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The Structural Dependence of Work Hardening in Low Carbon Steels

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(Ph.D. Thesis)

December 1991

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Prepared for the U.S. Department of Energy under Contract DE-AC03-76SF00098

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**The Structural Dependence of Work Hardening
in Low Carbon Steels**

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Ph.D. Dissertation

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This work was supported in part by the French government through the Genic Physique et Mécanique des Matériaux, INPG, Grenoble, France and by the Director, Office of Energy Research, Office of Basic Energy Science, Materials Science Division of the U.S.

Department of Energy under Contract No. DE-AC03-76F00098

MASTER

The Structural Dependence of Work Hardening in Low Carbon Steels

by

Patricia Elizabeth Johnson

Abstract

The influence of the dislocation cell structure on the work hardening behavior of low carbon steel sheets was investigated. Specimens were prestrained at low temperature to suppress cell formation and their subsequent behavior was compared with results of isothermal reference tests. It was found that the extent of cell development has little or no influence on the plastic behavior at room temperature and below. Interrupted temperature, tensile-shear tests demonstrated further that the transient behavior induced by loading path changes is also not strongly associated with the cell walls. In-situ straining studies indicate that the factor controlling the flow stress at room temperature is the limited mobility of screw dislocations moving in the cell interiors, and not dislocation interactions with the cell walls. The unique properties of $a/2\langle 111 \rangle$ screw dislocations are known to dominate low temperature deformation behavior in bcc metals. The current work indicates that these dislocations may still control the flow stress at intermediate temperatures, even in the presence of a developed cell structure.

Acknowledgements

The completion of this work would not have been possible without the extensive support I received from Professor Morris, my friend Dr. Dana Tribula, my mother Mary Zoeller, my brother Mark Johnson, and the members of my research group at LBL, particularly Dr. Jin Chan. I would also like to thank Professor Bernard Baudalet, Dr. Jean-Hubert Schmitt, Dr. Edgar Rauch, and the genial members of my host laboratory, GPM2, in Grenoble, France, whose input, support, and friendship made the last year of this research such a privilege, professionally and personally.

dedicated to
Amaelia, Paul, and Noah

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I. INTRODUCTION

In general, it is not yet possible to predict the behavior of a metal subjected to large plastic deformation by an appropriate description of the microstructural features which define the structure. Fundamental understanding of atomic scale behavior has developed immensely during the last two decades, yet the exceedingly complex problem still remains of expanding from the atomic scale to the macroscopic scale. Due to the multiplicity of effects, and the lack of a reference state, it is unlikely a "general" solution will ever exist in manageable form. A more realistic goal is the identification of deformation regimes in which the dominating structural features can be identified, and secondary or passive features ignored.

A common structural feature in plastically deformed metals is the formation of an extremely inhomogeneous arrangement of the dislocation population. Cell walls of high dislocation density delineate lower density cell interiors. Neither the cause of their formation nor their influence on behavior are thoroughly understood, and widely divergent opinions are present in the literature. Some consider the regular network of a developed cellular substructure as a recovery mechanism which necessarily influences the behavior, others see it as a passive feature of little consequence with respect to other structural developments.

At sufficiently high strain levels and temperatures, the cell walls may develop into subgrain boundaries. This development undoubtedly influences the behavior under specific conditions, for example during superplastic deformation, in which the deformation mechanism is controlled on the scale of the subgrain diameter. At the other extreme, early in the deformation, the structural dependence of the flow stress can often be adequately characterized simply by the total dislocation density and not its arrangement. In this case, the dominating mechanism occurs on a scale roughly equal to the average dislocation spacing.

However, metals are often subjected to deformations between these two extremes and it is unclear which mechanisms control, or in what manner they are mutually dependent. Therefore, it is of interest to clarify the role of the arrangement of the dislocation population during moderate (less than 100%) plastic straining at intermediate ($< 0.2 T_m$) temperatures. Many of the currently accepted models on plasticity hold that the flow stress is uniquely determined by the development of the cell walls. This work challenges the generality of these models by isolating the separate influences of cell formation and other strain hardening mechanisms.

During plastic deformation, the development of a cellular substructure occurs simultaneously with other structural developments. Any entirely monotonic loading path is therefore insufficient for monitoring the influence of cell wall formation, as the effect of the walls alone cannot be isolated. In bcc metals, the arrangement of the dislocation population can be substantially altered by changing the deformation temperature within a particular temperature range. Thus, the formation of the cell walls can be controlled by varying the temperature during an initial straining stage. If deformation is then continued at a reference temperature and strain rate, the behaviors may be compared between two structures with similar extents of work hardening, but dissimilar extents of cell formation, when both are subsequently deformed at a fixed set of conditions.

Specialized mechanical tests are necessary to allow the behavior to be studied out to reasonably high strain levels, beyond the necking strains of conventional tensile tests. This can be accomplished by performing the mechanical tests in pure shear, for which purpose it is most convenient to use the material in sheet form.

Low carbon, aluminum killed steel sheets are used in large quantities by the automotive industry as stampings for body panels. These sheets are cold rolled and annealed, polycrystalline α (bcc)-Fe. The temperature dependence of cell formation in these steels occurs near room temperature, which makes a large prestrain and secondary test matrix feasible for the present investigation.

To predict successful stamping operations, the automotive industry typically uses simplified constitutive equations which tend not to be based on knowledge of the structure during the deformation path of interest. When changes are made in the material or in the stamping, expensive failures often occur. Thus, due to their ability to provide an appropriate test material, as well as their great technological interest, low carbon steel sheets were selected for this investigation on the influence of dislocation cell formation.

To further understand the role of cell development in these steels, specimens were also prestrained at different temperatures to develop different initial substructures, and then subjected to a change in the loading path. It has been established that isothermal loading path changes in this material induce transient behavior during which the structure rearranges toward a new structure more appropriate to the second loading conditions. It is unclear to what extent the presence of a cell structure modifies the response to a loading path change, or on what scale the evolution of the structure influences the flow stress during the transient. Two loading path sequences were used for this purpose, each of which involve very different transient responses.

OBJECTIVE

This work investigates the influence on work hardening of a cellular dislocation substructure in low carbon steel at room temperature. Although the main motivation for this study was to isolate the role of dislocation cell formation in bcc metals at intermediate temperatures, the work was performed on a technologically interesting material, subjected to the deformation conditions in which it is commonly used. As such, the results are directly applicable to the development of structurally based constitutive equations in low carbon steel sheets, and of more general interest as it addresses a major ambiguity in the structural dependence of plastic behavior.

II. BACKGROUND

II. A LT bcc behavior

It is now generally accepted [2,8,72] that the plastic behavior of bcc metals at sufficiently low temperatures is determined by the unique properties of $a/2\langle 111 \rangle$ screw dislocations, whose low energy configuration is sessile. The behavior of these screws is the cause of one of the most distinctive properties of bcc metals, the strong temperature dependence of the flow stress below a critical temperature.

Several investigators [5,12,13,40,89,90,98,104,105] have used atomistic studies to model the atomic displacements around screw dislocations in various bcc transition metals. Although the detailed behavior is in most cases strongly sensitive to the assumed interatomic potentials used in the model, these studies are in agreement that the relaxed core of a $1/2\langle 111 \rangle$ screw dislocation in a bcc lattice is spread out onto three $\{110\}$ planes, and that each of these partial dislocations may further dissociate onto several $\{112\}$ planes.

The spatial extent of the dissociation is quite small, on the order of the Burger's vector, and direct observation of the dissociation is not possible with conventional methods. It is for this reason that the bulk of our current understanding on the core behavior under an applied stress is derived from computer simulations of atomic scale models. The credibility of these models has been strengthened by their successful ability to predict peculiarities in the plastic behavior of bcc metals. These include complicated and dissimilar orientational dependences among transition metals, and the strong temperature sensitivity commonly observed over certain temperature ranges.

Since the dissociated core is nonplanar, motion of the screws under an applied stress requires first constriction into a higher energy configuration, and the dissociation thus raises the flow stress. The flow stress is strongly temperature sensitive because the recombination is associated with only small atomic rearrangements, and can be assisted by thermal fluctuations. Above a critical temperature, which depends on the material but is

roughly 10-20% of the melting temperature, the constriction is easily accomplished. As it no longer controls the behavior, the flow stress becomes less temperature sensitive.

Edge dislocations in a bcc lattice do not dissociate in this way, and at low temperatures the mobility of edge dislocations is much greater than the mobility of screws. At the onset of plastic deformation, preexisting edge segments move very rapidly, become trapped at hard spots or grain boundaries, and leave behind the more or less homogeneous distribution of long straight screw segments which form the typical structure observed in bcc metals and alloys at low temperatures. Very little plastic strain is accomplished by the motion of the free edges initially present, and their supply is exhausted in the microstrain region [8,50,68,70,72,94].

Because of their limited mobility, the screws tend to remain straight, and move by the nucleation and propagation of double kinks. The kink segments are pure edge, and move with very little resistance, until they become pinned, at the dominant pinning centers (forest dislocations, precipitates and solute clusters, grain boundaries). The rate of successful kink nucleation is controlled by the current temperature, applied stress, and the length of mobile dislocation segments. The amount of strain accomplished by each nucleation event depends on the spacing of the pinning centers. In a typical application, this statement should be inverted: a deformation rate is imposed at a given temperature, and the flow stress to accomplish the imposed deformation is the stress level required to cause sufficient double kink nucleation and motion, which in turn depends on the density and distribution of mobile dislocations and pinning sites.

Although these same structural features, (the density and distribution of mobile dislocations and pinning sites) should influence work hardening at higher temperatures, the dependence on the current structure and temperature should change when the controlling mechanism no longer occurs on the nearly atomic scale associated with the individual motion of screws. At higher temperatures, the edge and screw mobilities are comparable,

recovery allows more rapid evolution of the structure, and larger scale structural features may influence the flow stress.

II.B Intermediate temperatures

At low temperatures the role of screws is dominant in bcc metals, and at sufficiently high temperatures, their unique influence is lost. In the temperature range between these two extremes however, the role of the screws is not as obvious, particularly with respect to the role of the arrangement of the dislocation population, since intermediate temperatures also mark the onset of nonuniform, cellular dislocation substructures.

Intermediate temperatures are defined here as the temperature range corresponding to the "knee" in a temperature versus yield or flow stress plot, where the flow stress is becoming less temperature sensitive, but is not yet representative of high temperature behavior. At these temperatures substantial motion of edge dislocations is observed, but the mobility of the edge segments is still much greater than that of screws [68,72]. Because of their dissimilar velocities, the dislocation segments at intermediate temperatures tend to be unmixed. The majority of the dislocations exist as either pure edge or pure screw, as at low temperatures, but now with larger edge segments present instead of only inter-atomic scale kinks.

It is possible that the flow stress may still be controlled by screw mobility even when a substantial amount of strain is accomplished by the motion of edges. If the movement of edge dislocations is coupled to the restricted movement of screws, the behavior of the screws remains the limiting step in the deformation process, and the screws retain their controlling influence. Compared to the behavior at low temperature, the flow stress is lowered at intermediate temperatures by the relative ease of constricting the screws, and an increased probability for kink nucleation, but the dominant mechanism is essentially the same.

The alternative possibility is that the mechanism controlling deformation at intermediate temperatures is distinctly different than at low temperatures. This would occur if the presence of the edges provided a deformation path independent of the screws, or if the formation of a cellular substructure substantially altered the behavior. It is the opinion of many researchers that the presence of dislocation cell walls necessarily influences the flow stress.

II.C. Dislocation Cell Structures

Provided the deformation temperature is not too low, the arrangement of the dislocation population of most metals is extremely inhomogeneous at strains above 5 or 10%. Sheets, braids, or "walls" of high dislocation density separate lower density cell interiors. As the deformation is continued, the dislocation density not only increases, its distribution becomes increasingly inhomogeneous. Tangles develop into sheets, the sheets become more well defined, more numerous, more uniform in size and shape. Reactions in the densely tangled walls can lead to a reduction in the thickness of the walls, which may eventually develop into subgrain boundaries.

Both the cause and influence of cell development is still a subject of much controversy, and a sampling of the diversity of opinions on the subject is presented in section II.C.2 and II.C.3. However, several experimental facts have consistently emerged regarding cell formation and its dependence on deformation conditions, these trends are reviewed first.

II.C.1 Dislocation Cell Formation -Experimental Observations

The average spacing between adjacent dislocation cell walls tends to decrease during deformation, at least over a certain strain range. A decrease in the average cell size

has been verified in nearly every effort at quantifying cell formation, in bcc as well as fcc metals, and for monotonic as well as cyclic loading [4,7,32,33,36-39,51-57,66,71]. Although this general trend is clear, several ambiguities in this type of measurements should be mentioned.

In a well developed cell structure, the shape of the dislocation cells tends to imitate the macroscopically imposed strain. Elongated cells develop during tensile straining, and more equiaxed cells develop during biaxial stretching. The shape of the cells for different types of loading has, for example, been studied in low carbon steel sheets by Korbel, Martin, Rauch, and Schmitt.

During most of their development, the size and shape of the cells are far from uniform and a large scatter in the measurements strongly smears out the overall trend. The cells begin as diffuse, randomly distributed tangles, and it is an arbitrary choice at what point the structure is described as cellular, or what the initial cell shape and wall thickness are. Further on in the deformation the cells become more clearly defined, and their shape begins to imitate the macroscopic shape change. Cell size measurements in general, and particularly comparisons for different loading paths, are complicated by the choice of which length, or average, is to be used when the cells are not equiaxed, and on which sections the measurements should be made.

Of particular interest is the point at which the average cell size becomes saturated, and whether this point occurs before work hardening has saturated. Within reasonable experimental uncertainty, different conclusions on the saturation point can easily be reached, even from the same data set. According to some, the cell size decreases monotonically, with no saturation before failure, others see the cell size saturate early in the deformation. These have distinctly different implications on the role of the cell structure during deformation.

The tendency for a cellular substructure to become more regular in shape and size as the deformation is continued, and the decrease in the *average* cell size implies that the

cell walls are rearranging as they form. The location and orientation of a particular cell wall therefore does not necessarily remain the same throughout its formation. Within a grain interior, cell walls are found to lie along different directions in different regions of the same grain, with, for example, one set of orientations near a grain boundary, and another set in the grain interior. Thus the development of the cell structure appears to occur in a continuous but rather complex manner, perhaps in response to the changing local stress field.

Schmitt and Rauch [84,85], and Ledezma [62] have independently analyzed cell wall habit planes in low carbon steel sheets for various loading paths. The work included three dimension analysis using cross-sectional TEM foils, and both the Sach's and Taylor models were used for predicting active slip planes in the polycrystalline sheets. The conclusion was made in both studies that, at least for some monotonic loading paths, the cell walls tend to lie on, or near, the primary slip planes.

If the limitations of the models used are taken into account, it is probably safe to assume generally that the cell walls lie near an active slip plane during monotonic loading. Since the development of a cellular structure appears to occur similarly for different monotonic loading paths, it is unlikely that a tendency for crystallographic habit planes would be clearly demonstrated in one case and not apply to another. The Schmitt and Ledezma results may be more general than is indicated by the data, if the cases which do not fit the trend are merely cases in which it is not possible to accurately predict a primary slip system.

However, it has been found that during cyclic loading, the cell walls tend to lie perpendicular to the active slip planes, instead of along them as in monotonic loading. This has been observed in aluminum alloys as well as steels, during steady state fatigue. It is therefore difficult to develop a cell formation model consistent with both cases, and the implications of a crystallographic habit plane are difficult to interpret.

It has been observed in bcc metals that the extent of cell formation at a given strain is strongly temperature dependent in certain temperature ranges, with higher temperatures promoting cell development [33,36-39,73-75,103]. Although designation of a structure as cellular or not is often subjective, distinctly dissimilar substructures have been observed over rather narrow temperature ranges.

The earliest major work for iron was done by Keh and Weissmann [37], who studied cell development in nearly pure iron single crystals. An attempt at quantifying cell formation from TEM micrographs was made by counting the grains in which substantial cell formation was seen as a function of strain and temperature. The structure was considered cellular, for example, after a 10% tensile strain at room temperature, but considerably less cellular at -100 °C. Vincent [103] shows similar results in low carbon steels, with a cellular structure present at 10% at room temperature, and not at all developed when the same strain is performed at -100 °C.

The temperature dependence of cell formation in a given bcc material can be substantially shifted to lower temperatures by the addition of solutes. The cell structure at room temperature in Fe-3Si single crystals [86] is less developed at a given strain than that observed, for example, in pure iron [37]. Similarly, Luft and co-workers have observed a strong suppression of cell formation when solutes are added to Mo single and polycrystals [74]. In that case, the majority of the solute content appeared to be contained in small coherent precipitates.

The manner in which the addition of solutes or precipitates impede or retard the formation of dislocation cell walls is not yet clear, and it is difficult to claim that the structures formed are actually "equivalent" to those formed, for example, at lower temperatures. However, the observations are unambiguous in that the appearance of the structure is qualitatively less cellular as the purity of the material is lowered, given the same deformation conditions (strain, loading path, temperature).

Higher melting point bcc metals tend to have the temperature dependence of cell formation shifted to higher temperatures. Extensive work has been done on molybdenum and niobium, single and polycrystals, by Luft and coworkers, Louchet, Christian, and others. A moderate ($\cong 10\%$) room temperature strain results in a typical low temperature (noncellular) structure, and cell formation is well established at higher temperatures (200 °C and above).

There is evidence that at a given temperature, the tendency for cell formation also depends on the type of dislocations present, and is apparently not simply an increasing function of the total dislocation density. Cell walls appear to be composed predominantly of edges [64], according to observations made, for example, in molybdenum by Mughrabi, and in variable temperature in-situ testing of niobium and iron by Louchet.

Comprehensive, direct observation of cell formation by in situ deformation in TEM is restricted by the influence of surface effects (a typical cell diameter is about the thickness of a conventional TEM foil) or, in thicker films, by irradiation damage at the higher operating voltages required for visibility [72]. Bcc materials offer a good compromise between these limitations since their flow stresses tend to be high, which reduces the relative influence of surface effects, and their damage threshold voltages not too low, allowing reasonably thick foils to be observed. Perhaps the most successful of these experiments are those in which only incremental changes in the cell structure are observed, following a prestrain with the material in bulk form.

In an in-situ study of Fe single crystals, Ikeda [28] finds that orientations which allowed substantial motion of edge dislocations resulted in more extensive cell development than orientations which caused predominantly screw motion. This was observed even for cases in which the total density of screws was much larger than the edge density. However, since changes in the orientation also change the flow stress, it is not clear from this study whether the tendency for cell formation was modified by the presence of the edges, or is itself a function of the stress level, or both.

Generally, any conditions which promote the use of multiple, non coplanar slip systems appear to enhance the formation of a cellular dislocation substructure. This has been verified by studying the deformation substructure of single crystals which were deformed in single slip and multiple slip orientations. Isolation or quantification of this trend is virtually impossible, since, again, the stress level is necessarily also altered by the orientation, and its potential role in cell formation cannot be assumed. Furthermore, as cross slip is easily initiated in bcc materials under some conditions, specimens nominally oriented for single slip may in fact deform on more than one slip system.

The distribution of internal stresses in cellular structures has been investigated by measuring the curvature of dislocations approaching the cell wall boundaries [79,80]. This data supports the claim that a polarity exists across the walls, caused by the building up of oppositely signed dislocations on opposite sides of the wall. This result is in keeping with some models on cell formation. The internal stresses associated with this polarity are thought by many to alter the behavior and, in particular, to be the major cause of the observed lowering of the flow stress following a stress reversal (Bauschinger effect).

II.C.2 Cell Formation-Models

Since the tendency for cell formation increases with strain at a given temperature, and increases with temperature at a given strain, many investigators have assumed that the formation of a cellular dislocation substructure is a dynamic recovery mechanism which allows reduction of the elastic strain energy during large deformations [51-55,58,64-66,80]. This is the physical basis of several models. Conversely, other investigators consider cell formation as a natural consequence of the deformation process itself, which is intimately related to the stress level, but not necessarily the result of a rearrangement which provides substantial recovery.

In the model supported by Kocks, for example, the cell structure is the result of the eventual linking up of hard regions which are initially present and grow during the deformation by the trapping or looping of mobile dislocations [41,42]. The structure observed in a typical TEM micrograph is misleading in that it only presents the structure left behind by the passage of the most recent dislocations. The implication is that the structure is in fact far more "open" during the deformation than a typical micrograph would indicate, and that dislocations are able to percolate through the connected soft regions of an open structure. The structure merely becomes increasingly more plugged up in the course of the deformation, and at some point could be described as cellular.

At perhaps the opposite extreme are the energy minimization models, such as those presented by Kuhlmann-Wildorf, and others. In this school of thinking, a cellular substructure is formed because it allows the dislocations to screen each others strain fields, and therefore provides an effective means of reducing the total strain energy. The long range internal energy of pile-ups is thought to be reduced by cell formation at the expense of an increase in short range energy resulting from closer dislocation-dislocation interactions. The periodicity (cell size) is selected by the material because this arrangement provides the lowest energy configuration given the current dislocation density.

The development of the Kuhlmann-Wildorf model involves the use of several empirically determined parameters, and only the physical reasoning will be described here. The structure is thought to remain geometrically "self-similar" throughout the deformation, changing only in scale, as the cell size decreases. The internal energy is written as a multiple of the elastic energy of an isolated dislocation, with the cell diameter used as the relevant size of the crystal. This is justified by the expectation that dislocations moving in the cell interior can no longer move when a cell wall is reached. Cell wall interactions thus control the flow stress in this model.

It is then claimed that the total strain energy is minimized by an agglomeration of the dislocations into the cell walls. The dislocation density scales with the strain, and the

current average cell size is determined by energy minimization for that strain level. This is thought to explain the experimentally observed decrease in cell size with deformation. The details of the behavior are worked out for a typical fcc material, but the basics of the model are claimed to be generally applicable.

Qualitatively, however, the structure does not necessarily appear to remain self-similar throughout the deformation, even for a given loading path. As described, the size, shape and habit plane of the initially diffuse tangles may change during the formation of the walls. Furthermore, this model stipulates that the flow stress is determined by interactions with the cell walls. Work hardening can only occur with continuously decreasing cell size in this model, yet the experimental evidence supporting this is weak.

A variation of this type of model is given by Mughrabi. The cell walls are composed of clusters of edge dipoles and multipoles which lie in close proximity to screen each others strain fields. Screw dislocations are eliminated by the annihilation of closely spaced pairs of screw dipoles, which are able to cross slip. The dislocations which form the walls contribute no long range stresses, but dislocations at the interface between the cell walls and cell interiors are required for compatibility in this model, and these interface dislocations cause a polarized structure with long range internal stresses.

Another type of analysis to explain cell formation involves writing balance equations which describe the coupled evolutions of the mobile and forest dislocation densities. These types of models have been reviewed for example, by Kubin. The governing coupled differential equations are solved for stable solutions which will predict a periodicity in the structure. Assumptions on the behavior of the controlling mechanisms are made, and the evolution is then typically modeled by computer simulation.

In the simplest case only the two dislocation populations are considered. Mobile dislocations become trapped, and as the stress level is increased, previously immobile forest dislocations are freed. Source and annihilation terms are included, as well as the

relevant lengths over which the governing interactions occur, and the evolution and rate of exchange between the mobile and forest populations are thereby described.

Some of these models have successfully predicted realistic periodic structures, but usually only after the appropriate selection of one or more free variables, whose value is difficult to justify on a fundamental basis. A physically appealing aspect of this type of model is that the behavior during deformation is treated as a dynamic process, rather than modeling the structure during deformation as a closed system seeking energy minimization. On the other hand there is perhaps no physical basis to assume that the scaling laws and evolution behavior should maintain the same form throughout the deformation, and a comprehensive accounting of all potentially relevant trapping, source, and evolution terms would be unreasonably unwieldy. Structurally based constitutive modeling of plastic behavior to include cellular structures has been analytically treated for example by Mecking and Estrin [76].

In an analysis proposed by Kratchovil [45,46] the mobile dislocation population is composed only of dipoles, which are extended or contracted, depending on their local stresses. The local stresses are modified by the applied stress, and also by the internal stresses associated with the arrangement of the dislocation population. He then considers the response to a hypothetical, internal stress distribution which is spatially periodic. The population of dislocation dipoles is now considered to be distributed throughout a periodic stress distribution, with all the dipoles now seeing a local stress gradient, which causes them to move.

In Kratchovil's model, an initially homogeneous distribution of dipoles is unstable to a particular periodic stress distribution. The motion of the dipoles in response to this distribution of local stresses causes the building up of an increasingly inhomogeneous structure with that periodicity, and the dislocation population becomes cellular. Although extended and contracted dipoles would indeed be expected to move up and down local stress gradients in the presence of such a stress distribution, is it not clear how their motion

would build up this distribution rather than deplete it. More generally, the initial assumption of a flow stress uniquely dominated by the motion of dislocation dipoles is perhaps too restrictive to explain the nearly universal observation of cell formation in most metals.

II.2.3 The Influence of Dislocation Cell Walls

Since the formation of cells is such a distinctive microstructural feature, and dislocations are known to respond to each other's stress fields, it may reasonably be expected that the presence of dislocation cell walls should modify the deformation process, and thereby influence the flow stress. Conversely, a cellular structure may merely be a passive feature of the deformation process. Any controlling mechanism which was independent of, or not restricted by, the presence of the walls would render them ineffective, and the walls themselves would have no influence. Both opinions are represented in the literature. They are briefly reviewed here, and analyzed more thoroughly in the discussion section.

In a model such as Kock's, the hard regions (precipitates or solute clusters, dislocation tangles and intersections) tend to become larger and stronger during the deformation, by the trapping of mobile dislocation segments. Passage of mobile dislocations through the soft regions becomes increasingly more difficult and work hardening results. This occurs in a continuous manner, regardless of whether or not the hard regions have yet connected to form cell walls. The cell structure itself plays no active role, in that it does not control the process, but is rather just a more developed version of the original structure. The extent of work hardening, and therefore the current structure, is best characterized by the total dislocation density, as this describes, on average, the resistance seen by the mobile dislocation population. Barring the presence of substantial

recovery mechanisms, the macroscopic flow stress is simply proportional to the square root of the dislocation density for monotonic loading in a typical material.

A necessary consequence of Kock's model, is that the walls are built up of dislocations of one sign on one side of the wall, and of opposite sign on the other side, causing a polarized structure, an orientation mismatch as a cell wall is crossed [41]. Within the wall itself, an even larger mismatch should be present, which would not necessarily influence the flow stress, but could modify the recrystallization behavior of the material, by providing preferential nucleation sites.

According to the Kuhlmann-Wilsdorf model, the cell structure uniquely controls the flow stress. The relevant "free length" of mobile dislocations is considered to be the cell diameter, or transverse length. The flow stress is then found to be proportional to the inverse of the cell size, which decreases during the deformation, and is thus responsible for work hardening.

The overwhelming majority of the dislocation population is assumed to be contained in the cell walls, and the cell interiors are considered virtually devoid of dislocations. Loops expand freely out to a critical radius, becoming pinned when the cell wall is reached. The "mesh length" associated with this critical radius is thus related to the current average cell size, and only the scale of the structure is reduced during deformation, in a geometrically "self-similar" manner. An extension of the model relates the cell size to the reciprocal square root of the total dislocation density [27], resulting in a flow stress proportional to the square root of the dislocation density, as found in other models.

The apparently bimodal distribution of dislocations, with a high density in the walls and a much lower density in the cell interiors, has led to the proposal of "two-phase" models to describe the behavior of cellular structures. The cell walls and cell interiors are considered to act as hard and soft "phases", respectively, and the structure during deformation is treated as a composite.

In the two phase model given by Mughrabi [78-79], the two "phases" are loaded in parallel, and therefore accommodate the same strain. The case of cyclic loading at saturation in an fcc material is presented, but the model is held to be qualitatively valid also for monotonic loading of any cellular structure. The cells are assumed to have already developed, and the response of both "phases" is perfectly elastic-plastic, with dissimilar yield points and flow stresses. As the structure is loaded, yielding occurs first in the cell interiors, while the walls continue to deform elastically, until their yield stress is reached. At this point the deformation becomes fully plastic, and occurs at a macroscopic flow stress equal to the flow stress of the "hard" walls.

During unloading, the dissimilar elastic strains in the walls and cell interiors result in a misfit at the boundaries. This misfit is accommodated by interface dislocations which maintain continuity. The presence of these interface dislocations is thought to contribute substantial long range stresses which influence the behavior, particularly during a stress reversal.

II.D Changes in the Deformation Path

Intentional, discontinuous changes in the deformation conditions are often used to isolate the influence of interacting effects. Strain rate sensitivity measurements using "jump" tests in which the strain rate is rapidly changed during the deformation is a common example. In this case, the development of structural features which may affect the stress level is held constant by fixing the pre-jump conditions, and the influence of strain rate alone can be isolated, at least for that structure. Here the implicit "reference state", which respect to which the measurements are made, is rather unambiguously defined, as the structure just before the change in strain rate.

In general, the choice of a reference state during plasticity is more difficult, and this complicates the interpretation of deformation path changes. Even when only the relative

influence of two paths are being monitored, the most appropriate means of comparison is often unclear. A method which has been used to determine whether the development of the structure can be adequately characterized by a single parameter following different deformation paths is described in this section, and path changes which are of interest to this study are presented.

II.D.1 Single Structure Parameter Models

A given material responds to a given set of deformation conditions in a manner which is determined by its structure, and perhaps, by the rate of evolution of the structure. The truth of this statement becomes obvious if the structure is described by the position, type, velocity, and forces acting on, each atom in the material. If the plastic behavior was well enough understood, the appropriate averages would be used instead, and the structure could be completely described by the smallest set of relevant structural features: lattice, grain size, texture, cell size, dislocation densities and distributions. If the deformation conditions were then specified (strain rate, temperature, geometry, loading), the subsequent behavior could be predicted, at least in principle.

This approach has lead several researchers to propose the existence of a mechanical equation of state in which a set of structural features and deformation conditions define the current state of the material, independent of the manner in which this state was arrived at. In the most simplified version of a mechanical equation of state, all the relevant features of the structure are described by the value of one scalar parameter ("S"), and the flow stress may be written as

$$\hat{\sigma} = \hat{\sigma}(\dot{\epsilon}, T, S) \quad (1)$$

A single structure parameter model does not require that only one structural feature be evolving, but it does imply that only one independent structural feature is controlling the

flow stress. All other developments of the structure either have no influence, or their evolution is dependent on the evolution of the single controlling feature.

If the strain rate and temperature are fixed, equation (1) may be inverted to give

$$\hat{S}|_{T,\dot{\epsilon}} = \hat{S}(\sigma) \quad (2)$$

and each stress level corresponds to a unique plastic state. If two specimens with different histories are subsequently deformed at the same temperature and strain rate, and compared from the same initial stress level, they will behave similarly if a single structural parameter model is valid. The structure, or state, is in a one to one correspondence with the stress and they evolve together in a manner determined by the strain rate and temperature. The converse is also true. If the two specimens described above do not behave similarly, it indicates that more than one structural feature is controlling the behavior.

This method may therefore be useful in analyzing the influence of deformation path changes in which the first path is varied during prestraining, and the deformation then continued at a common set of conditions. The appropriate comparison is then made from any common stress level obtained during the second path. Either the specimens given dissimilar prestrains subsequently behave similarly from a given stress level (a single parameter is adequate for describing the structure), or they don't (more than one structural parameter is needed).

Since the evolution of the structure is presumably different using different prestrain conditions, a common stress level will not necessarily be obtained at the same amount of strain along the second deformation path. To test for single parameter behavior then, the origin of a common strain axis should be set where the common stress level is reached during the second deformation path. Similar behavior from this point on indicates control by a single independent structural feature, but of course does not indicate what that structural feature is.

At the suggestion of Kocks, this method was used by Vincent in a study on the influence of dislocation cell structures in low carbon sheets [103]. Single parameter

behavior was observed for monotonic tensile loading, subsequent to a temperature change. This will be described more fully in the Discussion section, as the surprising results of Vincent's study were a major driving force for the current work.

A great deal of evidence to support this type of model has also been generated by Hart and coworkers [18-22] for monotonic loading in stress relaxation tests. These studies were done on a wide variety of fcc and bcc materials, including some precipitation hardened alloys. Very little strain is involved during the stress relaxation, and the tests generate stress and strain rate data taken at a constant structure:

$$\sigma|_{T,S} = \sigma(\dot{\epsilon}) \quad (3)$$

A series of tests were done at different structures, by varying the prestrain (or initial stress) level. It was found that a master curve for each material, at room temperature, could be generated by combining the curves. Since changes in the structure only changed the initial position on the master curve, the relevant development of the structure may be described by a single parameter.

In spite of its apparent oversimplicity, single parameter models are quite common. The Hall-Petch relationship relates the yield stress only to the grain size. Plastic behavior in which the flow stress is determined only by the overall dislocation density is another example. In the Kuhlman-Wilsdorf model described earlier, the only structurally relevant feature is the cell size. In fact, many researchers argue that in the absence of transient behavior associated with complex loading, single parameter behavior is usually expected.

II.D.2 Loading Path Changes

Following a loading path change, the favorably oriented slip systems are changed in a discontinuous manner, and mobile dislocations moving on the newly active systems are forced to interact with the work hardened prestrain structure. Loading path changes can therefore be used to uncover the structural dependence of work hardening by monitoring

the influence of these interactions. Isothermal, room temperature loading path changes in low carbon steel sheets have been studied extensively by Schmitt, and Rauch and coworkers [82-85, 15]. Loading paths from "hard" orthogonal path changes to "pseudo-Bauschinger" path reversals were studied at various prestrain levels.

These path changes were accomplished by a series of tensile and tensile-shear loading sequences. The flow stress following a loading path change was measured and compared to the flow stress of monotonically loaded reference specimens. A parameter was defined to characterize the magnitude of the loading path change and predict the subsequent stress level with respect to the monotonic reference curves. As expected, stress reversals resulted in a decrease of the subsequent flow stress, and orthogonal path changes raised the flow stress.

When an accounting was made of the expected influence of texture development during the deformation sequences imposed, it was found that the development of the crystallographic texture did not account for the observed behavior. The response was therefore determined to be due in part to some development of the intragranular structure formed during prestraining. It is not clear however, whether the relevant development was the formation of the cell walls during prestraining, the formation of a work hardened structure in the cell interiors, or a combination of both.

The transient behavior induced by a loading path change was also studied. Following a change in the loading path, the cellular prestrain structure rapidly evolves to a structure more appropriate to the second loading direction. It was observed that the previous structure is effectively "erased" by straining along the second direction an amount roughly equivalent to the prestrain amount. During this rearrangement, the stress strain curve shows transient behavior which depends on the prestrain level, and the magnitude of the path change.

Of particular interest is the structural dependence of the flow stress following a stress reversal. The lowering of the flow stress after a reversal was in many cases long

term, in the sense that the stress strain behavior did not approach the monotonic curve until well away from the point at which the loading path was altered. It has been proposed that the structural dependence of this Bauschinger effect may lie in the formation of a polarized cell structure during prestraining, and that a partial "unknitting" of the walls may provide an easy deformation path, which lowers the flow stress.

The orthogonal loading paths are essentially latent hardening experiments, but performed for the case of polycrystals at large plastic strains. For sufficiently large prestrain levels, it was found that the initially increased flow stress was followed by structural softening, seen as a plateau or a drop in the stress-strain curves. Since the second path was accomplished in shear [16], geometric softening was avoided, and a decrease in work hardening could be monitored over large strain increments without the onset of necking.

This softening has been identified with the formation of microbands in which the strain may be concentrated. As straining is continued, the microbands lose their role as paths of easy deformation, and work hardening is again observed. The role of the preexisting cell walls in the nucleation, propagation, and subsequent hardening of these microbands is under further investigation.

A similar, parallel study has been done by Rauch and co-workers on the response to a loading path change as a function of the development of the structure and type of loading in single crystals of Fe-3Si. In this case, slip system activity can be more adequately controlled by the choice of orientation during loading, and the habit plane of the microbands identified. During the portion of the tests in which a load drop occurred following an orthogonal loading path change, the observed microbands were easily identified as "alien" to the prestrain structure, but it was not clear to what extent they were responsible for structural softening in the form of strain localization. Equally perplexing is the observation that further in the deformation, when work hardening has again returned,

these microbands cannot be distinguished from the newly formed cell sheets (those which formed in a direction expected for the second loading).

The magnitude of the effect of a given path change on the stress level is an increasing function of the prestrain amount. Although cell formation is also more developed as the prestrain is increased, it cannot necessarily be concluded that the cell walls are responsible, since the extent of work hardening was not isolated from the extent of cell formation. It is for this reason that the current work was undertaken, in corroboration with Schmitt, Rauch and Baudalet at the GPM2 Laboratory at INPG in Grenoble, France. The equipment, and the techniques developed for the studies described above were used in the present work, where changes in the prestrain temperatures were also incorporated, to control the development of the prestrain structures, at the point of the loading path change.

II.D.3 Temperature Changes

The dependence of cell formation on temperature is exploited in this work to study the influence of an established cell structure. Specimens were prestrained at dissimilar temperatures to develop dissimilar extents of cell formation and the subsequent behaviors were compared. The potential information of this type of test sequence is described here.

Suppose a specimen is pre-strained at a given temperature, the temperature abruptly changed, and the deformation continued at a second temperature. To isolate the influence of the prestrain structure, the behavior is then compared with specimens strained entirely at the first temperature or entirely at the second temperature. If only the deformation temperature dictated the behavior, the prestrained specimen would be expected to behave after the temperature change as though it had been deformed continuously at the second temperature, and the behavior would be insensitive to the choice of prestrain temperatures. At the other extreme, if dissimilar developments of the structure during prestraining dictated the subsequent behavior irrespective of the temperature, the prestrained specimen

would be expected, at least initially, to continue as though deformed at the first temperature, even after a temperature change. The actual behavior should fall somewhere between these two extremes, with neither a purely structural nor purely thermal dependence expected.

This is essentially the reasoning used in the analysis of Cottrell-Stokes behavior, first observed in aluminum single crystals in 1955 [10]. As in the comparison described above, "thermal" or "short-range" structural dependence is measured by the extent to which a prestrained specimen follows the work hardening behavior characteristically observed at the second temperature (i.e. without the temperature change). This contribution to the flow stress arises from structural barriers to dislocation motion which can easily be overcome by thermal activation.

Insensitivity to the temperature change is measured by the extent to which the interrupted specimen continues to behave as though deformation were still occurring at the first temperature. This insensitivity is generally attributed to the development of "athermal", or "long range" structural features whose behavior is relatively unaltered by the deformation temperature. Barriers of this type are mainly overcome by raising the applied stress.

Although it appears that bcc metals do not show Cottrell-Stokes behavior (a constant *ratio* of athermal and thermal contributions over a wide range of strains), the relative influence of a temperature change with respect to an isothermal test can be similarly interpreted. In particular, an insensitivity to the prestrain temperature would indicate that the current deformation temperature plays a dominant role, and that the structural feature predominantly responsible for work hardening is therefore associated with a short range activation.

It is also possible that a change of temperature in a work hardened structure may drive an instability, or avalanche process. Luft and co-workers for example, have studied

instability processes in bcc transition metals and alloys, following a temperature change [36,73-75]. Their most extensive work was performed on molybdenum single crystals.

In molybdenum, a low temperature structure, characterized by the presence of long straight screws, was found in many cases to be unstable to an upward temperature change into the range where cell formation is observed. A large load drop, leading to failure in the tensile tests used, was accompanied by the formation of dramatically dislocation free "channels" cutting through the work hardened structure. This type of localization was also obtained for certain loading path changes, and also in some precipitation hardened structures. The early onset of this load drop, and its magnitude, were enhanced at higher prestrain levels, but even prestrains as low as 5% caused instability and channel formation.

However, when an equivalent set of experiments was done on iron single crystals, neither the load drop nor the formation of these channels was observed [74]. It is of course difficult to claim precise equivalence in the experiments on the two materials, even at the same homologous temperatures, but the results were sufficiently dissimilar to indicate very different responses between molybdenum and iron. For example, prestraining of the Fe specimens was performed at 153 K, a sufficiently low temperature to cause a homogeneous distribution of screw dislocations to dominate the structure. The second temperature was 353 K, where cell formation is well established. Even at prestrain levels much higher than those which caused channel formation and instability in molybdenum, the structure responded in a stable manner, with no channel formation or load drop observed. A satisfactory explanation for the greatly enhanced propensity for localization in the molybdenum experiments with respect to that observed in iron has not yet been found.

For the temperature change tests performed in the present work, polycrystalline low carbon steels were prestrained at one temperature to control the extent of cell development, and then continued at a second temperature. In all cases, the comparisons are made with specimens strained only at the same second deformation temperature (i.e. without a

temperature change). For tests in which a loading path change or strain rate change was also incorporated, the comparisons are made with specimens that were given the same sequence, except with all portions of the test performed at one temperature. Thus, the initial structure is controlled by the temperature during prestraining, and the subsequent behavior observed with respect to the behavior of a "reference" specimen having initially a different substructure, when both are given the same deformation conditions.

III. EXPERIMENTAL PROCEDURE

III.A Materials

Low Carbon, AKDQ (Aluminum Killed Drawing Quality) steel sheets were supplied by General Motors Corporation, and by Sollac (of USINOR SACLOR), a major European sheet steel supplier located in Metz, France. These sheets are hot rolled from slabs, cold rolled, annealed, and skin passed to suppress a room temperature yield point. The final sheet thickness is approximately 0.75 mm.

This processing yields a structure composed of pancake shaped grains, of approximately 30 μm average diameter, slightly elongated along the rolling direction. The grain structure of the as received General Motors Corporation sheets for example, is shown in the sheet plane in Figure 1.

Since the two suppliers use similar compositions and processing to produce these sheets, no substantial differences in their behavior are expected, nor were any observed. However, for internal consistency, each set of tests, wherein the deformation conditions were varied and the subsequent behavior compared, were performed with sheets from a given supplier. The interrupted temperature tensile-tensile testing, as well as the in-situ straining investigation, were performed on the GMC supplied sheets. A portion of the interrupted temperature tensile-shear tests, those performed for the lowest prestrain level studied for this sequence, ($\epsilon_p = 0.07$), were also done using the GMC sheets. The remainder of the tensile-shear sequences, including all loading paths at the higher prestrain levels ($\epsilon_p = 0.10, 0.17$), and all corresponding micrographs, were performed on the Sollac supplied steel.

III.B Interrupted Temperature Tensile Testing

Tensile specimens were cut, longitudinal to the rolling direction, from the GMC supplied sheets. These specimens had a 10 mm reduced width, a 50 mm reduced length, and a 25.4 mm gage length. Constant true strain rate tensile testing was performed by operating a digitally controlled test frame under strain control. The tests were done at strain rates of $1 \times 10^{-4} \text{ s}^{-1}$, $1 \times 10^{-3} \text{ s}^{-1}$, and $5 \times 10^{-2} \text{ s}^{-1}$.

Isothermal conditions were insured by performing all of the tests in a liquid bath. This step was taken following a preliminary investigation which demonstrated that strain induced heating at the higher of the strain rates used may slightly alter the behavior when the tests are performed in air.

Specimens were prestrained to a strain of 0.05 or 0.10, at room temperature (or 10°C) in ethyl alcohol, at -72°C in an ethyl alcohol-dry ice bath, or at 100°C in hot oil. Straining was then continued at a second temperature (room, -72°C , or 100°C). It was found that the behavior was not measurably altered by unloading the specimen during the temperature change, nor was the behavior altered by interrupting the testing for as much as 12 hours to allow the test equipment temperature to equilibrate. Constant temperature reference tests at each temperature and strain rate were also performed, and each test conditions was duplicated or triplicated.

III.C TEM (Conventional)

Specimens for TEM analysis were prepared from the test specimens by thinning to approximately 0.1 mm, either chemically in a 95%-5% mixture of hydrogen peroxide (30%)-hydrofluoric acid, or by mechanical grinding. No observable difference in the substructures was noted using the two techniques; they were therefore used interchangeably as a matter of convenience. Discs were punched from the central gage section and electro-polished to perforation in a solution of 95%-5% mixture of 2-

butoxyethanol and perchloric acid at 30V at 16°C. Observations were performed on a Philips EM301 operated at 100 kV for the tensile-tensile test specimens at UC Berkeley, or a JEOL 200CX operated at 200 kV for the tensile-shear test specimens at INPG, Grenoble, France.

III.D In-Situ (TEM) Straining

Tensile specimens were prestrained to 0.15 at -72°C or at room temperature, as described in Section III.B. The central gauge sections were cut out and thinned chemically to 100 microns in 95%-5% hydrogen peroxide (30%)-hydrofluoric acid. Micro-tensile specimens, with a 3 mm gauge length and 1 mm gauge width, were then etched out from the thinned sections. The orientation of the micro-tensile specimens with respect to the macro-tensile specimens is shown in Figure 2. Final thinning to perforation was done by electropolishing in 95%-5% 2-butoxyethanol-perchloric acid at 30V at 16°C.

In situ tensile testing was performed on a KRATOS EM-1500, equipped with a room temperature straining stage, and operated at 1.5 MeV. The straining stage was driven by excitation of a piezo-electric cell, or by a mechanical motor. However, in some cases, the piezo-electric cell was not operating, and as the motor caused excessive vibrations of the specimen, it was necessary in those cases to initiate yielding, shut off the motor, and record the resulting "avalanche" of dislocation motion.

Dislocation motion during deformation was recorded on a video camera. Additional plate photos were taken by stopping some of the tests in progress. In this way, higher resolution photos of the loaded structure could be recorded.

III.E Tensile-Shear Testing

An apparatus was built to allow low temperature tensile testing of larger sheet specimens. This apparatus was designed to be mounted on an existing Zwick tensile machine. Figure 3 shows the apparatus. Specimens were cut longitudinal to the rolling direction, 280 mm long x 100 mm wide. These were prestrained to $\epsilon_p = 0.07, 0.10$, and 0.17 at a strain rate of $1 \times 10^{-3} \text{ s}^{-1}$, in either an ethyl alcohol bath cooled with liquid nitrogen to -75°C , or at room temperature.

Smaller rectangular shear test specimens were sheared from the center sections of the prestrained sheets, at various angles with respect to the previous tensile axis. Shear testing was performed on an apparatus designed and built at the GPM2 laboratory in Grenoble, France. All shear testing was done at room temperature. The shear specimens used were 45 mm long, by approximately 20 mm wide, with a gauge width (between the grips) of 4.5 mm.

The loading path change for the tensile-shear test sequences is designated by the angle with which the shear specimen is cut with respect to the prestrain tensile axis, as shown in Figure 4. "Pseudo-continuous" testing was performed by reloading the prestrained specimen in shear at 45° , a stress reversal ("pseudo-Bauchinger" testing) by reloading at 135° , and an orthogonal loading path change by reloading at 90° or 0° . Except as noted, all of the orthogonal loading path sequences were performed on specimens cut along the prestrain tensile axis (0°).

IV. RESULTS

IV.A. Single Loading Path

IV.A.1. Interrupted Temperature Tensile-Tensile Testing

These tests were performed following a preliminary investigation by S. Vincent in 1987, which used two-step, interrupted temperature tensile tests [103]. The surprising result of the Vincent work was that the cellular substructure in low carbon steel did not influence the behavior. As this result is in sharp contrast to many of the currently accepted ideas on plasticity, and because no explanation of this result was proposed in the Vincent work, it was felt that the most appropriate method of beginning a more exhaustive investigation was to extend the test matrix, and make every possible effort at precision measurements.

Therefore, the testing included several variations in the prestrain level, temperatures, and strain rates. The tests were performed using isothermal baths, tested under constant true strain rate conditions, and all permutations were duplicated or triplicated. Larger prestrain levels were then made possible by combining tensile and shear loading. It should be added here that the results support the Vincent work. An explanation for this behavior, and the regime of its validity are given in the Discussion section.

IV.A.1.1 Prestrain Structures

Shown in Figure 5 is the dislocation substructure after a tensile strain to $\epsilon = 0.10$ at -72°C . The structure is composed predominantly of long straight screw dislocations more or less homogeneously distributed throughout the interior of the grains. Some diffuse tangles are present, but at this strain level no cellular substructure has developed. This type of structure was observed at this temperature and strain level for all of the strain

rates tested. It is typical of bcc materials in which the deformation temperature is sufficiently low.

Highly mobile edge segments initially present have moved through the structure during early microyielding, and left behind the relatively immobile screw segments which are then forced to move in response to the imposed deformation. As discussed in the Background section, the presence of these screws indicate that their mobility is sufficiently limited at -72°C to be the controlling factor in the deformation process. If this were not the case, and the edge and screw mobilities were more comparable, the dislocation population would be composed of bowed out, mixed dislocations.

The microstructure which develops when the same tensile strain is instead performed at room temperature is shown in Figure 6. The arrangement of the dislocation population here is distinctly less homogeneous, and a cellular substructure is well developed. The high dislocation density which composes the cell walls can be clearly seen (dark regions), and in this micrograph the cell interiors appear nearly dislocation free. The observation of apparently dislocation free cell interiors may be misleading however, and will be discussed further in the results of the in-situ studies.

The average cell shape has begun to imitate the macroscopic shape change imposed on the specimen and although they are not uniform, the majority of the cells are elongated. The dislocation density in the cell walls is already too high at this strain level for meaningful density measurements to be made. All regions of this specimen showed roughly the same extent of cell development, in that no regions showed the homogeneous arrangement seen at low temperature as in Figure 5.

The orientation of the walls is variable, even within a single grain. This probably indicates differences in the active slip systems in different regions of the same grain. All of the above observations are consistent with observations reported by other investigators on cell development in low carbon steel, as well as other bcc materials deformed at temperatures high enough for formation of cellular dislocation structures.

The surface slip lines corresponding to these two prestrains were also observed on specimens polished prior to straining. Wavy slip lines commonly observed in bcc metals can be seen following room temperature straining to $\epsilon = 0.10$, as shown in Figure 7. When straining is performed at -72°C , Figure 8, the surface slip lines tend to be a bit less coarse, implying that the surface deformation on the intragranular scale is slightly more homogeneous at the lower temperature. The overall surface roughness on the scale of the grain size appears similar in the two specimens.

No distinctive dissimilarities in the grain shape changes are observable in the two specimens, or if present, they are too subtle to be observed at this strain level. The grain morphology after tensile straining to $\epsilon = 0.10$ at -72°C and room temperature are shown in Figures 9 and 10, respectively. The most striking influence of the prestrain temperatures therefore, is on the arrangement of the dislocation population, with cell wall formation substantially suppressed at the lower temperature.

IV.A.1.2 Crystallographic Texture

The intention of prestraining at these temperatures was to develop dissimilar substructures, and thereby isolate the influence of cell development on subsequent behavior. To do this, it was also necessary to investigate the influence of crystallographic texture development (changes in the orientation distribution of the grains in a polycrystal during plastic deformation). In particular, it was necessary to identify any *dissimilarities* in the crystallographic texture developed during prestraining at low and room temperatures, which could cause dissimilar behavior not caused by the cell structure. Orientation distributions are usually measured in a series of x-ray diffraction experiments on a rotating goniometer, and the data is plotted as a pole figure.

Pole figures of the low carbon steel sheets used in this study were taken by Prof. R. Wenk of the UC Berkeley Geology Department. Specimens were strained to $\epsilon = 0.15$,

at room temperature, and at -72 °C. No pole figures were taken of the virgin sheets, as this data was not considered of interest. According to Wenk, an established expert in the field of texture, there was no significant difference in the crystallographic texture of the two specimens. It can therefore be concluded that in this temperature range the development of crystallographic texture has not been altered by the temperature, at least up to strains of 0.15. As the influence of texture is held constant, it is ignored in subsequent comparisons of stress-strain behavior.

IV.A.1.3. Tensile Test Data

Two stress strain curves, obtained at a strain rate of $1 \times 10^{-3} \text{ s}^{-1}$, are shown in figure 11. The specimen corresponding to the discontinuous curve was prestrained to $\epsilon = 0.10$ at -72 °C, then strained at room temperature, along the same tensile axis. For the continuous curve, the specimen was strained at room temperature only. It can be seen that at room temperature, the two specimens behave quite similarly, in spite of dramatic differences in their substructures at the point where the temperature was changed.

The same sequence was done (a low temperature prestrain to 0.10, followed by room temperature, and constant room temperature testing) with all portions of the tests at a strain rate one and half orders of magnitude higher ($5 \times 10^{-2} \text{ s}^{-1}$). The as-tested results are shown in Figure 12a. If the interrupted-temperature curve is shifted rigidly along the strain axis, (Figure 12b), it can be seen that the two curves coincide, when the comparison is made from a common stress level. The physical significance of this adjustment has been described in the Background section, where single structure parameter models are discussed. It corresponds to the selection of a "reference" stress level, from which the two curves may be compared. After this adjustment, the comparison can be made with both structures initially at the same level of work hardening, but obtained with different developments of the cell structure.

The particular choice of a reference stress level is of no significance, since the question is whether or not the behavior is determined generally by fixing the stress level and the deformation conditions (strain rate, temperature, and loading path). If the stress level and deformation conditions do fix the subsequent behavior, all the relevant features of the structure can be adequately characterized by the value of one scalar parameter. If this occurs with dissimilar extents of cell formation, it must be concluded that the development of the cell walls is not among the structurally relevant features required to fix the flow stress. The amount of shift along the strain axis, rather small in Figure 12b, shows the extent to which the controlling feature has developed during a low temperature prestrain with respect to its development during room temperature straining.

The results shown in figure 11 indicate that the presence of the cell walls has not influenced the work hardening behavior, since a specimen without the walls (prestrained at low temperature) strain hardens in the same manner as a specimen with developed cell walls. This is true both at the point of the interrupt, and, more importantly, throughout the remainder of the test. In addition, the results in figures 12a and b indicate that only one independent structural feature is controlling the flow stress for both substructures. Only the extent of development of this feature, which is not associated with the presence of the walls, has been altered by the dissimilar prestrain temperatures.

This test sequence was also performed at a lower strain rate ($1 \times 10^{-4} \text{ s}^{-1}$), and the results are shown in figures 13a and b. Figure 13a shows the as-tested data, after a slight shift to a common stress level (figure 13b), the curves coincide reasonably well, again indicating that the presence of the cell structure has had little influence on the behavior. The very slight mismatch present was consistently observed for these test conditions (room temperature deformation at the lowest strain rate tested). An explanation for this is given in the Discussion section. In any case, given the great dissimilarities in the substructures at the end of the prestrain, the presence of the cell walls has had little effect.

It was considered that the coincidence of the interrupted and continuous curves might merely be a consequence of weak work hardening. The curves are rather flat, and it was thought that perhaps fixing a stress level might be forcing spurious results. For this reason, the tests were duplicated using lower prestrain amounts, where the curves are steeper. These tests showed the same results: the work hardening behavior from a given stress level was not altered by the lack of cell formation during low temperature prestraining. Shown in Figure 14, for example, is the (as-tested) stress-strain curve for a specimen prestrained at $-72\text{ }^{\circ}\text{C}$ to $\epsilon = 0.05$, and continued at room temperature, at a strain rate of $1 \times 10^{-3} \text{ s}^{-1}$. The continuous room temperature test results are also plotted, showing that the room temperature behavior of the two specimens is quite similar.

The reverse sequence was performed, to monitor the influence of the prestrain structures when the deformation is continued at $-72\text{ }^{\circ}\text{C}$. A room temperature prestrain to $\epsilon = 0.10$ was followed by straining along the same tensile axis at $-72\text{ }^{\circ}\text{C}$. The stress-strain curves are compared with the results of continuous testing at $-72\text{ }^{\circ}\text{C}$. The results for a strain rate of 10^{-3} s^{-1} are presented in Figures 15a and b, where the as-tested and shifted curves are shown, respectively. These tests were also duplicated at strain rates of $5 \times 10^{-2} \text{ s}^{-1}$, and 10^{-4} s^{-1} , and the results are shown in Figures 16a and b, and 17a and b.

In possibly the only remaining permutation for tensile-tensile loading at these temperatures, a set of tests were done in which the prestrain rate was altered. The prestrain was performed at one strain rate and temperature (room or low), and deformation continued at a second strain rate and temperature (low or room). The comparisons were then made with results from continuous tests at the second temperatures and strain rates. In all cases, the curves coincide from a common reference stress, and changes in the rate of prestraining only change slightly the amount of shift required to cause the curves to coincide.

IV.A.1.4. Behavior Above Room Temperature (100°C)

A smaller subset of tests were performed at 100 °C. Specimens were prestrained at -72 °C to $\epsilon = 0.10$ as before, then continued in a hot oil bath. In Figure 18a, an (as-tested) stress strain curve is compared with the curve obtained by continuous straining at 100 °C, when all portions of the tests are performed at a strain rate of $1 \times 10^{-3} \text{ s}^{-1}$. The behavior in this case is quite different for the two specimens. As shown in Figure 18b, no amount of shift along the strain axis will cause coincidence of the two curves, as they cross over each other.

A single parameter model is therefore no longer adequate to describe the flow stress at this temperature, and the behavior at 100 °C has been significantly altered by the low temperature prestrain. The work hardening behavior may be altered by larger scale features, such as the cell walls, or the dissimilar behavior may be caused by intrinsically different dislocation interactions occurring in the two specimens on a smaller scale. As more than one structurally relevant feature is evolving, it is difficult in this case to isolate the influence of the cell walls alone.

IV.A.2. Higher Prestrains: Tensile-Shear Testing

The interrupted temperature test results presented thus far were obtained from single specimens in which the temperature and/or strain rate was changed during the test. Due to the onset of necking, interrupting the tests at larger prestrains and continuing in tension does not leave a sufficient portion of the curves to make a useful comparison. For this reason, a tensile-shear sequence was used for performing single loading path, interrupted temperature tests with higher prestrains.

Large specimens were prestrained in tension, at low or at room temperature, smaller specimens were cut from them, and deformation continued in shear loading along a 45° axis with respect to the tensile prestrain axis. The shear strain occurs over a constant

section and geometric softening is avoided, allowing data to be collected at much larger strains.

As verified by Schmitt, and Rauch, this test sequence results in monotonic loading, very nearly representative of uniaxial monotonic testing. The slight influence of cutting and changing the test mode is of particularly little significance in the present work, as the comparison is made between dissimilar prestrain structures which are then both subjected to the same cutting and loading paths. The equivalence was further verified for the interrupted temperature tests here by performing some of the tests at prestrains which overlap the tensile-tensile test conditions.

IV.A.2.1 Prestrain Structures

Figure 19 is a typical micrograph from a specimen prestrained to 0.17 at room temperature, at a strain rate of $1 \times 10^{-3} \text{ s}^{-1}$. As expected, cell formation is well developed, with extremely well defined, elongated cell walls present throughout the specimen. As described, continuing the deformation from this point, along a 45° direction in shear, should result in continuous development of this cell structure.

When the deformation is performed at -75°C , cell formation is always relatively suppressed compared to room temperature straining, but cells do develop at sufficiently high strains. In Figure 20 is shown one of the more cellular regions of a specimen strained at -72°C to $\epsilon = 0.17$. The cell walls are not as tight or uniform as the corresponding room temperature case, but clearly some of the loose tangles, which were present at strains of 0.10 at this temperature, have developed into cell walls. The extent of cell development is also less uniform throughout the specimen. Equally typical is the less cellular region shown in Figure 21, in which many of the walls are still loosely knit enough for individual dislocations to be identified, and the cell size is variable. Figures 20 and 21 roughly bracket the structures observed for these conditions, and both types of regions are

distributed throughout. Conversely, no ill-defined cell walls were observed in the room temperature specimen at this strain level.

In part because of their lack of uniformity, no effort was made at quantifying these structures from TEM micrographs, and perhaps any such measurements should be viewed with some skepticism. As cell walls are present at the low temperature at higher prestrains, this structure may be labeled as cellular. However, as even the most cellular regions are less developed than the typical room temperature cell walls, the two structures are easily distinguishable and it can be conservatively stated that the low temperature prestrain has relatively suppressed cell formation. Relative comparisons are in fact all that is ever possible, since even at the lowest temperatures, tangles are present early in the deformation.

Thus the same argument holds as for the lower prestrains where the structures were more easily labeled as cellular or noncellular: If the flow stress is controlled by the characteristics of the cell walls, it should become apparent when the subsequent behaviors of dissimilar structures are compared at the same deformation conditions.

IV.A.2.2 Shear Test Results

Shown in Figure 22 is the room temperature shear stress-strain curve for two specimens (Sollac sheets). The curves for the tensile prestrain (at $\gamma = 10^{-3}\text{s}^{-1}$) are not shown. The specimen corresponding to the curve with open symbols was prestrained to 0.17 at -75°C , and for the other curve (solid symbols), a specimen prestrained at room temperature to 0.17. As for the tensile-tensile tests, the two curves coincide, in this case without a shift, again indicating that the dissimilar extent of cell formation at the end of prestraining has had little influence on the subsequent behavior, even at higher prestrain levels.

The interrupted temperature tensile shear sequence for single loading paths was also performed at lower prestrains. Shown in Figure 23 are the room temperature shear tests results following a -72°C prestrain (open symbols) and room temperature prestrain (solid symbols) in tension to $\epsilon = 0.06$, performed on the GMC supplied sheets on which the tensile testing was done. Again, the curves coincide reasonably well without a shift. When the prestrain level and temperature are slightly altered (-80°C , and room temperature, to $\epsilon = 0.10$, performed on the Sollac supplied sheets), a slight shift is again needed to cause the subsequent shear stress-strain curves at 45° to be in registry. The curves corresponding to such a sequence are shown in Figure 24a (as tested) and b (shifted).

Specimens prestrained to different amounts at the two different temperatures will also coincide after a rigid shift along the strain axis. This is expected if a single structure parameter model is strictly obeyed, since only the extent of development of a single controlling feature may be altered by prestraining. A comparison of this type is shown in Figures 25a and 25b. For the lower curve (open symbols), the specimen was prestrained at -75°C to 0.17, then loaded in shear at room temperature. The lower curve (solid symbols) is for a specimen prestrained at room temperature to 0.10, and continued in shear at 45° . As expected, the low temperature prestrained specimen has work hardened further, and shows an initially higher stress level. However, when shifted to a common stress level, the curves coincide.

Variations of these conditions included changes in the shear strain rate ($5 \times 10^{-2} \text{ s}^{-1}$ to $1 \times 10^{-3} \text{ s}^{-1}$), prestrain level (0.06, 0.10, 0.17, 0.20) and prestrain temperatures (-80°C to room temperature), for sheets supplied by two sources. For single loading paths, the subsequent room temperature behavior could be made to coincide with an arbitrary room temperature reference curve (at the same strain rate and from the same supplier) by shifting rigidly along the strain axis to a common shear stress. As with the tensile-tensile test data, this demonstrates that room temperature work hardening in this material is dominated by

only one independent structural feature for these conditions, and that this structural feature is not sensitive to the extent of cell formation.

IV.B. LOADING PATH CHANGES

IV.B.1 Interrupted Temperature Testing

Specimens were prestrained at different temperatures to develop different substructures, and then subjected to a change in the loading path. As discussed in the Background section, constant temperature loading path changes in this material result in transient behaviors during which cell walls rearrange. The magnitude and duration of these transients are increased with the extent of work hardening at the point of the path change. It has been proposed that the macroscopic response is caused by the development of the cellular substructure during the initial straining path, yet the present study indicates that the cell walls have little influence during monotonic loading. The transient behaviors of the low and room temperature prestrain structures were therefore compared following two types of changes in the loading path.

A stress reversal was used to measure the relative magnitude of the internal stresses associated with the cell walls, as predicted by some orthogonal loading path changes were also studied, in which the direction of maximum shear during the second loading sequence had a shear stress of zero during prestraining. This represents the "hardest" loading path change, as slip systems which were most latent during the prestrain are forced to become more active, and the previously active systems are no longer favorably oriented.

The tests made using monotonic loading paths demonstrated that a single structure parameter model was adequate, independent of the prestrain temperature and extent of cell formation. The comparison was made by rigidly shifting one of the stress-strain curves along the strain axis to a common stress level. Due to the transient behavior induced,

incorporation of a loading path change complicates the comparison, and such an adjustment may not be appropriate.

The rearrangement of the structure following a change in loading should in general evolve with strain in a manner which is itself dependent on the current stress level. A more accurate statement of this dependence is that the observed changes in the flow stress indicate the ease with which the current structure can accommodate the imposed change in the deformation path. In any case, considering as an example orthogonal loading changes, the transient involves an increase in the flow stress, followed by softening and subsequent hardening. If a rigid shift along the strain axis to a common stress level causes a comparison between transient and post-transient behaviors, the influence of cell development alone cannot be isolated and the comparison may be meaningless.

It is therefore not expected that a given stress strain curve should necessarily coincide with an arbitrary curve obtained at the same conditions by performing such a shift, particularly for conditions in which result in substantial transient behavior. If the *initial* flow stress, at the point of the loading change, is similar for two specimens however, a comparison of their corresponding curves is justified. The role of the current stress level on transient and post transient behavior is then held constant and if dissimilar behavior is subsequently observed, it must be caused by dissimilarities in the prestrain structures.

Thus, a single structure parameter model as described in section II.D.1 is not expected to be valid during a loading path change, as at least one more parameter is needed to specify the path change. In spite of this, and with the added restriction described above, loading path changes will provide supplementary information on the influence of a cellular substructure. Similar behaviors from an initially common stress level for example, will indicate that even the transient behavior is unaltered by the development of the cell walls.

IV.B.2 Stress Reversals

The comparisons made in this section are of specimens which had been prestrained in tension (to various amounts, at a strain rate of $1 \times 10^{-3} \text{s}^{-1}$), at different temperatures to control cell wall formation, as in the monotonic loading sequences. They were subsequently both subjected to the same stress reversal, by loading in shear at 135° with respect to the prestrain tensile axis, or continued along the same loading direction by loading in shear at 45° , as described in the Experimental Procedure and shown in Figure 4.

Shown in Figure 26 are the room temperature shear test results following a stress reversal. for a tensile prestrain to $\epsilon = 0.07$ at room temperature (curve with solid symbols) and at -72°C (open symbols). The specimens respond similarly to the path change. The influence of the stress reversal itself, compared to continued deformation along the same loading path, is shown for the low temperature structure in figure 27. Here a -72°C prestrain to $\epsilon = 0.07$ at a strain rate of 10^{-3}s^{-1} is followed by room temperature shear testing either at 45° with respect to the prestrain tensile axis ("pseudo-monotonic" loading path), or by room temperature shear testing at 135° ("pseudo-Bauschinger" loading). The stress reversal has caused measurable softening, even in this noncellular structure.

The same test sequences were performed following a higher prestrain level ($\epsilon = 0.17$). The influence of the isothermal stress reversal alone is shown in Figure 28, where a room temperature prestrain is followed by "pseudo-monotonic" loading in shear (45°) at room temperature, or by a reversal (135°) at room temperature. It can be seen that the path change has caused a substantial, and long term, lowering of the flow stress.

Figure 29 demonstrates the effect of a stress reversal on the low temperature (less cellular) substructure following prestraining to $\epsilon = 0.17$ at -80°C . Again, a strong Bauschinger effect is observed, and it appears that extensive development of a cellular substructure is not necessarily required for the softening to occur.

The relevant comparison for observing the role of the cell structure alone on the effect of a stress reversal at the larger prestrains is shown in Figure 30, where both a specimen prestrained at low temperature and a specimen prestrained at room temperature are subjected to the same stress reversal (open and solid symbols, respectively). The curves are not in perfect coincidence, but their dissimilarities are not dramatic, considering the large dissimilarities in cell structure development, at the end of pretraining. The relative suppression of cell formation in the specimen corresponding to the (open symbol) low temperature structure has again not substantially altered the response to a stress reversal.

IV.B.3 Orthogonal Path Changes: Results

The role of cell wall development in the transient behavior caused by orthogonal (hard) loading path changes was studied by duplicating the initial loading path at lower temperatures where cell formation is relatively suppressed. Figure 31 shows the room temperature shear stress-strain curves following an orthogonal path change for specimens given the lower prestrain level ($\epsilon = 0.07$) at -72°C (curve with open symbols) and room temperature (closed symbols). The shear loading was done along the prestrain tensile axis (0°), at a strain rate of 10^{-3}s^{-1} .

Little influence of the prestrain temperature is seen; both low and room temperature substructures respond similarly. The effect of the path change alone is shown in Figure 32 where it can be seen that a hard change in the loading has raised the flow stress with respect to that observed for monotonic loading. As expected from previous work, the response occurs in a stable manner at this prestrain level, and no load drop is observed.

When the prestrain level, sheet supplier and prestrain temperature are modified, the curves corresponding to the low and room temperature prestrains vary, but only by a slight rigid shift along the strain axis, as was observed in the monotonic loading sequences.

Figures 33a and 33b show the behavior for the Sollac sheets, prestrained to $\epsilon = 0.10$, at -80°C and at room temperature and continued in shear at 0° (at a strain rate of 10^{-3}s^{-1}). After a slight rigid shift along the strain axis, the room temperature response of the two structures is similar. As described in Section IV.B.1, a single structural parameter model should not be strictly valid with this loading path, but the lack of an instability in the transient behavior at these lower prestrains, and the small shift required in this particular case allow coincidence of the curves nonetheless.

When the prestrain amount is increased the initial stress level is raised further, and an instability, seen as a load drop, is observed. Continued deformation from this point then causes the return of work hardening. This softening, and its tendency to increase with increased prestrain levels has been observed previously by other researchers, for isothermal loading path changes. As shown in Figure 34 for a prestrain of $\epsilon = 0.17$, this sequence of events occurs similarly in the low temperature (less cellular) and the room temperature (very cellular) prestrain substructures.

This softening is similarly enhanced by raising the strain rate following a given prestrain. The curves in Figure 35 correspond to two specimens which were both prestrained at -75°C to $\epsilon = 0.17$, and then given an orthogonal path change at shear strain rates of $1 \times 10^{-3}\text{s}^{-1}$ (lower curve) or $5 \times 10^{-2}\text{s}^{-1}$ (upper curve).

It has also been observed that if this loading path change is performed by shear loading at 90° with respect to the prestrain tensile axis, instead of 0° , the magnitude of the load drop is greater (for a given prestrain). The curves for such a comparison are shown in Figure 36, where, following a room temperature tensile prestrain to $\epsilon = 0.20$, one specimen has been loaded in shear at 90° , and another in shear at 0° , both at strain rates of 10^{-3}s^{-1} .

IV.C Evolution of the Structure After Change in Temperature

To identify the controlling structural features which cause dissimilar degrees of cell formation to subsequently behave similarly, it was necessary to study the evolution of the substructure following the temperature change. The upward temperature change test sequence, in which a more homogeneous distribution of dislocations is developed at low temperature and continued at room temperature, is of primary interest since the evolution should be most rapid for this case, yet the initial extent of cell wall formation had no observable effect. If, for example, the low temperature arrangement is highly unstable at room temperature, and rapidly evolves to form a more cellular structure after the temperature change, the coincidence of the curves could be explained as the result of comparing essentially similar substructures.

However, TEM analysis indicates that the evolution of the structure is rather sluggish at room temperature, and is presumably even more sluggish at lower temperatures. Duplicate tests were performed, at a strain rate of $1 \times 10^{-3} \text{ s}^{-1}$, and stopped at intermediate strains for TEM analysis. A specimen was strained to $\epsilon = 0.1$ at low temperature, then strained 0.1 more at room temperature, to an accumulated strain of $\epsilon = 0.2$. Another specimen was strained continuously at room temperature to $\epsilon = 0.2$. As demonstrated in Figure 11, the curves corresponding to these conditions coincide, without a shift. Therefore, the relevant features of these two specimens are equivalent at equal strains, in the sense that they behave similarly, from the point of the temperature change onward.

Figure 37a is a TEM micrograph of the specimen strained continuously to 0.2 at room temperature. As expected, the cell structure is more developed than it was after room temperature straining to $\epsilon = 0.1$ (Figure 2). The average cell size is apparently smaller, and more uniform, and the walls are extremely well defined. This degree of cellularity was found throughout the specimen.

A region of the specimen strained at low temperature and then at room temperature is shown in Figure 37b. Cell formation is much less extensive, and appears to be more or less superimposed on the low temperature structure, rather than having erased it. The substructure is also much less uniform throughout the specimen. Much of the specimen is typical of the micrograph shown in Figure 37b, other regions are more cellular, but nowhere is the cell structure as developed as that corresponding to continuous room temperature straining to 0.2.

Although some cellularity is present in both specimens, it is clear that they are not equivalent with respect to the extent of cell development at a point well after the temperature change. Since their corresponding stress-strain curves are similar, it must be concluded that the characteristics of the cell walls are not significantly altering the flow stress.

The sluggish evolution of the structure following an upward temperature change to room temperature is shown again in Figures 38 and 39. Here a specimen was prestrained in tension at -72°C to a strain of 0.07, and continued along the same loading direction at room temperature, this time by shear loading along the appropriate axis (45°). The room temperature shearing was stopped at 0.09. Again the structure appears rather chaotic, particularly when it is considered that the total strain at this point is over 0.15 which, if carried out at only room temperature, would result in much more extensive cell formation (between that shown in Figure 6 at $\epsilon = 0.1$, and in Figure 37a at $\epsilon = 0.20$).

A more subtle argument on the influence of the evolution of the structure after a temperature change is presented by the stress-strain data itself. If the evolution of the structure to a more similar arrangement was responsible for the shape of the curves, it would be expected that a noticeable transient would be observed at the point of the temperature change, since it is unlikely that a cellular structure can form instantaneously during an upward temperature shift, nor dissolve instantaneously during a downward temperature shift. The curves show that such a transient is extremely weak, if it is present at all.

Furthermore, the evolution should be even more slow when the temperature is decreased, compared to when it is raised, yet the corresponding curves coincide quite well. Similarly, the evolution to a cellular structure should be most rapid when the second temperature is raised to 100 °C, yet the curves don't coincide well at all. Thus, conditions which accelerate the evolution of a cellular structure after a low temperature prestrain are not associated with more closely similar behavior at the second temperature.

In conclusion, a rapid evolution of the substructure following a temperature change to room temperature or below does not appear to explain the observed behavior. The rearrangement of a low temperature structure toward the more cellular structure characteristic of room temperature deformation appears to occur in a slow and stable manner in this material. This rearrangement does not measurably alter the work hardening behavior, with respect to the behavior of a continuously evolving, and initially more cellular, substructure.

IV.D In-Situ Straining at Room Temperature

A direct investigation on the mechanisms operating during room temperature deformation that cause the cell walls to have little or no influence on the flow stress, was performed in an in-situ (TEM) straining study. This work was done in collaboration with Dr. J.-H. Schmitt, then of the GPM2 laboratory at the Centre National Recherche Scientifique in Grenoble, France.

To avoid the influence of thin film effects on cell formation, specimens were prestrained as bulk specimens to establish the characteristic low temperature, or cellular room temperature, structures before making the in-situ tensile film specimens. The prestraining was done to strains of 0.10 or 0.15, at -72 °C and at room temperature. The specimens were then mounted on a straining stage and pulled in tension at room temperature. The dislocation activity during deformation was recorded on video film, and

in some cases the deformation was stopped during the tests to allow plate micrographs to be taken of the loaded structure.

The observations made for specimens prestrained at room temperature and continued in-situ at room temperature along the same loading direction are as follows. Expanding loops, generated in the cell interiors and at the cell walls, soon become rectangular in shape, composed of pure edge and pure screw segments. The edge segments move very rapidly and become incorporated in the neighboring cell walls. The rapid passage of the edges leaves behind a dipole composed of the two straight screw segments which traverse the cell interior. These segments then typically separate, and move with a much lower velocity. They move in a somewhat jerky manner, but tend to remain straight as they move. Their limited mobility indicates that the stresses required to move them is much higher than that of the edge segments, yet their motion appears to be a necessary step in the deformation.

Due to the extremely high dislocation density in the walls, it was not possible to monitor reactions in the walls caused by the incorporation of new edge segments. It was typically observed however, that after a slight delay, during which the screw segments in the cell interior continued to move, the cell wall would become active. New dislocations would be emitted into the adjacent cell interior, which then moved in a similar manner, generating a new pair of screw segments as the neighboring cell interior was crossed by the more mobile edge segments. Thus the interactions in the walls following the addition of new dislocation segments may be one of altering the stresses in the regions of the walls, and causing sources to be activated.

The rearrangement at the walls, and subsequent emission of dislocations allows strain to be accommodated across the cell walls. Although an individual dislocation does not "pass" through the wall, it cannot necessarily be concluded that the walls provide substantial resistance. If easy sources are present in the wall and only slight alterations are necessary to activate them, the walls may in fact be rather "transparent". The relative

influence of this interaction with respect to the macroscopic flow stress is particularly diminished if the stress to move screw dislocations in the cell interior is substantially higher than the stress required to activate cell wall sources.

Regardless of whether the source originates in a wall or a cell interior, the outcome is similar. Edge segments rapidly move to the neighboring wall and a pair of screws are left to move in the cell interior. Although a substantial amount of straining is accomplished by the easy motion of the edge segments (they are sometimes as long as half a cell diameter), continuing the deformation once the edge segments are "captured" at the cell walls appears to require movement of the remaining screws. The motion of the edges is thus seen to be coupled to the restricted motion of the screws, which can therefore be identified as the rate controlling step.

Shown in Figure 40 is a sequence of photos which were taken from screen prints of a video tape recorded during in-situ deformation. The specimen shown was prestrained at room temperature, and the location of the region shown was such that it has been subjected to nearly monotonic loading, with respect to the prestrain axis. The cell walls seen in the micrographs were formed during the prestrain.

The sequence begins at micrograph A, where a dislocation loop expanding in the central cell interior has been marked with an arrow. The loop becomes progressively elongated in B and C. The lower edge component of the loop has traveled rapidly to the adjacent cell wall in D, where the remaining screw segment can be seen, lying parallel to other screw segments which traverse the same cell interior.

A plate micrograph shows more clearly the loaded structure during the deformation. Such a micrograph is shown in Figure 41, for a specimen which had been prestrained at room temperature, strained in-situ, and then stopped without removing the load. Again, the cell walls were formed during prestraining. It can be seen that the interior of the cells is quite active, with a relatively high density of dislocations present. The majority of these

dislocations are of screw character, and they traverse the cell interior. Typically, two families of these screws are observable in a given cell interior.

When a specimen which had been prestrained at low temperature to develop the more homogeneous distribution of long straight screws typical of low temperature deformation was then strained in-situ at room temperature, no rapid rearrangement of the dislocation population was observed. As discussed in the previous section, a rapid evolution to a more cellular structure was not expected. The motion of individual dislocations was observed to occur in a manner quite similar to that observed in the cell interiors of specimens prestrained at room temperature. Screw oriented segments tended to move in a slow and jerky manner, and the growing edge segments moved much more rapidly.

In-situ straining at room temperature was limited to strains below which well developed cell walls were able to form out of the low temperature prestrain structure. The mobile edge segments tended to become tangled during straining, but in a rather disorganized manner. This was undoubtedly the beginning stages of cell formation in an already work hardened low temperature structure. The observed slow and stable agglomeration of the more mobile edge segments to form a cellular room temperature structure more or less superimposed on the low temperature prestrain structure was thus the expected evolution following a temperature change, as predicted by the mechanical test data and the conventional TEM observations.

The operating voltage used for these experiments (1.5 MeV) was well above the damage threshold voltage which is thought to be about 700 kV for this material. Although this allowed viewing of thicker regions perhaps most indicative of bulk behavior, the damage at higher operating voltages may have caused an increase in the apparent mobility of edge dislocations. For this reason, quantitative data from the in-situ observations would probably be inaccurate, and was not attempted.

However, conventional TEM micrographs taken at lower voltages provide evidence that the mechanisms observed in-situ are in fact typical of room temperature deformation, and the general conclusions reached on the rate limiting step of screw dislocation mobility appears to be valid. In Figure 19 was shown the dislocation cell structure after room temperature tensile straining to $\epsilon = 0.17$, and observed at 200 kV, well below the damage threshold. The activity in the cell interiors, seen in Figure 19 can now be understood as similar to that observed in-situ. It can be seen that a relatively high density of straight screw oriented dislocations traverse the cell interior, and that the greatly dissimilar mobilities between edge and screw dislocations, which is responsible for the retention of these screws in the cell interior is not an artifact of above-threshold voltage damage.

Due to the slight orientation mismatch as a cell wall is crossed, some cell interiors are in sharp contrast, while others are out of contrast and appear to be dislocation-free. As the specimen is tilted, it can be observed that a typical cell interior contains these dislocations. The presence of these screws indicate that their mobility is limited, as in the structures observed after low temperature straining. The in-situ experiments indicate further that the motion of these screws is required to accomplish the deformation.

V. DISCUSSION

V.A. General

The relative suppression of cell wall formation during low temperature straining with respect to room temperature deformation, has been demonstrated. Furthermore, the more macroscopic features, such as crystallographic texture and grain shape changes, appear to develop similarly. If the extent of cell wall formation is necessarily a factor in work hardening, it is expected that these two structures should behave differently when the deformation is continued at the same second temperature. Conversely, a similar response of each of these structures to a fixed set of deformation conditions disproves, at least for these conditions, models in which the flow stress is necessarily controlled by the development of the cell walls.

The coincidence of the curves corresponding to the two prestrain structures for the case where the deformation is continued at the lower temperature is perhaps not surprising. The structure observed at -72°C indicates that the deformation process at this temperature is uniquely controlled by the limited mobility of the screws. The reasons for this dependence were discussed in the Background section. When the more cellular room temperature structure is deformed at -72°C , it is therefore expected that the initial supply of mobile edge segments, present during the room temperature strain, would rapidly become exhausted. After exhaustion of the free edges, the flow stress should be controlled by the remaining screws, as dictated by the current temperature. Since this mechanism operates on a scale much smaller than the cell wall spacing, and represents the limiting step in the deformation process, the cell walls themselves would not necessarily modify the behavior, other than possibly providing temporarily easy sources immediately after the temperature change.

This is in fact what was observed in Figures 15 through 17, where the cell walls present at the end of a room temperature prestrain have not influenced the subsequent

behavior at the lower temperature, compared to specimens deformed only at -72°C . The absence of a significant transient at the point of the temperature change indicates further that the influence of any excess edge segments initially present has also had negligible effect. Either the supply is exhausted during microyielding, or they are not able to provide an easy deformation path independent of the screws, even immediately after the temperature change.

In summary, the cell walls have little influence when a cellular structure is "frozen in" by continuing the deformation at sufficiently low temperature. The flow stress is determined by the limited mobility of individual screw dislocations at these temperatures. The screws are presumably moving in the cell interiors of the prestrain structure, and their motion controls the flow stress in much the same manner as it does during continuous low temperature straining. As the dominant mechanism occurs on the nearly atomic scale, the behavior is insensitive to larger scale features such as cell walls formed during prestraining and should in general be insensitive to the choice of prestrain temperatures.

Although the controlling mechanism at low temperature is well understood, the occurrence of similar behavior at a higher temperature in which cell formation is enhanced is more surprising. These are the cases in which a low temperature less cellular structure is continued at room temperature, and behaves just as a specimen in which cell walls are already well formed. The in-situ study best demonstrates the microscopic cause of this macroscopic observation. The evidence, from the mechanical test data and the in-situ study, is that screws dislocations moving in the cell interiors retain their controlling influence as the rate limiting step in the deformation process well into the range of conditions in which the arrangement of the dislocations into cell walls is thought to become influential.

A single structural parameter model appears to be adequate only up to about room temperature, and no longer works at 100°C . In fact, the limit of validity may already be approached at the lowest strain rates at room temperature. Although the misfit seen for

example in Figure 13 is slight, it was consistently observed when the tests were duplicated. The most precise fits between interrupted and continuous temperature test curves occur at the highest strain rates and lowest temperatures. The controlling feature is therefore most dominant in this regime.

This trend in the data further identifies the screws as the dominating feature. Room temperature is known to be an intermediate temperature with respect to the mobility of screw dislocations in this material [68,72], and 100°C a sufficiently high temperature for easy recombination of the dissociated screws, at which point they would no longer be expected to control the flow stress. The regime of their dominance therefore, in low carbon steel, is defined roughly by room temperature and below, at strain rates of 10^{-3} s^{-1} or more. Although a large fraction of the strain is accomplished by the relatively easy motion of edge dislocations at room temperature, compared to that at lower temperatures, their motion is coupled to the still restricted motion of the screws.

It is clear from the results here that extensive formation of cell walls does not necessarily mean they are influential, as assumed in many models. In this particular case, a bcc material is deformed at low to intermediate temperatures, and the sluggish motion of screw dislocations can be identified as the rate controlling mechanism which causes the cell walls to have little or no influence. This of course raises the question of whether this lack of influence is specific to bcc materials at low and intermediate temperatures, or whether the development of a cell structure may also be intrinsically uninfluential in other cases.

There are other documented cases in which the extent of development of a cellular substructures was observed to be uninfluential at higher temperatures, and in fcc materials. Kassner et al [34] have isolated the separate influences on the flow stress of subgrain formation, and forest hardening in the subgrain interiors, in 304 stainless steel. It was determined that the dislocation density within the subgrains dominates the flow stress at elevated temperatures. Similarly in pure Al, at large strains, the character of subgrain boundaries were dramatically altered with no influence on the flow stress [35], indicating

that accomodating the imposed deformation across the boundaries was not associated with the rate limiting step of the deformation. Recently, Henshall et al [26] report similar results in an Al-5.8 wt% Mg alloy, finding that subgrain formation did not significantly influence the flow stress.

Hasegawa et al [23,102] have compared the subsequent behavior of a cyclically pre-deformed aluminum alloy with the behavior following monotonic loading to the same stress level. Cell formation was more extensive in the cyclically deformed specimens due to larger accumulated strains, but, like the work here, the comparison was made with the dissimilar structures at the same initial extent of work hardening. Although the high temperature recrystallization behavior was altered by the extent of cell wall formation, no influence on the subsequent work hardening behavior was observed at moderate temperatures.

Obviously, the unique properties of screw dislocations in a bcc lattice are not responsible for the lack of influence in these cases. Rather, it appears that in a duplex structure composed of cell walls (or subgrain boundaries) and cell or subgrain interiors, interactions with the cell walls are not neccesarly responsible for work hardening, and that the rate limiting step in the deformation will in many cases occur in the interiors. Any mechanism which sufficiently impedes dislocation motion in the cell interior will decrease the relative influence of the walls, and, under some commonly used deformation conditions in both bcc and fcc alloys, they may have no measurable effect on work hardening.

As will be discussed in section V.E on modeling the role of cell walls, for this to be true, continuity requires that the cell walls be relatively soft, with respect to the more difficult, rate controlling step in the cell interior. Although the extremely high dislocation density of a typical cell wall appears virtually impenetrable, it may be that they are in fact "soft", in the sense that they rearrange easily, and thereby accommodate strain.

V.B. Controlling Structural Feature in Single Parameter Model

A rigid shift along the strain axis causes initially dissimilar curves to coincide, and therefore a single scalar parameter is adequate for describing the structure of this material, at least for single loading paths at room temperature and below. All structural developments relevant to work hardening must depend on the development of only one independent structural feature. Any other development is either dependent, or it is uninfluential. An obvious candidate for the controlling structural feature is the screw dislocation density, since the easy motion of the edges appears to be coupled to the restricted motion of the screws. The cell walls have been seen to be uninfluential, as their suppression has left no observable effect.

For bcc materials deformed at low temperature, the deformation is accomplished by double kink nucleation and propagation on screw dislocations. Although the detailed description may be a subject of continued investigation, the general form of the rate equation governing this behavior has been established. The strain rate is expected to be proportional to the length of the mobile segments, since this increases the rate of kink nucleation, inversely dependent on the distance between pinning sites, and exponentially dependent on the stress level and temperature [72,99].

All the explicit structural dependence is contained in the pre-exponential term, which depends on both the mobile dislocation density, and density of pinning sites. Description of the structure at a given stress level, in a deformation regime controlled by double kink formation and propagation requires two structural parameters.

It is apparent from the data that in the regime of interest here, the relevant pinning sites are those occurring in the cell interiors. Reactions at and within the cell walls are not associated with the rate limiting step in the deformation process. In low carbon steels, at strain levels well beyond yield, the overwhelming majority of the pinning sites are assumed to be forest dislocations. Demonstration of single structure parameter behavior therefore

indicates that the mobile and forest densities in the cell interiors are coupled, or more precisely, that they are macroscopically indistinguishable.

The direction of the shift required to cause the curves to coincide is of interest, as it represents the relative extent of development of the controlling structural feature during prestraining. Except at the lowest strain rates, which are perhaps ambiguous as described, the low temperature structure shows an initially lower stress level (in the cases which require a shift), whether the deformation is continued at low or at room temperature (Figures 12, 15, 16, 24). Apparently the development of the relevant parameter with strain has been slightly retarded at low temperature with respect its development at room temperature.

Since the imposed strain rate is fixed, the product of the mobile dislocation population and the average velocity must be similar during both prestrains ($\gamma = \rho_m b \langle v \rangle$). It is not clear however that these may be separated to allow a conclusion on the relative mobile populations at the end of the low and room temperature prestrains. At lower temperatures the screw mobility is more restricted, yet the higher stress level may raise the average velocity, or may drive the activation of more sources, increasing the mobile density.

As described, evidence of single structure parameter behavior in the deformation regime of interest here implies that the mobile and forest densities are coupled, so that either one is sufficient for "fixing" the state of the material. An attempt was not made to examine in more detail the developments which cause the average density in the more homogeneous structure to respond in an equivalent manner to the density in the cell interiors of the more cellular structure. Performing this analysis would require an accounting of the mobile and forest dislocation populations, at various strain levels, preferably measured on the as-loaded structures. It can only be concluded that the populations have not saturated, since work hardening is still occurring at these strain levels ($\cong 1.0$).

The relevant populations are those in the cell interiors, and not the total population which would include the dislocations which comprise the cell walls. The dislocations at the cell walls are removed from a description of the state in this regime, as they act neither as effective pinning sites, nor as a mobile population carrying out a portion of the strain.

VI.C Loading Path Changes

VI.C.1 Stress Reversals

As seen in Figures 26 through 30, the sensitivity to a stress reversal does not appear to be strongly related to the extent of cell wall formation. The magnitude of the softening is however, dependent on the extent of work hardening at the point at which the stress reversal was applied. This bias develops in the work hardened structure during prestraining, and is more apparent as the prestrain level is increased. However, as discussed in Section IV.B.2, this bias does not result from cell wall formation.

In a typical Bauschinger test, a stress reversal causes a lowering of the flow stress, which is identified with the build up of internal "back stresses" during the initial loading path. These back stresses cause a portion of the work hardening to behave as though it were reversible. A biased structure is formed which impedes further dislocation motion in the "forward" direction but assists dislocation motion in the "reverse" direction much in the manner in which dislocation pile-ups are typically modeled (although the behavior is not restricted to structures in which substantial pile-ups are observed). The magnitude of this directionality in work hardening is measured by the extent of softening observed when the load is reversed.

The development of cell walls appears to result in a polarized structure, being built up generally of dislocations of one sign on one side of a wall, and of opposite sign on the other. Whether the internal stresses associated with the development of the walls are significant or not depends on the relative magnitudes of the stresses required to move

dislocations in the cell interiors, and the stresses associated with rearranging the dislocations inside the walls. If the internal stresses associated with the walls are sufficiently high, as predicted for example in Mughrabi's model, the extent of cell wall formation at the point of the stress reversal would necessarily influence the magnitude of the Bauschinger effect.

However, an influence of this sort would have been inconsistent with the results of the single loading path tests, as it would imply that the walls themselves play an active role. If a substantial build up of back stresses, measured during a stress reversal, is associated with the walls, these back stresses should also influence work hardening in the forward direction. Suppression of cell wall formation by prestraining at lower temperatures would then have a measurable influence during monotonic loading, yet this was not observed.

It has been observed [25,58,80,82] that the softening following a stress reversal in cellular substructures is accompanied by a partial unknitting of the cell walls during the reversed loading sequence. This lends credibility to the hypothesis that the dislocations which compose the polarized cell walls are able to provide an easy deformation path during a stress reversal, and are thereby responsible for the softening observed.

The insensitivity to the extent of cell wall formation observed here indicates that although the unknitting of the walls may initially provide easy sources, its role in determining the macroscopic flow stress is limited in the deformation regime of interest here. This result is consistent with the interpretation of the results of sections IV.A and IV.D, that the flow stress is ultimately determined by the sluggish motion of dislocations moving in the cell interiors, at least at room temperature and below.

V.C.2 Orthogonal Path Changes

As in the monotonic and stress reversal loading sequences, the response to an orthogonal path change does not appear to be closely related to the formation of the dislocation cell walls, at room temperature and below. However, the preferential elongation of the grains in the prestrain direction during prestraining does appear to affect the response. The influence of this grain shape texture can readily be investigated in these loading sequences. Both the initial raising of the flow stress, and the susceptibility to unstable flow, seen as a load drop when the higher prestrain level (0.17) is followed by orthogonal loading, are increased when the orthogonal path change is accomplished by shearing perpendicular to, rather than along, the prestrain tensile axis (see Figure 36).

The orthogonal loading path change causes increased dislocation activity on previously latent, or at least less preferentially oriented slip systems. Dislocations moving on these newly activated systems must cut through an already work hardened microstructure, in which the previously mobile population has become oriented as an array of "forest" dislocations. The subsequent increase in the flow stress is therefore expected, and has been observed in most single crystal latent hardening experiments, as well as in polycrystalline steels sheets.

Previous work [82-85], has included similar orthogonal loading path changes in low carbon steels, for prestrain levels of up to 0.3, without the temperature change. For sufficiently high prestrain levels, an increase in the flow stress was followed by structural softening, which was been identified with the formation of microbands. The transient response was found to increase with the prestrain amount, and was also more pronounced when the shearing was performed perpendicular to the prestrain axis (0° , in the notation used here) rather than along it at 90° .

A sketch of this geometry is shown in Figure 42, where the symmetry of the two loading sequences can be seen. The untested sheets have only a slight texture along the

rolling direction, and the behavior is nearly isotropic in the sheet plane. If it is assumed that the slight initial texture is overwhelmed by plastic strains above 15%, the only nonsymmetry between the two sequences (90° vs 0°) is in the orientation, during the second loading path, of the elongated grains and of the elongated cell walls in the grain interiors.

It has been demonstrated here that the response to orthogonal loading changes is not dominated by the extent of development of the cell walls (Figures 31-34]. Therefore, the enhanced response of the 90° specimen with respect to the 0° specimen at a given prestrain level appears to be related to the orientation of the grain boundaries during the second loading path.

V.D Cell Formation

An explanation for the temperature dependence of cell formation in bcc metals and alloys must be consistent with the experimental data here that the extent of cell formation does not necessarily affect the flow stress. It must also account for the experimental observation made by many researchers that the flow stress at a given temperature is related to the extent of cell formation (characterized for example by the cell size).

The present work indicates that cell formation is perhaps controlled by the stress level, rather than the converse, as proposed by some of the models currently found in the literature. At low temperatures the stress level required to move the screws is high, and a strong temperature dependence of the flow stress is observed. Edge segments, the majority of which are in the form of kinks at these temperatures, move very rapidly with respect to the screw segments. Due to the high stress level, they have a high tendency to pass through preexisting hard spots, such as tangles and solute clusters, and relatively few of them become trapped at a given site. The development of tangles into cell walls is thus

retarded, predominantly by the stress level, and also by the inadequate supply of edge segments which compose the walls.

At room temperature, the motion of screw dislocations in the cell interiors is still sufficiently difficult to be the controlling factor in the deformation path, but easier recombination of the screws allows the flow stress to be greatly reduced compared to that at lower temperatures. Larger edge segments are present, and the stresses moving them are lower, both of which enhance the tendency for these edge segments to become trapped, and accelerates the development of a cellular structure. If the strain accomplished by the motion of edges is coupled to the sufficiently restricted motion of the screws, the potential influence of the walls as effective pinning sites will not be a factor in work hardening, and the development of the cell structure will not itself influence the behavior.

When a given deformation path is imposed at a fixed temperature, both the flow stress and the extent of cell formation will increase monotonically. In this respect, they are in a one-to-one correspondence, and, provided the temperature and path are not altered, the structure might be adequately described by any parameter which characterizes the extent of deformation, including the current average cell size. A relationship between the flow stress and the chosen structural feature (cell size for example) will be observed even for cases in which the feature (the cell walls) plays only a passive role and does not directly affect the flow stress at all. To isolate the influence of the walls themselves during a given deformation sequence, it is necessary to vary the the extent of cell formation, preferably making the comparison at a fixed degree of work hardening, as was done here.

The increase in cell wall development with strain at a given temperature appears to be a natural consequence of the fact that continuity requires an incremental increase in screw density be accompanied by an increase in edge density, even if the edge "segments" are in the form of kinks. At a given temperature, work hardening is observed as the straining is continued because increases in the forest density further impede the motion of the screws. These interactions are the result of increased activity on secondary slip

systems. As the total density increases, and more and more edge segments moving on multiple slip systems are forced to interact, cell formation will eventually become unavoidable at sufficiently large strains, even at lower temperatures, unless failure intervenes first. The transparency of the walls, or their lack of influence, in the cases studied here, is due to the fact that the rate controlling step in the deformation is neither associated with the preexistence of the walls, nor with their formation.

It has been observed in-situ that the cell walls rearrange during the deformation, while new walls are forming. The experimentally measured decrease in the *average* cell size during isothermal, monotonic deformation is caused by a combination of this rearrangement and new formation. If the walls are hard, yet forced to rearrange, their absence (for example in the specimens here prestrained at low temperature) would necessarily have an influence. The results for the deformation regime here indicate they do not.

Furthermore, the observation that the cell wall habit plane tends to lie on or near an active slip plane during monotonic loading and perpendicular to the active slip planes during cyclic loading may also indicate the relative ease of rearrangement. Since many materials neither cyclically harden or soften, it is not plausible, at least in those cases, to conclude that the arrangement of the cell walls is a dominant source of work hardening.

In conclusion, it is proposed here that the tendency to form a cellular substructure is dependant on the flow stress, and on the type of dislocations present in the mobile population. The dependance is not mutual: the walls do not necessarily affect the flow stress. Whether or not the presence of the cell walls has an influence on the plastic behavior depends on the relative magnitude of the stresses to move dislocations in the cell interiors, compared to the interaction stresses with the cell walls.

V.E. Implications to Models

In the models in which the cell structure plays an active role in controlling the flow stress, the walls are considered as hard regions. In the Kuhlmann-Wilsdorf model, they act as the dominant pinning site for dislocations moving in the cell interiors. In for example the Muhgrabi or Hansen models, the walls themselves deform with plastic properties different than those in the cell interiors, and these properties determine the flow stress. In the results found here, the presence of the cell walls, had no observable influence even in a regime in which their formation is extensive. If the cell walls are "hard" their behavior will control the flow stress, unless an easier deformation path exists independent of the walls. Therefore the only way to explain the present results in manner consistent with the predications of a two-phase, or hard cell wall type model is to identify an easier deformation path, independent of the cell walls, which is plausibly available at these strain levels.

In the two-phase models, the cell interiors and cell walls are loaded in parallel, and accommodate the same strain. This type of loading, or a variation of it, is in fact required by continuity. During large plastic deformations, the cells cannot be responding independent of each other, as void formation is not observed at conditions of interest here. Furthermore, the temperature range ($< 0.2 T_m$) is well below creep conditions where accommodation stresses might be easily relieved. Therefore, a deformation path independent of the cell walls does not appear to exist, and the cell interiors and the walls must be deforming together. It follows then that the absence of an influence of the cell walls on the flow stress implies that the transmission of strain in the walls is the easier step in deforming a "two-phase" structure, which consists of a more difficult, and therefore rate-limiting step in the cell interiors, and a rearrangement process in the developing cell walls.

Regarding the apparent "hardness" of the dense cell walls, as observed in-situ a given dislocation does not pass through the wall without resistance. Rather, incoming dislocation segments are incorporated into the wall, and "new" dislocations eventually emitted at the far side. In this manner some strain is accommodated in the walls, as they are forming. This strain is apparently accomplished by a rearrangement of the dislocations which compose the walls, followed by "transmission" to the neighboring cell interiors, and is not necessarily associated with high stress levels.

A Kuhlmann-Wilsdorf type model in which the flow stress is controlled by dislocation interactions with the cell walls is clearly inconsistent with the tensile and tensile-shear test measurements. Even if a modification of the model is made to account for the non-circular bowing expected in bcc metals, and any reasonable definition used for the cell size during the interrupted temperature tests, it is not possible to fit a dependence of the flow stress on the cell size to the data. As discussed in the previous section, a correspondence of this type will work however for any strictly monotonic, isothermal deformation path, since the extent of cell formation increases monotonically with the relevant structural feature, the cell interior dislocation densities. This does not imply a dependence on the cell structure, but just represents the use of a secondary feature to account for the evolution of the structure for the case of monotonic deformation.

It is quite common to express the plastic flow stress as proportional to the square root of the dislocation density (either total or forest), with a proportionality factor which is thought to account for the arrangement of the dislocation population, plus an additive frictional term which is not structurally dependent:

$$\sigma_f = \alpha G b \rho^{1/2} + \sigma_0(\epsilon, T) \quad (5)$$

This is a two parameter model; both the dislocation density and the constant are needed to fix the structure at a given temperature and strain rate.

As dislocation density measurements were not attempted in the work here, the validity of such a model cannot be directly verified. However, if the consequences of this

model are applied to the results, for example the behavior demonstrated in Figure 3, it can be shown that consistency requires that the proportionality factor to be the same for the dissimilar prestrain structures. In terms of the model, this indicates that the arrangement of the total densities are similar, which they are obviously not, or that the arrangement is of no consequence. More probably, it is not the total dislocation density which belongs in such an expression for the case here, and a proper accounting of the dislocation population, using only the screws moving in the cell interiors, would more accurately predict the behavior.

Essentially such an analysis is provided by Luft and co-workers, who measured separately the total dislocation density and the straight screw density in low temperature Molybdenum structures in which extensive tangles were observed. When the total density was used to fit an equation of this form, the fit was not good at large strains, where tangles were more developed. If instead only the long straight screw density between the tangles was used, the fit was good at all strain levels. Thus the tangles, which are the embryos of cell wall development, did not influence the flow stress. Furthermore, saturation was reached for some conditions, and density of the screw population remained constant during a plateau in the flow stress, independent of further development of the tangles.

In general, similarities in the tendency for cell formation in most fcc and bcc materials, combined with the dissimilarities in their temperature dependence and work hardening characteristics, makes it difficult to defend the generality of models in which the cell walls uniquely control the flow stress. The deformation of a cellular substructure consists of two steps: that of transmitting strain at or through the cell walls, and that of accommodating strain in the interiors between the walls. It can be seen here that either step in this process may be rate limiting, and an appropriate description of the structure will depend on which step is controlling the flow stress.

VII. CONCLUSIONS

The cellular dislocation substructure which forms during plastic deformation of low carbon steel sheets appears to have little or no influence on the room temperature work hardening of these materials. The flow stress at room temperature and below is dominated instead by the sluggish motion of screw dislocations which move in the cell interiors. It is already well established that the low temperature behavior of most bcc metals is determined by the unique properties of the $\langle 111 \rangle$ screws, whose low energy configuration is sessile. This investigation indicates that the range of conditions over which this behavior dominates extends into the temperature range at which well developed cells are formed, and that cell walls do not necessarily influence the flow stress.

A single structural parameter, independent of cell wall formation, is adequate for describing the flow stress at and below room temperature, for monotonic loading paths. Prestraining at a different temperature only changes the extent of development, and not the nature, of the controlling structural feature. As the limiting step in the deformation process appears to be the restricted motion of screw dislocations in the cell interiors, this parameter must be associated with the cell interior screw density, and not the total density, which would include the predominantly edge dislocations which compose the cell walls. Above room temperature (100°C), a single structural parameter model is no longer adequate.

Following sufficiently large prestrains (at least 10%), an orthogonal change in the loading path at room temperature causes the initially increased flow stress to be followed by structural softening, seen as a load drop. This response is also not closely associated with the development of the dislocation cell structure, but is affected by the prestrain texture.

A transient behavior in work hardening is observed when the loading path is reversed. Softening following a stress reversal increases with prestraining, and is long term, in that the behavior does not approach monotonic behavior well away from the point at which the load was reversed. This transient occurs similarly in well developed cell

structures and in structures in which cell formation was substantially suppressed. The magnitude of the "back stresses" thought to be present as a result of cell wall formation are therefore relatively small with respect to the flow stress required to move screw dislocations in the cell interiors in this material at room temperature.

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100 μm

Figure 1. Optical micrograph showing grain morphology of the as received low carbon steel sheet, supplied by General Motors Corporation. The rolling direction is horizontal in the photo.

XBB 899-8069

in-situ specimen
strained in
High Voltage TEM

Figure 2. Specimen configuration for tensile testing and in-situ straining experiments. The central gauge section of the macroscopic specimen was thinned prior to cutting the in-situ specimen.

Figure 3. Apparatus for low temperature tensile testing of large sheet specimens.

Figure 4. Configuration of shear specimens cut from large tensile prestrained specimens.

Cutting angles with respect to tensile axis define the loading path change:

0° , 90° = orthogonal path change, 45° = continuous loading, 135° = stress reversal

0.5 μm

Figure 5. TEM micrograph of dislocation structure after tensile straining at -72°C to $\epsilon_p = 0.10$. Note homogeneous distribution of long straight screw dislocations, typical of low temperature deformation.

XBB 892-1302

0.5 μm

Figure 6. TEM micrograph of dislocation structure after tensile straining at room temperature to $\epsilon_p = 0.10$, showing extensive cell formation.

XBB 892-1300

100 μm

Figure 7. Optical micrograph showing surface slip on a specimen polished prior to straining at room temperature to $\epsilon = 0.10$.

XBB 890-8486

100 μm

Figure 8. Optical micrograph showing surface slip on specimen polished prior to straining at -72°C to $\epsilon = 0.10$.

XBB 890-8485

100 μm

Figure 9. Optical micrograph showing grain morphology at the end of room temperature straining to $\epsilon = 0.10$. The tensile axis is parallel to the sheet rolling direction, and is horizontal in the photo.

XBB 899-8971

100 μm

Figure 10. Optical micrograph showing grain morphology at the end of straining at -72°C to $\epsilon = 0.10$. The tensile axis is parallel to the sheet rolling direction, and is horizontal in the photo.

XBB 899-8970

Figure 11. Tensile stress-strain curves at $\dot{\epsilon} = 10^{-3}\text{s}^{-1}$. The discontinuous curve corresponds to a specimen prestrained at -72°C to $\epsilon = 0.10$, then continued at room temperature, and the continuous curve to a specimen strained at room temperature only.

Figure 12. Tensile stress-strain curves at $\dot{\epsilon} = 5 \times 10^{-2} \text{s}^{-1}$. The discontinuous curve corresponds to a specimen prestrained at -72°C to $\epsilon = 0.10$, then continued at room temperature, and the continuous curve to a specimen strained at room temperature only.

(a) As tested, and (b) After shifting the discontinuous curve along the strain axis.

Figure 13. Tensile stress-strain curves at $\dot{\epsilon} = 10^{-4} \text{s}^{-1}$. The discontinuous curve corresponds to a specimen prestrained at -72°C to $\epsilon = 0.10$, then continued at room temperature, and the continuous curve to a specimen strained at room temperature only.

(a) As tested, and (b) After shifting the discontinuous curve along the strain axis.

Figure 14. Tensile stress-strain curves at $\dot{\epsilon} = 10^{-3}\text{s}^{-1}$. The discontinuous curve corresponds to a specimen prestrained at -72°C to $\epsilon = 0.05$, then continued at room temperature, and the continuous curve to a specimen strained at room temperature only.

Figure 15. Tensile stress-strain curves at $\dot{\epsilon} = 10^{-3}\text{s}^{-1}$. The discontinuous curve corresponds to a specimen prestrained at room temperature to $\epsilon = 0.10$, then continued at -72°C , and the continuous curve to a specimen strained at -72°C only. (a) As tested, and (b) After shifting the discontinuous curve along the strain axis.

Figure 16. Tensile stress-strain curves at $\dot{\epsilon} = 5 \times 10^{-2} \text{s}^{-1}$. The discontinuous curve corresponds to a specimen prestrained at room temperature to $\epsilon = 0.10$, then continued at -72°C , and the continuous curve to a specimen strained at -72°C only. (a) As tested, and (b) After shifting the discontinuous curve along the strain axis.

Figure 17. Tensile stress-strain curves at $\dot{\epsilon} = 10^{-4}\text{s}^{-1}$. The discontinuous curve corresponds to a specimen prestrained at room temperature to $\epsilon = 0.10$, then continued at -72°C , and the continuous curve to a specimen strained at -72°C only. (a) As tested, and (b) After shifting the discontinuous curve along the strain axis.

Figure 18. Tensile stress-strain curves at $\dot{\epsilon} = 10^{-3} \text{ s}^{-1}$. The discontinuous curve corresponds to a specimen prestrained at -72°C to $\epsilon = 0.10$, then continued at 100°C , and the continuous curve to a specimen strained at 100°C only. (a) As tested, and (b) After shifting the discontinuous curve along the strain axis.

XBB 913-1733

1 μm

Figure 19. TEM micrograph showing dislocation cell formation after tensile straining at room temperature to $\epsilon = 0.17$.

Figure 20. TEM micrograph showing dislocation structure after tensile straining at -75°C to $\epsilon = 0.17$. Compare with Figure 19, which shows the structure after room temperature straining to $\epsilon = 0.17$.

XBB 913-1732

Figure 21. TEM micrograph showing dislocation structure after tensile straining at -75°C to $\epsilon = 0.17$, as in Figure 20.

XBB 913-1731

Figure 22. Room temperature shear stress- shear strain data on Sollac supplied sheets, tested at $\dot{\gamma} = 10^{-3} \text{ s}^{-1}$. The specimen corresponding to the open symbol curve was prestrained in tension at -75°C to $\epsilon = 0.17$, for the second (solid) curve the same prestrain was performed at room temperature. Specimens were cut at 45° from the tensile prestrain axis, giving monotonic loading when continued in shear.

Figure 23. Shear stress-strain curves on GMC supplied sheets, following prestraining to 0.07 at -72°C (open symbols) and room temperature (solid symbols). Shearing was performed along 45° with respect to tensile prestrain axis (monotonic loading), at $\dot{\gamma} = 10^{-3}\text{s}^{-1}$.

Figure 24. Shear stress-strain curves on Sollac supplied sheets, following prestraining to 0.10 at -80°C (open symbols) and room temperature (solid symbols). Shearing was performed along 45° with respect to tensile prestrain axis (monotonic loading), at $\dot{\gamma} = 10^{-3}\text{s}^{-1}$. (a) As tested, and (b) After shifting along the strain axis.

Figure 25. Shear stress-strain curves on Sollac supplied sheets, following prestraining to $\epsilon = 0.17$ at -75°C (open symbols) and at room temperature to $\epsilon = 0.10$ (solid symbols). Shearing was performed along 45° with respect to tensile prestrain axis (monotonic loading), at $\dot{\gamma} = 10^{-3}\text{s}^{-1}$. (a) As tested, and (b) After shifting along the strain axis.

Figure 26. Shear stress-strain results following a stress reversal (135°), for GMC supplied sheets prestrained at 10^{-3}s^{-1} to $\epsilon = 0.07$ at room temperature (solid curve) and at -72°C (open symbols). Room temperature shearing was performed at $\dot{\gamma} = 10^{-3}\text{s}^{-1}$.

Figure 27. Shear test results showing the influence of a stress reversal following tensile prestraining at -72°C to $r = 0.07$, and continued at room temperature in shear at 45° (monotonic loading, upper curve), or at 135° (stress reversal, lower curve).

Figure 28. Shear test results showing the influence of a stress reversal following tensile prestraining at room temperature to $\epsilon = 0.17$, and continued in shear at 45° (monotonic loading, upper curve), or at 135° (stress reversal, lower curve).

Figure 29. Shear test results showing the influence of a stress reversal following low temperature tensile prestraining at -75°C to $\epsilon = 0.17$, and continued at room temperature in shear at 45° (monotonic loading, upper curve), or at 135° (stress reversal, lower curve).

Figure 30. Relative influence of stress reversal on specimens previously prestrained at room temperature (solid symbols) and at low temperature (-75°C , open symbols), then continued in shear at room temperature at 135° (stress reversal).

Figure 31. Relative influence of orthogonal loading path change (sheared at 0° with respect to the prestrain tensile axis), for specimens prestrained to $\epsilon = 0.07$ at room temperature (solid symbols) and at low temperature (-72°C , open symbols). Room temperature shear testing was performed at $\dot{\gamma} = 10^{-3}\text{s}^{-1}$.

Figure 32. Shear stress strain test results showing influence of orthogonal loading path change on specimens prestrained at -72°C to $\epsilon = 0.07$, and continued at room temperature in shear at 0° with respect to the prestrain tensile axis (upper curve), or at 45° (monotonic loading, lower curve). GMC sheets, at $\dot{\gamma} = 10^{-3}\text{s}^{-1}$.

Figure 33. Relative influence of loading path change following prestraining to $\epsilon = 0.10$ at -80°C (open symbols), and room temperature (solid symbols). Sollac sheets, at $\dot{\gamma} = 10^{-3}\text{s}^{-1}$. (a) As tested. (b) After rigid shift along the strain axis.

Figure 34. Shear stress strain curves at $\dot{\gamma} = 10^{-3}\text{s}^{-1}$, corresponding to specimens prestrained to $\epsilon = 0.17$, and continued at room temperature in shear at 0° (orthogonal loading path change). Sollac sheets, prestrained at -75°C (open symbols), and at room temperature (solid).

Figure 35. Shear stress strain curves showing influence of strain rate, following orthogonal loading path change, here applied to specimens prestrained at -75°C to $\epsilon = 0.17$, and continued at room temperature at $\dot{\gamma} = 5 \times 10^{-2} \text{ s}^{-1}$ (upper curve), or at $\dot{\gamma} = 10^{-3} \text{ s}^{-1}$ (lower curve).

Figure 36. Shear stress strain curves showing influence of cutting at 0° versus 90°, with respect to the tensile prestrain axis. Both orthogonal loading path changes shown were applied to specimens prestrained at room temperature to $\epsilon = 0.17$, and continued at room temperature at $\dot{\gamma} = 10^{-3}\text{s}^{-1}$, either by loading in shear along the tensile axis (0°, lower curve), or perpendicular to it (90°, lower curve).

1.0 μm

Figure 37. TEM micrographs showing evolution of the structure following a temperature change. Upper photo: Structure after continuous room temperature straining to $\epsilon = 0.20$. Lower photo: Structure after prestraining at -72°C to $\epsilon = 0.10$, followed by room temperature straining to an accumulated strain of $\epsilon = 0.20$.

XBB 898-6631

Figure 38. TEM micrograph showing evolution of structure following a temperature change. The specimen was prestrained in tension at -72°C to $\epsilon = 0.07$, then continued in shear, at 45° (monotonic loading), at room temperature to $\gamma = 0.09$ (total shear strain of approximately 0.19).

XBB 913-1729

Figure 39. TEM micrograph showing evolution of structure following a temperature change. As in Figure 27, the specimen was prestrained in tension at -72°C to $\epsilon = 0.07$, then continued in shear, at 45° (monotonic loading), at room temperature to an accumulated $\gamma = 0.19$.

XBB 913 1730

Figure 40. Sequence of TEM micrographs taken from the in-situ straining film, on specimen prestrained at room temperature.

NBB 897-5991A

XBB 895-3859

Figure 41. TEM micrograph of loaded structure taken during in-situ straining, at 1.5 MeV.

Figure 42. Schematic of symmetry between orthogonal loading path changes accomplished by shear loading along, and perpendicular to, the tensile prestrain axis.

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