DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

DISCLAIMER

This book was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

TECHNICAL PROGRESS REPORT

Strain Hardening and Ductility of Iron:

Axisymmetric vs. Plane Strain Elongation

Submitted to:

U. S. Department of Energy
Materials Science Program
Division of Basic Energy Sciences
Washington, DC 20545

Submitted by:

George Langford
George Langford, Sc.D.
Drexel University
Department of Materials Engineering
Philadelphia, PA 19104

May 1980

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product or process disclosed or represents that its use would not infringe privately owned rights.

Approved:

S. Herbert Raynes
S. Herbert Raynes

Vice President for Academic Affairs

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED *gb*

TABLE OF CONTENTS

	Page
Summary	
I. Introduction	1
A. Main Concepts	1
B. Scope	2
C. Main Accomplishments	3
II. Progress	5
A. Experimental	5
1. Mechanical Properties of Plane Strain Elongated Iron	5
a. Mechanical Characterization of Monotonically Deformed Strip	6
b. Obtaining True Flow Stress from Mean Compressive Pressure in the Plane Strain Compression Test	10
c. Flow Properties of Strip in Reverse Deformation	16
d. Results of Flow Characterization of Plane Strain Elongated Fe-0.17 w/o Ti; Comparison with Axisymmetrically Elongated Wire	20
2. Characterization of Microstructure	24
a. Wire (Axisymmetric Elongation)	31
b. Strip (Plane-Strain Elongation)	33
c. Chip (Pure Shear)	37
3. Progress Expected in the Remainder of the Current Contract Term	39
a. Analysis of High-Voltage Electron Microscope Plates	39
b. Further High-Voltage Electron Microscopy	39
c. Mechanical Testing of Wire, Wire Drawn Strip and Strip Drawn Wire	39
B. Theoretical Progress	40
1. Stability of Microstructure and Mechanical Properties of Heavily Deformed Iron	40
2. Strengthening Mechanisms	42
C. References	46
D. Time Devoted to Project by Principal Investigator	47
E. Financial Statement of Estimated Costs	48

TABLE OF CONTENTS (Continued)

III. Appendices

- A. G. Langford and A. Razavi: Comparison of STEM and HVEM Microdiffraction as a Tool for Accurate Orientation Determination in Submission Cellular Substructures of Heavily Deformed Iron. Informal report submitted to Dr. A. Taylor, Director of the ANL-HVEM Facility.
- B. D. Apelian and G. Langford: Annual Progress Report for DARPA project DAAG 29-77-C-0028, "Rapid Cycle Steel Casting".

G. Langford - Strain Hardening and Ductility of Iron: Axisymmetric vs. Plane Strain Elongation

SUMMARY

The strain hardening of iron at high strains in plane strain elongation (strip drawing) is shown to fall increasingly below that of drawn iron wires at true strains above 2, going through zero strain hardening at $\epsilon = 3.0$, and becoming strongly negative thereafter, at least up to $\epsilon = 4.1$. High resolution selected area electron diffraction (using the Argonne National Laboratory's High Voltage Electron Microscope) has been used to map the size, shape and crystallographic orientations of a group of about 50 contiguous cells centered around an incipient shear band in an edge section of a strip drawn to $\epsilon = 1.8$. This map shows no textural instability or change in orientation associated with the shear band, although it does show substantial misorientations throughout the group of cells, the axis of these misorientations lying in the same direction as the trace of the shear band. No other large misorientations (i.e. 5° or so) are present. The only instability seen in this shear band is microstructural: the slip distances in the two primary slip directions are quite anisotropic, being much longer along the trace of the shear band. Note that this shear band has appeared during the linear hardening portion of the high-strain strain hardening curve of the drawn strip. It is therefore considered a direct weakening mechanism rather than a result of another weakening effect. Further such orientation maps have been or are being prepared for more highly deformed strip, and for wire and pure-shear specimens as well.

Characterization of the flow properties of our Fe-0.17 w/o Ti has been or will be completed for monotonic plane strain elongation (as described above), monotonic axisymmetric elongation, and completely reversed deformation in the

above plane strain and axisymmetric cases. We have already found that completely reversed deformation in plane strain gives a considerably reduced flow stress but no mechanical instability, whereas it has been shown previously that reversed deformation of drawn iron wires gives initially high flow stresses followed by drastic flow instability. We also expect to complete the processing of material with less drastic changes in mode of deformation of strip drawn wire and wire drawn strip.

I. Introduction

A. There are three main concepts on which the present work on strain hardening and ductility of iron is based:

1. At high strains strain hardening is due to the accumulation of excess dislocations of one sign, which is physically equivalent to lattice curvature. Strain hardening vanishes when the iron crystals no longer have to bend or curl to accommodate each other, and is persistent when lattice bending is a necessary part of the deformation process, as in the well-known curly grain structure of drawn body centered cubic and hexagonal close packed wires.
2. The symmetry of the combination of slip systems active for a particular deformation process (wire drawing, rolling or strip drawing, free compression, pure shear, etc.) controls the rate of generation of lattice curvature. Body centered cubic and hexagonal close packed wire have two fold symmetry of their slip systems and form ribbon shaped grains which must interfold (curl) to maintain grain boundary continuity. The $\langle 111 \rangle$ component of face centered cubic wire has three fold symmetry leading to the formation of triangular grains; the $\langle 111 \rangle$ component of freely compressed bcc metals would be expected to form triangular grains analogously to $\langle 110 \rangle$ textured fcc compression specimens which form curly grains like $\langle 110 \rangle$ textured bcc wire. However, the curvature developed by the shape-change interactions between grains in wires is accentuated by the concurrent reduction in cross sectional dimensions (hence the linear strain hardening of bcc wire) whereas the curvature developed similarly in compression specimens would

be reduced by the ongoing increase in cross-sectional dimensions.

3. The symmetry of the shape change during deformation affects the microscopic homogeneity of the deformation; non-uniform strains (shear bands) consequently limit the residual macroscopic ductility of heavily deformed metals. Axisymmetric elongation appears to have the least tendency to form shear bands, plane strain elongation may rank second best, while pure shear may be the most prone. Axisymmetric (free) compression is an unranked unknown in this respect. Direct comparisons are difficult because the hydrostatic component of the stress state involved in each deformation mode is quite different.

B. The scope of the present work is to test these theses by comparing the strain hardening, ductility, and microstructure of iron in plane strain vs. axisymmetric elongation of wire, sheet, and tubes, and to apply the results to the manufacture of tubes. The deformation texture of tubing has long been known to be sensitive to the mode of deformation used to reach the final dimensions; it can be a wire texture if the tube diameter and wall thickness have both been reduced in the same proportion, and it can be a sheet texture if either the wall thickness or tube diameter is kept constant during the prior elongation. It is now clear that the microstructure and mechanical properties of such tubes are also going to be sensitive to the mode of deformation: iron tubing with a wire texture will have a curly grain structure with persistent and substantial strain hardening, but the strength will be susceptible to catastrophic flow softening and localized deformation if the tube is loaded and deformed more than a few percent in uniaxial compression; iron tubing with a sheet texture will have ribbon-shaped grains without curling or persistent strain hardening, with great

mechanical stability against catastrophic deformation in uniaxial compression, but the microstructure will be filled with many micro- and possibly macroscopic shear bands which could be the loci of ductile failure in monotonic (tensile) loading. What is not clear is whether there is a compromise microstructure/texture/mechanical condition when the tubing is made by some intermediate deformation mode. This is what is being evaluated in the current contract period.

C. The following report summarizes the experimental progress of the past year within this project. The main accomplishments are:

1. The plane strain flow stress of strip drawn iron has been evaluated at strains up to $\epsilon = \ln t_0/t = 4.0$. This extension of the results reported previously for this project reveals that the flow stress goes through a maximum at $\epsilon = 3.0$ and thereafter decreases at an increasing rate with increasing strain. This flow softening phenomenon during monotonic deformation of a pure metal below $T/T_m = 0.5$ has been observed before only in pure shear, to which it appears to be related.
2. The plane strain compression test used to obtain the above results for strip drawn iron has been fully characterized so that the actual flow stress of the specimen can be obtained, free of any frictional exaggeration, by simple, paired compression tests on geometrically related specimens. This test enables exact true flow stress - true strain data to be obtained from specimens as small as 0.6 mm (gage length) by 5 mm (width) by 0.2 mm (thickness).

3. High resolution selected area diffraction has been used in the Argonne National Laboratory's High Voltage Electron Microscope to obtain maps showing the size, shape and complete orientation in formation (within ± 0.2 to 0.5°) for 50 to 100 contiguous cells in the cellular dislocation substructures of drawn wire, drawn strip, and an orthogonally cut chip of Fe-0.17 w/o Ti.
4. The orientation map for an edge section of drawn strip at $\epsilon = 1.8$ (before the end of linear hardening) reveals an incipient shear band in which there is no sign of any orientation change within the band, but in which there is a clear anisotropy of slip distance between cell walls. This supports the results of previous workers who found a similar situation in shear bands in fcc metals. It also leads one to the speculation that shear bands are a cause of mechanical instability, not a result of it.
5. An explanation based on dislocation interaction has been developed to explain the strong propensity for curly-grain formation in drawn bcc wires instead of axisymmetric deformation of each grain. It is shown that axisymmetric deformation of each grain would lead to even more vigorous strain hardening due to the formation of sessile $a[001]$ dislocations which are unlikely to form during the curly-grain deformation mode.

II. Progress

A. Experimental

1. Mechanical Properties of Plane Strain Elongated Iron

We have completed the plane strain elongation of our Fe-0.17 w/o Ti to a total natural strain of $\epsilon = \ln t_0/t = 4.1$. Most of this strain was accomplished by pulling the strip of 0.50 in. (12.7 mm) original thickness through an adjustable carbide strip drawing die with side constraint and with an included angle of 10° . The deformation zone geometry was chosen to give a minimum amount of redundant deformation according to the work of Green and Hill (1); 16% reduction in thickness per pass and 30% reduction per pass were used for the two different sets of specimens. These are hereinafter referred to as $\Delta = 1.0$ (16% reduction per pass) or $\Delta = 0.5$ (30% reduction per pass). These geometries give slip-line fields consisting of one or two pairs of (characteristic) slip lines crossing each other at 90° . The strip was finally rolled in a four high mill in two passes from $\epsilon = 3.7$ to $\epsilon = 4.1$, finishing at 0.008 in (0.20 mm) thickness. The width was 0.500 in (12.7 mm) for all the drawn strip, and only spread to 0.507 in (12.9 mm) during rolling. The deformation zone geometry during rolling was equivalent to $\Delta = 0.29$ (the roll diameter was 1.50 in (38 mm)). In all cases a proprietary MoS_2 lubricant was applied to the strip by spraying before each deformation pass. We were forced to use the rolling mill for the final reductions because of the difficulty of accurately indenting the very thin strip before insertion into the strip drawing die; too much indentation led to breakage because of the reduced load-bearing area; too little indentation gave incorrect deformation zone geometry. We stopped at $\epsilon = 4.1$ only because to go farther would have required progressively tinier and less precise tooling for the plane-strain compression test used to evaluate the flow stress of the deformed strip. More about this later.

a. Mechanical Characterization of Monotonically Deformed Strip

We have used a plane strain compression test to measure the flow properties of the strip; the active portion of the compression test specimen was arranged so that the elongation of the specimen was in the same sense and direction as that of the strip, and so that no strain occurred in the long transverse direction. This constraint was accomplished by the inactive portion of the specimen and, in some cases, by direct action of the tooling. The deformation modes are different only in that strip drawing is a steady state process in which the same deformation zone geometry is propagated through the specimen sequentially, whereas in plane strain compression the deformation zone changes shape throughout the test so that progressively greater deformation is applied to the same test section. The stress states are also different, of course, there being no tensile stress anywhere in the test section during a plane strain compression test whereas there is a tensile stress at the die exit during strip drawing. Therefore the mean hydrostatic stress is somewhat less compressive in the latter case. Ductile fracture did not intervene in either the strip drawing or in the plane strain compression tests, although some shear cracks started to form from the corners of the specimen edges during plane strain compression tests carried to strains beyond unity.

Strains were measured with a special gage designed by the Principal Investigator for tests done with the plane strain compression subpress in this and previous work (2). This gage used a linear variable differential transformer as the transducer and follows the general arrangement of a Tinius Olsen extensometer. The smaller subpresses generated much lower forces than could be measured with our Tinius Olsen Universal testing machine, so we had to use an Instron to apply these smaller loads. This necessitated adaptation of an Instron (Model S1-11) strain-gage extensometer as shown in Fig. 1, because of

Figure 1. Subpress and extensometer used for plane strain compression tests, shown as set up in Instron.

the incompatibility of the Tinius Olsen extensometer and the Instron electronics. A great deal of time was spent in arriving at the seemingly simple method of offering the Instron extensometer to the specimen - the extensometer is extremely compliant and gives erroneous signals when the forces used to support it change during the test. The Tinius Olsen-type extensometer is about two orders of magnitude less sensitive to the support forces at the same strain sensitivity.

The plane strain compression test used here has a constant load bearing area and constant gage length, so that the load-elongation curve is also a true stress-true strain curve. Even though this is a great simplification, there are two major complications which must be considered in interpreting the results: (1) the inactive portion of the specimen (that which lies outside the gage length) causes substantial rounding of the stress-strain curve up to a strain of about 15%, probably because it takes this much deformation to develop fully the deformation zone; (2) there is a significant friction hill, not only over the load bearing area of the specimen, but also between the edges of the specimen and the side constraints (when these are used).

The first of these complications has been handled in the present work by using the 20% offset flow stress to characterize the strain-hardened condition of the specimen; we have simply ignored the first 20% of the plane strain compression flow curve, although we do include the 20% strain in the total strain reported from the tests. In other words, the extreme rounding of the initial portion of the stress-strain curve is considered to be an artifact of the plane strain compression test; note that our strains are measured as elongations of the specimen itself, not by changes in thickness of the specimen or by cross head motion. The actual length change and thickness change of the specimen were measured by a micrometer after the tests and were always consistent with the extensometer readouts on the chart record.

The second complication is considered in detail in the following section; the analysis is based on Woodward's clever method for accounting for friction in free-compression tests (3).

Three subpresses were used to perform the plane strain compression tests; these used specimens with thickness (h): gage length (l): width (w) measurements shown in Table I. Some tests were run with other geometries as described in the following section.

Table I

Dimensions of Plane Strain Compression Test Sections
(inches: 1 in. = 25.4 mm)

	Specimen		
	S _A	S _B	S _C
h	0.028	0.014	0.007
l	0.084	0.042	0.021
w	0.500	0.250	0.200

Specimen S_C could not be made from any strip thinner than 0.007 in (0.18 mm) for obvious reasons; therefore, we did not attempt to make or test any thinner strips; however, we feel that the general method could be extended to suppresses another order of magnitude smaller if meticulous attention is paid to accuracy and alignment of the anvils, the active parts of the subpress. Length-change and load measurement would not be limiting factors - only the accuracy of the deformation zone. This would increase the range of total true strain for which flow properties could be characterized to about $\epsilon = \ln t_0/t = 6.4$, not a large enough improvement to be worth the time and expense at this stage.

b. Obtaining True Flow Stress from Mean Compressive Pressure in the Plane Strain Compression Test

A slab analysis of plane-strain compression between smooth dies yields the following expression (see, for example, Backofen (4)):

$$\frac{\bar{p}}{2k} = 1 + \frac{\mu_h}{2\Delta_h} + \frac{\mu_w}{4\Delta_w} \quad (1)$$

where $\bar{p}$ is the compressive load divided by the area ($\ell \times w$) of the anvils, k is the shear flow stress of the specimen material, μ_h and μ_w are the apparent friction coefficients (assuming Coulomb friction) between the specimen and the anvils and between the specimen and the side constraints, respectively. The quantities Δ_h and Δ_w are given by the following ratios:

$$\Delta_h = \frac{h}{\ell} \quad (2)$$

$$\Delta_w = \frac{w}{\ell} \quad (3)$$

where h , ℓ , and w are the active dimensions of the specimens as listed in Table I (in most cases - variants are described later in this section).

The derivation of Eq. (1) assumes a Tresca-type yield criterion, uniform stresses across the differential slab element, and is evaluated for the first three terms of a Taylor expansion of $\exp(x)$; one term cancels out, leaving an (apparently linear) approximation.

When the side constraints offered by the screws visible in Fig. (1) are omitted, the $\frac{\mu_w}{4\Delta_w}$ term of Eq. (1) vanishes, and a Woodward-type analysis (3) can be performed on the results of a pair of plane strain compression tests on two specimens of the same material with different Δ_h (obtained by using two different specimen thicknesses, h_A and h_B). See Figure 2. The frictionless compressive pressure P is given by the relationship:

Figure 2. Effect of anvil friction on apparent flow stress in plane strain compression test; $\Delta_h^A = 0.50$; $\Delta_h^B = 0.33$. No side constraints used.

$$P = \frac{\bar{P}_A \Delta_h^A - \bar{P}_B \Delta_h^B}{\Delta_h^A - \Delta_h^B} \quad (4)$$

if the widths w of each specimen are the same or are long enough to constrain the specimen fully against spreading. Eq. (4) is derived directly from Eq. (1) without the $\frac{\mu_w}{4\Delta_h^w}$ term. In the present work, $\Delta_h^A = \frac{1}{2}$ and $\Delta_h^B = \frac{1}{3}$ were used to do this analysis, so that

$$P = 3\bar{P}_A - 2\bar{P}_B \quad (5)$$

The resulting curve of P vs. true strain ϵ is then representative of the frictionless compressive flow stress of the material, Fig. 3. The apparent friction coefficient μ_h is given by

$$\mu_h = \frac{\bar{P}_A - \bar{P}_B}{\left[\frac{\bar{P}_B}{2\Delta_h^A} \right] - \left[\frac{\bar{P}_A}{2\Delta_h^B} \right]} \quad (6)$$

if there are no side constraints. We found $\mu_h = 0.031$ for Teflon plumber's tape, 0.001 in (0.025 mm) thick, and $\mu_h = 0.033$ for the proprietary MoS_2 lubricant. The friction hill raised the apparent flow stress by about 5%. Figure 4 shows the effect of completely removing the frictional components from the flow stress.

In some of the tests, side constraints were used to prevent spreading of the specimen (we have since found that spreading without the side constraints is negligible); these cause another friction hill to form. This friction hill formation provides additional compression pressure, which is higher for thicker specimens and reverses the specimen shape effect of the anvil's friction hill (Figure 5). We made a special subpress with $h:l:w = 0.028 \text{ in}:0.084 \text{ in}:0.200 \text{ in}$ (1 in = 25.4 mm); h could also be 0.042 in. This enabled comparison with the largest subpress where $h:l:w = 0.028 \text{ in} \text{ (or } 0.042 \text{ in)}:0.084 \text{ in}:0.500 \text{ in}$. If

Figure 3. Same compression stress strain curves as Fig. 2, $C = 2k = \text{actual flow stress} = 3P_A - 2P_B$, where P_A and P_B are the mean compression pressures for specimens A and B respectively. No side constraints used.

Figure 4. Plane strain compression test data for strip drawn Fe-0.17 w/o Ti; solid line and squares represent mean compression pressure $\bar{P}$; dotted line represents the actual flow stress, corrected for friction.

Figure 5. Effect of combined anvil friction and side-constraint friction on mean pressure in plane strain compression test; $\Delta_h^A = 0.5$; $\Delta_h^B = 0.33$; $\Delta_w^A = \Delta_w^B = 6.0$.

$\Delta_h^A = \Delta_h^B$ and $\mu_h^A = \mu_h^B$, manipulation of Eq. (1) leads to

$$\left(1 + \frac{\mu_h}{2\Delta_h}\right) P = \frac{\bar{P}_A \Delta_w^A - \bar{P}_B \Delta_w^B}{\Delta_w^A - \Delta_w^B} \quad (7)$$

which reduces to the equation below for $\Delta_w^A = \frac{0.500 \text{ in}}{0.084 \text{ in}} = 6$ and $\Delta_w^B = \frac{0.200 \text{ in}}{0.084 \text{ in}} = 2.4$ when $\mu_h/2\Delta_h \approx 0.05$

$$P = \left(\frac{1}{1.05}\right) \frac{6.0 \bar{P}_A - 2.4 \bar{P}_B}{6.0 - 2.4} \quad (8)$$

This analysis assumes that the contact area between the specimen and side constraints is confined to the active gage length only; that is a lower bound; the upper bound is impossible to determine, so the value of μ_w will be exaggerated; in fact, $\mu_w \approx 0.1$ even though the same lubricants were used for the side constraints as for the anvils. Figures 6 and 7 show the effect of specimen width on the apparent flow stress.

The contribution of side constraint friction to the inflation of the mean compressive pressure $\bar{P}$ is only about one tenth of the anvil friction (Figure 8).

c. Flow Properties of Strip in Reversed Deformation

Langford and Cohen (5) have already shown that drawn Fe-0.007 w/o C wire is extremely unstable against reversed deformation, even though the initial compressive flow stress (in free compression) is nearly as high in magnitude as the monotonic flow stress. We have performed the analogous experiment for our plane-strain elongated Fe-0.15 w/o Ti, by doing plane strain compression tests in which the test elongation is in the thickness direction and the test compression is in the direction of original extension of the strip. This is not a trivial experiment since the small as-drawn thickness of the strip demands a very short gage length for the plane strain compression test specimen.

Figure 6. Effect of friction against side constraints during plane strain compression tests. A' = narrower specimen, A = wider specimen; $\Delta_h^{A'} = \Delta_h^A = 0.50$; $\Delta_w^{B'} = \Delta_w^B = 2.33$.

Figure 7. Effect of friction against side constraints during plane strain compression tests. B' = narrower specimen; B = wider specimen; $\Delta_h^{B'} = \Delta_h^B = 0.33$; $\Delta_w^{B'} = 0.40$ $\Delta_w^B = 2.33$.

Figure 8. Frictional effect on apparent flow stress in plane strain compression test. $A' = \bar{P}$ = mean pressure vs. true strain; $C = 2k$ = flow stress corrected for frictional constraint.

We were therefore limited to strip thicknesses of greater than 0.03 in (0.8 mm) because the smallest subpress has a gage length of 0.021 in (0.50 mm). The extra length of 0.03 in vs. 0.02 in. was necessary to facilitate attachment of the extensometer. The results of these reversed-deformation tests are shown in Figure 9. The compression between monotonic and reversed plane strain compression is shown in Figure 10. Both curves are corrected for friction.

d. Results of Flow Characterization of Plane Strain Elongated Fe-0.17 w/o Ti; Comparison with Axisymmetrically Elongated Wire

The overall results of these mechanical tests are summarized in Figure 11. Here, the comparison is facilitated by plotting equivalent stress and equivalent strain. The nominal and equivalent stresses and strains are identical for wire, of course; for strip the relationships are

$$\sigma_e)_{\text{strip}} = \frac{\sqrt{3}}{2} P \quad (9)$$

$$\epsilon_e)_{\text{strip}} = \frac{2}{\sqrt{3}} \ln t_0/t \quad (10)$$

These equations assume von Mises yielding behavior. The frictional component of P has already been removed for both monotonic and reversed deformation. The remaining differences between wire and strip are therefore due to true differences in mechanical properties and not to the test method; however, the flow stress in compression in annealed iron and even in hardened steel is greater in magnitude than the tensile flow stress; this is known as the strength-differential (SD) effect (6). It is related to the dislocation core structure rather than to any difference in microstructure as developed by different shape changes and so it will not be given further consideration. The SD effect is not insignificant, however.

Figure 9. Results of reversed plane strain compression tests on drawn strip of Fe-0.17 w/o Ti.

Figure 10. Monotonic vs. reversed plane strain compressive flow stress of strip drawn Fe-0.17 w/o Ti.

Figure 11. Strain hardening rate of wire vs. stripdrawn Fe-0.17 w/o Ti, compared on the bases of equivalent stress and equivalent strain.

2. Characterization of Microstructure

During the present contract period, the Principal Investigator was awarded twelve working days on the Argonne National Laboratory's High Voltage Electron Microscope as well as two additional days for evaluation of its efficiency for the intended purpose (See Appendix A for results - the answer was "yes"). So far, six of the twelve days have been used, with a total output of nearly 700 electron diffraction patterns and about 100 bright-field photographs of the associated microstructures. About 100 of these have been analyzed so far.

Orientation maps like those first obtained by Langford and Cohen (7) were obtained for thin foils of drawn wire ($\epsilon = 2.0$) and drawn strip ($\epsilon = 1.8$) in our Fe-0.17 w/o Ti. Additional data and diffraction patterns were obtained to make orientation maps for the following specimens, which have not yet been analyzed quantitatively, but which will be discussed qualitatively: Wire ($\epsilon = 4.00$) - one area of 88 cells; Strip ($\epsilon = 2.55$) - three areas totalling about 160 cells; and a chip ($\gamma = 1.6$, $\epsilon = -0.8$) - two areas totalling about 185 cells.

This work is proving extremely productive in terms of acquisition of data; the main instrumental obstacle is the relatively long time required to reload the photographic film in the electron microscope. No difficulties were encountered with the accuracy of area selection, exposure time, keeping track of which diffraction pattern goes with which cell, or accuracy of cellular orientation as read from the diffraction pattern. No difficulty was experienced with obtaining good thin-foil orientation in the microscope as anticipated in last year's progress report; the high repeatability of the specimen stage tilts enabled rapid determination of the optimum orientation and contrast of the thin foil. In all cases, large areas with similar orientations were found, greatly simplifying subsequent data analysis because all cells in each area gave the same basic $[hk\ell]$ pattern. So far, so good.

— 1.0 μm —

Figure 12(a). Bright field image of cellular substructure of Fe-0.17 w/o Ti wire drawn to a total true strain of $\epsilon = 2.0$ & $D_0/D = 2.0$. Transverse section. Double exposure with image of selected area aperture over cell number 1.

— 1.0 μ m —

— BASE LINE OF PLATE —

Figure 12(b). Sketch of same area as part (a), showing outlines of cell boundaries.

Table of θ orientation of cells; θ is measured counterclockwise from the baseline of the bright field plate to the $[1\bar{1}]$ direction of the cell's $[110]$ diffraction pattern.

Cell Number	θ Degrees	Cell Number	θ Degrees
1	128.0 ± 0.1	13	135.0 ± 0.1
2	129.3 ± 0.1	14	132.7 ± 0.1
3	127.8 ± 0.2	15	130.6 ± 0.1
4	136.8 ± 0.2	16	130.2 ± 0.1
5	134.7 ± 0.2	17	$121 \text{ to } 129$
6	134.7 ± 0.3	18	131.1 ± 0.1
7	129.1 ± 0.1	19	140.4 ± 0.1
8	129.8 ± 0.1	20	133.2 ± 0.4
9	133.4 ± 0.1	21	128.7 ± 0.1
10	134.9 ± 0.1	22	123.7 ± 0.3
11	137.9 ± 0.3	23	125.0 ± 0.3
12	140.1 ± 0.4	24	123.1 ± 0.6
		1 (repeat)	128.3 ± 0.1

Figure 12(c). δ -orientation of cells; each point corresponds to exact location of the cell's $[110]$ pole; frame of reference same as (a) and (b).

Figure 12(d)

Figure 13(a). Bright field image of cellular substructure of Fe-0.17 w/o Ti wire drawn to a total true strain of $\epsilon = 2.0$ $\ln D_0/D = 2.0$. Transverse section.

—1.0 μm —

Figure 13(b). Sketch of same area as part (a), showing outlines of cell boundaries.

Table of θ orientation of cells; θ is measured counterclockwise from the baseline of the bright field plate to the $[\bar{1}10]$ direction of the cell's $[110]$ diffraction pattern.

Cell Number	θ Degrees
1	100.6 $\pm$ 0.1
2	101.2 $\pm$ 0.2
3	101.6 $\pm$ 0.3
4	102.2 $\pm$ 0.2
5	111.7 $\pm$ 0.3
6	112.0 $\pm$ 0.1
7	112.0 $\pm$ 0.1
8	111.6 $\pm$ 0.3
9	103.9 $\pm$ 0.1
10	104.4 $\pm$ 0.2
11	104.7 $\pm$ 0.1
12	104.8 $\pm$ 0.1
13	99.5 $\pm$ 0.5
14	100.4 $\pm$ 0.2
15	101.4 $\pm$ 0.3
16	102.2 $\pm$ 0.4
2 (repeat)	101.2 $\pm$ 0.2

Figure 13(c). δ -orientation of cells; each point corresponds to exact location of the cell's (110) pole; frame of reference same as (a) or (b).

Figure 13(d).

The primary obstacle to rapid progress in the field of orientation mapping is the time required to index the diffraction patterns (i.e. measure the θ orientation and two δ -orientation angles - as defined in (7) - to 0.2° accuracy if possible) and to prepare the orientation maps. The manual skills and technical training of a Ph.D. tool and die maker are required, together with the patience of an idiot savant. The Principal Investigator knows only one individual with these characteristics - himself - but most of his time (about 20% during the academic year and 50% in the summer) went towards project administration, consultation with students, theorizing, literature searches, report writing, and miscellaneous interruptions. Therefore, the Principal Investigator expects that preparation of quantitative orientation maps for these and additional specimens will continue to lag far behind the raw data for at least another year, or until additional (and highly motivated) manpower is obtained.

Once the orientation maps are produced, quantitative comparisons can be made between the different specimens and shape changes by means of the method developed by Langford and Cohen (7). The misorientations and cell sizes within the orientation map are converted into an equivalent (lower bound) dislocation density. This has not yet been done for the three maps presented below, but the necessary computer program used in (7) is in hand and ready for use. Further development of the computer analysis is discussed in the enclosed renewal proposal.

a. Wire (Axisymmetric Elongation)

Two orientation maps were finished for Fe-0.17 w/o Ti wire drawn to a true strain of 2.0 (Figures 12 and 13). These maps are qualitatively similar to those obtained by Langford and Cohen (7) for drawn Fe-0.006 w/o C wire. However, it is necessary to repeat this work on the present material because the cell sizes of drawn iron alloys are known to be sensitive to alloying even though the rates of strain hardening are only weakly dependent

on alloying (8,9). This will enable us to determine whether the combination of cell size and cellular misorientation follows the same alloy-insensitive variation with strain hardening as in the previous work. We will also be able to make direct comparisons between the various shape changes and flow stresses.

Figure 13 shows a sharper [110] texture than Figure 12, but substantially smaller misorientations, both in terms of rotations about the polar (microscope and wire) axis and also in terms of rotations about transverse directions. These misorientations are read from the orientation in terms of two parameters: (1) $\Delta\theta$ between cells (i.e. across the cell wall between them), which comes from Figure 12d; and (2) $\Delta\delta$, also between cells, which comes from the great-circle (at this scale, the straight line) angular distance between the [110] poles of the two cells in Figure 12c. For example, the $\Delta\theta$ misorientation between cells 1 and 23 is $2^\circ \pm 0.4^\circ$ and the $\Delta\delta$ misorientation between the same cells is 1.5° ($\pm$ about the same error). The $\Delta\theta$ misorientation in this example is pure tilt about the [110] axis, whereas the $\Delta\delta$ misorientation happens also to be approximately tilt about the [002] axis (remember, θ is the angle measured from the base line to the cell's $[\bar{1}10]$ direction). In the present computer program to be used in the quantitative comparisons between strains and shape changes, these two misorientations are simply added together; detailed analysis of the type of cellular misorientation prevalent in each substructure should be carried out eventually, but no increase in the sophistication of the orientation map is necessary; these maps contain fundamental information.

The raw data is on hand for an orientation map of our Fe-0.17 w/o Ti wire at $\epsilon = 4.0$. Since few surprises are expected, preparation of this map will be begun after those for strip and chip.

b. Strip (Plane-Strain Elongation)

One orientation map was finished for an edge section of Fe-0.17 w/o Ti strip drawn to a true strain of 1.8 (Fig. 14). The diffraction patterns were all [111]; the poles shown in Fig. 14c are therefore [111]'s, not [110]'s as in Figs. 12c and 13c. However, the θ orientations are all measured from the base line to the same $[\bar{1}10]$ direction in the cell as in the case of wire. The longitudinal direction in this specimen is approximately parallel to the base line of the map; the short transverse (thickness) direction is vertical in the plane of the map. Hence, the preferred orientation (deformation texture) within this particular tiny region of the specimen corresponds to $(11\bar{2})[\bar{1}10]$, the same as component D in Figs. 30 and 31 of Barrett (10). As can be seen from Fig. 14c, there is considerable spread in this orientation about the longitudinal direction within the observed region - 9° between cells 6 and 23.* The $[\bar{1}10]$ direction only varies ± 2 or 3° from the longitudinal direction of the strip. The $\Delta\delta$ misorientations are dominant in this map, and are mainly rotations about $[1\bar{2}1]$. This area was selected deliberately to include what was thought to be a shear band (running approximately in the $[1\bar{2}1]$ direction (i.e., upper right to lower left)). However, there is no pronounced clue as to the origin of this shear band (admittedly a weakly developed one) in either the $\Delta\theta$ or $\Delta\delta$ misorientations in this map. The only actual hint is visual: the slip distance in the direction of the $\frac{a}{2}[1\bar{1}1]$ burgers vector appears to be somewhat longer than that in the direction of $\frac{a}{2}[\bar{1}11]$ burgers vector for cells 22, 25, 17, 4, 6, 1, 13, 18, 19, 35, 43, 44, 33 and 32. These two slip directions make equal angles with the longitudinal axis, but the displacements caused by

* Note: Because iron is magnetic, there is some doubt about the orientation of the foil itself about the transverse microscopic axes; hence, no attempt is made here to correct for the slight tilt (about 6°) of the specimen holder used to obtain good contrast.

Figure 14(a). Bright field image of cellular substructure of Fe-0.17 w/o Ti strip drawn to a total true strain of $\epsilon = \ln t_0/t = 1.8$. Longitudinal (edge) section.

1.0 μm

1.0 μ m

BASE LINE OF PLATE

Figure 14(b). Sketch of same area as part (a) showing outlines of cell boundaries.

Table of θ orientation of cells; θ is measured counterclockwise from the baseline of the bright field plate to the $[1\bar{1}]$ direction of the cell's $[110]$ diffraction pattern.

Cell Number	θ Degrees	Cell Number	θ Degrees	Cell Number	θ Degrees
1	184.5 ± 0.4	16	183.6 ± 0.2	31	183.8 ± 0.1
2	185.5 ± 0.4	17	183.6 ± 0.1	32	181.0 ± 0.3
3	184.5 ± 0.3	18	184.8 ± 0.3	33	180.9 ± 0.1
				1(repeat)	184.1 ± 0.2
4	183.4 ± 0.5	19	183.2 ± 0.3	34	182.8 ± 0.3
5	184.2 ± 0.2	20	182.5 ± 0.1	35	185.3 ± 0.1
6	185.0 ± 0.5	21	182.4 ± 0.2	36	182.9 ± 0.1
7	184.8 ± 0.3	22	182.1 ± 0.1	37	183.3 ± 0.4
8	185.6 ± 0.2	23	182.9 ± 0.1	38	183.6 ± 0.2
9	184.7 ± 0.3	24	183.4 ± 0.5	39	184.0 ± 0.1
10	184.3 ± 0.1	25	184.7 ± 0.1	40	183.8 ± 0.3
11	184.3 ± 0.1	26	184.8 ± 0.1	41	182.7 ± 0.2
12	184.7 ± 0.2	27	184.4 ± 0.1	42	183.3 ± 0.4
13	184.5 ± 0.2	28	184.4 ± 0.1	43	183.7 ± 0.2
14	181.2 ± 0.1	29	183.7 ± 0.3	44	184.0 ± 0.2
15	184.0 ± 0.2	30	183.7 ± 0.1	45	182.9 ± 0.1
				1(repeat)	184.8 ± 0.1

Figure 14(d)

Figure 14(c). δ -orientation of cells; each point corresponds to exact location of the cell's (111) pole; frame of reference same as (a) and (b).

$\frac{a}{2}[\bar{1}\bar{1}1]$ are more compatible with the overall shape change. There does not appear to be a textural instability, only a microstructural one. This is consistent with the results of Grewen, et al. (11) for rolled copper as discussed in last year's progress report.

We have on hand raw data for three orientation maps for our Fe-0.17 w/o Ti strip drawn to $\epsilon = 2.6$. More will certainly be needed to survey properly the many kinds of cellular arrangements seen so far. The wire microstructure is far less complicated, to be sure.

c. Chip (Pure Shear)

Data were obtained for two areas within an edge section of an orthogonally cut chip (shear strain = 1.6, longitudinal strain = -0.8), which has been our approximation of pure shear. Figure 15 shows an area near the tool-chip interface where this approximation is more nearly correct. The diffraction pattern is from the cell marked "5" on the bright field image, and it has been rotated CCW by 169° to bring the diffraction pattern into the correct relationship to the bright field image. This is a $[110]$ pattern; note that the $[\bar{1}\bar{1}2]$ and $[\bar{1}11]$ directions in cell 5 are parallel and perpendicular, respectively, to the long axes of the cells, which are aligned in the direction of shear. The $\frac{a}{2}[\bar{1}\bar{1}1]$ slip vector is aligned 20° to the long axis of the cells. This orientation is typical, with minor variations (nevertheless crucial to the average dislocation density and the flow stress) from cell to cell.

Quantitative comparison of this mode of pure shear and its microstructure to the microstructure of shear bands formed during plane strain elongation must await completion of the orientation maps and the acquisition of data from more specimens. However, a qualitative similarity exists between the two: (1) the microstructure of the chip can be described as having two characteristic slip distances: a very short one in the direction of the inactive $\frac{a}{2}[\bar{1}\bar{1}1]$ burgers vectors, and a very long slip distance between cell walls in the $\frac{a}{2}[\bar{1}\bar{1}1]$

Figure 15(a)

Bright field image of cellular substructure of Fe-0.17 w/o Ti chip cut with 30° rake angle; shear strain $\gamma = 1.6$. Longitudinal (edge) section. $[1\bar{1}1]$ direction is also the cutting direction.

Figure 15(b)

$[110]$ selected area electron diffraction pattern from cell number 5 marked on part (a). $[1\bar{1}1]$ and $[111]$ directions marked on part (a) represent the orientation of the majority of the cells in the field of view.

direction; (2) the microstructure of a shear band appears to have a longer slip distance in the $\frac{a}{2}[\bar{1}11]$ direction than in the $\frac{a}{2}[\bar{1}1\bar{1}]$ direction as in the chip. Hence, the similarity is that the structure of each is weaker in the direction of greater shear deformation. We don't know yet what stabilizing influence there is - note that the "shear band" in Fig. 14 is in a strip drawn specimen still exhibiting vigorous strain hardening (see Section II.A.1.d).

3. Progress Expected in the Remainder of the Current Contract Term

a. Analysis of High-Voltage Electron Microscope Plates

The Principal Investigator expects to complete the remaining orientation maps for which data already exists as discussed above during May, June and July.

b. Further High-Voltage Electron Microscopy

The Principal Investigator will consume the remaining six of the twelve days on the Argonne National Laboratory's High Voltage Electron Microscope during June and July. From past experience, this should be enough time to obtain data for about twelve orientation maps. These will be distributed among the following specimens: (1) drawn strip, $\epsilon = 1.8, 2.6$ and 4: seven maps; (2) drawn wire, $\epsilon = 1, 2, 4$ and 6: five maps. No further work is to be expended on chips in deference to specimens deformed in pure shear by more sophisticated means as explained in the accompanying renewal proposal. No quantitative electron metallographic studies will be made on specimens with complex deformation histories until we know what to look for as found by conventional transmission electron microscopy and from the results of the mechanical tests.

c. Mechanical Testing of Wire, Wire Drawn Strip and Strip Drawn Wire

We expect to complete the mechanical testing of specimens deformed monotonically in axisymmetric elongation - to be compared with those deformed

monotonically in plane strain elongation. We also expect to complete the two tests of the stability of mechanical properties (and microstructure) against change in the mode of monotonic elongation: strip drawing to wire drawing and vice versa. The strip has already been wire drawn; we expect to strip draw a wire specimen, and we will complete both the tensile testing of the wire drawn from strip and the plane strain compression testing of strip drawn from wire. This will enable us to predict the schedule of deformation of tubing necessary to achieve either a wire texture (and microstructure) or a sheet texture (and microstructure) when the starting and finishing dimensions are the same.

B. Theoretical Progress

1. Stability of Microstructure and Mechanical Properties of Heavily Deformed Iron

Our results for the strain hardening of strip drawn iron look surprisingly similar to the results of Walker and Shaw (12) for the work hardening of steels in pure shear, in that the strain hardening is at first vigorous and linear, falls to zero, and then becomes negative (hereafter called "flow softening"). In the case of pure shear with small normal stress on the plane of shear, the strength peaks much earlier than in the case where a normal compressive stress comparable in magnitude to the shear stress is applied on the plane of shear (12). In strip drawing, the normal stress on the plane of maximum shear is also comparable in magnitude to the shear stress. Therefore, we have come upon a chicken/egg dilemma: is the flow softening phenomena in pure shear and plane strain elongation (strip drawing) a consequence of microstructural instability or a cause of it? We see incipient shear bands in strip drawn iron before the strain hardening even begins to drop off; in strip drawn pearlite there is abundant evidence of shear bands during increasing strain

hardening (i.e. exponential strain hardening) (2). Walker and Shaw (12) attribute the flow softening in pure shear to microscopic shear cracks which reweld and thereby remain subcritical, the more so the greater the normal compressive stress (hydrostatic pressure component of the stress state). It is clear from our work that the shear bands in our strip drawn iron at $\epsilon = 1.8$ and 2.6 do not involve such microcracks - the bands have finite thickness - but we haven't made any thin foils of the strip drawn to $\epsilon = 4.0$ yet. Nor do we have any convenient way of changing the normal stress during strip drawing except by changing the deformation-zone geometry, and that is not an effective technique. Even our chip with a stress strain of 1.6 does not show any microcracks, but the results of Walker and Shaw wouldn't predict flow softening at that strain anyway. Higher strains via orthogonal cutting are out of the question because the mechanical forces are too large for the specimen to support without deformation ahead of and below the chip. Therefore, definitive work on the question of the stability of microstructure and flow properties will depend on first, a repetition of the Walker and Shaw experiment (12), in which for one set of specimens sufficient normal compressive stress is applied to inhibit flow softening, and for a second set of specimens the normal compressive stress is much less so that flow softening does occur. We would then use transmission electron microscopy to study the resulting microstructures. The second step would be to do a fundamental study of the effect of hydrostatic pressure on plane strain elongation of our Fe-0.17 w/o Ti to determine whether the flow softening phenomenon can thereby be postponed to a higher total strain. Although no tubing will ever be made commercially in a Bridgman-type hydrostatic pressure cell, it might be quite practical to manufacture steel tubing by extrusion; we would therefore be establishing fundamentally whether the cold extrusion process would be worth developing and we would also establish the

mechanical conditions necessary to achieve the desired effect. Note that a combination of drawing and extrusion may be necessary to accomplish this without overloading the die - the question is - can we do this without defeating our purpose of optimizing the mechanical properties of the tube?

2. Strengthening Mechanisms

The formation of a "curly grain" structure in drawn bcc wire as first observed by Peck and Thomas (13) has been interpreted by Hosford (14) in terms of the number of slip systems available to cause elongation of the wire. There are only two such slip directions in the [110] bcc wire texture, of course; Hosford (14) has pointed out that it would take as much as 50% extra stress to activate the other two $\langle 111 \rangle$ slip directions and thereby force completely axisymmetric elongation rather than the observed (locally) plane strain elongation on the scale of the grain dimensions.

This interpretation leads to the following paradox: the bcc wire strain hardens so much, as a result of the resulting curling or interfolding of these ribbon-shaped grains (necessary to maintain grain-boundary continuity), that it becomes much stronger than it would be if it simply "paid the 50% penalty" and its grains deformed axisymmetrically. Since this Principal Investigator has observed quite equiaxed cells (equivalent to grains) at the highest strains observed in iron wires ($\epsilon = 10$) (9), why wouldn't they begin to avoid further strain hardening by switching to an axisymmetric mode of deformation at sufficiently high strains, thereby saturating their strain hardening capacity? No such saturation of work hardening or even any slackening of the observed linear rate of work hardening has been observed, however.

The Principal Investigator has conceived the following explanation of the curly grain structure in drawn bcc wires which eliminates the above paradox.

Figure 16a shows a transverse section of a curly grain in the $[110]$ bcc wire texture; the $[110]$ direction is therefore perpendicular to the paper whereas the $(\bar{1}\bar{1}0)$ plane containing the $[111]$ and $[1\bar{1}\bar{1}]$ slip directions (those which do contribute to elongation of the wire) is vertical in the sketch and perpendicular to the plane of the ribbon-shaped grain. Also shown in Fig. 16a are the $[\bar{1}11]$ and $[\bar{1}\bar{1}\bar{1}]$ slip vectors which must be activated, either by curling of this grain, as shown in Fig. 16b, or by sidewise compression (giving axisymmetric elongation when combined with the ongoing plane strain elongation) as shown in Fig. 16c. The bending deformation in Fig. 16b results in the storage of excess dislocations of one sign ρ_{xs} according to the relationship

$$\rho_{xs} = \frac{1}{r} \left(\frac{\alpha_1}{b_1} + \frac{\alpha_2}{b_2} \right) \quad (11)$$

where r is the radius of curvature of the grain, α_1, α_2 are geometrical factors (approximately equal to 1) and b_1, b_2 are the burgers vectors of the xs dislocation of one sign, either $\frac{a}{2}[\bar{1}11]$ or $\frac{a}{2}[1\bar{1}\bar{1}]$ in this case. The sidewise compression in Fig. 16c fails to give rise to lattice curvature and would, therefore, be expected not to lead to substantial strain hardening. However, the $\frac{a}{2}[\bar{1}11]$ and $\frac{a}{2}[1\bar{1}\bar{1}]$ slip vectors meet at an angle of less than 90° in Fig. 16c, and so the following dislocation reaction will occur:

$$\frac{a}{2}[\bar{1}11] + \frac{a}{2}[1\bar{1}\bar{1}] = a[001] \quad (12)$$

This reaction is energetically favorable because the sum of the squares of the burgers vectors on the left hand side of Eq. (12) is greater than the square of the $a[001]$ burgers vector. Note that the $a[001]$ dislocation is likely to be far less mobile than the $\frac{a}{2}\langle 111 \rangle$ type in spite of having a (110) slip plane because of the longer burgers vector. Therefore it can be considered sessile and a serious obstacle to further deformation. In fact, such formation of

(a)

(b)

(c)

Figure 16. Dislocation configurations in deforming $[110]$ textured grain in bcc wire. (a) Possible slip vectors and their relationship to the grain. (b) Dislocations activated by bending during plane strain elongation of the grain. (c) Dislocations activated by sidewise compression during axisymmetric elongation of the grain.

sessile $a[001]$ dislocations at the intersection of each pair of slip planes ($\{112\}$ assumed here for convenience) would lead to the classical Stage II linear Taylor hardening because the slip distance would decrease in inverse proportion to the dislocation density, requiring ever-greater increases in the latter to maintain the strain rate.

The dislocations on intersecting slip systems in the bending case of Fig. 16b meet at an angle of greater than 90° , so the corresponding (hypothetical) dislocation reaction would be:

$$\frac{a}{2}[\bar{1}11] + \frac{a}{2}[\bar{1}1\bar{1}] = a[\bar{1}10] \quad (13)$$

This reaction is not energetically favorable; so the bending produces no sessile dislocations.

The original paradox is therefore solved - axisymmetric elongation of the $[110]$ wire textured bcc grain would lead not only to 50% greater flow stress but also to substantially greater strain hardening than does the observed deformation mode: plane strain elongation plus curling.

C. References

1. A. P. Green and R. Hill: Calculations on the influence of friction and die geometry in sheet drawing, J. Mech. and Physics of Solids, Vol. 1, 1952, p. 31.
2. G. Langford: Deformation of Pearlite, Met Trans A, 1977, Vol. 8A, p. 861.
3. R. L. Woodward: Communication: A Note on the Determination of Accurate Flow Properties from Simple Compression Tests. Met Trans A, 1977, Vol. 8A, p. 1833.
4. W. A. Backofen: Deformation Processing, Addison Wesley, Reading, Mass. pp. 162-165.
5. G. Langford and M. Cohen: Strain Hardening of Iron by Severe Plastic Deformation, Trans ASM, 1969, Vol., 62, p. 623.
6. W. F. Hosford and T. J. Allen: Twinning and directional slip as a cause for a strength differential effect, Met Trans., Vol. 4, 1973, p. 1424.
7. G. Langford and M. Cohen: Microstructural Analysis by High-Voltage Electron Diffraction of Severely Deformed Iron Wires, Met Trans A, 1975, Vol. 6A, p. 901.
8. H. J. Rack and M. Cohen: Strain hardening of iron-titanium alloys at very high strains, Mater. Sci. and Eng. 1970, Vol. 6, p. 320.
9. G. Langford, P. K. Nagata, R. J. Sober, and W. C. Leslie: Plastic flow in binary substitutional alloys of BCC iron - effects of wire drawing and alloy content on work hardening and ductility, Met Trans, 1972, Vol. 3, p. 1843.
10. C. S. Barrett, Structure of Metals, 2nd ed., McGraw Hill, New York, 1952, p. 469.
11. J. Grewen, T. Noda, and D. Sauer: Electron Microscope Investigation of Shear Bands, Z. Metallkunde, 1977, Vol. 68(4), p. 260 (in German).
12. T. J. Walker and M. C. Shaw: On Deformation at Large Strains, in Advances in Machine Tool Design and Research 1969, Proceedings of the 10th International MTDR Conf., U. of Manchester, Inst. of Science and Technology, Sept. 1969, p. 241.
13. J. F. Peck and D. A. Thomas: A study of fibrous tungsten and iron, Trans. Met. Soc. AIME, 1961, Vol. 221, p. 1240.
14. W. F. Hosford, Jr.: Microstructural changes during deformation of [011] fiber-textured metals, Trans. Met. Soc. AIME, 1964, Vol. 230, p. 12.

D. Time Devoted to Project by Principal Investigator

		Man Months
Summer	50% from 8/1/79 through 9/15/79	0.75
Fall	16.7% from 10/1/79 through 12/31/79	0.50
Winter	18.8% from 1/1/80 through 3/31/80	0.56
Spring	16.7% from 4/1/80 through 6/30/80	0.50
Summer	100% from 7/1/80 through 7/31/80	1.00
Total Time Spent		3.31 man months

E. Financial Statement of Estimated Costs

Drexel University

Contract EY-76-S-02-4072

For the Period 1 August, 1979 through 31 July, 1980

1. Estimated Total Project Costs for the Current Period	\$ 44,040
2. Total Amount Chargeable to DoE (95% of line 1)	41,837
3. Cumulative Support Costs	86,163
4. Estimated Total Cumulative Support Costs	128,000
5. Cumulative Support Ceiling	128,000
6. Anticipated Difference between Items 4 and 5	-0-

III. APPENDICES

College of Engineering

Department of
Materials Engineering
(215) 895-2322

June 27, 1979

Dr. A. Taylor, Manager
ANL - HVEM Facility
U. S. Department of Energy
Argonne National Laboratory
9700 South Cass Avenue
Argonne, Illinois 60439

Dear Tony:

Thank you for your hospitality and attention during our recent stay at Argonne. The experimental work was quite successful, thanks to the assistance of Al Philippedes, Larry Lehman, and Pete Baldo. They are all highly competent, helpful, and cheerful workers - and made our work fun as well as fruitful. Larry deserves special thanks for his persistence in working with the particularly recalcitrant STEM, which enabled us to get positive results even when we were told to expect virtually zero, due to its recent move.

We have compared the ANL High Voltage Electron Microscope and two scanning transmission electron microscopes, a Philips 300 (retrofitted) and a JEOL-100C (non-dedicated), with respect to their relative abilities to make accurately resolvable single-crystal electron diffraction patterns (SAD) from submicron crystallites in a cellular dislocation substructure in highly deformed iron.

My conclusions follow:

1. The HVEM is more valuable for pioneering scientific work in quantitative orientation mapping of cellular dislocation substructure because:
 - a. The SAD crossed-grid spot patterns are easily recognizable so that the operator can qualitatively survey the structure for characteristic cellular arrangements (which are otherwise indistinguishable without the aid of microdiffraction).
 - b. Bright field/dark field imaging is available at the push of a couple of buttons so that high-resolution transmission electron micrographs can be made and so that SAD patterns can easily be correlated with microstructures.

A-1

College of Business and Administration
College of Humanities and Social Sciences

College of Engineering • Evening College
School of Library and Information Science
Nesbitt College of Design, Nutrition, Human Behavior, Home Economics • College of Science

- c. SAD patterns can be obtained wherever adequate contrast is obtainable in bright field imaging modes.
2. Both the HVEM and STEM can make quantitative orientation maps of the type described in reference (1); however, no one has published STEM maps equivalent to the HVEM maps in reference (1), although M. D. Ball and P. L. Morris have used STEM to make an orientation survey presented as a series of histograms of single-parameter misorientation. (see reference 2 - attached). Figure 1 shows bright field and SAD patterns for different pairs of cells. We have not yet analyzed our patterns quantitatively, so I cannot comment on the relative accuracy of STEM vs. HVEM from our recent work. However, I claimed about 0.5 degree resolution of misorientations between the cells in wire drawn iron in my HVEM work reported in reference (1); Ball and Morris in reference (2) dismiss misorientations below 5 degrees measured with a STEM in as-rolled Al-6% Ni as perhaps arising within single subgrains. Their histograms show 2 degree resolution for as-deformed specimens and 5 degrees for annealed ones.
 3. The STEM SAD'S contain Kikuchi bands, which are sufficiently well resolved in the Philips 300 STEM (at least) to promise better than 1 degree accuracy of misorientation measurements if they can be correctly indexed; however, the field of view of each Kikuchi pattern may be too small to ensure correct interpretation, and only a small fraction of such patterns are sufficiently bright and symmetrical on the reviewing screen to be recognizable during "live" examination of the specimen.
 4. Contamination of the thin foil specimen by the stationary microbeam in the STEM'S SAD mode is positively useful because:
 - a. The mark is clearly visible in bright-field STEM or TEM mode, simplifying identification of cells as they are surveyed (Fig. (1) b and Fig. (1) c).
 - b. The contamination spot greatly decreases the intensity of the nearly useless SAD cross-grid spot pattern, making the Kikuchi pattern easier to interpret. Usually, too few spots are seen for quantitative or even qualitative indexing.
 5. The STEM has one distinct advantage over the HVEM and that is with respect to the size of the smallest area from which single-crystal selected area diffractive patterns can be obtained: 0.002 μm to 0.02 μm with the STEM, versus 0.02 μm to 0.1 μm with the HVEM. The former is limited by microscope performance and cell shape (as it relates to foil thickness), whereas the latter is limited by the availability of field limiting apertures (0.7 μm needed to select 0.02 μm , 3 μm to select 0.1 μm and so on), not by spherical aberration. This should not be a significant criterion for the proposed work because our cell sizes are rarely below 0.05 μm , and 2 μm field-limiting apertures are readily available.
 6. Neither STEM can conveniently be switched from STEM to TEM mode; the Philips 300 STEM requires a pole-piece change, and the JEOL-100C STEM must be optically realigned, which is not possible unless the specimen is removed

from the optical axis and replaced with a holey carbon film. This prevents convenient high-resolution recording of the bright-field image of the area surveyed. This is a serious hindrance for the present work, since bright-field STEM photomicrography does not produce sufficient resolution of the dislocation contrast within the cellular substructure; i.e. conventional TEM and HVEM both are vastly superior in resolving power for this purpose.

7. The HVEM is essential for conventional SAD via coherent (non converging) electron beams. The improvement in quality of area selection is substantial between 500 kV and 1000 kV, since the number of extraneous spots in SAD patterns of a 0.4 μm cell decreases greatly over this range of accelerating voltage even with the 0.3 μm selected area used so far.
8. Radiation damage in the HVEM was not a serious problem at the low beam current (measured at the specimen) of 10^{-7} amp. None of the microscopes caused appreciable general contamination.
9. The tilt stages of the HVEM and Philips 300 STEM are both sufficiently accurate that no trouble was experienced in properly orienting the specimen; this was indeed a pleasant surprise, since it formerly was a serious obstacle. We did not tilt the specimen in the JEOL-100C STEM, because it was already OK upon first insertion in the microscope.
10. None of the microscopes was adequately calibrated for what ought to be a basic mode of operation; the rotations between the bright field "(SA)" and "diffraction" modes were unknown. For greatest accuracy, the individual researcher should probably check this rotation and the magnification for every day's running; however, it is time consuming to do so, as two specimen changes and several plates must be expended. A service instrument like the HVEM should have a current calibration of magnifications and image rotations for all conventional modes of operation, and this calibration should be "dated" by the range of plate numbers for which it is applicable. Otherwise, quantitative work is made much more tedious.
11. The HVEM is particularly easy to use once it is up and running. Al Philip-pedes is probably responsible for its excellent performance as far as alignment and reliability are concerned. However, we discovered a serious limitation for our purposes, and that is the long time required to pump down the camera after reloading. We would like to consume about 100 to 200 plates in a typical "productive" day; this would require up to three reloads.
12. Our productivity in terms of cells surveyed is presently further reduced by the lack of facilities for taking Polaroid photographs of the imaging screen.* We will look into this problem on our own; I believe that we can photograph the screen with a long focal length (4x5) press camera with Polaroid back and close-up lenses.

* These are needed so that we can identify which cells correspond to which SAD patterns; sketches take too long and are not very reliable.

I hereby request that you grant me the 100 hours (12 - 8 hour shifts) requested in my original proposal. I would prefer that the time be granted at least two days at a time and preferably three within a given work week. The first couple of visits should have a day or more of "rest" in between HVEM sessions so that we can collect our thoughts. Later, three days in a row would not be unreasonable, especially if we can be taught (and trusted) to shut down after the end of the normal working day (which is rather restrictive now).

I am making separate request for travel money from the Argonne Universities Association to supplement the present travel budget; the recently completed trip consumed almost \$600.

Yours truly,

George Langford
Associate Professor
Materials Engineering

GL/cc

References

1. G. Langford and M. Cohen, Microstructural Analysis by High-Voltage Electron Diffraction of Severely Drawn Iron Wires, Met. Trans. A, vol. 6A, 1975, p. 901.
2. M. D. Ball and P. L. Morris, The Use of STEM Microdiffraction for the Study of Misorientation Between Subgrains, Ninth International Congress on Electron Microscopy, Toronto, 1978, vol. 1, p. 622.

cc: Dr. R. O. Scattergood
Dr. D. B. Williams (Lehigh University)
Dr. S. M. Wolf (DOE)

Figure 1. Representative electron micrographs illustrating the relative utilities of two scanning transmission electron microscopes (STEM) vs. a conventional (high voltage) transmission electron microscope (HVEM) for the purpose of determining the orientations of crystallites in the cellular dislocation substructure of transverse sections of heavily drawn iron wire (true strain = 4.0).

- 1A. Philips 300 Retrofitted STEM (Lehigh University), 100 kV.
- B. JEOL 100 C STEM (Argonne National Laboratory), 100 kV.
- C. AEI HVEM (Argonne National Laboratory), 1000 kV.

Bright Field, STEM Photomicrograph, 40000 x

SAD, Cell 1, C.L. $\approx$ 2 m

SAD, Cell 2, C.L. $\approx$ 2 m

Figure 1A. Philips 300 Retrofitted STEM (Lehigh University), 100kV

Bright Field, STEM Photomicrograph, 30000 x

SAD, Cell 1, C. L. ~ 5 m

SAD, Cell 2, C. L. ~ 5 m

Figure 1B. JEOL 100 C STEM (Argonne National Laboratory), 100kV

Bright Field, HVEM Photomicrograph, 25000 x

SAD, Cell 1, C. L. = 0.8 m

SAD, Cell 2, C. L. = 0.8 m

Figure 1C. AEI HAVEM (Argonne National Laboratory), 1000 kv.

M. D. Ball and P. L. Morris

Alcan Laboratories Limited, Atlantic Region, Research Centre, Banbury, Oxon., England

While there have been studies of the relationship between misorientation in worked structures and the development of recrystallisation nuclei⁽¹⁻⁴⁾, most have lacked accuracy owing to the positional uncertainty of selected area diffraction. However the fine electron probe forming capability required for STEM operation enables diffraction information to be obtained from regions of the order of 20 nm or less in diameter. We have applied this technique to an examination of recrystallisation *in situ*, where the early stages of grain development are arrested by the presence of particles.

The microdiffraction studies were carried out on specimens prepared from an Al-6% Ni alloy which had been given various rolling and annealing treatments. A JEOL JEM 100C/ASID-4D electron microscope was used in the STEM mode with the operating conditions such that the spot size was about 20 nm with a beam divergence of 10^{-3} rad. For each of the specimens studied, a suitably thin region with a typical subgrain structure was selected and photographed. Microdiffraction patterns were obtained from each of the subgrains in this chosen area. It is interesting to note that despite the thinness of the areas studied, Kikuchi lines were strongly excited. This is believed to be a consequence of the diffuse scattering which occurs in the contamination marks which form during the recording of the patterns. The presence of this Kikuchi line information greatly increases the accuracy with which the sub-grain orientations can be determined. The contamination marks also serve to indicate the exact positions at which the diffraction patterns were obtained.

Figure 1 shows part of an area from which diffraction patterns were recorded. The contamination spots are numbered and the corresponding microdiffraction patterns are shown. For each diffraction pattern, the positions and orientations of the Kikuchi lines were measured and the results were analysed using a specially developed computer program. Using this program and applying self-consistency constraints it was possible to assign unique orientations to most of the subgrains in the areas of interest, and therefore to obtain unambiguous values for the misorientations between adjacent subgrains. Inevitably, however, there were some ambiguities which could not easily be resolved. For example, some of the diffraction patterns had insufficient information for orientation determination. In other cases patterns were recorded which could not be indexed in terms of a single subgrain, and therefore could be attributed to regions of subgrain overlap. This effect was more prevalent in the finer subgrain structures and limits this technique to areas of the specimen where the thickness is small compared to the subgrain size.

Some results of these investigations are presented in the form of histograms (Figure 2) in which the number of misorientation measurements falling within a given range has been plotted against the misorientation angle. In the as-rolled specimens the mean subgrain size was about 0.3 μm and the results obtained indicate that the misorientations were generally below 15° . In each of the histograms, the very low misorientations ($< 5^\circ$) are of limited value since they may in fact arise when two diffraction patterns are recorded from different parts of the same subgrain. Similarly the results may be distorted slightly by subgrains being overlooked during the microdiffraction. After the annealing treatments a much wider spread of misorientations was recorded. During the 250°C anneal the mean subgrain size was about 0.5 μm and misorientations up to about 45° were measured. Similarly, after the 400°C anneal, the mean subgrain size had increased to about 1 μm and misorientations up to 50° were measured. It was found that in almost every case, high misorientations could be associated with sub-boundaries which were pinned by the NiAl_3 particles. Boundaries which were not decorated by the NiAl_3 particles generally had low misorientations. The results presented here demonstrate that a high volume fraction of second-phase particles can inhibit recrystallisation by immobilising high-angle "subgrain" boundaries.

References

- (1) D. T. Gawne and G. T. Higgins. Textures in Research and Practice. Springer-Verlag, Berlin (1969), p.319.
- (2) R. K. Ray, W. B. Hutchinson and B. J. Duggan. Acta Met. 23, 831-840, (1975)
- (3) F. J. Humphreys. Acta Met. 25, 1323-1344 (1977)
- (4) J. W. H. G. Slakhorst and M. J. ten Bouhuijs, Scripta Met. 11, 947-952, (1977)

Figure 1. The substructure of the Al-6% Ni alloy after annealing for 1 h at 400°C. The microdiffraction patterns were obtained from the positions indicated by the numbered contamination marks.

Figure 2. Histograms showing the distribution of misorientations which were measured between adjacent subgrains

PROGRESS REPORT /

(TWENTY COPIES REQUIRED)

1. ARO PROPOSAL NUMBER: _____
2. PERIOD COVERED BY REPORT: July 1, 1979 - March 31, 1980-
3. TITLE OF PROPOSAL: Rapid Cycle Steel Casting

4. CONTRACT OR GRANT NUMBER: DAAG 29-77-C-0028
5. NAME OF INSTITUTION: Drexel University
6. AUTHOR(S) OF REPORT: D. Apelian, G. Langford
7. LIST OF MANUSCRIPTS SUBMITTED OR PUBLISHED UNDER ARO SPONSORSHIP DURING THIS PERIOD, INCLUDING JOURNAL REFERENCES:

A paper titled "Performance of a Porous Refractory Valve for Liquid Metals" has been accepted for publication in Metallurgical Transactions, B. A copy of this paper is enclosed. In addition, there are seven papers under preparation (continued on attached page).

8. SCIENTIFIC PERSONNEL SUPPORTED BY THIS PROJECT AND DEGREES AWARDED DURING THIS REPORTING PERIOD:

Dr. D. Apelian (up until December 31, 1979)
Dr. G. Langford (up until December 31, 1979)
Mr. M. Paliwal (up until March 31, 1980)
Mr. H. Sreshta (up until March 31, 1980)

A BRIEF OUTLINE OF RESEARCH FINDINGS IS GIVEN IN THE ATTACHED APPENDIX.

D. APELIAN & G. LANGFORD
DREXEL UNIVERSITY
PHILADELPHIA, PA 19134

15284-MS

7 (continued)

(i) Casting Manufacture by Diffusion Solidification, (ii) Rapid Heating of Unbonded Steel Powders and Porous Compacts, (iii) Design of Investment Molds for Casting by Diffusion Solidification, (iv) Partitioning of Third Elements During Diffusion Solidification in the Iron-Carbon System, (v) Solid-Liquid Interface Motion in Diffusion Solidification, (vi) Process Economics of Diffusion Solidification, (vii) Manufacture of Low Oxygen Atomized Shot.

Copies of these papers will be sent to ARO as they are prepared.

APPENDIX

The original DARPA proposal listed a series of twelve objectives to be completed so as to "develop the design of a rapid cycle casting machine" i.e., to enable a realistic analysis of the economic basis of diffusion-solidification casting as a processing principle.

These objectives and our progress in achieving them are listed below:

1. Measure the attainable rate of heating of low-carbon steel shot in a refractory mold.

This objective has been attained. Two direct methods have been proven feasible and their economic parameters established.

2. Establishment of the operating characteristics and reliability of the quick-acting valve for controlling liquid metal flow into the shot-filled mold.

This objective has been attained. A complete analysis of the particle valve has been made, together with fundamental experimental measurements of break-through pressures. The particle valve is in everyday use for infiltration measurements, inert gas atomization runs, and also for filtration of the melt. A classic paper on the particle valve is in press (Met Trans B.).

3. Development of a method of controlling mold permeability to waste gases, pressurizing gas, and liquid metal.

This objective has been attained qualitatively; quantitative work is in progress. Investment molds have been proven feasible.

4. Measurement of the tensile properties of partially frozen diffusion-solidified castings.

The "hot tensile test" method has been proven; we have obtained finite strength of diffusion-solidified steel castings at the casting temperature within five minutes of infiltration. Any intermediate test time is also possible; further work is in progress.

5. Demonstration of avoidance of hot-metal channeling in the shot during infiltration.

This objective has been attained by proving with decanting experiments that channelling does not occur when the liquid cast iron is at its liquidus temperature (i.e. saturated with respect to solid austenitic iron).

6. Check the validity of the theoretical expression for infiltration pressure.

Work to experimentally determine the infiltration equation is in progress; we routinely use the present theoretical expression successfully to predict operable combinations of shot size, casting depth, and infiltration pressure.

7. Work out the detailed techniques for making suitable non-permanent molds.

We have made our own investment molds successfully and made diffusion-solidified steel castings with them. Excellent surface finish and density have been obtained.

8. Establish the specifications and manufacture of permanent molds.

Several recent and process-simplifying revelations regarding the casting logic render this objective relatively unimportant; existing molding techniques such as the Shaw process phosphate bonded alumina or other refractories bonded with colloidal silica would probably be easily adapted.

9. Cast selected engineering parts by the diffusion-solidification process.

There has been no response from mission agencies to justify diversion of our limited manpower to production of a specific part despite repeated contacts. This objective is therefore deferred to later projects after these agencies make their needs known.

10. Development of the atomization technique and economic parameters.

We have demonstrated the range of utility of the Drexel inert gas atomization apparatus; the particle valve works routinely, about 30 runs have been made, 0.003% oxygen in the shot has been attained, and an adequate supply of high quality shot has been made.

11. Ductility-controlling variables

Decarburization of high-carbon commercial shot has been demonstrated with control of carbon activity in CO/CO₂ mixtures. This enables ductile castings to be obtained with \$0.20/lb. shot. The relationship between oxygen content and ductility of heat treated castings has been established. Further work on structure-sensitive ductility is in progress.

12. Failure Analysis

Scanning electron microscopy has been used to confirm the efficacy of solid-state vacuum-carbon deoxidation of the shot during heating to the casting temperature - such castings do not fail at the shot-melt interface. Fundamental studies of the origin of ductile fracture are in progress.

Several supplementary experiments conducted by senior thesis students are highly pertinent to this project; the following tasks are in progress:

- Heat treatment. One student is using two-dimensional diffusion-solidified welds in steel to study the heat-treat sensitivity of the last metal to freeze.
- Impact transition temperature. Another student is making massive (100 by 500 by 1 mm thick) welds from which U notch Charpy impact specimens can be made to study the impact behavior of the infiltrant, diffusion-enriched, and base metal zones in diffusion-solidified castings.
- Adiabatic solidification. A third student is precompacting loose shot to higher packing densities to increase the fraction solidified adiabatically from 75% to 100% in order to determine the resulting changes in infiltration rate and mechanical behavior.