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William J. Salesky
(Ph.D. thesis)

November 1982

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ON FUNDAMENTALS OF SLIDING WEAR IN STEEL

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ON THE FUNDAMENTALS OF SLIDING WEAR IN STEEL

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ON THE FUNDAMENTALS OF SLIDING WEAR IN STEELS

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ABSTRACT

The mechanism of unlubricated sliding wear in ferrous metals has been investigated utilizing the techniques of both scanning and transmission electron microscopy. The deformation structures formed in ferritic, pearlitic, spherodized, and martensitic steels as the result of sliding wear have been characterized. Diffraction contrast in TEM was used to image the deformation structures in cross-sections made perpendicular to the wear surface. Fine substructures of high dislocation density were observed in the near surface regions. Subsurface cell dimensions suggest shear strains of at least 4. In several instances, a 200-300nm wide zone of lower dislocation density indicative of recovery was noted (immediately below the surface). Cracks appeared to be formed at the boundary of this zone along dislocation cell walls.

The mechanism of wear particle formation during unlubricated sliding wear has also been investigated. Many hundreds of particles were collected from sliding wear experiments. Using SEM automatic image analysis, the mean of the size distribution was determined to be 1-2microns in width. Detailed study of individual debris particles has revealed that they are platelike--typically 200 to 400nm thick. The particles are generally iron oxides except when testing was done at low tempera-
tures or in inert atmospheres.

The results of this study suggest that for several iron-based materials, tested in air, wear particles are formed by crack initiation and propagation along the boundary of dislocation cells. In materials that undergo recovery, crack initiation is more likely at the boundary of the recovered region adjacent to the wear surface. Oxidation then occurs during separation of the platelet from the parent material. The transformation from metal to oxide during separation of the debris from the substrate is a consequence of the highly pyrophoric nature of thin metal flakes. A new mechanism for sliding wear will be discussed in light of these considerations.

The results of the study suggest the definite connection between the material microstructure and the observed wear behavior. Materials with a fine-scaled microstructure and a high yield strength in the absence of large undeformable particles, appear to be the most wear resistant. Rapid thermal cycling through such surface treatments as laser and electron beam lock the alloying elements in solution and refine the microstructure sufficiently to effectively increase the sliding wear resistance. Surface treatment appears to be a viable and cost-effective method for increasing the wear resistance of a component or wear system.
I. INTRODUCTION

A) Significance of the Study

This significance of wear in every day life often goes unnoticed until a catastrophic failure occurs. The economic expense of all forms of wear in the United States, for just the materials involved, is estimated to be over 15 billion dollars annually with the total 'wear' expense (including lost productivity and labor) to exceed several tens of billions of dollars (ref. 1). With the national debt exceeding 1 trillion dollars, reduction of this expense would be of great stimulus to the economy. Hence, control and elimination of wear is of immediate importance.

Wear is not a new problem. Research in the field has been conducted for hundreds of years, involving such notable individuals as Di Vinci and Coulomb (ref. 2). Tribological investigations have engaged a wide cross section of academic and industrial professionals including materials scientists, chemists, physicists, biologists, as well as mechanical, civil, chemical, and lubrication engineers. Thus, there are many approaches and perspectives to wear problems. Correspondingly, there is a large volume of literature published with widely divergent views on the subject. Yet to this date there is no full understanding of the mechanisms of wear. Though surprising at first, this ignorance is understandable as one reviews the history of the field.

The absence of a fundamental understanding stems from two characteristic problems of the field. First, from an engineering standpoint, when a problem with wear arises in an industrial setting,
the motivation due to economic pressures is to quickly find a solution rather than to reach an understanding of the underlying fundamentals. Hence, a large volume of the wear literature is engineering data obtained through experimentation to solve a problem, often by trial and error.

The second problem relates to the nature of most forms of wear. Generally, wear is defined as the loss or displacement of material from a surface. By such a broad definition, the study of wear has evolved into a vast field. It is often stated that the number of classification systems in wear is not less than the number of researchers (ref. 1). But, the classification system most widely accepted is that which categorizes wear by the general system in which it occurs such as:

(a) sliding
(b) abrasion
(c) fretting
(d) corrosion
(e) rolling-fatigue
(f) galling
(g) erosion
(h) impact

Unfortunately the problem of classification is further complicated by more than one form of wear occurring in the system at one time. This is frequently observed in industry where, for example, abrasion, erosion, or impact wear may take place in a corrosive environment, such as water.

Wear is not a property of a material but a characteristic of a system. Consequently there are many variables which affect wear such as:
(a) geometry
(b) loading (or stress distribution)
(c) atmosphere
(d) presence of lubricants
(e) moisture (or relative humidity)
(f) material couple
(g) speed of relative motion
(h) ambient temperature
(i) load cycle
(j) duration etc.

Understanding the individual influence of each variable in such complicated systems is a monumental task. Isolating the individual influence of one variable is often impossible without affecting a second variable. Therefore, design of critical and definitive experiments is often difficult.

Unfortunately, wear theory has largely developed by implication and conjecture rather than by direct experimental evidence. Only a few detailed experimental investigations have been systematic and sufficiently sophisticated experimentally to characterize the specific details of the wear system, especially the microstructural influence. In depth scientific investigations to elucidate the influence of material parameters are therefore desperately needed to gain an understanding of sliding wear from the most fundamental perspective possible. However, due to the large number of variables which significantly affect wear, it is unlikely that there exists for all materials a unique mechanism, even for a particular form of wear.

B. Objectives

The principal objective of this study is to gain a deeper understanding of unlubricated metal on metal sliding wear through systematic investigation using the most advanced analytical techniques
available. The approach to the problem is based on a fundamental material perspective. Characterization of the 'worm' microstructure is performed. The study focuses on iron-based materials due to their industrial significance and lack of detailed study. The emphasis is to relate the changes in the material microstructure to the observed wear behavior and establish a more fundamental understanding of unlubricated sliding wear.

C. Background and Theory

To understand developments in the sliding wear field a review will be made of the mechanisms which have attracted attention over the years. The major points as well as the limitations of each theory will be presented. Since approaches to elucidating the sliding wear mechanism encompass different philosophies, the literature review is broken into three categories:

(a) Adhesion
(b) Fatigue
(c) Delamination

This structure will simplify the presentation of the mechanisms.

(a) Adhesion

The adhesion theory of wear proposed by Archard (refs. 3 and 4) was largely based on the theory of friction of Bowden and Tabor (ref. 5). Bowden and Tabor recognized that two contacting surfaces touch only at a number of contact spots constituting a small fraction of the total geometric contact area. Contact spots occur at protruberences on the surface, termed asperities. Bowden and Tabor
suggested that surface asperities adhere (or weld) at these contact spots to form junctions. The frictional force is then directly related to the forces needed to shear these junctions. However, since the model assumes homogenous and isotropic properties of the materials, it is somewhat unrealistic. Yet, it has provided important insights into both friction and wear (ref. 6).

Archard's theory was intended for prediction of wear. The amount of wear \( W \) was related to the applied load \( L \), sliding distance \( S \), and the flow pressure of the material \( P \) as follows:

\[
W = \frac{KL}{3P} S
\]

Archard's theory assumes that the true contact consists of a number of circular contact points, the area of which is determined by a plasticity relation and that hemispherical wear particles are formed with the probability \( k \) (ref. 7). Generally, for mild wear \( k = 10^{-7} \) to \( 10^{-8} \); for severe wear \( k = 10^{-3} \) to \( 10^{-4} \).

Archard's linear rate law indicates that wear is proportional to sliding distance and load, yet is inversely proportional to the hardness of the material. This is more easily seen if one approximates the relation between hardness and flow strengths to be: \( H = P/3 \). This results in the well-known form of Archard's law:

\[
W = \frac{KL}{H} S
\]

There are several major problems with this theory. First, no allowance in the theory is made for loose wear debris, characteristic of sliding wear. Second, hemispherical particles are rarely observed
in sliding wear except in lubricated systems, for which the spherical geometry is attributed to actions after formation (Most frequently, plate-like or wedge shaped particles are observed in sliding wear). Third, by Archard's theory, the size distribution of the debris would be on the scale of the asperity size, which is not in agreement with the wide size ranges typically observed. Fourth, there is some question whether adhesion would occur in the presence of surface oxides, which form to minimize the surface energy on most metal surfaces. Archard has suggested that the action of sliding would be sufficiently violent to break up the oxide. Finally, Archard's theory is linear, yet in reality wear is often non-linear. Most often there is a non-linear break-in period before a steady state wear rate is reached.

Although there are several problems with Archard's theory, the significant and long standing outcome of Archard's theory was the inverse relationship of wear rate with hardness (which has been experimentally observed). The shortcomings of the details of Archard's theory have, however, motivated other researchers to become acquainted with the wear field.

(b) Fatigue

The repetitive nature of practical sliding systems has lead several investigators to suspect fatigue mechanisms are operative in sliding wear. Application of the principles and concepts of fatigue has lead to interesting developments in wear theory. From Archard's probability \( (k) \) of wear particle transfer, Rozeanu suggested that repeated interaction of the asperities results in cumulative damage,
leading to eventual fracture (ref. 8). As noted by Kimura, a successive damage concept of wear, complimenting the adhesion theory, allows for a weak interaction to cause fracture of the asperity, resulting in detachment of a wear particle. This eliminates one of the essential shortcomings of the adhesion theory (i.e., formation of loose particles).

The asperity fatigue concept of wear was augmented by the work of Kraghelsky who postulated that wear results from a combination of stress and deformation, the magnitude of which depend on geometrical factors and adhesion forces (ref. 9). The Coffin-Manson damage accumulation (low-cycle fatigue) relationship was used to relate the deformation to the number of cycles (the reciprocal of which is the probability factor (k)).

Halling further contributed to this concept through the application of Greenwood and Williamson's probabilistic treatment to asperity contact (ref. 10). Based on an idealized mechanics approach, both contacting surfaces were assumed to be covered with asperities of hemispherical features. Deformation and fracture were to occur in one surface only. The model, however, only considers the normal approach of the surfaces.

Finkin criticized that Halling's assumption of all contacts participating in wear particle formation through a low cycle-fatigue process neglects the effects of elastic interactions. Finkin suggested that elastic interactions make a significant contribution to supporting the load but do not participate in the wear process. Finkin expounded on Halling's model assuming a more general contact model
(i.e., hill against a plane) allowing either elastic or plastic asperity interactions to contribute to wear through a strain ratio fatigue failure law (ref. 12). Finkin showed that this broadened analysis predicted the linear rate law. Significantly, the contact model used by Finkin does not limit the size of the wear debris to a value close to that of the asperities, but that failure within the substrate can generate any size particle (ref. 11). Nevertheless, the analysis is basically a mechanics approach for which the only material parameters employed were the strain to failure (in one cycle) and the strain to begin plastic deformation. Microstructure and hardening characteristics, which have been shown to strongly influence the amount of wear (refs. 13 and 14), were not addressed in this theory.

Distinctly different from that of Finkin is the approach of Kimura (refs. 7 and 15) which includes consideration of the state of the subsurface and its relation to wear particle formation (issues not considered in any previous mechanism). Kimura noted that the non-linearity of wear rate is not well understood, especially in the early stages of wear, and recognized that the surface is typically in a deformed state prior to wear, due to specimen machining and/or grinding. Damage in sliding was noted to extend into the substrate farther than the thickness of a single debris particle. Consequently, when a layer of particles is removed some damage has already accumulated in the surface which is newly exposed (ref. 7).

From the above observations, an idealized model of the surface layers consisting of uniformly sized blocks which detach one by one
was introduced (ref. 16), a concept akin to the formation of dislocation cells. The block size is determined by the material properties and mechanical conditions. Utilizing the principles that damage must accumulate to a critical point in the near surface regions before detachment of a block could occur, the probability \( P \) of the block detachment as a function of sliding distance \( n \) was derived:

\[
P(n) = \exp\left(\frac{-(E(h) - G(h))}{bF}\right).
\]

The maximum amount of damage the material can withstand is defined by \( E \); \( G \) denotes the amount of damage at the \( N \)th unit sliding distance; \( bF \) denotes the average surface damage per unit sliding; and \( h \) the position of the surface at the \( N \)th unit sliding distance. Though the values in the parameters are arbitrary, Kimura showed that various wear distance relations could be obtained (ref. 16). He interpreted this to mean that interaction of the damage caused by the sliding with that initially present can result in a non-linear relation even if there are no changes in the sliding conditions (ref. 7). Within the limitations of this model, it generally addresses the observed characteristics, although largely omitting the influence of microstructure.

(c) Delamination

This is a broad category that basically will encompass several other prominent theories for which the concept of delamination is a central theme.

It is often observed that the particles produced during unlubricated sliding wear are either partially or totally oxidized. Quinn proposed an oxidation mechanism of sliding wear (refs. 17, 18.
and 19) which involved oxide layer growth to a critical thickness followed by spallation of the oxide under the motion of the asperities thereby producing oxide wear debris.

The essence of Quinn's model is the linear wear law (of Archard). The parameter (k) is suggested to represent the reciprocal of the average number of asperity encounters needed for the critical oxide thickness to form atop an asperity. No failure mechanism related to fatigue or unstable fracture was proposed in the theory. Specifically, oxidation was suggested to follow the parabolic rate of oxidation in transition metals. The wear rate (W) was related to load (L), sliding speed (S), hardness (H) (of the material below the oxide), and the sliding distance (D) by:

\[ W = \frac{L O D}{S H f p C} \]

The oxidation rate constant (O) is given by an Arrhenius equation (containing the parabolic oxidation constant); (f) the fraction of oxide in the film; (p) the density of the oxide; and (C) the critical oxide thickness.

Although there is reasonably good agreement between the model and experiment, expressions for wear rate have a large number of parameters, some of which cannot be determined experimentally. In addition, although this model may clearly be valid at high sliding speeds where frictional heating is appreciable, at slower speeds oxide wear particles are also produced for which the oxide debris particle is several orders of magnitude thicker than the oxide on the wear surface. Quinn's proposals would not appear to apply here. This
situation has been observed in this study and will be discussed in greater detail later.

One of the most stimulating mechanisms developed in recent years has been that of Suh and his co-workers (ref. 18). Their delamination theory of wear model essentially suggests that wear occurs through deformation of the surface layer, nucleation of cracks in the subsurface regions, and propagation of the cracks nearly parallel to the worn surface to form loose sheet-like wear particles (refs. 20, 21, and 22). It was proposed in the original paper (ref. 22) that a soft layer existed beneath the immediate surface due to dislocation image force influence. Voids were said to form in the underlying heavily deformed layer and to coalesce to form cracks running parallel to the surface. Crack propagation occurs when the crack exceeds the critical crack length. Eventually, the crack propagates to the surface to form a plate-like wear particle (ref. 6).

The 'soft layer' aspect of this theory was later deleted due to the lack of experimental proof combined with the knowledge that image forces have a short range influence (on the order of 100's of angstroms) especially in the presence of surface oxides. TEM investigations by Doyle and Turley (ref. 23) and van Dijck (ref. 24) showed the dislocation density to be highest at the surface. Selected area electron channeling experiments on the worn surface, by Ruff (ref. 25), showed the strains at the surface to be the largest. Thus, these results dispute the soft-layer concept.

Suh cites the x-ray and electron diffraction work of Mitchell and Laufer (ref. 26) which he interprets to support the depleted layer
concept. However, there is some question of Suh's interpretation that the absence of dislocations within a well-defined dislocation substructure supports the existence of a depleted or soft layer. Clearly from dislocation mechanics in a well defined structure the dislocations that form within the grain eventually move and become entrapped in the cell wall so as to minimize the net Burgers vector of the dislocation cell wall. However, in any case, later updates of the theory by Suh concentrated on plastic deformation, strain accumulation, and fracture aspects of sliding wear (ref. 21).

Voids resulting in cracks were said to form some distance beneath the surface due to the triaxial compressive stress state imposed on the surface layer under the applied load during sliding. In a material containing hard carbides and/or inclusions, crack nucleation via matrix decohesion or particle fracture was suggested by Jahanmir and Suh to account for crack initiation (ref. 27). Although void nucleation, leading to cracking, is known to occur in the presence of carbides and/or inclusions in both uniaxial tension and pure shear (refs. 28-31), it was recognized by Jahanmir that his theory of crack initiation does not apply to single phase metals without inclusions. Consequently, the crack nucleation aspects of the delamination theory are still largely unclear for many metals. However, the fracture surface studies of Suh et al. certainly confirm that delamination effects are important in sliding wear.

A notable aspect of Suh's work was the calculation of the state of stress beneath a sliding asperity. Calculations (ref. 27) were made for an elastic-perfectly plastic plane sliding past a rigid,
stationary, curved, surface (the asperity). Using the analysis of
Merwin and Johnson (ref. 32) it was found that the regions ahead of
the slider, in the deformable elastic-perfectly plastic plane, were
plastic and in compression. The region behind the slider was found to
be in the elastic region but in tension. These calculations were
consistent with that of Smith and Liu (ref. 33) who calculated the
stress field under an asperity. But, neither calculation accounted
for plowing observed on the wear surface, which would substantially
modify the state of stress near the surface. Hence, these analyses
are apparently incomplete.

It is clear there is a stress cycle from plastic compression to
elastic tension upon passage of an asperity beyond a fixed point.
These results are significant in that they give an indication why
cracks open and propagate parallel to the surface. In the absence of
adhesion, a tensile stress is apparently necessary to cause separation
of the debris particle from the underlying materials.

As mentioned, delamination cracks were suggested by Fleming and
Suh (ref. 34 and 35) to grow incrementally rather than in one step as
in static fracture. Crack growth was suggested to occur in only the
tensile portion of the cycle (ref. 34). Growth was indicated to be
the limiting step in wear failure. The growth rate of the crack was
calculated to be less than or equal to $10^{-5}$ mm/cycle. A correlation
between wear rates and mode I fatigue crack propagation was suggested
from studies on 2024-T3 and 7075-T6 aluminum (ref. 35).

Ritchie (ref. 36) expressed that gross correlations between wear
rate and crack propagation rates in fatigue may be subject to
criticism. The wear crack propagation rates quoted by Fleming (ref. 35) were noted by Ritchie to be in a near threshold regime (Stage I), where the influence of microstructure is of paramount importance, rather than in Stage II as suggested by Jahanmir and Suh (ref. 27).

Experiments designed to examine the correlation between fatigue crack initiation and wear resistance found wear resistance to decrease as the resistance to near threshold propagation increased, for the ultra high strength steel tested. Whereas, an excellent correlation was found between increased fatigue limit, as measured in smooth bar tests, and wear resistance (ref. 36). Since the scale of the wear cracks is expected to be on the scale of the debris (i.e., <20 microns), Ritchie suggested that threshold cracks (as calculated from linear elastic fracture mechanics) are >65 microns which is significantly larger than the wear particle scale. Hence, wear cracks are too small to be treated as macrocracks and therefore will not correlate with macroscopic fatigue crack propagation rates. Rather, subsurface wear damage in steels must be related to the initiation and growth of microcracks as best described by the fatigue limit. In essence, Ritchie showed for high strength steels that wear rate and fatigue are intimately related but not through the conventional measures of crack initiation or propagation due to the smaller scale of the wear cracks.

Gross attempts toward correlations of fatigue data without regard to the testing model was suggested by Ritchie to be hazardous (ref. 36). The data of Suh et al. (refs. 27, 34, and 35) only
involved mode I crack propagation testing. Sliding wear, however, is known to involve large shear strains. Crack propagation in shear is widely known to occur. Ritchie suggested that combined mode I and II loading would be expected to show the best correlation with wear data.

Taking into account the above discussion and qualifications, a summary of the Delamination theory is as follows (after Suh, ref. 21):

(a) Initially, surface contact occurs at isolated points through which the normal and tangential loads are transferred. Fracture in the softer asperities readily occurs, while that in the harder occurs more slowly. The initial result is smoother surface.

(b) Surface traction (or friction) exerted at the contact points induces incremental plastic deformation after each pass of an asperity. Damage accumulates with repeated loading.

(c) Deformation continues until subsurface cracks nucleate. Cracks do not form at the surface due to the triaxial compressive stress state at the contact surface.

(d) Crack propagation occurs upon further loading parallel to the worn surface. The depth at which cracks propagate is suggested to be governed by material properties and loading conditions. Stress concentrators at or near the tip of the propagating crack cause arrest. Cracks propagate under the locally applied tensile loading of a passing asperity.

(e) When the crack shears to the surface, long thin wear sneets delaminate.

Utilizing the concept of delamination wear, Rosenfield (refs. 38-41) explored the relevance of fracture mechanics to the study and prediction of wear. Rosenfield recognized the importance of considering shear crack propagation of a delamination crack. Due to the compressive stress field ahead of a moving asperity, he suggested that there is an additional friction force imposed by the faces of the
crack rubbing together. The effective shear stress \( (T_{\text{eff}}) \) of the face of the crack is then:

\[
T_{\text{eff}} = T - Ft\tan(B),
\]

where the faces of the crack are in compression \( (F) \) and shear \( (T) \). As the asperity approaches the cracks, the shear stress decreases relative to the compressive stress until \( T_{\text{eff}} = 0 \) (due to the large frictional force). After the asperity passes over the crack, the compressive force becomes a tensile force (ref. 38). The crack can then open up and grow by combined tensile/shear loading. Rosenfield emphasized that crack propagation in shear while under compression is significant and that Flemming's contention that the tensile part of the cycle solely causes crack propagation is inconsistent with the smooth underside of the wear debris observed by Rigney (ref. 38). Though this question is largely unresolved, the work of Hills and Asheleby (refs. 42) suggest that both tension and shear are important to crack propagation.

Rosenfield points out that most of the literature involves elastic calculations. Yet clearly there is some question of their applicability. As suggested by Shie (ref. 37), the disparity between linear elastic fracture mechanics (LEFM) fatigue crack growth equations and that for shear crack growth is large, indicating that LEFM may not be applicable. In turn, Rosenfield has suggested an elastic-plastic analysis for which the formation of wear particles is by separation along dislocation cell boundaries. Although the model of the deformed
region is over idealized, the fracture criterion was the attainment of a critical crack tip displacement. Thus, crack growth is assumed to occur when a critical condition is reached at a microstructurally significant distance beneath the wear surface.

Estimating the strain distribution from displacement of pearlite lamellae and the stress from a microhardness measurements, an expression was derived for wear rate analogous to that of Archard (ref. 3):

\[ W = \frac{kF}{E e} \]

where \( k \) is a proportionality constant; \( F \) the force exerted by a point load on a surface; \( E \) the elastic modulus; and \( e \) the wear strain. The wear strain was proposed to be a function of the yield strain and the critical strain to form a wear particle (ref. 41). Note that in relation to Archard's law, hardness has been replaced by the product of the elastic modulus and the wear strain. Unfortunately it is unclear how to determine the critical strain to form a wear particle.

Rosenfield acknowledges that this derivation relies on several tenuous assumptions, yet suggests that the model has virtue since it is based on physical observations.

Although the perspectives of both Suh (refs. 20–22) and Rosenfield (refs. 29, 38–41) are mechanics oriented, that of Rigney and Glaeser has been toward investigating the physical metallurgical aspects of wear (refs. 43–46). In the regions adjacent to the wear surface of ductile metals well defined dislocation cell structures have been
observed, in TEM, to form in several FCC materials (refs. 43-44). Rigney et al. (ref. 43 and 45) have suggested that fine dislocation cell structures produced near to the wear surface are intimately connected with delamination wear. The non-linearity of wear rate in the break-in period is attributed to the large degree of plastic deformation required to form a cell structure. Thus, microstructural changes are suggested to be important in the initial stages of wear.

The subsurface cracks observed by Suh (ref. 22), Kimura (ref. 15), and others have been suggested, though not observed, to be formed within or near the dislocation cell walls in the highly deformed surface regions (ref. 43). Planar wear debris particles are thought to form except when phase transformations are involved (ref. 45, 46). Thus, the thinnest particle was suggested to be one unit thickness of the cell structure, with the thickest being that of the entire deformed region. This model is analogous to the brick model of Kimura. However, Rigney suggests a gradient of brick sizes rather than one single size as suggested by Kimura (ref. 16).

Stacking fault energy was noted by Hirth and Rigney (ref. 46) to significantly effect wear. Materials with low stacking fault energy were suggested to show a clear trend of lower wear rate compared to that of high stacking fault energy materials. The work of Pavlov et al. (ref. 47) and that of Blomberry and Perrot (ref. 48) substantiate the conclusions of Hirth and Rigney (ref. 49). It was suggested that the apparent correlation with stacking fault energy was an indication of the importance of dislocation dynamics in wear.
Though most of these concepts have gained popularity, they are nevertheless descriptive and largely unproven. Rigney has suggested the importance of further investigation along these lines employing TEM to characterize the dislocation cell structures.

The preceding review was intended to highlight the major contributions to the field of sliding wear. As mentioned earlier no attempt was made to review all the work in the field. Recently, several detailed reviews and other proposed mechanisms have been published (refs. 49–62). The motivated researcher is referred to them for further detail.
II. EXPERIMENTAL APPROACH

A) Overview

The experimental approach was designed to systematically determine wear characteristics of ferrous materials and characterize the resultant structures by using advanced analytical methods. Due to the complexities of the wear process, in general, the attempt was to minimize the number of variables while maximizing the information derived from the experiments. Hence, the majority of the tests performed were carried out at room temperature, in air, under the conditions of un lubricated sliding. It was not the intention of this study to model any specific wear system, but to study the fundamentals of the wear phenomena in ferrous metals.

B) Materials Selection

The materials and their heat treatments selected for this study were as follows:

<table>
<thead>
<tr>
<th>Material</th>
<th>Composition</th>
<th>Heat Treatment</th>
<th>Structure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ferrovac E</td>
<td>99.999 Fe</td>
<td>800°C Aust. (1 hr) + Air Cool</td>
<td>Ferrite</td>
</tr>
<tr>
<td>AISI 1020</td>
<td>0.1 C/0.5 Mn/Fe</td>
<td>900°C Aust. (1 hr) → 690°C (2 hrs) + Furnace Cool</td>
<td>Pearlite and Ferrite</td>
</tr>
<tr>
<td>AISI 1020</td>
<td>0.1 C/0.5 Mn/Fe</td>
<td>900°C Aust. (1 hr) + Air Cool + 690°C (24 hrs) + Air Cool</td>
<td>Spherodized Carbide + Ferrite</td>
</tr>
<tr>
<td>Experimental</td>
<td>3 Cr/2 Mn/0.3 C/0.5 Mo/Fe</td>
<td>1100°C Aust. (1 hr) + Oil Quench + 200°C Temper + Air Cool</td>
<td>Lath Martensite + Retained Austenite Films</td>
</tr>
</tbody>
</table>
Ferrovac E was obtained from U.S. Steel Research Laboratory in Monroeville, Pennsylvania, in cast, forged, and homogenized scale-free rods, 0.635 cm (0.25 in) diameter. After machining the 0.635 cm (0.25 in) diameter hemispherically tipped pins, they were encapsulated in (argon-filled) quartz tubes and heat treated. The structure was entirely ferritic with an average grain size of 50 microns.

AISI 1020 was purchased from a commercial metal supplier in the normalized condition. As described above, pins were machined from hot swaged 0.635 cm (0.25 in) rod. A coarse pearlitic microstructure was formed through heat treatment. This established a pearlite spacing of 0.15 microns. The pearlite colonies and ferrite grain sizes were both approximately 10 microns. Banding of the pearlite was observed. The banding was oriented parallel to the axis of the pin. The banding was useful for determining the strain imposed by sliding wear.

The same AISI 1020 was spherodized establishing a particle size of 0.6 to 0.8 microns. The ferrite grain size was approximately 10 microns.

Air melted 3 Cr/2 Mn/0.5 Mo/0.3 C experimental alloy was obtained from Certified Alloy, Los Angeles, California, in large ingot form. After upset and cross forging to break up the cast structure homogenization was performed at 1200°C for 24 hours. After heat treatment, the structure consisted of dislocated lath martensite with 500 Å wide interlath films of retained austenite. The prior austenite grain size was 250 microns.

For comparison and contrast several specimens of the martensitic steel were laser surface treated in an attempt to increase their wear
resistance. The surface treatments were done at Coherent Inc., Palo Alto, CA. All surface treatments were done using a 500 watt continuous carbon dioxide laser. The feed speeds were 0.5 cm/sec (0.2 in/sec) and 0.89 cm/sec (0.35 in/sec). These speeds were selected so that little or no surface melting occurred. The heat treated zone was approximately 300-450 microns deep and 4000-5000 microns wide. The prior austenite grain size was 35-50 microns.

C. Wear Testing

All wear testing was done using a pin-on-disk wear machine designed and built at the Lawrence Berkeley Laboratory, as shown in Figure 1. The test specimens were 0.635 cm (0.25 in) diameter hemispherically-tipped pins. In all cases the test pins were worn against a spherodized AISI 52100 flat steel disk having an indentation hardness of 62.5 Rockwell "C" scale.

Tests were performed in air under dead weight loads of 500g and 2,000g without lubrication. The pins traversed the freshly ground surface of the AISI 52100 steel disk in the same wear track for a distance of 750m. The amount of pin-wear was monitored and continuously recorded during all tests by measuring the downward vertical displacement of the pin as a function of time using a linear variable displacement transformer (LVDT), as indicated in Figure 2. To complement the LVDT data, the volume loss resulting from wear was determined by measuring the weight change of the pin. Both methods yielded mutually consistent results.

An exception to the above testing procedure was that of the laser surface-treated martensitic steel. Because of the inherent
Fig. 1. Pin-on-disk machine for testing in air. Pin specimen, disk dead weight, and load are shown.
Fig. 2. Modified pin-on-disk wear machine for testing in inert gas atmospheres. Helium gas was pumped through a 50 ft coil immersed in liquid nitrogen prior to entry into the enclosure. The enclosure was purged for 20 min prior to testing. An LVDT is shown for recording continuously the amount of wear.
difficulties in laser treating a curved surface, a flat-ended 1.27 cm (0.5 in) rod configuration was employed for these experiments. The entire surface of the rod was laser treated. To remove start and stop marks produced by laser treatment, a 0.32 cm (0.125 in) land was machined. To provide a basis of comparison for these treatments conventionally heat treated specimens were made and tested under identical conditions.

The wear debris produced during all sliding wear experiments was recovered from the surface of the disk using a slip-covered permanent magnet. A dry nitrogen gas jet was directed across the wear track to ensure that no debris remained in the track after the pick-up. After the test, the wear debris was carefully lifted from the magnet by removing the glassine paper slip-cover and placed in a static-free container pending analysis.

To ensure reproducibility of the data, only one steel disk was used for all tests. One wear test was made on each side of the disk followed by surface grinding under flood cooling prior to the next series of tests. Grinding to remove approximately 150 microns ensured a fresh surface, avoiding interference that might have been caused by prior tests. Grinding was always done on the same grinding machine and diamond wheel.

To investigate the influence of ambient atmosphere on the wear rate and the character of the debris, several experiments were performed in inert gas atmospheres. The testing platform of the wear machines was enclosed in a clear vinyl bag into which dried helium was pumped, as shown in Figure 2. The helium was dried by circulating the
gas through a 15.25 m (50 ft) copper coil immersed in liquid nitrogen to condense water vapor present in the gas. Heat tape was used to attempt to reduce the amount of moisture in the enclosure.

To investigate the influence of temperature on the character of the wear debris, testing was also performed in liquid nitrogen. As shown in Figure 3, the test disk was placed in a polyethylene dish into which liquid nitrogen was poured. Once the pin and disk reached liquid nitrogen temperature the wear experiment was performed in the same manner as at room temperature.

D) Metallography

1.0 Wear Pins

The surface of the worn pins were initially characterized by optical and scanning electron microscopy. Relevant surface features were examined and recorded. Tilting experiments in the SEM were performed to aid measurement of the surface feature thickness.

To preserve the wear surface, the pins were nickel plated prior to cross sectioning. Using a slow cut diamond saw sections were made parallel (longitudinal) and perpendicular (transverse) to the sliding direction. Metallographic mountings were made with the cross section for optical and scanning electron microscopy. Cold mounting was preferred over hot mounting in bakelite to prevent decohesion of the nickel plating. Diamond polishing (6 and 1 micron) followed by 0.05 micron alumina polishing and light etching in 1 percent nitric acid in ethanol served to reveal the microstructure. Lastly, to facilitate examination of the mountings and discourage discharging in the SEM, a conductive coating of gold was RF sputtered onto the
Fig. 3. Pin-on-disk wear machine modified to test in liquid nitrogen. Liquid nitrogen was placed in the dish cover sufficient to the disk. The system was allowed to cool 30 min before testing.
mountings to a thickness of 0.1 microns. The interfacial regions of the specimens were then examined.

Thin foils for transmission electron microscopy (TEM) were prepared from cross sections cut from the nickel plated pins (see Figure 4). Sections typically 250 microns (0.010 in) in thickness were mechanically abraded to 50 microns (0.002 in).

The specimen was prepared for mechanical thinning by attaching it to a flat aluminum block, using double-sided tape, and then surrounding the specimen with 75 micron (0.003 in) thick brass shims to ensure flatness and aid in gauging the thickness during grinding. Mechanical abrasion was performed on silicon carbide papers always keeping the nickel plating in compression to prevent spalling. Final grinding with 600 grit paper produced the finish required for electro-polishing.

Removal of the thin section from the double-sided tape was difficult. Xyelene was used as a solvent with good results. The entire thinning process was repeated on the other side of the specimen to produce a section 50 microns thick.

Electro-polishing was performed, after both surfaces of the specimen had been ground, using conventional window techniques and a glacial acetic-sodium chromate anhydrous thinning solution. This solution was prepared by mixing 3.785 litres (1 gallon) glacial acetic acid slowly with 0.5 kg (1.1 pounds) of anhydrous sodium chromate.

Thinning conditions of the specimen varied with the size of the specimen but ranged from 25-35 volts at 0.1 to 0.2 amperes in a moderately stirred solution. Thinning continued until a hole formed
Fig. 4. Schematic diagram of the sectioning of the nickel plated worn surface. Slices 250 microns were cut for longitudinal and transverse cross sections.
along the wear interface. The chemical reaction was stopped by immersion in water (prior rinses were only with anhydrous methyl alcohol). The specimen was then dried and removed from the tweezers.

To create a 3mm TEM foil, the thinned specimens were sectioned with a scalpel and mounted onto an oval-holed copper grid using silver paint. Extreme care was taken not to deform the specimen during cutting or mounting.

Argon ion machining was required to obtain regions at the wear interface sufficiently thin to transmit electrons. This additional step is required as the chemical polishing solution does not attack nickel and iron at the same rate. Optimum conditions to maximize milling efficiency, while minimizing ion damage of the iron and steel, were found to be 4-5 KV with an ion current of 10-25 micro-amperes. Typical milling times were 5-15 hours.

The specimens were then examined at 100KV in a Phillips EM301. Due to the fine scale of the deformation structures, microdiffraction was carried out on the Phillips 400 STEM. Due to the difficulties in thinning, examination was also made at high voltage in both the Hitachi HU-650 and the AEI/HAFELEY EM1500 microscopes.

2.0 Wear Debris

Wear debris were produced and collected from the wear experiments. To characterize the wear debris, high resolution SEM studies were carried out at Berkeley on an ISI DS-130 microscope. The characteristic thickness of the debris was determined in several tilting experiments. Specimens were tilted to high angles (80-90 degrees) so that the thickness of the particles could directly be observed.
The debris was also examined in TEM. Foils suitable for TEM were prepared by mechanically depositing the debris on to the opposite side of a holey carbon grid. Carbon was then vapor deposited over the debris, to a thickness of .1 microns, to hold it in place. The particles were examined at 1 MeV and at 200 KV at U.S. Steel. Electron diffraction was employed to elucidate the nature of the particles.

TEM and SEM are selective techniques which only survey a small portion of the particles. It was important, therefore, to determine the particle size distribution to ensure that observations were made on statistically significant particles.

To characterize the size and shape distributions of the debris formed under various conditions, automatic image analysis was performed at U.S. Steel using a Tracer-Northern quantitative metallography system on an ETEC SEM. This system enabled over 300 particles per specimen to be accurately sized and counted in a relatively short time.

To determine the through-section chemistry of the debris, sputter-depth profiling was performed by Auger spectroscopy. The specimens used for this experiment were the same as that used in the SEM studies. A debris particle 0.5–2.0 microns across was found and argon ion sputtered. Auger spectra were taken regularly until the particle disintegrated. Even under the action of sputtering, the deposition technique adequately fixed the particle to the filter. The time to sputter through the particle was found to be consistent with the measured thickness.

Due to the submicron nature of the debris and the tendency of the debris to remain as aggregates, a technique was developed at U.S.
Steel to disperse the debris uniformly over a surface suitable for examination optically as well as in an Auger or SEM microscope. The technique consists of suspending the debris in filtered acetone and vacuum filtering the solution through a 0.2 micron cellulose acetate filter (trade named Nucleopore). The filter containing the wear debris is then removed from the filtering frit and mounted on a flat aluminum SEM stud using dispersed amorphous graphite. To avoid charging the mounts are either carbon or gold coated.

A schematic diagram of the deposition process is shown in Figure 5.
WEAR DEBRIS PREPARATION FOR SEM EXAMINATION

LOOSE DEBRIS → SUSPEND IN ACETONE VIA ULTRASONIC VIBRATION → VACUUM FILTER SOLUTION TO DEPOSIT DEBRIS ON SMALL PORE FILTER MATERIAL → MOUNT FILTER ON AN ALUMINUM STUB USING D.A.G.

RF SPUTTER COAT WITH GOLD → EXAMINE IN SEM

Fig. 5a. Schematic diagram of the specimen preparation process for the wear debris.
WEAR DEBRIS PREPARATION FOR TEM EXAMINATION

Loose debris particles are mechanically deposited on carbon coated grids, then evaporated with EXM-11 NE carbon. The specimen is examined in the TEM.

Fig. 5b. Schematic diagram of the specimen preparation process for the wear debris.
III. RESULTS AND ANALYSIS
A) Wear Testing
1.0 Wear Pins

The unlubricated sliding wear data for all ferrous materials tested is plotted in Figure 6. The use of an LVDT in addition to weight loss measurements on the pin enabled the continuous measurement of wear. Overall, dislocated lath martensite was found to be the most wear resistant of the conventionally heat treated materials. (Laser heat treated lath martensite steel was found to have a 30-40% greater resistance to wear than the conventionally heat treated steel.) Initially, under low loads (0.5 KG), however, martensite was observed to exhibit a higher wear rate than either pearlite or ferrite. But, a plateau in wear rate was rapidly reached. This is to be expected due to the rapid work hardening rate of lath martensite—a consequence of the fine scale of the microstructure and the initially high dislocation density.

Under large loads (2 and 3 KG), martensite clearly wears less than either ferrite or pearlite. In addition it reaches a constant wear rate more rapidly than at lower loads. Ferrite, however, never reaches a constant, instead the wear rate increases approximately linearly with time at all loads. Pearlite, on the other hand, exhibits a wear rate intermediate between ferrite and martensite. Under small loads a constant wear rate is reached which is above that of martensite but below that of ferrite. Yet as the load is increased, the wear rate steadily increases such that a constant wear rate is not reached in
Fig. 6. Amount of wear vs sliding distance for all the materials tested in this study. a) Tests under 0.5 KG load. b) Tests under 2.0 KG load.
the length of the test. This has been commonly referred to in the literature (refs. 21) as 'break-away.'

The shapes of the wear curves are expected to reflect the modes of deformation and the mechanism(s) of wear particle formation. As has been reported herein, for all the materials tested, these curves have essentially the same form even though the rate of wear varied. The significance of these results will be discussed in greater detail in the Discussion.

For the materials concerned, the gross differences in wear rate are believed to be related to differences in yield and work hardening behavior. Pure iron, in the ferritic condition, has a low yield strength and slow hardening rate, resulting in a high wear rate. Whereas martensite with a high yield and rapid work hardening rate exhibits a low rate of wear. This inverse relationship between wear rate and yield strength appears consistent with the well known relationship between wear rate and hardness established for abrasive wear (refs. 56-58). Yet, no clear relationship exists between sliding wear rate and hardness. This is not surprising due to the complex relation of hardness to ultimate tensile strength and work hardening rate.

2.0 Counterface

Because sliding wear is generally not a material property but is characteristic of a system, it is essential to characterize the mode and rate of wear in the counterface material. As mentioned in the Experimental Approach, a spheroidized AISI 52100 (bearing steel) disk was used as the counterface material. The load range used in this
study was selected on the basis of obtaining appreciable wear on the pin with little or no wear on the disk. Disk wear was measured by determining the depth of the wear scar (or groove) via a diamond stylus profilometer. The profilometer measurements were made at U.S. Steel Research Laboratories and revealed that no detectable amount of material was removed from the surface of the disk in tests with ferritic, pealitic, and spherodized pins, see Figure 7. The limit of detectability was 0.025 microns (1 micro-inch).

As indicated by the trace in Figure 7b, the martensite was observed to noticeably modify the surface roughness of the disk. Vertical displacement of the trace relative to the surrounding region would indicate that the disk had worn. No such net vertical displacement was observed. Rather, material was displaced but remained attached to the disk. This deduction was supported by optical examination of the wear scar revealing no evidence of delamination which would have suggested that the disk had lost material. Ridges were observed in the wear scar thereby suggesting displacement had occurred.

Strictly speaking by the definition of 'wear' (the loss or displacement of material from its place of original) the disk was definitely worn by the martensitic pin. Yet, because no material was lost and because the magnitude of wear was so small, it is reasonable to conclude that it is negligible. This suggests that the wear debris formed and collected entirely came from the pin. Hence, there was essentially no contamination of the wear debris by the disk material.

As discussed in the Experimental Approach, the majority of the wear experiments were carried out in air. Several experiments were
Fig. 7. Profilometer measurements on the wear disk. Traces represent the surface profile across the wear scar. The bars indicate the width of the scar. 

a) Scar made by ferrite (Ferrovac E) typical of that made by pearlitic and spherodized steels. No wear apparent. 

b) Scar made by martensite (Quatough). Note the substantial disturbance of the metal, with little being removed.
also done in inert gas atmospheres. The wear rate was observed to be approximately the same in air as in the inert atmosphere. The significance of this result will be discussed in a later section.

B. Metallography

1.0 Wear Pins

1.1 Worn Surface

SEM micrographs of the worn surface of the pins showed that large amounts of plastic deformation had occurred (see Figures 8-10). Platelike features were observed on the surface of all the materials involved in the study. Apparently material was being removed in sheets under the action of sliding. The larger of these plate-like features appeared to be aggregates of the wear debris. This mode of wear failure has been termed delamination wear (ref. 22), due to failure appearing to occur by separation of the surface layers from the bulk in a manner superficially similar to that of the mineral mica.

In association with the delamination events (or craters), debris particles were profuse, distributed uniformly across the wear surface. As seen in Figure 10, for a pin observed immediately after testing, several of the particles in the field of view appear to be near the craters from which they were formed.

No change in the appearance of the surface was observed when the load was increased. The crater size averaged between 0.02 to 5 microns. As suggested by the plate-like appearance of the surface features, plastic flow of the materials was observed to be parallel to the surface. Evidence for this is shown in Figure 10d, for which a
Fig. 8. SEM micrographs. Worn surface of ferrite (Ferrovac E). a) Center of the worn pin. b) Enlargement of area indicated in a). Large amounts of plastic deformation are evident from the grooving of the surface. c) Enlargement of area indicated by double arrows in a).
Fig. 9. SEM micrographs. Worn surface of pearlite (AISI 1020).

a) Low magnification of the worn pin.  b) Central region of the pin.
c) Enlargement of the region indicated in b).  d) Same region as b).
e) Enlargement of the region indicated in d).  Both c) and e) show
plate-like features. Appears to reflect delaminatin wear is occurring.
Fig. 10. SEM micrographs. Worn surface of ferrite (Ferrovac E). A large amount of the wear debris is seen on the pin surface. a) Typical region of the worn surface. b) Same region as in a) but tilted 50 degrees about an axis in the plane of the figure. c) Enlargement of the central region of b). Craters and the associated debris are seen on the surface of this feature. d) An example of the layered nature of the worn surface. Layer thickness is approximately 150-300 nm (1500-3000Å).
tilting experiment in an SEM on a typical wear plate, revealed a layered appearance of the feature. It is interesting to note that the thickness of the layer was measured to be approximately 150-300 nm (1500-3000Å). Roughly this is on a scale consistent with the wear debris.

Chemical depth profiling of the pin surface was done. An oxide layer, apparently Hematite (Fe₂O₃) was found to be 10nm (100Å) thick. This suggested that the oxide was formed under the normal influence of the atmosphere since no other oxide was found beneath this layer.

1.2 Cross Sections - SEM

The structures that form as the result of sliding wear are seen in cross sections parallel to the sliding direction, as shown in Figures 11-20. In all of the figures the sliding direction is left to right unless otherwise indicated. A prominent characteristic of the sliding wear deformation is the apparent flow of material parallel to the direction of sliding. This region near the surface has been termed the heavily deformed layer (HDL), Bilby layer, white etching layer (addressing the fact that little structure has historically been observed), friction layer, or the micronized layer (refs. 6, 20, 43, 45 and 59). Most commonly this region is called the HDL. The extent of the HDL for tests performed under 2KG load is as follows:
Fig. 11. SEM micrograph. Plastic replica of a cross section of nickel plated ferritic iron. Bulk plastic deformation apparent from the ferritic iron. Bulk plastic deformation apparent from the distortion of the grains. The deformed zone extends 14-19 microns from the surface. Sliding direction is left to right.
Fig. 12. SEM micrograph. Plastic replica of cross section of a nickel plated ferritic iron. a) Striations are seen to be parallel to the surface. Their width is 0.25-0.80 microns. b) Cross section through a feature soon to be a wear particle. Note the crack at the base of this feature.
Fig. 13. SEM micrograph. Polished and etched metallographic cross section of pearlite (AISI 1020). Aligned pearlitic structure is to be noted. Pearlite colonies adjacent to the interface are broken up by the sliding wear deformation. Voids or holes along the interface are where the carbides pulled out during specimen preparation.
Fig. 14. SEM micrograph. Polished metallographic cross section of pearlite (AISI 1020). Enlargement of the interfacial regions. Pearlite lamallae break up into individual carbide particles 0.1 to 0.3 microns in diameter. Carbides do not appear to act as sites of crack initiation.
Fig. 15. SEM micrograph. Polished metallographic cross section spherodized AISI 1020. a) Low magnification of the polished section. b) Enlargement of the area indicated in a). Extent of deformation is 15-17 microns. c) Region near the interface shows that cracking near the carbides common. d) Example of cracking associated with the carbides.
Fig. 16. SEM micrograph. Polished metallographic cross section of spheroidized AISI 1020. (a) and (b) both show that cracks initiate between the carbides as well as by decohesion at the carbide matrix interface, as depicted in (b).
Fig. 17. SEM micrograph. Polished metallographic cross section of spherodized AISI 1020. a) Low magnification--overview. b) Oxides are dispersed randomly throughout the specimen. c) The oxides are seen to act as sites for both initiation and propagation of cracks. These oxides are believed to have been rolled into the steel during the hot swaging operation.
Fig. 18. Optical micrograph. Polished metallographic cross section of the martensitic steel. a) Etched. Cracks are parallel to the striations. The extent of the deformed zone is 8-12 microns wide. b) Unetched. Same region as in a). Cracks apparent even before etching.
Fig. 19. SEM micrograph. Polished metallographic cross section of the martensitic steel. a) Secondary electron image of interface. b) Backscattered electron image. Enlargement of the area indicated in a). Cracking seen parallel to the length of the striations. Etching with nitric-ethanol solution has made the cracks appear larger than actual. Extent of deformation 8-10 microns.
Fig. 20. SEM micrograph. Polished metallographic cross section of the martensitic steel. Enlargement of 18b near the crack tip. The striation spacing ranges from 0.05 to 0.1 microns.
Ferrite has the largest HDL (see Figures 11, 12) and martensite has the smallest (see Figures 18-20). Clearly the extent of this region appears to be inversely proportional to the yield strength of the material, consistent with the hardness relationship previously discussed.

A feature common to all the cross sections observed in the SEM was the existence of a 'striated' region in the HDL. By the waviness of the lines in this region and the propensity of iron to undergo wavy slip, these features are believed to be wavy slip lines. Careful metallography and judicious etching, along with the use of plastic replicas observed at high magnification in the SEM, enabled the imaging of this layer.

At the immediate surface in sections parallel (longitudinal) to the sliding direction the slip lines are observed to be parallel to the surface. They are observed to evolve gradually from the undeformed grains, as seen in Figures 11, 13, 15, and 19. The widths observed for the slip lines, summarized in the table above, ranged from 0.05 to 0.8 microns (5-800nm) wide while being several microns long. Cracks were observed to initiate 0.10 to 3.0 microns (100 to

<table>
<thead>
<tr>
<th>Material</th>
<th>Depth of deformation (microns)</th>
<th>'Striated' line width microns</th>
</tr>
</thead>
<tbody>
<tr>
<td>ferrite</td>
<td>17-20</td>
<td>0.10-0.80</td>
</tr>
<tr>
<td>pearlite</td>
<td>12-15</td>
<td>0.07-0.3</td>
</tr>
<tr>
<td>spheroidized</td>
<td>15-17</td>
<td>0.08-0.4</td>
</tr>
<tr>
<td>martensite</td>
<td>8-12</td>
<td>0.05-0.1</td>
</tr>
</tbody>
</table>
3000 nm) below the surface and propagate parallel to the length of the lines (see Figures 11, 16 and 19). The significant point regarding the slip lines is the observation of crack initiation and propagation parallel to them (note—etching in 19 has caused the crack to appear wider than actual size). This type of cracking behavior has been observed in all the material examined.

It has been proposed by Suh (refs. 20-22, 27, 34) and others (refs. 1, 42, 52, 53, 59 and 64) that subsurface cracks initiate by the fracture of inclusions or the decohesion of second phase particles. It is suggested by these workers that carbides, inclusions, or oxides are necessary for the cracks to initiate. In the case of the spheroidized steel, this certainly appears true as seen in Figures 15 and 16. Similarly, oxides which apparently were rolled into AISI 1020 during processing are seen in Figure 17 to also aid in initiating wear cracks.

However, by this reasoning there is no explanation for the origin of subsurface cracks formed in sliding of pure iron, which apparently contains neither carbides, oxides nor inclusions. It will be suggested from the results presented in the next section that cracks can and do initiate at dislocation cell boundaries near the surface.

The slip lines are found to be on the same scale as these dislocations near the wear interface in ferrite and pearlite. In martensite the deformed laths themselves appear on the same scale as the slip lines. In summary, the SEM evidence suggests that crack initiation indeed does occur at cell boundaries.
1.3 Cross Sections - TEM

Due to the expectations that the dislocation structures play a significant role in the wear mechanism, imaging of the dislocation structures in TEM was essential. Figures 21-28 show representative regions from ferritic, pealitic, spherodized, and martensitic foils, respectively. Notable, discrete dislocation structures were observed in ferrite, pealite, and spherodized structures. However, a cellular structure was not observed in martensite. In the martensitic case, as seen in Figure 26 and 27, the dislocated lath martensite elongates in the direction of sliding resulting in the laths necking down from an initial width of 0.5 microns to less than 0.5 microns (50 nm). Dislocation cells 0.025 microns (25nm) wide were observed within the laths adjacent to the interface. The cell structure within the laths was more easily resolved 1 micron from the interface (see Figure 26).

No retained austenite was observed at the immediate wear surface in the martensite steel. Due to the implicitly high strain, which is in the neighborhood of 4 to 6 true shear strain, the austenite would certainly be expected to transform, by shear, to martensite. The estimates of strain were made by calculating the plastic strain from the bending of the microstructural features. However, approximately 2.5 microns from the interface where the strain is estimated to be less than a true shear strain of 1, retained austenite was observed.

In sections parallel to the sliding direction (longitudinal) a dislocation structure was observed adjacent to the wear interface in ferritic specimens with a width of 40-60nm (400-600Å, see Figures 21 and 22). A region 2.5 microns from the interface, shown in Figure 21,
Fig. 21. TEM micrograph. Longitudinal cross section of ferritic iron. a) Arrow indicated the interface as determined by EDS. Circle encloses the region exhibited in b). b) Representative dislocation structure 2.50 to 5.50 microns from the wear interface. The width of a typical cell at 3.0 microns from the interface is 0.19 microns. This is indicative of a shear strain of at least 5.
Fig. 22. TEM micrograph. Longitudinal cross section of ferritic iron. 
a) Bright field. Interfacial regions are exhibited. The width of the dislocation cells is 75-150 nm (750-1500Å) wide. 
b) Centered aperture dark field of a nickel reflection highlights the interface. The nickel plating is in the lower half of the frame as indicated.
Fig. 23. TEM micrograph. Longitudinal cross section of pearlitic steel. a) Low magnification of the interface and adjacent region. The arrow indicates the Fe-Ni interface. b) Enlargement of a). Recovered zone exists adjacent to the interface. The heavily dislocated layer is adjacent to this zone. Cracks are indicated at the junction of recovered region and the heavily dislocated region. c) Schematic diagram of the micrograph in b).
Fig. 24. TEM micrograph. Longitudinal cross section of pearlitic steel. Carbide embedded in the matrix. a) EDS of the matrix. Note the nickel in the spectra from the nickel plating which is 2 microns away. b) Bright field micrograph. The particle is indicated by arrows. c) EDS of the particle. Shows the high Mn content of the particle without S being present. Thus, particle is a carbide and not a sulfide.
Fig. 25. TEM micrograph. Longitudinal cross section of pearlitic steel. Feature appears to be a debris particle in the process of forming. Arrows indicate the Fe-Ni interface. a) EDS from region 1-nickel. b) Bright field. Region 2 is the debris. Region 3 is the heavily deformed region. c) EDS from region 2-indicates iron. Cracking is observed along the dislocation cell boundaries.
Fig. 26. TEM micrograph. Longitudinal cross section of martensitic steel. Structure formed in martensite from sliding wear. Note that the laths appear to have necked down with little evidence of a dislocation structure.
Fig. 27. TEM micrograph. Longitudinal cross section of martensitic steel. Enlargement of the region near the interface. The diameter of the laths appear to be 50 nm.
has a cell structure 200nm wide. The size of the dislocation structure compared to the work of Embury et al. (ref. 60), indicates that the region supported a shear strain of 5.

Longitudinal cross sections of the pearlitic steel near the wear surface revealed a cell structure 10-30nm (100-300Å) wide by several microns long (see Figures 23 and 24). In cross sections perpendicular to the sliding direction (transverse) the shape of the dislocation cells, formed under the same conditions, was observed to be 10-30nm (100-300Å) by 20-40nm (200-400Å), see Figure 28. Thus, the shape of the dislocation cells is evidently rectangular with the dimensions of 100-200Å thick by 200-400Å wide by 8000-20,000Å long.

An interesting finding was the existence of a zone, 2000-3000Å wide at the immediate surface, with a lower dislocation density than the surrounding area (see Figures 22 and 23). This zone was observed in both ferrite and pearlite but not in martensitic or spherodized steels.

Within the zone, the recovered cells are typically 40nm (400Å) wide and 60nm (600Å) long. This is distinctly different from the adjoining region where the width of the cells is 12-15nm (120-150Å) by 1-5 microns long. In addition, what appears to be cracks were observed at the boundary of the recovered zone and the heavily dislocated layer beneath (see Figure 23 where indicated). The cracks appear to form along the boundaries of the cells, consistent with the SEM observations. Utilizing the data of Embury et al. (ref. 60) the true shear strain in this region is estimated to be greater than 4.
It is believed that this zone indicates recovery is occurring. Several investigators have observed what they have termed recrystallized or dynamically recrystallized regions near the wear surface, typically in copper (refs. 24, 61-63). From the terminology it is unclear in some of the literature whether dynamic recovery or recrystallization is actually occurring. Due to the low temperatures at the contact surface implicit in slow speed (0.5 cm/sec) sliding and the large extent of deformation, recovery would be favored in lieu of recrystallization.

It should be noted that the cracks observed along dislocation cell boundaries or at the junction of the recovered zone are not believed to be artifacts—they have frequently observed in several foils, thousands of angstroms from the specimen edge.

As referenced in the SEM section, crack initiation has been suggested to occur by either cracking or decohesion of carbides, intermetallics, or oxides. However, as shown in Figure 24b for coarse pearlite, a particle, apparently an iron carbide, is observed in the TEM micrograph. The carbide is believed to be formed from the pearlite lamellae broken up during sliding. Note that the ferrite matrix around the particle has deformed substantially without a crack initiating. In fact, the ellipsoidal shape of the particle implies that the particle itself has deformed. EDS analysis was used to analyze the particle. The results (see Figure 24) show the particle to be rich in Mn but deficient in S. Thus, the particle is not MnS. Because Mn will substitute for Fe in cementite, it is likely that the particle is a carbide.
Consistent with the suggestion that crack initiation does occur along dislocation cell walls is the observation of a wear particle (or debris) apparently in the early stages of formation, as shown in Figure 25b. The nickel plating is shown as region 1, the particle as region 2, and the heavily deformed bulk as region 3. EDS analysis confirmed the location of the interface as shown in Figure 25b. Note that the dislocation cell structure within the particle is similar to that in the recovered zone, as shown in Figure 23. Also note that the thickness of the particle is 100-400nm (1000-4000Å), very close to the observed thickness of the debris and the width of the recovered zone previously discussed. Finally, Figure 25 shows a crack has propagated along dislocation cell boundaries, consistent with Figure 23.

2.0 Metallography - Wear Debris

2.1 SEM Observations

To obtain a full understanding of the wear system it is important to consider all the components. Hence, the following are the results of postmortem studies on the wear debris.

Since the number of particles observed in TEM is limited, automatic image analysis in an SEM was first performed. Figure 29 summarized the results of these studies. Note that more than 90 percent of the particles are less than 4.0 microns across. The ratio of length to width was found to average 2:1. In comparison, the size of wear particles quoted in the literature ranges from 0.2 to 20 microns (refs. 6, 43, and 59). Although particles larger than 4.0 microns were observed in this study, they were found to be less than 1 percent of the total number.
Fig. 29. Summary of the automatic image analysis (AIA) data on the wear debris formed from conventionally heat treated steel.
Fig. 30. Summary of the AIA data for laser treated steel.
AUTOMATIC IMAGE ANALYSIS
INFLUENCE OF LOAD

<table>
<thead>
<tr>
<th>LOAD (Kg)</th>
<th>TOTAL PARTICLES</th>
</tr>
</thead>
<tbody>
<tr>
<td>1/2</td>
<td>296</td>
</tr>
<tr>
<td>2</td>
<td>299</td>
</tr>
</tbody>
</table>

Fig. 31. AIA data for the wear debris as a function of applied load (and material).
Fig. 32. SEM micrograph. Tilting series on wear debris produced from martensitic steel. Debris are deposited on cellulosic acetate filter. Note in d) the particle thickness is approximately 250-350 nm.
Fig. 33. SEM micrograph. Tilting series of wear debris produced from ferritic iron. Note the plate-like nature of the debris. The thickness is 300-400 nm.
Fig. 34. SEM micrograph. Tilting series on the debris from the laser treated materials. The thickness of these particles is 200-225 nm, which is slightly less than the conventionally heat treated materials.
Since more than 300 particles were counted for each datum point in this study, the data are statistically significant. Little mention is typically made in the literature of the number of particles counted.

Figure 29 shows that the peaks of the size distributions are as follows: martensite 1.0-1.7 microns, pearlite, 0.2-0.5 microns, spheroidized 1020 0.2-0.5 microns, and ferrite 0.5-1.0 microns. Figure 30 shows that the debris from the laser treated martensite was approximately the same size as that conventionally heat treated.

Because the HDL in martensite is the smallest one might expect the debris to be the smallest. But, due to its high strength it is plausible that the debris are more cohesively bonded than the other materials and are consequently larger. In the case of pearlite, fracture of the pearlite lamellae occurs and particles 0.2-0.7 microns are formed as can be seen from Figure 14. This would explain the peak in the pearlite size distribution being pushed toward the 0.5 micron side relative to the results of ferrite.

An indication of the size dependence of the debris as a function of load is seen in Figure 31. Increasing the load from 0.5 to 2.0KG causes the size distribution to shift to smaller sizes. Basically, all the materials exhibit a peak at 0.5-1.0 microns. The aspect ratio of the debris did not appear to change. Intuitively the shift to smaller sizes can be understood from the increased load causing the wear debris to fracture into smaller particles rather than remain as a cracked mass.

Tilting experiments were performed in the SEM to determine the thickness of the debris. Figures 32, 33, and 34 suggest that the
particles of the conventionally heat treated materials are clearly plate-like having a thickness of 250-350nm (2500-3500Å). For the laser treated martensitic steel, the debris were observed to be slightly thinner, 200-225nm (see Figure 34). This might be expected for the finer scale of the microstructure and the shallower HDL in the laser treated steels.

2.2 AES Results

Auger electron spectroscopy (AES) studies were performed on the debris from wear tests on all the materials tested in air. The results are presented in Figure 35 for chemical composition as a function of depth from sputter-profiling experiments. The significant outcome of this experiment was that a particle 300nm (300Å) was found to be entirely oxide throughout the thickness. Though deformation of the cellulose acetate filter did occur, causing the particle to appear thicker than actual, the debris were adequately fixed so that the same particle could be continuously sputtered. Each experiment was repeated at least four times with similar results being obtained each time.

These results are significant in that atmospheric oxidation of iron would not be expected, at room temperature, to result in an oxide thickness greater than 10nm (100Å). Hence, for a 300nm (3000Å) particle to oxidize through thickness, events other than atmospheric oxidation are obviously occurring.

Utilizing the Auger survey and the machine sensitivity factors, the atomic concentration of a constituent in the survey can be semi-quantitatively determined. Figure 36 is a survey taken after 2
Fig. 35. AES sputter profiles. Performed on debris from ferritic iron. Indicates the particle is oxide through thickness. All three frames are part of the profile. The trace trails (falls) off as the beam drifts off the particle.
Fig. 36. AES surveys. a) After 2 min sputtering. Suggests the oxide to be hematite. b) Survey after 20 min sputter. The oxide appears to be magnetic. The substrate deformed and caused to particle to appear thicker than actual.
minutes of sputtering. Having initially calibrated the sputtering rate at 8KV to be 55nm/min (550Å/min), the survey suggests that approximately 110nm (1100Å) in to the particle, Hematite (Fe₂O₃) is found. After 20 minutes of sputtering the same particle, the stoichiometry of the oxide was found to be that of Magnetite (Fe₃O₄). For atmospheric oxidation the entire oxide would be expected to be Hematite, the equilibrium oxide. However, below 400°C, as one increases the temperatures at a given partial pressure of oxygen, the thickness of Magnetic relative to Hematite increases. Above 400°C, Wustite (FeO) composes 95 percent of the scale with 4 percent Magnetite and 1 percent Hematite. Hence, by implication, the temperature at the contact surface is above room temperature but below 400°C. The significance of the observations will be discussed in a later section.

Wear tests performed in liquid nitrogen on pure iron produced wear debris which were found to be predominately metallic. Sputtering experiments on debris particle 2-10 microns across revealed that only the outer surface of the particles were oxide with the interior still being metallic.

Wear tests performed in helium gas purged atmosphere were found by AES to yield predominately metal particles. These particles, like that of the liquid nitrogen experiments, were found to be metallic at the center, especially in the larger particles (see Figures 37). This result suggests that oxidation occurs after the particle is formed and not before, as implied by an oxidative mechanism of wear.
Fig. 37. AES sputter profile of debris particle formed from an iron pin worn in an inert gas (helium) environment. The particle exterior is an oxide while the interior is still metallic. This proves that the debris form as metallic particle which oxidize, depending on the atmosphere, during separation from the bulk.
2.3 TEM Observations

TEM observations made on debris from each of the materials generally confirmed the AES findings. The smaller particles, less than 3.0 microns, were found to be transparent to electrons at 100KV and 200KV. Larger particles were examined at 1MeV. Electron diffraction from the smaller debris formed in air found them to be entirely oxide (see Figure 38). However, selected area diffraction at high voltage on the larger particles showed strong evidence of metallic iron as well as for Magnetite. This is indicative of incomplete oxidation.

TEM observations on debris produced under 0.5KG load showed a significant amount of cracking within the particles, especially those at the larger end of the size distribution (see Figure 38). Debris produced on the same material but under 2.0Kg loads, however, were generally, smaller and exhibited fewer cracks in the particle.

Debris from tests in liquid nitrogen examined using high voltage were found to be predominantly metallic (see Figure 39). Diffraction information obtained from testing of ferritic iron in liquid nitrogen revealing the particles were metallic, substantiate the AES results. Similarly, the diffraction information obtained from debris formed in a helium atmosphere is also found to contain mainly metallic particles but in a somewhat lower proportion than those in the liquid nitrogen experiments.

Microdiffraction experiments in the STEM on cross section of the wear pin indicated a relative rotation of approximately 15–20 degrees between adjacent dislocation cells within 100nm (1000Å) of the
Fig. 3. Electron diffraction of a typical debris particle formed during wear testing in air. Note the oxide character.
Fig. 39. Electron diffraction of a particle formed at liquid nitrogen temperatures. Note the metallic character.
interface. Due to the extremely fine nature of the cell structure it was impossible to unambiguously characterize the texture developed as the result of sliding wear. Qualitatively, a large fraction of the cell orientations observed in transverse cross sections in ferrite and pearlite were found to be (100) and (110).
IV. DISCUSSION

A) Overview of the Mechanism

Generally, sliding wear occurs through the creation of loose debris particles from two mating surfaces in sliding contact. The use of scanning and transmission electron microscopy on thin foils prepared from body centered cubic iron and steel alloys, tested under slow sliding speeds at relatively high loads on a pin-on-disk machine, has lead to several insights into the fundamental processes of unlubricated sliding wear in BCC materials.

Incremental plastic shear strains are believed to be induced into the subsurface during sliding by asperity interactions. Strain accumulates in the deformed material proportional to applied load and sliding distance. The outward manifestation of the accumulated strain is the gradual formation of dislocation cell structures in the near surface regions, typical of large strain deformation. Well defined dislocation cells were observed, in all the materials examined in this study, to have a width proportional to the distance from the wear surface. The non-linearity of the wear rate in the initial stages of sliding wear is attributed to the formation of a stable or steady state dislocation cell structure.

Deformation continues until a critical degree of strain is reached in the immediate surface regions. Experiential observations revealed the critical failure strain, in the ferrous materials studied, ranged from 4-6 true shear strain. In materials that contain second phase particles or inclusions wear failure initiates by cracking parallel to the surface in the highly deformed region, preferentially at or around
the inclusions, or second phase particles. Yet, in materials without stress concentrating particles, fracture initiation is associated with dislocation cells subjected to extremely large strains (akin to the formation of wedge cracks in grain boundaries during high stress, low temperature creep deformation). The cracks propagate principally along the dislocation cell walls, in shear, while under compression, assisted by the cyclic tensile component imposed immediately after passage of an asperity over the crack. Once the crack has reached critical length, the tensile forces imposed by an asperity plus that of adhesion, when applicable, serve to separate a planar particle from the bulk. Consequently, the particle oxidizes due its pyrophoric nature, degree of plastic deformation, and exposure of fresh surface to oxygen. Predominately oxide wear debris particles, which remained unchanged throughout the test, were formed and averaged 0.2-2.0 microns wide and 0.2-0.4 microns thick. The extent of oxidation, however, is dependent on the thickness of the particle, with the thinner particle being fully oxidized and the thicker being only partially oxidized. Hence, both metallic and oxide loose wear debris particles are produced in sliding wear experiments in air. A schematic diagram of the wear mechanism is presented in Figure 40. The next several sections will discuss the details of the foundation of the mechanism.

B. Foundation

1.0 Degree of Deformation

The concept of cumulative deformation introduced into the wearing solids by surface asperity interactions in well known (refs. 7, 10,
Fig. 40. The pyrophoric mechanism of wear. a) Dislocation cells form from surface interactions. b) Recovery occurs and cracks initiate at the dislocation cell walls. c) Particle separation occurs concurrently with particle oxidation.
Largely unanswered, however, is the issue of crack initiation. Before consideration of the possible initiation mechanisms it is important to address the state of the near surface regions. Using the method of Dautzenberg and Zatt as shown in Figure 41, calculation of the plastic strain \( e(p) \) as a function of depth (assuming plane strain deformation) was made in metallographic cross section parallel to the sliding direction from the bending of aligned pearlite colonies as well as grain boundaries in ferrite, spherodite, and martensite (see ref. 15 for the details of the calculation). The strain distribution was noted to decrease exponentially with depth near the surface and hyperbolically with larger distance. Microhardness measurements, as shown in Figure 42, indicate the same general trend.

It was seen that the (true) shear strains 1 micron from the surface are extremely large and range from 4, calculated for martensite, to 6, calculated for ferrite. A problem with this technique is the large disturbance of the microstructure within the first micron of the surface. In addition the tangent of the angle of the sheared boundary approaches infinity as the angle approaches 90 degrees. Hence, Dautzenberg's technique is unable to describe the degree of strain in the immediate surface layers.

As first suggested by Rigney and Glaeser (ref. 43), the dislocation texture formed during rolling operations is similar to that formed during sliding wear. Since the deformation textures formed during wire drawing and rolling are principally \( <110> \), the wire drawing data of Embury, Keh, and Fisher (ref. 60) may be used to
Fig. 41. Strain as a function of depth derived from the ending of the microstructure. Note the true shear strain at the immediate surface ranged from 4 to 5. The strain decreases exponentially with distance from the surface.
Fig. 42. Hardness as a function of distance from the wear surface derived from micro-hardness measurements.
estimate the shear strains within 1 micron of the surface (from the dislocation structures observed in TEM). As shown in Figure 21 for ferritic iron 3.0 microns from the interface, the width of the dislocation cells average 0.19 microns (1900Å) indicating the region supported a shear strain of at least 4. This is reasonably close to that calculated by the method of Dautzenberg. At the surface, the dislocation cell widths observed in Figure 22, is 0.075 microns (750Å) indicative of shear strains exceeding 5.5. The strain distribution as a function of depth, derived from these measurements, is summarized in Figure 43. It is clear that the shear strains at the worn surface are large by any form of measurement.

2.0 Crack Initiation

(a) Particle Associated

Due to hydrostatic compression at the surface, Suh and Jahamir (ref. 27) suggested that voids initiate subsurface typically at second phase particles (or inclusions), to grow and coalesce under cyclic tensile loading imposed by asperity motion, resulting in subsurface cracks. Hills and Asheleby (ref. 42) concurred with this approach and calculated the interfacial tractions on an inclusion in a contact stress field, concluding inclusions and second phase particles oriented perpendicular to the surface to be the most detrimental in sliding wear. Both the approaches outlined above require the presence of particle for the initiation of the cracks. In the most liberal case, this view would require that at least one particle be involved in initiating the critical crack.
Fig. 43. Strain as a function of distance from the wear surface derived from dislocation cell measurements.
The particles characterized in this study were found to be platelets with average linear dimensions of 0.2 by 0.5 by 1.0 microns. In a typical experiment, 2mg of debris were collected and calculated to contain over 2.5 billion particles. By these theories, the wearing material must contain an inclusion or second phase particle density of $10^{13}$ particles/cubic centimeter for one particle to be involved with each wear event. Translated into tangible terms, this would roughly mean a maximum particle center spacing of 2000Å. Certainly this is possible in a spherodized medium carbon steel. Yet, such a high particle density does not occur in many other materials that contain second phase particles or inclusions. Additionally, iron carbides such as in pearlite are deformable, as compared to that of a spherodized iron or vanadium carbide, so that cracks may not form at or around the carbide even if it is near to the wear interface. This has been observed in this study, as shown in Figure 24. Hence, factors other than cracking associated with carbides or oxides appear to contribute to crack initiation.

(b) Dislocation Associated

The theories of Suh and Hills et al. acknowledge their inability to explain crack initiation in the absence of particles (i.e., failure in single phase materials). Certainly cracking does involve dislocation dynamics. However, the classical mechanisms of crack formation at dislocation pile-ups as proposed by Cottrell and Stroh (refs. 66 and 67) are clearly inappropriate in this case of plane shear deformation while under compression. Moreover, the extent of deformation is so large that a complete dislocation cell structure has formed.
Rigney and Glaeser first recognized the association of dislocation structures in the wear process by suggesting that cracks would be favored to initiate at dislocation cell boundaries. They suggested that the similarities of the dislocation structure to fatigue and the observation of wear particles one or several cells thick substantiates this claim. However, no mechanism for crack initiation was proposed by them.

Microscopic investigations into several BCC materials in this study suggest the formation of wear cracks at dislocation cell walls is similar to the mechanism of Rigney (ref. 45) proposed for FCC materials. As noted in SEM studies on cross sections of the wear pins, the highly deformed layer was composed of slip line-like features. The width of the features was found to be consistent with the scale of the dislocation cells measured in TEM. Cracks were observed to form and propagate parallel to the surface along the boundaries of the features within 2 microns of the surface. This evidence as shown in Figures 11-13 and 18-20. In conjunction with that observed in TEM, these results appear to show that cracks do indeed propagate parallel to the boundaries of dislocation cells.

Supporting evidence for the formation of cracks at or close to dislocation cell walls is offered by Gardner, Pollock, and Wilsdorf whom investigated glide deformation in the necked region of single crystals deformed in tension (ref. 68). In situ deformation studies in the high voltage transmission microscope produced results which reveal the initiation of microcracks at cell boundaries in particle free single crystal materials (ref. 69). Microcrack initiation at
Fig. 44. A summary of the crack initiation mechanisms.
these interfaces was suggested to be a logical consequence of the high surface free energy of cell walls in stage III of tensile deformation. Gardner et al. suggested that surface effects, such as image forces, do not appear influence and dislocation behavior unduly when their density is high, especially for in situ deformation at high voltage for which specimens up to 5 microns thick may be deformed and remain electron transparent, implying that thin foil effects are minimized.

It is proposed here that critical strain to nucleate a crack is related to the misorientation between dislocation cells near the surface. In sliding wear the degree of deformation near the surface has been shown to be large. The experiments of Langford and Cohen (ref. 70) on heavily deformed iron wire found that at low strains misorientations between adjacent cells were similar to low angle tilt boundaries, being 3-5 degrees. At higher drawing strains, approaching that of wear, misorientation between cells increased, to 15-20 degrees, comparable to high angle grain boundaries. The misorientation measured between cells in worn iron approximately 1 micron from the surface was found here to be 15-17 degrees. Due to the ultra fine nature of the dislocation cells near the surface combined with the difficulties in preparing thin specimens from worn materials, measurements closer to the interface could not be made. It is expected that closer to the surface, in the absence of recovery or recrystallization, the misorientations would be greater due to the higher degree of strain. It is believed that there is a critical misorientation at which cracks initiate.
Gardner and Wilsdorf (ref. 69) suggested the energy approach to decohesion across any interface is applicable to dislocation structures. For a single phase metal the energy of fracture \( E(f) \) is defined:

\[
E(f) = 2E(s) - E(b)
\]

where \( E(s) \) is the surface energy and \( E(b) \) is the energy of any boundary. For perfect registry \( E(b) = 0 \), suggesting the work of fracture is equal to the energy of the new surfaces created. As the degree of strain and misorientation increase, so does \( E(b) \), effectively lowering the fracture energy. Hence, cell boundaries will be the preferred site for crack initiation. This argument also would apply to cracks formed near the wear surface. Due to the very large shear strains imposed in the materials for sliding wear, crack nucleation would be the most likely at triple point sub-grain junctions of dislocation cells, since the localized stress along the dislocation walls would be highest due to the incompatibility strains induced by one layer of cells sliding past that below aided by the tensile stress component. This concept of crack initiation is analogous to the mechanism of wedge-like crack formation by grain boundary sliding during high temperature creep deformation.

Although energy models of fracture typically are often unsatisfactory due to plastic deformation expending energy on heat, the regions at the immediate surface are at the plastic limit, no longer being able to support further plastic deformation, hence,
decohesion with little further plastic flow would then be expected. Gardner and Wilsdorf (ref. 69) suggested that the process of decohesion at subcell boundaries is fundamental to the mechanism of mechanical failure in ductile pure metals. Clearly this suggestion appears relevant to sliding wear.

Hence, the nucleation of cracks at dislocation cell walls is primarily expected in single phase pure metals such as ferritic iron. Yet for the two-phase materials crack initiation at inclusions, oxides, or second phase particle as suggested by Suh et al., and Ashelby et al. competes with initiation at dislocation cell walls. Crack initiation in AISI 1020, composed of hypo-eutectoid pearlite colonies and pro-eutectoid ferrite grains, is expected by both decohesion at the carbides as well as at dislocation cell boundaries in the ferrite grains. Whereas in spherodized steel, containing carbide particles dispersed throughout the ferrite grains, initiation principally occurs by decohesion or shear of the particles, as shown in Figures 16 and 17.

Wear crack initiation in ferrous lath martensite is more complex than above due to the existence of three phases: iron carbide, retained austenite, and lath martensite. Due to autotempering, Widmanstatten carbides are observed to form within the lath even during a rapid quench (ref. 68). Logically these would be expected to initiate the crack. In situ tensile deformation in the high voltage electron microscope of martensitic foils (ref. 72) revealed cracks to initiate and propagate within the martensite laths, presumably indicating their initiation at the carbides. However, in the martensite microstructure there are other possible sites for crack nucleation,
such as the martensite-retained austenite boundary. In the as quenched state Rao (ref. 71) and Sarikaya (ref. 73) have shown this boundary to be coherent, consistent with the Kurdjumov-Sachs and Nishiyama-Wasserman orientation relationships. In this study, the observation of retained austenite no closer to the wear surface than 1 micron implies the metastable austenite transformed to martensite under the large shear strain induced by sliding wear. The possibility exists that the newly formed martensite may act as a stress concentrator causing the crack to form at the lath boundary. Thus, wear cracks may initiate in association with carbides or with the lath boundaries.

(c) Influence of Recovery

To complete the discussion of crack initiation it is necessary to consider the influence of recovery and recrystallization. Regions of adjacent to the wear surface of dislocation density lower than that 0.5 microns away from the interface were observed in ferritic and pearlitic materials as shown in Figures 22 and 23. These observations have been interpreted in this study to be indicative of dynamic recovery occurring during sliding wear.

The influence of recovery on the initiation of wear cracks is seen from the decreased dislocation density of the surface relative to the bulk applying a tensile stress on the boundary sufficient to assist crack nucleation in the already highly misoriented region. Dynamic recovery, being a dislocation cross slip and climb process, occurs rapidly during coldwork for annealed pure iron between 50-200°C (ref. 74) but is generally better known for steels to occur between
300–600°C, depending on composition, structure, and prior strain (ref. 75).

Recrystallization, being a nucleation and growth process, typically occurs at higher temperatures and is not expected to occur in iron and steel alloys tested under the stated conditions of this study. In low alloy steel recrystallization has been shown to occur at 650°C in 1 minute (ref. 76), yet at 480°C in more than $10^3$ minutes (16.7 hrs). Therefore, effectively below 600°C recrystallization in iron does not occur. Even though coldwork reduces the recrystallization temperature by increasing the stored energy and the number of nucleation site, a 50 percent reduction in area was shown only to decrease the temperature, for 50 percent recrystallization, by 100°C (ref. 77). Thus, for recrystallization to occur in iron and steel during sliding wear, the temperature at the contact must be maintained in the range of 400–600°C.

In sliding wear the length of asperity contact is short, being well less than one second. Spurr (ref. 78) has shown for steels that contact temperatures range from 70–420°C as derived from potential drop studies and measured by thermocouples under a wide range of sliding conditions. Hence, for iron and steel alloys only limited recovery would be expected under unlubricated sliding wear conditions.

The conclusion of recovery causing a lower dislocation density observed near the surface is contrary to that of Dautzenberg (ref. 63), Kuhlman-Wilsdorf (ref. 79), and Ives (ref. 80). All of these investigators have studied chiefly copper and suggested that
dynamic recrystallization (and not dynamic recovery) occurs at the contact surface. Since the recrystallization temperature for 50 percent deformation of copper cold rolled 98 percent has been shown to be slightly above 100°C. In view of the apparent contact temperatures suggested by Spurr, the degree of strain, the threshold recrystallization temperature, and the time length of the sliding contact, recrystallization would be expected for copper but not for iron or steel.

Hence, it is concluded that recovery does indeed occur for iron and steels in unlubricated sliding wear. Dynamic recovery is therefore the by-product of the extreme plastic deformation in dry sliding wear and not a prerequisite for failure along dislocation cell boundaries. The observance of recovery in ferrite and pearlite treated steels and the absence in martensite is due to the high initial dislocation density, the small microstructural scale, and the high carbon content combined with the shear transformation of austenite to martensite preventing cross slip and climb, thereby discouraging recovery.

3.0 Crack Propagation

The principal point in considering crack propagation is the explanation of plate-like wear particle formation. The stresses involved in crack propagation in sliding wear have received a great amount of attention. The calculations of Smith and Liu (ref. 33) suggested that beneath a passing asperity the stress cycled from compression to tension. Further calculations by Flemings and Suh (ref. 27) suggest the stress to cycles from plastic compression to
elastic tension on the passage of a surface asperity past a fixed point in the contacting plane. Suh et al. reasoned that hydrostatic compressive stress prevented surface cracks from growing, allowing subsurface cracks to propagate parallel to the surface in the tensile part of the applied cyclic stress. This view, however, allows the influence of shear stresses and strains endured during sliding, to produce sharp, well defined dislocation cell structures parallel to the sliding surface, reflecting exceedingly high (greater than 4) strains.

From studies on predominately FCC materials Hirth and Rigney (ref. 45) first suggested that cracks would form parallel to the surface. The principal plastic shear acts on a plane in the subsurface region due to the hydrostatic compressive stress state of the immediate surface layers. Subsurface cracks tend to follow a path along dislocation walls. From both the SEM and TEM observations made in this study, crack propagation in BCC materials does indeed appear to occur along dislocation cell walls, for the materials studied.

Since the dislocation cells have been observed to be oriented parallel to the sliding surface (see Figures 21-28), crack propagation predominately along (or in) the cell walls would yield the plate-like particle observed (see Figures 33-34). The debris particle thickness (oxide) measured by tilting experiments in the SEM are comparable to the width of the recovered zone observed in ferritic and pearlitic materials, taking in account the Pillings-Bedworth ratio. In TEM the recovered zone is seen to be 2000-3000Å wide (see Figure 23). Whereas the oxidized planar wear particle measures 4000-4200Å thick. Hence,
plate-like wear particles of oxide character would only form if crack propagation were to occur parallel to the recovered zone along dislocation cell boundaries. Thus, the observations in this study on several BCC materials confirm the suggestions of Hirth and Rigney (ref. 45) that crack propagation does indeed follow dislocation walls resulting in planar wear particles.

Due to the well defined cell structure, high degree of shear strain near the surface, as well as the large misorientation between adjacent dislocation cells, the contribution to crack propagation by mode I is believed to be secondary to that of mode II. Fleming and Suh considered mode II crack propagation using the same assumptions as for mode I. The crack faces were suggested to lock under the compressive stress allowing crack propagation only in the tensile portion of the cycle (ref. 35). Whereas Hirth and Rigney (ref. 45) suggested that in mode II the crack would propagate in the intense plastic shear region ahead of the load contact where the compressive stresses act on the surfaces of the crack. Shie (ref. 37) successfully produced under controlled conditions a shear crack under compressive loading along the maximum shear direction associated with crack tip shear displacement, substantiating the conclusions of Hirth and Rigney. Hence it is clear from this study and that of others that wear cracks form along dislocation cells and propagate by shear while under compression.

4.0 The Pyrophoric Effect

Wear particle formation occurs when the subsurface cracks are sufficiently long that they cannot tolerate the applied loads.
Assuming the wear debris are a fundamentally critical size, wear failure is imminent for particles 1-2 microns in diameter. Failure occurs by separation of the platelet during the tensile phase of the asperity passage cycle. Plastic deformation during particle separation evolves heat. Due to the exposure of fresh highly deformed material in the presence of oxygen combined with the poor thermal conductivity of air and the high specific area of the debris, oxidation readily occurs. Moreover, oxidation of the debris occurs during formation (separation) of the wear particles. Heat cannot be drawn away from the particle thereby motivating oxidation. Upon separation of the plate from the substrate, heat is evolved from the plastic deformation and exposure of fresh highly deformed material occurs, causing the underlying layers to recover as shown in figure 40c.

Experimental studies on the reactivity of fine iron powders by Evans, Boland and Mardon (ref. 81) determined that iron particles with specific areas (area/mass) greater than 5 m²/g spontaneously oxidize at room temperature. Whereas at 0.80 m²/g spontaneous oxidation occurs at 285°C. The particles in this study were typically planar ranging from 0.2 to 5.0 with a 1:2 aspect ratio. The specific area was calculated to range from 0.79-0.893 m²/g, with the smaller particles having a higher specific area. For oxidation to occur in wear, the data of Evans et al. would suggest that the wear surface temperature locally needs to be in the range of 250-300°C. From the results of Spur (ref. 78) combined with the observed recovery in iron the local temperatures near the surface is in the range of 100-300°C, most likely close to 300°C.
Oxidation, however, may occur at substantially lower temperatures if the heat evolved during the separation of the particle (heat of deformation) is not rapidly drawn away from the particle. Since the process of oxidation itself produces heat, oxidation may become autocatalytic. Only a flash in temperature into the 250-300°C range would then be necessary to initiate oxidation. In unlubricated sliding wear it is most reasonable to conclude that in the vicinity of an asperity (or propagating wear crack) peak temperatures in the 300°C range would be experienced. Hence the entire sliding surface would not have to reach 300°C for oxidized wear debris to be formed, only small regions locally.

To determine whether oxidation occurred before or after platelet separation as well as the influence of environmental factors on the pyrophoric mechanism of debris formation, several experiments were carried out in an inert gas atmosphere and at low temperatures. If the pyrophoric mechanism is to apply to wear both temperature and availability of oxygen should affect the particle oxidation. When the temperature was lowered, as in experiments performed in liquid nitrogen, metallic wear particles were observed. Similarly in experiments performed in helium, wear particles were predominately metallic (Figure 39). In comparison, AES studies on the surface of the worn pin show only a 50Å (500nm) thick oxide (insufficient to account for the debris character). Hence, the experimental evidence strongly suggests that the plate-like wear particles were initially metallic and oxidized in air upon separation from the substrate. This deduction is significantly different from that of Quinn (ref. 17-19) who
suggested that the oxide forms on the wear surface and that particles form through spallation of the oxide. Hence, it is clear from this study that in slow speed sliding wear the pyrophoric mechanism of wear particle formation is significant.

c) **Significance of the Mechanism**

There are several key points to the mechanism proposed herein which makes it unique:

(a) relates changes in ferrous microstructures to the observed changes in the wear characteristics;

(b) a relationship is established between the deformation induced into the parent material (the substrate) and the formation of the debris particle;

(c) the pyrophoric mechanism of oxidation of the debris accounts for the observed oxide wear debris produced in air as well as the effect of atmosphere and temperature.

Unlike other mechanisms reviewed in the Introduction, the mechanism herein accounts and explains the influence of the major variables in the sliding wear process leading to a deeper fundamental understanding.
V. CONCLUSIONS

1. Metal composition and microstructure have a significant affect on the degree of wear.

2. The strains produced during sliding wear exceed 5.5 true shear strain. Debris in ferrous materials particles form at a critical shear strain of 5-6.

3. Wear particle formation in ferrous metals is associated with dislocations structures. Cracks initiate at or around dislocation cell boundaries, second phase particles, or inclusions. Crack propagation typically occurs along cell boundaries.

4. Sliding wear debris of ferrous materials are pyrophoric, for which oxidation occurs during platelet separation from the substrate.

6. The degree of wear particle oxidation is dependant on the availability of oxygen, the extent of deformation, the rate of heat removal, and the specific area of the wear particle.
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REFERENCES


72. G. Thomas, unpublished work.


78. R. T. Spurr, "Temperatures reached by sliding thermocouples,"
Wear, 61, 1980, pp. 175-182.

79. D. Kuhlmann-Wilsdorf, "Dislocation concepts in friction and wear,"
in ref. (7), pp. 119-185.

80. L. K. Ives, "Microstructural changes in copper due to dry abrasion
246-256.

81. J. P. Evans, W. Borland, and P. G. Mardon, "Pyrophoricity of fine
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