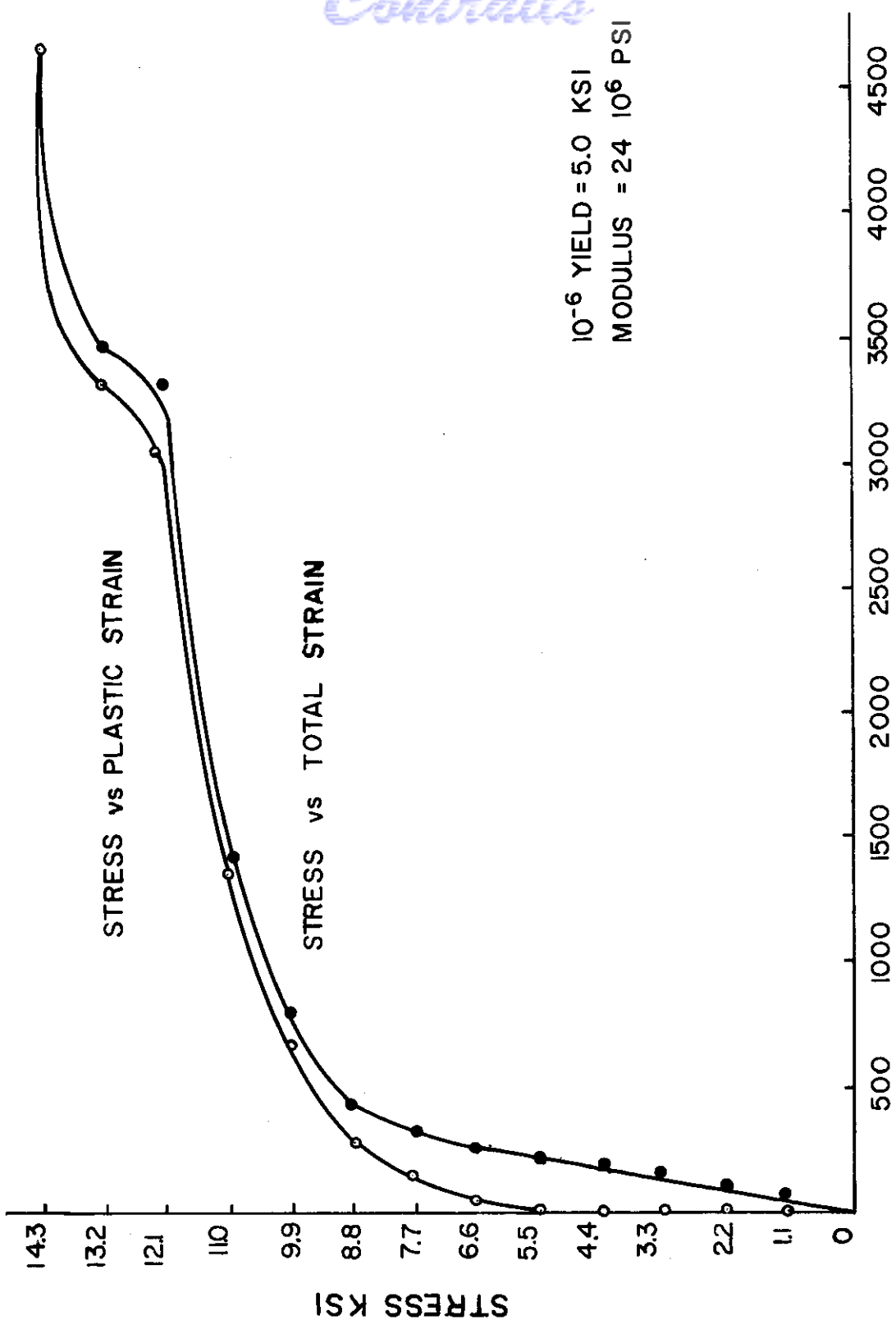

FIG. 2 STRESS STRAIN CURVE

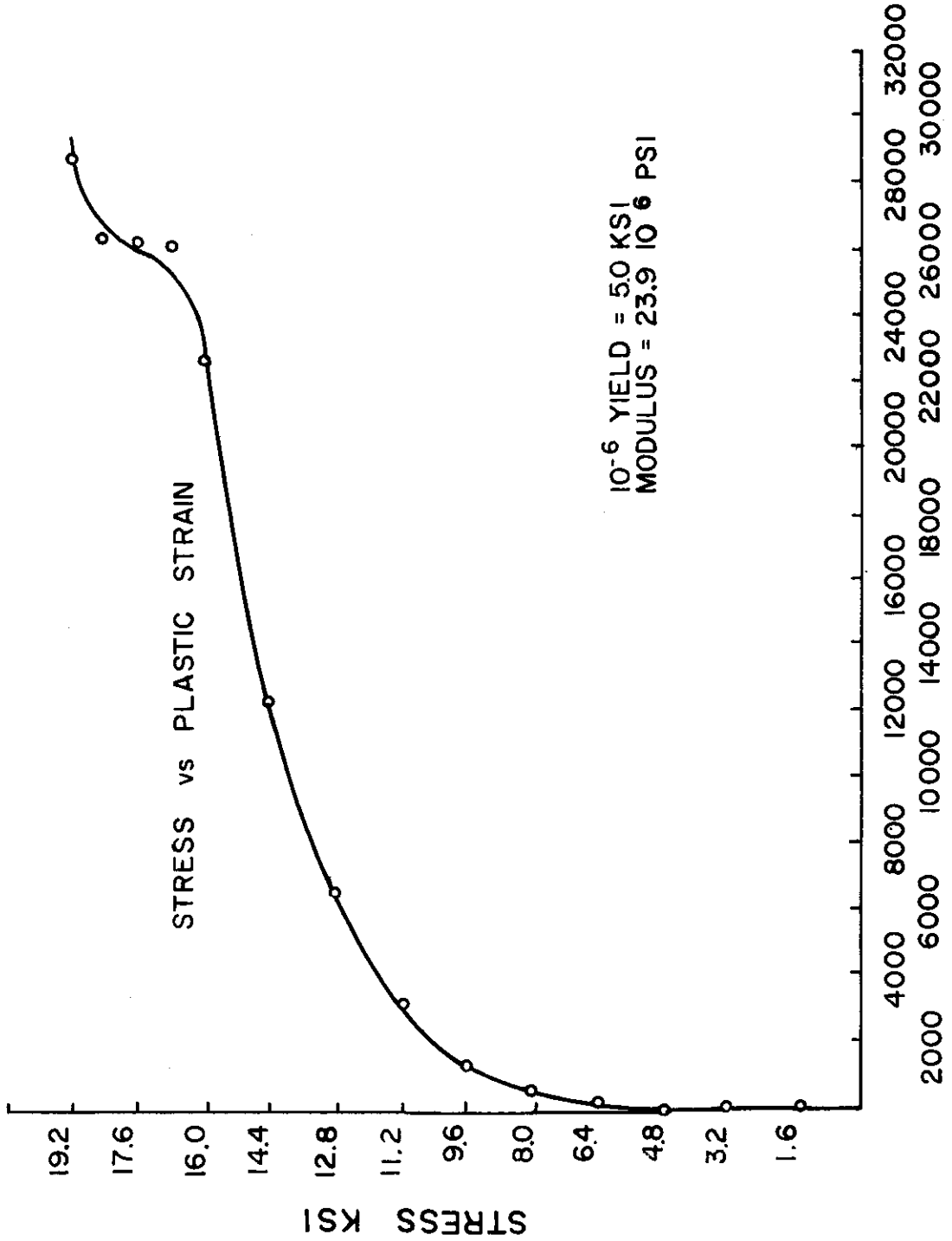

FIG. 3 STRESS STRAIN CURVE

and for the (211) [110] condition with the stress also applied along the [110] axis, we obtain

$$\sigma_{[011]} = 80 \times 10^{6} \epsilon_{[211]}$$

Shifts in the rocking curve corresponding to the latter relation have been qualitatively observed. No measurable effect of elastic strains on the shape of the rocking curve has been observed.

In the annealed sample, rocking curve half-breadths of the order of 60 seconds have been observed. The curves persist essentially unchanged to a stress of the order of 10,000 - 12,000 psi when the first observation of plastic flow is made.

2. Plastic Strains

The effect of plastic straining has been observed by pictures of the diffracted beam, rocking curves, and electron microscopic observation of prepolished specimens.

a. Qualitative Description

The initial application of stress sufficient to produce plastic flow causes a rotation of the sub-lattice and a sharpening of both the rocking curve and the diffracted beam. The continued application of stress produces large increases in strain for little increase in the stress level. General broadening of the rocking curve is observed. A constant work hardening coefficient is found during both these initial stages of deformation. The amount of strain at a given stress level is dependent on the mode of deformation.

b. Results

Three general observations which have been found to be independent of straining conditions are the elastic modulus, the 10^{-6} yield point, and the work hardening coefficient. The values of these are tabulated in Table 3 for several selected specimens. The selection was based solely on the unstrained rocking curve half breadth which was in the range of 60 to 120 seconds of arc.

Table 3

Tabulation of the Constants of the Stress-Strain Relationship Obtained by the Load-Unload Method on the Double Crystal Spectrometer

Specimen	Elastic Modulus (10^6 psi)	10^{-6} Yield (psi)	Work Hardening Coefficient
3	24.0	5500	.154
4	27.2	4293	-
5	23.9	4884	.185
7	24.7	5022	.099
8	14.0	5404	.171
10	27.5	6380	-
11	20.8	4176	.149
12	24.5	4735	-
13	24.7	-	.235
14	14.6	4875	.240

IV. RESULTS AND DISCUSSION

The results discussed below will be concerned with specimens having initial half-breadths between 60-90 seconds of arcs for the (211) diffraction plane. For every tensile specimen satisfying this criteria one or more others becomes in some way unsuitable. The amount of data obtainable from each suitable specimen, however, is large.

A. Constants of the Tensile Data

It has been observed that certain features of the stress-strain curve produced by the load-unload procedure are reproducible. The elastic modulus, the 10^{-6} yield, and the work hardening coefficient are all constants for the material. The modulus is of interest because of the ability to correlate the stress-strain data obtained by our techniques with the modulus calculated from elastic constant data for pure iron. This correlation is felt to be an important check on the tensile apparatus developed for this investigation.

The constancy of the work hardening coefficient is felt to be of more fundamental importance. While it is realized that this coefficient is a rather insensitive measure, its constancy does imply that a single mode of strengthening is operative during the initial stage of deformation irrespective of the apparent discrepancy in the stress-strain curves as shown in Figs. 2 and 3. Due to the insensitivity of the observation, however, it is clear that such an observation would not preclude the existence of other deformation mechanisms, but it does limit the possible deformation mechanisms available.

As employed in this discussion the 10^{-6} yield is a measure of the elastic limit of the material. The elastic limit is therefore defined as the stress associated with the initial observation of permanent deformation as measured by strain gages. The precision is necessarily low because of the rather large increments in stress employed. It is not fundamental to the observation that this "elastic limit" is accurately known since this data is available in the literature. We are using these constants as a measure of the merit of the straining apparatus.

B. Pictures of the Diffracted Beam

From taking pictures of the diffracted beam, it is possible to make a definite evaluation about the nature of the particular rocking curve under observation. In particular, it is possible to decide whether or not the subgrain that one is diffracting from is essentially contiguous. The deformation of this subgrain and the observed changes in the rocking curve are, therefore, directly related to this one subgrain. A confirmation of the constancy of the irradiated area in deformed specimens is two-fold. These are related to both the shape of the observed rocking curve and in a very obvious way the shape of the diffracted beam taken at the peak of the rocking curve. From the shape of the rocking curves alone, the constancy of irradiated area can be determined. Because of this fact, confidence in the reported data is high.

On the other end of the spectrum, for higher values of strain, approximately 20 percent, pictures of the diffracted beam indicate the formation of a cellular

structure. The nature of the picture is analogous to that developed by annealing a specimen which had been strained of the order of 3 percent. The development of a cellular structure has been reported in thin films of iron strained in the electron microscope. The formation of the cellular structure is identified in this case with a decrease in the dislocation density in subgrain areas with the application of plastic strains of the order of 20 percent.

C. Observations of Changes in Rocking Curve as a Result of Plastic Deformation

The changes in the half-breadth of the rocking curve, as a function of plastic strain, can be followed with considerable confidence to strains between 0.2 to 0.5 percent and can be estimated in some cases to somewhat higher strain values. A correlation between the half-breadth and the area under peak divided by the peak height has indicated essential agreement between these two methods of evaluating the changes in the rocking curve with strain. The results of such a comparison are given in Fig. 4. One of the other features of this plot is the initial decrease in half-breadth with plastic strain. The initial plastic straining is therefore accompanied by a decrease in dislocation density within the subgrain area. This sharpening is therefore associated with the annihilation or motion to subgrain boundaries of already existing dislocations. It is of interest here to observe that the number of dislocations which are available to take part in this process is small compared to the number produced by the multiplication mechanisms. In particular, the dislocation density of the as-grown material is increased an order of magnitude by the application of about 1 percent plastic strain, whereas a conservative estimate indicates that the active number of dislocations in as-grown material is less than one-tenth of the total. We have therefore a large change in the number of active dislocations as a result of relatively small amounts of strain.

D. Correlation of Dislocation Densities of As-Grown Crystals Obtained from Etch Pitting Techniques and Rocking Curve Analyses.

Without ascribing to all the assumptions inherent in the simplified theory, it may be of interest to correlate dislocation densities for an annealed crystal as determined by etch pitting and by x-ray rocking curve techniques. The rocking curve half-breadth is given by the expression:

$$\beta = 4b^2 N_D \quad (1)$$

where β = Breadth of rocking curve at 1/2 peak intensity

b = Burger's vector

N_D = Dislocation-density/cm^2

For the range of half-breadths considered in the present investigation, 60-90 seconds of arc, we obtain dislocation densities in the range of 3.5 - 8.2 10^6 dislocations/cm^2. This corresponds well to etch pit observations of 6-9 10^6 dislocations/cm^2.

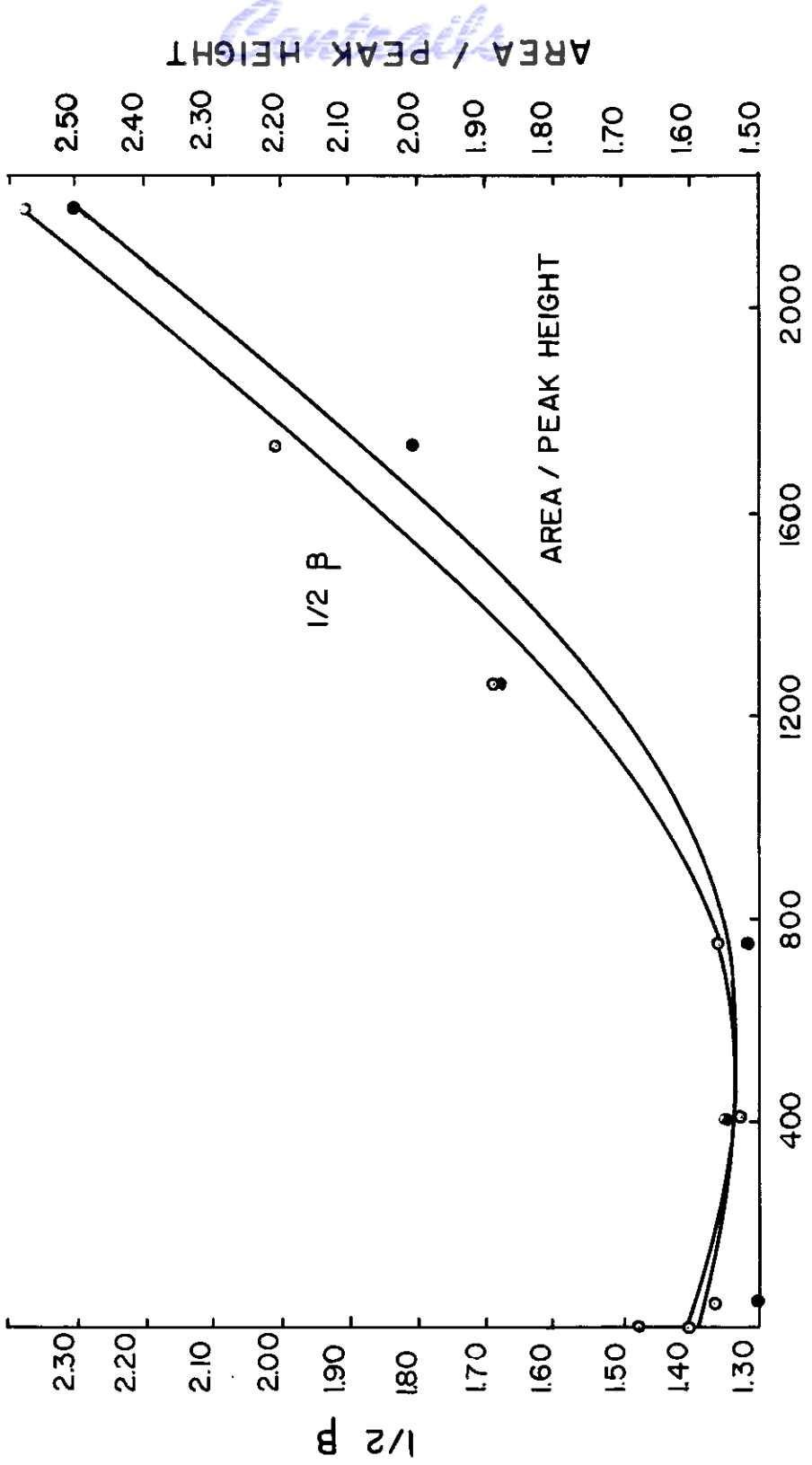

FIG. 4 ROCKING CURVE BROADENING vs PLASTIC STRAIN

E. The Effect of Strain on the Number of Active Dislocations

We have initially n_o active dislocations. Further if we maintain a constant strain rate we can expect some multiplication of dislocations dependent on the instantaneous number of dislocations. We can also expect that a certain number of active dislocations will in some way become inactive. The frequency of this latter process would be expected to depend more strongly on the number of dislocations. Johnston and Gilman[2] give the following relation:

$$\frac{dn}{dt} = \alpha n - \beta n^2$$

to express the situation as to total number of dislocations both active and inactive. If on the other hand one is concerned with only the active dislocations at any given time, it is clear that the total number must pass through a maximum. The nature of a plot of n vs t should therefore have the form in Fig. 5.

In a qualitative way, therefore, the data in Fig. 5 can be interpretated on the basis of Stein and Low's[3] dislocation velocity data to yield a fairly representative stress-strain relationship for iron including the yield point effect.

This program is attempting to supply information on the regions of plastic strain as defined by A and C in Fig. 5.

F. Synthesis of a Stress-Strain Relationship Based on the Dislocation Velocity Model

If we define n as the number of active dislocations within the subgrain areas, the production of dislocations obeys the relationship:

$$n' = 10^{11} \epsilon \quad (4)$$

where ϵ = plastic strain

and n' = total number of dislocations produced by multiplication processes.

We can relate n' to n for low values of strain, and from the work of Johnston and Gilman[2] note that

$$\dot{\epsilon} = b\, n\, v$$

where $\dot{\epsilon}$ = Strain rate

b = Burger's vector

v = Dislocation velocity.

According to Stein and Low[3] the dislocation velocity is related to the shear stress in silicon iron at 298°K by the relation:

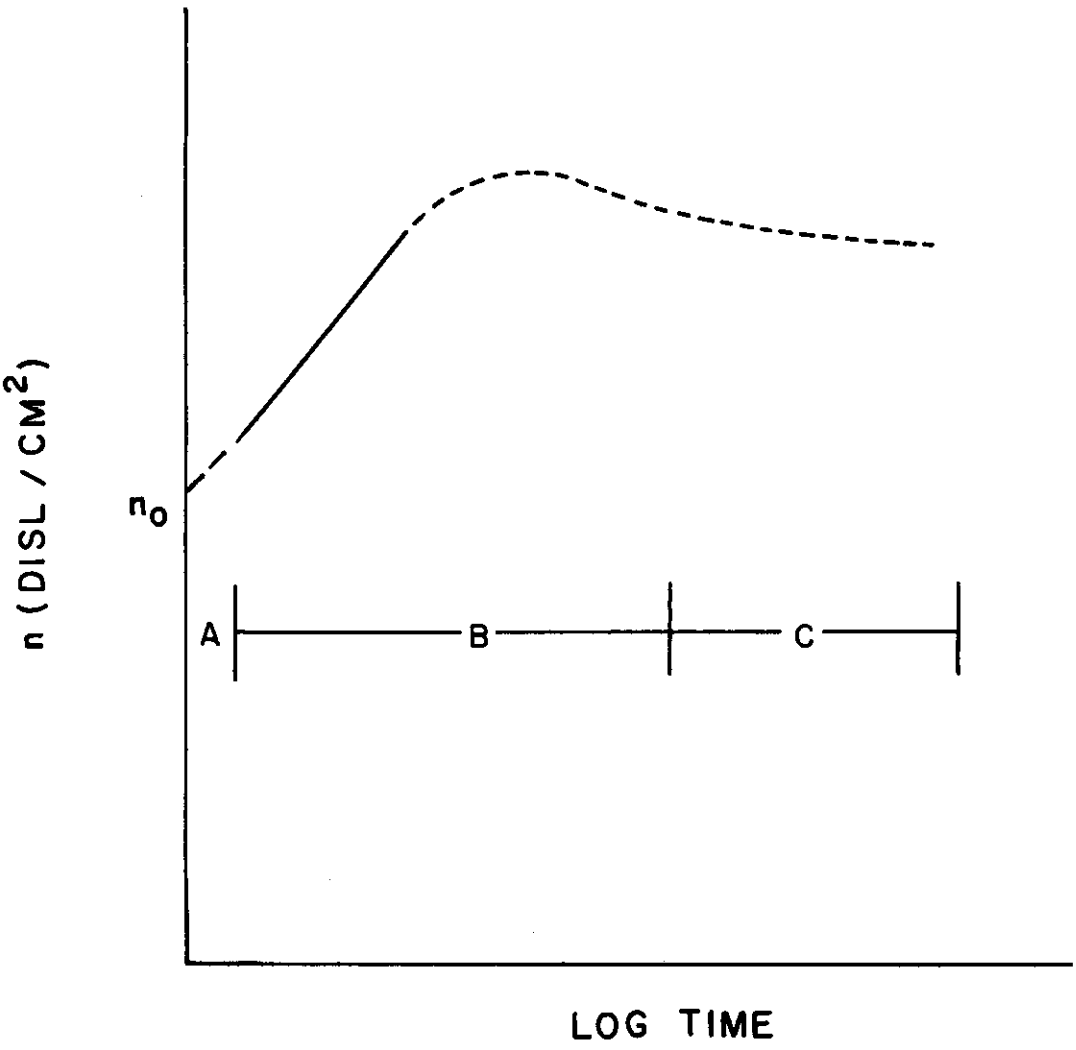

FIG. 5 - SCHEMATIC REPRESENTATION OF THE NUMBER OF DISLOCATIONS AS A FUNCTION OF TIME FOR A CONSTANT RATE OF STRAIN.

$$v = (\tau/210^9)^{35}$$

For a constant strain rate, an increase in the number of active dislocations is compensated for by a decrease in the velocity of these dislocations. This in turn is reflected by a decrease in stress. If we relate the various strains from 10^{-5} - 10^{-1} to the calculated stress in the equation

$$\epsilon = 10^{11}\, b\, \epsilon\, (\tau/210^9)^{35} \quad (\text{Const } \epsilon)$$

and if we define τ_o as the elastic limit, we are able to calculate the ratio τ/τ_o vs. plastic strain. The data is tabulated in Table 4.

As indicated in the previous section the number of active dislocations should pass through a maximum with strain. The decrease in active dislocations for higher strains is related to the annihilation and intersection of dislocation due to the increased number of dislocations. It is not difficult, in a qualitative way to predict the shape of the stress-strain curve for the deformation process. In order to indicate the required increase in flow stress for higher strain values it is only necessary to postulate some decrease in the number of active dislocations by an annihilation process.

To extend the above to a more quantitative description would require more detailed work. In particular, the shape of the dislocation density versus strain must be evaluated to a higher order accuracy than the linear relationship employed above. In addition, an evaluation of the velocity vs. flow-stress relationship must be made for higher and lower strain values. While the latter is dependent on further work becoming available in the literature, it is felt that some quantitative extrapolation based on observations made in this investigation will be able to supplement this data.

Table 4

Calculated Stress-Strain Relationship

Strain	τ/τ_o
10^{-5}	.99
10^{-4}	.98
10^{-3}	.925
10^{-2}	.87
10^{-1}	.815

V. SUMMARY AND CONCLUSIONS

The results of a large number of runs in the strain anneal growth of single crystals of vacuum melted iron has indicated that a number of factors are of critical importance. These factors and the interrelationship between some of them have been discussed. For the consistent growth of single crystals of vacuum melted iron, the conditions as tabulated in Table 5 should be approximated. The carbon content, cold reduction, gage, annealing (recrystallization) temperature, percent strain, polygonization temperature, and peak gradient temperature are the critical factors in the growth of single crystals of vacuum melted iron. Other factors in Table 5 are not critical within the limits investigated.

Two concurrent gradient anneals are proposed to facilitate the selection of the recrystallization and peak gradient temperatures respectively. The applicability of at least the latter to silicon iron has given useful results as to the peak gradient temperature. Similar application to other metals would presumably give useful information.

The introduction of the polygonization anneal following critical straining has increased the probability of obtaining a single crystal by gradient growing. While the nature of this anneal is not definitely understood, it is felt that a form of subgrain growth to some critical size is involved. These subgrains then grow when passed through the gradient furnace.

Attempts to grow single crystals of silicon iron have met with only limited success. The two stage reduction method of Dunn and Nonkin[5], on doped silicon iron, has produced large grained crystals only in 0.015 in. thick material. The doping is accomplished by the addition of small quantities of manganese and sulfur to the melt. Carbon does not seem to be critical to the growth process.

Apparatus has been constructed whereby a tensile specimen can be strained in place as the second crystal of a double crystal spectrometer. It has been confirmed that a single area of the specimen is maintained in the x-ray beam throughout the straining operation.

The excellent correlation of the elastic modulus with that calculated from the elastic constants of iron indicates that the tensile data is valid.

The yield strength and yield elongation are shown to be a function of the mode of deformation (Fig. 2 and 3). The work hardening coefficients calculated for these markedly different stress-strain curves are essentially the same. The interpretation associated with this observation is that work hardening is not a direct function of the dislocation density.

The sharpening of the rocking curve during the initial stage of deformation and the formation of a cellular structure for strains approaching about 15-20 percent are the major results of this phase of the investigation.

Table 5

Data Tabulated for Each Run in the Strain Anneal Growth of Single Crystals

Sample Designation	Cr-3
Number	207
Gage	.044
Cold Reduction (pct.)	89.4
Annealing	
Temperature (°G)	825
Time (hr.)	1/2
Atmosphere	Argon + Hydrogen
Method of Cooling	Air
Straining	
Percent	1.9
Luders	Yes
Yield Point (lbs.)	460
Fracture Load (lbs.)	540
Polygonizing	
Time (hr.)	1/2
Temperature (°G)	750
Atmosphere	Wet Hydrogen
Method of Cooling	Air
Growing	
Gradient	Moderate
Peak Temperature	870°C
Speed (cm/hr)	1.7
Atmosphere	Argon + Hydrogen
Remarks	Bi-crystal

An interpretation based on the dislocation velocity concept developed by Johnston and Gilman in LiF and extended to silicon iron by Stein and Low gives a good qualitative explanation of the two major observations. In particular, the sharpening of the rocking curve is associated with a decrease in dislocation density which according to these concepts is capable of producing a sharp yield point effect for a favorably selected strain rate. The development of a cellular structure for higher values of strain is also associated with a decrease in dislocation density within the subgrains and therefore, according to the same concepts would produce an increase in the stress level. The intermediate ranges including the lower yield range depend on the rapid multiplication of dislocations. Depending on the rate of strain, this multiplication of dislocations would initially decrease the stress level; but, eventually by various annihilation processes, would so lower the number of active dislocation as to cause the stress level to rise.

VI. FUTURE WORK

A more quantitative evaluation of the stress-strain relationship will be of primary importance during the next contract period. Within the framework of techniques developed during the course of this investigation, attempts will be mainly directed toward refining the techniques and of being even more selective in the choice of tensile specimens having "suitable" rocking curve half-breadths. In particular, efforts will be made to decrease the initial dislocation densities below the $6\text{-}9\ 10^6$ disl/cm^2 thus far attained.

Because of the importance of having a quantitative evaluation of the relationship of plastic strain to the dislocation density, it is proposed to strain well-annealed specimens in increments of 0.1, 0.2, 0.5, 1.0, and 2.0 percent plastic strain. The determination of the resulting dislocation densities will be carried out by etch pitting techniques.

It will be of interest to correlate the results of the analysis presented in this report to the observed stress-strain relationship taken on a hard tensile machine and at a constant rate of strain. It will be necessary to grow single crystals of circular cross section to best carry out such an investigation.

The growth of silicon-iron single crystals will be of particular importance. Subsequent deformation of these crystals will follow much the same procedure as employed with iron crystals.

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