EFFECT OF SLIDING VELOCITY ON THE TRIBOLOGICAL BEHAVIOR OF COPPER AND ASSOCIATED NANOSTRUCTURE DEVELOPMENT

DISSERTATION

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By
Andrew Emge, M.S.

The Ohio State University
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Dissertation Committee:
Professor David Rigney, Adviser
Professor Glenn Daehn
Professor John Morral
Professor Isamu Kusaka

Approved by
Adviser
Graduate Program in Materials Science and Engineering
ABSTRACT

The unlubricated sliding of metals is important in many mechanical devices covering a wide range of sliding velocities. However, the effect of sliding velocity on the tribological behavior of unlubricated metals has not been widely studied. Similarly, the relationship between microstructures developed at high sliding velocities and tribological behavior has not been studied in depth. Microstructures produced at low sliding velocities have been studied extensively and commonly include nanocrystalline or fine grained material near the sliding surface with heavily deformed microstructures further from the surface.

The current research relates two aspects of the sliding friction of ductile metals, the effect of sliding velocity and the production of nanocrystalline tribomaterial. The project focused on the effects of sliding velocity on the frictional behavior of oxygen free high conductivity (OFHC) copper sliding against 440C stainless steel, Nitronic 40 stainless steel, and copper. Low velocity tests were performed with a pin on disk tribometer. High velocity tests were performed with a rotating barrel gas gun (RBGG) which combined impact with sliding. The RBGG provides sliding velocities as high as 5.5 m/s and impact velocities as high as 12 m/s while maintaining sliding times on the order of tens of microseconds. Changes in the coefficient of friction, microstructure, and composition were studied.
Surface and subsurface microstructures of the worn samples were characterized with a range of instruments including scanning electron microscopy (SEM) with energy dispersive X-ray spectroscopy (EDS), focused ion beam (FIB) milling and imaging, transmission electron microscopy (TEM) with EDS, orientation imaging microscopy (OIM), and nanoindentation.

In the case of self-mated copper the sliding velocity had little effect on the coefficient of friction for both experimental apparatuses. For the case of copper sliding against 440C stainless steel on the pin on disk system the friction was found to increase with sliding velocity and was strongly influenced by material transfer from the copper to the steel pin. An increase in the coefficient of friction with sliding velocity was observed for the sliding of OFHC copper against Nitronic 40 steel in RBGG tests. The increase in the coefficient of friction was correlated to an increase in subsurface plastic deformation and grain refinement.

The growth of the nanocrystalline tribolayer in copper after sliding against 440C stainless steel at varying times was studied at sliding velocities of 0.05 and 1.0 m/s. A sliding velocity of 0.05 m/s produced a consistent nanocrystalline layer in as little as 10 s. The thickness of the nanocrystalline layer grew to an average thickness of 3 µm after 10 ks of sliding, but large variations in thickness were observed. A sliding velocity of 1.0 m/s produced a continuous nanocrystalline layer after 10 s of sliding. Ledges developed on the wear tracks at longer sliding times which greatly influenced the tribolayer thickness making it difficult to quantify. Dynamic recrystallization of the tribolayer also led to difficulties in measuring its thickness.
Dedicated to

My wife Angela and my entire family
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VITA

Oct. 19, 1979..................................................Born in Emporia, Kansas
2002................................................................B.S. Chemistry, Louisiana Tech University.
2006.............................................................M.S. Materials Science and Engineering,
The Ohio State University
2002 – present..............................................Graduate Research Associate,
The Ohio State University

PUBLICATIONS


FIELDS OF STUDY

Major Field: Materials Science and Engineering

Study in Tribology:

   Prof. D.A. Rigney

Study in materials characterization:

   Prof. D.A. Rigney and Prof. M.J. Mills
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CHAPTER 1

INTRODUCTION

The understanding of the sliding behavior of metals at high sliding velocities is important in mechanical parts such as bearings, brake liners, gears, and friction clutches, and also in some microelectromechanical systems (MEMS) [1,2]. Despite the large body of work exists on the unlubricated sliding of metals there is a more limited volume of work on the influence of high sliding velocities on frictional behavior. Molecular dynamics (MD) simulations by Hammerberg et al. on the sliding of self-mated copper have predicted that the coefficient of friction depends markedly on the sliding velocity [3]. Early work by Bowden et al. focused on the effect of sliding velocity on the coefficient of friction for unlubricated metals at sliding velocities of hundreds of meters per second and reported a decrease in the coefficient of friction with increasing sliding velocity [4, 5]. However, characterization of the samples was rather limited.

A common theme in much of the work in the literature on high sliding velocity tribological behavior is a lack of characterization of microstructures produced by high sliding velocities. Furthermore, the relationship between microstructure and frictional behavior at high velocities is not well understood. Deformation substructures have been
characterized for many lower sliding velocity tests of ductile metals and commonly include nanocrystalline or fine grained material near the surface and heavily deformed microstructures further below the surface [6-8].

The appearance of finely structured material, also known as the tribolayer, produced near the surface during sliding has been widely reported. The tribolayer has been shown to be the source of wear debris and has a strong effect on tribological behavior. In some cases the development of a stable tribolayer has been suggested to result in a decrease in the wear rate and coefficient of friction for the unlubricated sliding of metals [9, 10]. Although development of the tribolayer has been widely reported, the means by which it is produced are somewhat unclear. Some authors have likened tribolayer formation to severe plastic deformation processes [11]. Recent work by Karthikeyan et al. on the unlubricated sliding of materials has suggested that vorticity may be responsible for tribolayer formation [12].

The work presented here focused on two particular aspects of the unlubricated sliding of metals. The first was the effect of sliding velocity on the coefficient of friction and microstructural evolution. This was studied with four series of experiments. Pin on disk tests were performed for self-mated copper in air and copper sliding against steel in vacuum. Higher velocity tests were performed with a rotating barrel gas gun (RBGG) for comparison. RBGG tests were performed for self-mated copper and for copper sliding against stainless steel in air.

The second focal point of the research was on the development and growth of the nanocrystalline tribolayer produced in copper at low and high sliding velocities. The tribolayer development and growth was studied using two series of tests, one at a high
sliding velocity and one at a low sliding velocity, on the pin on disk system. The sliding time of the tests was varied to allow for microstructural characterization of the tribolayer at different stages of development.

This research project was a collaborative endeavor between the wear group at The Ohio State University (OSU) and two groups at Los Alamos National Laboratory. Sliding tests were performed on two experimental apparatuses. The first was a modified pin on disk system designed and constructed at OSU. This system allowed for low sliding speeds and relatively long contact times.

The second apparatus was a novel rotating barrel gas gun which was located at Los Alamos National Laboratory. It was capable of performing high sliding velocity, short term tests through impact and sliding loading. In an improvement over some other high speed tribometers, the RBGG allowed for independent control of the impact pressure and sliding velocity.

Characterization of all samples from both test apparatuses was performed at OSU taking advantage of the electron optics facility. Characterization was focused on surface and subsurface deformation induced by sliding and was carried out using a wide range of techniques including scanning electron microscopy (SEM), transmission electron microscopy (TEM), and ion channeling contrast imaging with a focused ion beam (FIB).
CHAPTER 2

LITERATURE REVIEW

2.1 Introduction to tribology

The word ‘tribology’ comes from the Greek word ‘tribos’ which means rubbing [13]. The field of tribology is described as the science of interacting surfaces in relative motion and includes the study of friction, wear, and lubrication [14,15]. Friction is a dissipative process and can be described as the resistance to motion of one body moving tangentially across another body in which it is in sliding or rolling contact. The friction coefficient is the ratio of this resistive tangential force to the normal force holding the two bodies together and is expressed as

$$\mu = \frac{F_T}{F_N}$$

The coefficient of friction can range from values of about 0.001 for lightly loaded roller bearings to greater than 10 for self-mated clean metals under vacuum [13].

Two empirical “laws” of friction were initially presented by Leonardo da Vinci, and were additionally discovered and proposed by Guillaume Amontons and are known as Amontons’ equations [16, 17]. A third law was later added by Coulomb. These three laws are as follows:
1. The friction force is proportional to the normal load

2. The friction force is independent of the apparent contact area

3. The friction force is independent of the sliding velocity.

It is important to emphasize that these are empirical laws based on observations, and that there are various exceptions to each of them. The first law can be expressed by the above equation and holds for many materials pairs and sliding conditions. However, it does not always hold for the unlubricated sliding of metals. One example comes from the work of Whitehead on the sliding of copper on copper in air [18]. The coefficient of friction versus the normal load was not found to be constant as shown in Figure 2.1. The transition in the coefficient was explained in terms of an oxide film that on the surface of the copper. The oxide was proposed to affect the coefficient of friction by preventing direct contact of the two metals. This oxide layer survived at low loads, but was destroyed at higher loads leading to true metallic contact and an increase in the coefficient of friction.

The second law tends to hold for most materials. However, polymers and other elastic and viscoelastic materials do not follow the second law [13, 15]. This is in part due to the fact that the real area of contact approaches the apparent area of contact for soft materials such as polymers [16].

The third law is typically not true. The coefficient of friction generally decreases with an increase in sliding velocity. This may be due in part to frictional heating which causes softening or melting of the material near the surface [4, 5]. Experimental work on the unlubricated sliding of metals has shown a decrease in the coefficient of friction at very high sliding velocities as can be seen in Figure 2.2 [4].
2.1.1 Adhesion and ploughing mechanisms of friction

Early work by Bowden and Tabor proposed that the friction force could be broken down into two components [18]. For unlubricated sliding they suggested that friction has an adhesion component and a deformation component [19, 20]. The coefficient of friction, $\mu$, was therefore written as the sum of the adhesive term $\mu_{adh}$ and the deformation term $\mu_{def}$,

$$\mu = \mu_{adh} + \mu_{def}$$

The deformation component of friction is the result of asperities of the harder material ploughing grooves in the surface of the softer material or fracturing the softer material. This case was simplified by considering a single rigid asperity sliding over a planar surface. The asperity was assumed to be conical in shape with a semi-angle $\alpha$ for further simplicity. The friction force needed to move the asperity was set equal to the product of the flow pressure, or hardness, of the softer material and the cross-sectional area of the groove. This approach led to the following equation for the deformation component of friction.

$$\mu_{def} = \frac{2 \cot \alpha}{\pi}$$

According to Bowden and Tabor, the adhesion component of friction resulted from the attractive forces that are present at asperity contacts. The adhesive forces for metal pairs tend to be greater if the mated surfaces are free from oxides, surface films, and adsorbed gases. Ductile metals such as gold and copper have been found to exhibit adhesive forces sometimes greater than the load used to hold the materials in contact under ultra-high vacuum (UHV) conditions [13]. The adhesive forces at the asperity
contacts can be sufficiently strong such that junctions are formed and the two materials effectively become a continuous solid at the contact area [21]. For such a case Bowden and Tabor expressed the adhesive component of friction in terms of the mean shear strength $\tau_m$ and the yield pressure of the softer material $p_0$, or the indentation hardness $H$, as

$$\mu_{adh} = \frac{\tau_m}{p_0} = \frac{\tau_m}{H}$$

The adhesive component of the coefficient of friction tends to be about 0.2 for metals, and the deformation component for metals tends to be less than 0.1 if the previous two equations are used [13]. This gives a coefficient of friction of 0.3 or less for metals which is significantly less than experimentally measured coefficients of friction for many sliding metal pairs [16]. Hutchings suggested that the difference in theoretical and experimental values could be explained by work hardening and junction growth. The effect of work hardening would be to increase the mean shear stress $\tau_m$ relative to the indentation hardness $H$ which will lead to an increase $\mu_{adh}$ based on the expression for the adhesive component [13].

Tabor later modified their adhesion model to account for junction growth and also addressed the effect of surface contamination [22]. This resulted in a new expression for the coefficient of friction:

$$\mu = \frac{1}{\sqrt{\alpha \left(k^{-2} - 1\right)^{1/2}}}$$

where $\alpha$ is a constant and $k$ is defined as
\[ k = \frac{\tau_i}{\tau_m} \]

where \( \tau_i \) is the critical shear stress of the contaminant film and \( \tau_m \) is the critical shear stress of the metal. This model yields a coefficient of infinity for clean surfaces (when \( \tau_i = \tau_m \)) and quickly drops to low values of friction as surface contaminant films decrease in shear strength.

One of the problems with the above theories and expressions for the adhesive component of friction is that the mean shear strength is not clearly defined. This leads to some ambiguity in the literature as other researchers used the adhesion theories, but interpreted the mean shear strength differently. In some work it has been interpreted to be proportional to the material yield pressure while in others it is taken to be equal to the yield shear stress of the material [23]. Furthermore, it is unclear whether the mean shear stress is that of the harder or softer material or of the junction.

In addition to the confusion over the mean shear stress, this approach only focuses on the materials properties of hardness and flow stress and does consider modification of the materials during sliding. It does not take into account work hardening, and also ignores the effect of energy dissipation through plastic deformation of subsurface regions of one or both of the tribopair materials. The approach also fails to include microstructural properties which are widely known to affect material behavior.

2.1.2 Energy-based friction models

Rigney and Hirth introduced a friction model based on the idea that frictional energy is mostly dissipated during plastic deformation. It more specifically focused on the work done in the heavily deformed region of metals that occurs just below the sliding
surface [24]. This region commonly contains a very fine microstructure that includes dislocation cells [25]. The model was developed for steady state sliding conditions under which the cell region of the material has a constant thickness $t$. It is assumed that the frictional work is the result of plastic deformation that is confined to the cell region. The plastic work during the process is equated to the frictional work during the process which gives

$$V \tau \varepsilon = F \delta x$$

where $V$ is the volume of the plastically deformed region, $\tau$ is the shear stress in the cell region in the sliding direction, $\varepsilon$ is the strain per cycle in the cell region, $\delta x$ is a displacement, and $F$ is the friction force. The deformed volume $V$ is given by $wt \delta x$ where $w$ is the width of the deformed layer. The coefficient of friction takes the form of

$$\mu = \frac{wt \tau \varepsilon}{W}$$

The term $W$ in the above equation is the normal force. This simple model was found to predict coefficient of friction values for copper that are quite close to experimentally determined values and can be used for materials pairs where both materials deform plastically by taking the sum of the coefficients of friction for each material [24].

Further work by Heilmann and Rigney, again based on the idea that frictional work is dissipated primarily through plastic deformation under steady state conditions, led to the extension of the energy-based friction model previously discussed [26]. The mechanical behavior and properties of materials were introduced through stress-strain curves which led to the following expression for the coefficient of friction.
\[ \mu = \frac{NA}{W} \tau_{\text{max}} F \left( \frac{\tau_s}{\tau_{\text{max}}} \right) \]

where \( N \) is the total number of asperity contacts, \( A \) is the average area of each contact, \( W \) is the load, \( \tau_s \) is the surface stress, and \( \tau_{\text{max}} \) is the ultimate shear strength of the material. The function \( F \) is a monotonic function defined by

\[ F = 1 - 2 \frac{\ln(1+u) - u}{\ln(1-u^2)} \]

where \( u = \frac{\tau_s}{\tau_{\text{max}}} \) for the range \( 0 \leq u \leq 1 \). This model is fairly general and can be used for many sliding conditions and materials pairs including materials with coatings.

Some of the difficulties with using the above energy based friction models are that both the surface stress and real area of contact are difficult to determine during steady state sliding. Furthermore, the ultimate shear strength of the sliding materials may not be available, and is complicated by the presence of oxide layers and contaminants. These models also focus on steady state sliding conditions and do not address friction at short times.

Wang and Rigney modified the above model further to apply it to ductile materials and materials with coatings, and to allow for the calculation of the coefficient of friction as a function of sliding distance, including short sliding distances such as in run-in periods [27]. This modified model was applicable to any materials system for which stress-strain curves, displacement profiles, and hardness were known. This model again used stress-strain curves to incorporate materials properties, but also included the hardness and displacement profiles, all of which are a part of the integral \( I \) in the following equation for the coefficient of friction.
Displacement profiles can be determined by measuring the displacement of intrinsic markers, such as lamellar structures or grain boundaries, or by inserting artificial markers such as thin films.

The above equation can be used for any sliding system including materials with coatings and materials undergoing chemical reactions, phase transitions, or mechanical mixing as long as the appropriate stress-strain curves, displacement profiles, and hardness values are available. The difficulty with using this model again includes a lack of experimental data to feed into the model. Appropriate stress-strain curves and displacement profiles may be difficult to find or determine for the sliding materials.

2.1.3 Wear

Wear is defined as “the surface damage or removal of material from one or both of two sliding surfaces in a sliding, rolling, or impact motion relative to each other” [16]. Wear can operate by many different mechanisms including adhesive, abrasive, fatigue, impact, chemical, and fretting. Adhesive and abrasive wear are estimated to account for nearly two-thirds of the wear in industrial applications [16]. Adhesive, abrasive, corrosive and fatigue wear will be discussed in later sections.

2.1.3.1 Wear measurement

The extent of wear is often measured in terms of the volume of material removed during sliding or rolling. In the case of sliding wear of unlubricated metals it has been found that the wear volume \( v \) is generally proportional to the applied load \( W \) and sliding distance \( x \) and inversely proportional to the hardness of the material undergoing
wear. A theoretical analysis for such wear was developed by Archard and Holm and yielded what is known as the Archard wear equation,

\[ \nu = \frac{KWx}{H} \]

where \( K \) is termed the wear coefficient [13]. The wear coefficient is dimensionless and always less than unity [16]. The wear coefficient varies over a large range \( (10^{-2} \text{ to } 10^{-7}) \) in order to fit experimental data [28]. The Archard wear equation does not hold for many materials pairs and sliding conditions, but the wear coefficient is a useful parameter for the comparison of the severity of wear in different systems [29, 30].

### 2.1.3.2 Adhesive wear

Adhesive wear is thought to occur when asperities from the two surfaces adhere or cold-weld to each other and are subsequently sheared by sliding [31]. This may result in the removal of material from one surface and attachment to the other. This material can then be transferred back to its initial surface or break free to form wear particles. Some authors have broken adhesive wear of metals down into subcategories of mild and severe wear [32]. Severe wear has been categorized by some researchers as having large metallic wear particles on the order of 1-100 µm in size and a specific wear rate between \( 10^{-7} \) and \( 10^{-5} \) mm\(^2\)/N [33]. Mild wear has been characterized as producing small oxide wear particles ranging from 10-100 nm in size and specific wear rates less than \( 10^{-8} \) mm\(^2\)/N.

### 2.1.3.3 Fatigue wear

Fatigue wear is defined as wear resulting from repeated sliding or rolling contact of a surface. During repeated sliding the materials undergo repeated loading and
unloading. This causes the initiation of surface and subsurface cracks. Further loading will cause the cracks to propagate which will eventually cause the removal of surface material and the formation of wear debris. Wear rates for fatigue wear can be quite low for an extended period of time and experience sudden increases [19].

2.1.3.4 Abrasive wear

Abrasive wear occurs when the asperities of a hard surface or hard particles slide on a softer surface causing damage [16]. The abrasive wear of the softer surface by the asperities of the harder surface is called two-body abrasion. Abrasive wear caused by hard particles caught between the two surfaces is called three-body abrasion and may damage both sliding surfaces. In some cases the break-up of hard oxide layers on metallic surfaces can result in abrasive wear by particles of the oxide. Abrasive wear may occur via both plastic deformation and brittle fracture [13]. Wear debris produced by abrasion commonly contains cutting chips [34, 35].

2.1.3.5 Corrosive wear

Corrosive wear is unlike other types of wear in that it is affected by chemical reactions between the environment and the material surface. Corrosive wear occurs when one or both of the sliding materials react with components in the environment causing dissolution of the surface or the formation of reaction products [36, 37]. Once reaction products are formed on the surfaces they will influence the sliding behavior. In some cases metal oxides will be formed that help protect the surface from pure metal on metal contact. In other cases the reaction products may break apart and be mixed into the base material, thus affecting the sliding behavior.
2.2 Unlubricated sliding behavior of metals

The sliding of unlubricated metals is capable of producing chemical changes and extensive structural changes [38, 39]. Some phenomena commonly observed in unlubricated sliding of metals include transfer of material from one sliding surface to the other, mechanical mixing, production of wear debris, and surface and subsurface plastic deformation [40-42]. Large plastic shear strains, sometimes in excess of 100, and strain gradients are also common in the subsurface regions of slid metals [43-45].

The subsurface region was subdivided into three zones by Rice et al. as shown in Figure 2.3 [46]. Zone 1, consisting of the base material, was the furthest away from the sliding interface, and was considered to be unaffected by the sliding. Zone 2 contained the base material that had undergone plastic deformation. Material near the top of Zone 2 was more heavily deformed than material near the bottom and has commonly been found to exhibit a refined structure. Zone 3 included the contact surface as well as the initial material and possibly species from the environment and the counterface material that were mixed in during sliding. Zone 3 may also be called the tribolayer or mixed layer and will be discussed in more detail in the following section.

The descriptions of the subsurface zones fit well with the findings of other researchers. Zone 2 in the description from Rice corresponds to the heavily deformed region found just below the tribolayer. This region can extend up to hundreds of micrometers from the surface or be as shallow as a few micrometers [47]. The depth of this region can be determined by using markers such as grain boundaries, lamellar microstructures, and implanted markers. Markers that are initially perpendicular to the surface tend to bend over in the direction of sliding and can be used to obtain estimates of
the strains and strain rates [47, 48]. Elongated subgrains and equiaxed cells have been observed in the Zone 2 region in high purity copper [47]. Similarly, dislocation cells have been observed in low-carbon steels [49].

In addition to microstructural changes, sliding is capable of inducing phase transformation, formation of metastable phases, and chemical reactions. For example, strain-induced martensite has been observed in type 304 stainless steel subjected to sliding [47]. Oxidation of sliding metallic surfaces and wear debris has also been reported [50, 51].

2.2.1 Tribolayer

The tribolayer has been referred to by many names in the literature, some of which are the third body, mechanically mixed layer, transfer layer, Beilby layer, and nanocrystalline layer. Some of the characteristics of the tribolayer as described by Godet et al. are that it flows, transmits load, and separates the tribopair materials [42]. Tribolayer material may also be driven far from equilibrium both compositionally and structurally. The tribolayer is important to understanding and controlling friction because it is the region where a majority of the plastic deformation occurs, and it has been shown to have a strong effect on sliding behavior [52, 53]. Furthermore, for two materials sliding against each other, it is the tribolayer, not the base material, that is actually in contact. Many researchers have also shown the tribolayer to be the source of wear debris [42, 54-55]. Work by Heilmann et al. looked at wear debris formed from the sliding of various copper based metals sliding against steel and found the wear debris to have the same structure and chemical composition as the tribolayer [56].
The tribolayer commonly consists of finely structured or even amorphous material and may include atomic species from both tribopair materials and the environment. The existence of nanocrystalline and/or amorphous material in the tribolayer has been reported in numerous findings [45, 57, 58]. The formation of nanocrystalline material in the tribolayer has been likened to the formation of nanocrystalline material through processes such as ball milling and other methods of severe plastic deformation [42]. The formation of nanocrystalline material by ball milling has been shown to evolve from dislocation structures. At early milling times dislocation cell structures or low angle boundaries are observed. As the milling time increases the cells decrease in size and the boundaries become high angle boundaries with nanoscale spacing [59].

More recent work by Karthikeyan et al. has suggested that vorticity may be responsible for nanocrystalline formation [12]. Molecular dynamics simulations of the sliding of unlubricated metals has shown vorticity formation along the sliding interface with a size scale similar to that of nanocrystals produced in experimental work. The presence of vorticity may also help to explain mechanical mixing within the tribolayer. Intermixing of atomic species in the tribolayer has been commonly reported and was thought to require lateral flow, but the mechanisms behind this flow were somewhat unclear [42]. Vorticity could provide a means for material to flow in a direction normal to the sliding interface and this would result in intermixing of the tribopair materials.

2.2.2 Material transfer

The sliding of unlubricated metals often results in transfer of material from one surface to the other [56, 60-62]. Material transfer has been found to be unidirectional or bidirectional and depends on the material pair in contact [57, 63]. Material transfer has
also been shown to have a significant effect on sliding behavior. Several researchers have attributed material transfer to adhesion and suggest that simple adhesion is responsible for transfer. However, adhesion alone is not sufficient in explaining transfer. For example, Heilmann et al. detected iron crystals within the nanocrystalline tribolayer on a copper sample after sliding against steel [56]. From these results it is obvious that material does not simply adhere to the opposing surface, but undergoes a complicated process involving transfer and mixing. Adhesion theories also ignore modification of the contacting material during sliding whether it is due to work hardening, mechanical mixing, or other mechanisms.

Early work by Kerridge and Lancaster focused on the formation of a transfer layer resulting from brass pins sliding against much harder Stellite or tool steel rings [63]. Radioactive brass and autoradiographs were used to measure transfer from the brass pins to the rings. Brass was found to transfer to the ring after short sliding times and reached steady state values early on as well. The wear rate of the pin was also measured and was found to reach a constant wear rate around the same time that the transfer layer reached a steady state. They concluded that equilibrium was reached between the rate of transfer from the pin to the transfer layer and the rate of loss of material from the transfer layer as wear debris. This equilibrium corresponded to a constant wear rate of the pin. They also found that the transfer fragments were many times smaller than the wear debris and suggested that agglomeration of transfer patches must occur.

A mechanism for material transfer was suggested by Rigney under which accumulated deformation and strain during sliding results in heterogeneous deformation structures [42]. The heterogeneous structures are then susceptible to shear instabilities
which promote transfer. Work by Chen et al. on various metal pairs has shown that
transfer starts in localized regions after short sliding times [64]. He also found that the
thickness of transfer fragments of both copper and aluminum were similar to the subgrain
size adjacent to the surface suggesting that the heterogeneity of the deformation
structures played a role in transfer.

2.3 Tribological behavior of copper

Heilmann et al. tested oxygen free high conductivity (OFHC) copper in sliding
against 440C stainless steel in a block on ring geometry [56]. Tests were performed with
a 66.6 N load, sliding velocities of 1 and 5 cm/s, and sliding distances of 0.12 to 12 m.
The coefficient of friction was found to vary between 0.3 and 0.6. Transmission electron
microscopy (TEM) was used to characterize the subsurface deformation in the copper.
An ultrafine grained layer with a grain sizes ranging from 3-30 nm was found closest to
the surface and can be seen in the upper left-hand corner of Figure 2.4 which is a
longitudinal section [47]. X-ray energy dispersive spectroscopy (EDS) revealed that the
layer was primarily copper, but did have small amounts of iron mixed in from the
counterface material.

Directly below the ultrafine grained region subgrains with sharp cell walls were
observed to be elongated parallel to the sliding direction. Further away from the sliding
surface the subgrains were equiaxed and the cell walls were less sharp. The transition
between equiaxed and elongated subgrains occurred when the average diameter of
subgrains decreased to 1 µm or less. Another interesting feature was a grain boundary
which appeared to be bent over in the direction of sliding. This grain boundary can also
be seen in Figure 2.4.
Additional work was performed to study the disorientation between the subgrains [65]. It was found that the disorientation of the cells increased as the surface was approached. Disorientation angles of over 50° were found near the surface while disorientations of less than 10° were found a few micrometers below the surface. The rotation of the cells relative to a reference cell was also measured. Rotations about the x-, y-, and z-axes were measured, where the x-axis was anti-parallel to the sliding direction, the y-axis was perpendicular to the sliding direction and parallel to the surface, and the z-axis was normal to the surface. The rotation was found to be predominately around the y-axis and decreased with depth. The depth of the rotations was found to increase with increasing sliding time as can be seen in Figures 2.5a and 2.5b for 1 and 100 cycle sliding times, respectively.

Further work Rigney et al. used the bent grain boundary in Figure 2.4 to estimate shear strains and strain rates [47]. It was assumed that the grain boundary was initially straight which allowed for measurements of the deflection of the grain boundary as a function of depth. The displacement versus depth profile was fit to an exponential function for depths greater than 4 µm. Extrapolation of the function to the surface estimated shear strains of 11.4 at the surface. The shear strain rate was calculated using the strain profile and sliding velocity giving a shear strain rate of $3.7 \times 10^3$ s$^{-1}$. Although there is some uncertainty in the displacement profiles near the surface due to disappearance of the bent grain boundary in the ultrafine grained layer, this method gives a general estimate of the high shear strains present near sliding surfaces.

Later work by Divakar focused on substructure development and orientation changes in OFHC copper after sliding against M2 tool steel [66]. Similar substructures
were reported to those found by Heilmann et al. including an ultrafine grained layer near the surface and dislocation cells further away from the surface [56]. Larger equiaxed grains were found at depths of a few microns from the surface and were concluded to be recrystallized grains. One other interesting finding was regions of the ultrafine grained region that appeared to be pressed into the surface of the copper.

Hughes et al. used the sliding of high purity copper against steel as a means to generate nanostructured material within the copper [45]. The test geometry consisted of a block of copper sliding against a sheet of steel with parallel sets of asperities. The sliding was confined to a single pass for a total sliding distance of 127 mm under an applied normal pressure of 22 MPa. The microstructures produced by the sliding were characterized using TEM.

The resulting microstructure is shown in a TEM bright field image in Figure 2.6. The left edge of the micrograph is the sliding surface. A very fine structure was produced near the surface with boundaries parallel to the surface and boundary spacings of 3-30 nm. The boundaries were not referred to as grain boundaries, but instead were termed geometrically necessary boundaries. The volume between boundaries was reported to contain very few dislocations. The spacing of cell boundaries was observed to increase further away from the surface reaching spacings of 50 nm at a depth of 2 µm and 170 nm at a depth of 20 µm. The boundaries were determined to be a mixture of both high and low angle boundaries at depths of less than 100 µm. Beyond depths of 100-200 µm high angle boundaries were no longer found. Strains were estimated based on microstructural details yielding an estimated strain of over 100 at the surface. The microstructures reported in this work are similar to those reported by Heilmann et al [56].
The estimated shear strains at the surface are much higher than those reported in [47], but may likely be due to the different experimental apparatuses.

Dryzek et al. used positron annihilation to study the effects of load, sliding distance, temperature, and sliding velocity on subsurface zones in copper after sliding against a steel ball in a pin-on-disk system [40]. Technical purity copper was used for the tests although there is no mention as to what is meant by technical purity. The tests were all performed under vacuum of $10^{-5}$ Torr. Positron annihilation was used to measure the S-parameter, which is a quantity that depends on vacancies, as a function of depth into the samples. The vacancies were considered to be a relative measure of the extent of deformation of the subsurface regions by comparison with untested specimens.

An increase in load from 1.14 to 2.49 N was reported to give an increase in the depth of detected vacancies from 300 to 800 µm as well as an increase in the defect concentration near the surface. Increasing the sliding time from 30 to 60 minutes showed an increase in the depth of defects, followed by a decrease after 90 minutes. The decrease in defect depth was proposed to be the result of dynamic recrystallization, but there was no measurement of temperature during the test or post-test microstructural characterization that could verify this proposition. An increase in sliding velocity from 5.3 to 15.3 cm/s showed an increase in both the depth of defects and the defect concentration near the surface. The effect of temperature was tested from -50 °C to room temperature, but there were no appreciable difference in vacancy depth and concentration.

Although this technique can yield some interesting results, it is fairly limited in terms of microstructural details. It doesn’t give any indication as to whether or not grain
refinement occurs as is commonly observed in copper [42, 45, 56]. Furthermore, there is no mention of whether or not species mixed in from the counterface can be detected.

Sadykov et al. investigated the effect of grain size on the wear intensity for copper shoes sliding against a hardened steel disk [67]. Tests were performed at a sliding velocity of 0.78 m/s, normal pressure of 5 MPa, and sliding distance of 500 m. Grain sizes ranging from 0.2-80 µm were tested. The wear intensity “I” was calculated from

\[ I = \frac{\Delta m}{LS} \]

where \( \Delta m \) is the mass loss, \( L \) is the sliding distance, and \( S \) is the area of contact.

A minimum wear intensity was reported for the ultrafine grained copper with a grain size of 0.2 µm followed by a steep jump to a maximum value for specimens with 2-3 µm grains. The wear intensity then decreased with increasing grain size to about 50µm, but did not reach the low values of the ultrafine grained materials. The minimum measured wear intensity for the ultrafine grained copper was related to microhardness values which decreased with increasing grain size from 1.4 GPa for the ultrafine grained copper to 0.7 GPa for the coarse grained copper. The increase in wear intensity with the decrease in hardness follows the general trend that would be expected from Archard’s wear equation [16].

Characterization of the sliding surfaces with scanning electron microscopy revealed grooves and ridges typical of ploughing processes in sliding wear. No compositional characterization was performed to check for transferred material. Microhardness measurements were taken as a function of depth for the ultrafine grained and coarse grained specimens. In both cases the material near the surface showed an
increase in microhardness to about 2.5 GPa. The increased hardness was localized to within about 10 µm of the surface for the ultrafine grained specimens, but extended about 50 µm from the surface in the coarse grained material. No explanation was given for this observation.

Later work by Sadykov et al. focused on the effect of texture on the wear intensity for copper over a range of grain sizes [68]. Coarse grained, fine grained and ultrafine grained copper was tested with the sliding direction either parallel or perpendicular to the extrusion direction. The tests’ geometry and sliding velocity were the same as in [67], but the normal pressure was fixed at 2 MPa and the sliding distance was varied from 0.5-10 km.

The reported wear intensities followed a similar trend to those in [67] in regards to grain size, but the difference between the ultrafine grained copper and larger grain sizes was significantly different. The wear intensity for the ultrafine grained material was reported as nearly five orders of magnitude lower than that of the copper with larger grain sizes. Orientation of the extrusion direction parallel to the sliding direction showed a decrease in wear intensity of about 10% for 2-3 µm grains as compared to perpendicular orientation. The effect of orientation was much more pronounced for grain sizes of 0.1-0.2 µm where the parallel orientation resulted in a wear intensity of 1.9 times less than the perpendicular orientation. The effect of orientation on the wear intensity was proposed to be a result of differing mechanical properties in different crystallographic directions. However, no experimental evidence was given that could verify this. Besides the extrusion direction, there was no mention of the type of texture of the samples.
Characterization of the surfaces with scanning electron microscopy and subsurface microhardness measurements gave similar results to those in [67].

The work by Sadykov et al. in [67] and [68] is useful in relating wear intensity to hardness, but it does not address effects of microstructure on the coefficient of friction or fully explain the effect of texture on wear intensity. It would also be interesting to see if the measured subsurface microhardness values corresponded to microstructural features such as grain size.

Biswas et al. studied the frictional behavior of OFHC copper pins sliding against alumina discs at varying sliding velocities [69]. The samples were allowed to run-in at a sliding velocity of 0.05 m/s for 120 minutes to ensure good contact between the pin and disc. Data were not recorded during the run-in period. The wear rate and steady state coefficient of friction were measured. The coefficient of friction was found to be 0.45 ± 0.05 regardless of the sliding velocity in the range of 0.1 to 4.0 ms\(^{-1}\). The wear rate was found to be the greatest at the lowest sliding velocity and quickly dropped off to much lower values with an increase in sliding velocity. This work ignored the transient behavior and the effect of sliding velocity on the transient behavior. No details were given as to the length of the transient period or how the coefficient of friction behaved during the transient period.

2.4 Effect of sliding velocity on friction – high velocity friction

Although there is a large body of research on friction a much smaller body of work is focused on dry sliding friction of metals and related structural changes at high sliding velocities, high loads, and short contact times [70]. The difficulty of reaching high sliding velocities on traditional tribometers has led to the development of several
high speed tribometers as well as the use of computational methods, such as molecular
dynamics, to study tribological behavior at high sliding velocities. This section will
focus on high sliding velocity test apparatuses, results obtained from such apparatuses,
and results from molecular dynamics simulations.

Work by Bowden et al. focused on high sliding velocity tests under low loads for
various metal pairs including steel on copper, aluminum, bismuth, antimony and
molybdenum and copper on molybdenum [4, 5]. The tribometer they used suspended
and rotated a steel ball with the use of magnetic fields and could reach sliding velocities
above 600 m/s. The ball was rotated between three vertical specimens set 120° apart.
One specimen was spring loaded and was used to apply the load to the spinning ball. The
balls were marked such that the rotational velocity could be measured with the use of a
photomultiplier. The deceleration of the ball was used to calculate the friction force as
the ball slowed to a stop.

Bowden et al. found that the coefficient of friction decreased with an increase in
sliding velocity for all metal on metal pairs tested. The decrease in the coefficient of
friction was attributed to frictional heating and eventual melting of the material. It was
also found that different initial sliding velocities resulted in differing coefficients as the
ball decelerated to lower velocities as can be seen for the case of copper sliding against
steel shown in Figure 2.2. Velocities below about 140 m/s for copper sliding on steel
resulted in seizure of the specimens and thus did not yield results at the lower velocities.
Characterization of the copper after sliding, starting at 150 m/s, showed flow of the metal.
After sliding, starting at 600 m/s, the surface of the copper was found to be smooth and
shiny with a very small grain size.
One of the problems with this method is that the coefficient of friction is not measured at a constant sliding velocity, but over a decreasing range of velocity. In addition, the coefficients measured at lower velocities are likely affected by structural and possibly chemical changes to the samples that occur at higher velocities. The surface structures reported for different velocities are most likely affected by continued contact during the deceleration period. Therefore the coefficients and associated structural changes measured pertain to a range of sliding velocities and may not be indicative of the behavior that would be observed when testing at a constant sliding velocity.

Philippon et al. designed a tribometer capable of reaching sliding velocities up to 60 m/s with an air-gun setup as shown in Figure 2.7 [71]. In their apparatus a dynamometer ring was used to apply a normal load of up to 230 MPa on materials $M_A$ and $M_B$. A projectile fired from an air gun impacted material $M_B$ which caused it to slide relative to material $M_A$. Strain gauges on the tube were used to measure the tangential, or frictional, forces.

Philippon et al. tested steel on steel tribopairs at different velocities and normal pressures. They found an increase in the coefficient of friction from 0 to 3 m/s, and a decrease for velocities above 13 m/s. Characterization of the specimens was not conducted, and no explanation for the trend in the coefficient of friction was given.

Various modifications to the traditional torsional Kolsky bar have been made by different researchers in order to study dynamic friction events. Rajagopalan and Prakash used a modified torsional Kolsky bar to study dynamic friction at high sliding velocities and normal loads, and short slip distances [1]. Their apparatus was capable of reaching sliding velocities as high as 15 m/s and normal pressures up to 125 MPa. Slip distances
were about 10 mm. The modified torsional Kolsky bar is shown in Figure 2.8. The normal load was applied through the incident bar with a hydraulic actuator. A hydraulic pulley was used to store torque which was released to provide a torsional pulse. The addition of a frictional clamp was used to hold the torque and it allowed for the release of a pulse wave with a sharp front. Strain gauges on the incident tube were used to measure the torsional strains from both the incident waves and the reflected waves from the specimen. The measured shear strains and applied axial stress were used to calculate the frictional stress, slip distance, and slip speed.

The materials tested consisted of either 6061-T6 Al or 7075-T6 Al thin-walled tubes sliding against a disk of 1018 steel or Carpenter Hampden tool-steel (CH). The steel disks were used for the rigid support shown in Figure 2.8. Rajagopalan and Prakash studied both the effects of surface roughness and sliding velocity for the material pairs. Steady state coefficients were achieved immediately after sliding was initiated. Rajagopalan and Prakash found that an increase in surface roughness of both sliding surfaces resulted in an increase in the coefficient of friction for both sets of materials. They also found that the surface roughness of the harder material controlled the friction stress. Variations in the surface roughness of only the softer material had little effect on the coefficient of friction.

The effect of sliding velocity was tested at slip speeds from 3-6 m/s for the CH/7075-T6 Al tribopair. Rajagopalan and Prakash found little variation in the coefficient of friction at different velocities. A coefficient of friction value of about 0.22 was found at all velocities. Scanning electron microscopy was used to examine the sliding surfaces of the materials. Parallel grooves aligned in the sliding direction were
observed on the 7075-T6 Al surfaces. Such grooves are indicative of sliding wear, and result from the harder material plowing through the softer material. Significantly less wear was found on the steel surfaces, which is to be expected, as it is has the higher hardness of the tribopair. Rajagopalan and Prakash did find craters in the steel surfaces and stated that they were the result of pulling out of large inclusions during sliding. If this were the case one would expect some of the inclusions to be embedded in the softer material. However, there was no compositional characterization, such as X-ray Energy Dispersive Spectroscopy (EDS), to determine if this were the case. There was also no mention of whether material was transferred from the softer material, the Al alloys, to the harder materials as is commonly seen in the sliding of unlubricated metals [42, 61, 72]. Characterization of subsurface microstructures was not performed, although it would have shed some light onto the extent of deformation.

Espinosa et al. used a similar torsional Kolsky bar to study dynamic friction of several materials pairs including both SAE 4340 steel and Ti-6Al-4V sliding against a WC/Co cermet [73]. The applied normal pressure values for the two material pairs were about 28 MPa and 40 MPA, respectively. A special C-clamp was used to hold and quickly release the stored torque. Tests were performed at sliding velocities of around 2-4 m/s and yielded contact times of about 200 μs. The coefficient of friction for SAE 4340 steel sliding against WC/Co was found to decrease from about 0.3 to 0.2 as sliding velocity increased from 2-4 m/s. The coefficient of friction of Ti-6Al-4V sliding against WC/Co was found to decrease from about 0.34 to 0.23 as sliding velocity was increased from 1.8-4 m/s.
The contact areas of the specimen surfaces were characterized by Atomic Force Microscopy (AFM) following testing. The WC/Co cermet was found to exhibit only minor changes in the surface topography, while the steel and titanium alloy specimens showed reductions in surface roughness. Deep scratches were observed on the Ti-6Al-4V surfaces, but not on the SAE 4340 steel surfaces. The surface characterization seems to agree with the measured coefficients of friction for the two material pairs. If one considers that the majority of frictional energy is dissipated as plastic work as mentioned in section 2.2, the materials exhibiting a higher degree of plastic deformation, the Ti-6Al-4V in this case, would be expected to have a higher coefficient of friction, which agrees with the results. No attempt was made to explain the decrease in the coefficient of friction with increasing sliding velocity.

Ogawa modified a torsional Kolsky bar in order to study dynamic friction during impact [74]. A schematic of the device is shown in Figure 2.9. Three of the key features of the apparatus were the striker tube, the input tube, and a rotating output tube. The striker tube was used to strike the input tube which was free to move in the axial direction. This resulted in the input tube traveling toward and striking the output tube which was already rotating at a desired velocity. The input tube velocity was varied to control the normal stress. The resulting contact was composed of an axial stress from the impact, and a shear stress resulting from the frictional forces between the tubes. Normal and shear stresses were measured by strain gauges mounted on the input tube. This apparatus has the distinct advantage of allowing for independent control of impact velocity and sliding velocity which is not possible for other impact friction tests such as the plate-impact pressure-shear technique which will be discussed later.
Ogawa tested brass sliding against brass at sliding velocities up to 5 m/s. Contact times of about 200 µs were obtained. Results showed that the coefficient of friction was nearly constant with a value of about 0.2 at sliding velocities up to 5 m/s. The coefficient of friction was also found to be independent of the normal stress for normal stresses ranging from about 40-100 MPa. Post-test optical examination of the surfaces was reported to revealed light scratches. Neither the size of the scratches nor the amount of the surface that was covered by scratches was mentioned. There is also no mention of whether or not the scratches were distributed uniformly on the surfaces which would indicate a good uniform contact. Further characterization of the surfaces with SEM, or the subsurface regions with SEM or TEM was not conducted.

Rajagopalan et al. and Okada et al. have used a plate-impact pressure-shear technique to study dynamic friction at high sliding velocities and normal pressures and short contact times [75, 76]. A schematic of the apparatus is shown in Figure 2.10. It uses a gas gun to propel a flyer plate towards a stationary parallel target plate. The target and flyer plates are tilted relative to the direction of travel of the flyer plate resulting in combined longitudinal and transverse forces. The two plates constitute the tribopair. The longitudinal and transverse velocities of the flyer plate are measured with a laser interferometry technique and are used to calculate the normal and shear stresses during the tests. Contact times for the tests reported in [75] and [76] are on the order of a few microseconds.

Rajagopalan et al. tested Ti6Al4V flyer plates against Carpenter Hampden tool-steel target plates [76]. The tests were performed at impact velocities near 60 m/s, normal pressures of 1-2 GPa, and with varying surface roughnesses of the flyer and target
plates. The authors found an increase in the coefficient of friction with surface roughness which agrees with work by Rajagopalan and Prakash on a modified torsional Kolsky bar, as previously discussed [1]. The reported coefficients of friction varied from about 0.14 for a low surface roughness pair to 0.6 for a material pair with higher surface roughnesses. As with much of the high velocity friction work there was no mention of surface or subsurface characterization and no connection between microstructural behavior of the materials and the observed coefficients of friction. Calculations of the expected temperature rise of the specimens during contact predicted higher temperatures for the higher surface roughness pairs.

Rajagopalan et al. concluded that the increased surface roughness and temperature were responsible for an increase in the coefficient of friction, but there was no explanation as to why this might be. Furthermore, higher temperatures are commonly considered to cause thermal softening of the materials and a decrease in the coefficient so it seems unlikely that an increase in temperature is responsible for the increased coefficient of friction [4, 5].

Okada et al. tested 7075-T6 Al flyer plates sliding against CH tool-steel target plates at impact velocities of 115 and 217 m/s, which corresponded to normal loads of 1-2 GPa, in order to study friction at near-melt and fully-melt conditions [76]. The lower velocity test yielded a sliding velocity that varied from about 30-50 m/s. The corresponding coefficient of friction fluctuated from 0.14 to 0.2 during different stages of the contact. The coefficient of friction for the higher velocity case quickly rose to about 0.09 followed by a decrease to 0.045 after about 1.5 µs of contact.
Characterization of the lower velocity specimens revealed grooves running parallel to the sliding direction on the surfaces. Grains sheared in the direction of sliding were observed below the sliding surface as shown in Figure 2.11. These observations are consistent with the findings of other researchers for the sliding of ductile metals at longer sliding times [47, 48]. Characterization of the high velocity specimens showed substantial deformation of the 7075-T6 Al surfaces including wear tracks and cracks that appear to propagate from the surfaces into the specimens. Additionally, molten aluminum was reported to be smeared along the wear track. EDS results of the two materials’ surfaces showed transfer of material in both directions.

One of the primary drawbacks of the plate-impact pressure-shear technique is an inability to independently vary the normal load and sliding velocity. In order to decrease the normal load the impact velocity must be reduced which in turn reduces the sliding velocity. It is possible to vary the sliding velocity at a fixed impact velocity by altering the skew angle, \( \theta \), shown in Figure 2.10, but any such changes result in different loading geometries and conditions which may not be comparable.

Winter et al. developed an experimental technique termed FN6 which utilized explosive charges to reach sliding velocities of 50-200 m/s with contact pressures of 5-15 GPa [77]. The apparatus consists of two cylinders, each having a face cut at 45° to the cylinder axis, as shown in Figure 2.12. The two cylinders are placed in contact within an aluminum sheath, and the upper cylinder, the slider, is driven against the lower cylinder, the anvil, with the use of an explosive charge placed above the slider. Although this technique can provide high sliding velocities, high normal loads, and short contact times, it does not provide a way to measure the sliding velocity or normal and shear stresses,
and thus it is incapable of measuring the coefficient of friction. The loading geometry is essentially the same as the plate-impact pressure-shear technique and as such it results in an unavoidable coupling of the normal pressure and sliding velocity.

Winter et al. tested an extruded aluminum alloy slider against a stainless steel slider. Grains in the aluminum were initially elongated parallel to the cylinder axis as shown in Figure 2.13a. Post-test characterization of these grains showed bending of the grains and boundaries in the direction of sliding, as shown in Figure 2.13b. Kim estimated the shear strains from the bent grains in the aluminum alloy and also characterized the subsurface microstructures with focused ion beam techniques and transmission electron microscopy [78]. For a sliding velocity of about 180 m/s (18 mm charge) shear strains were measured to be as high as 3000% near the surface. Grain refinement was observed immediately below the surface with grain sizes ranging from 50 nm to hundreds of nanometers. Similar results were seen for a 12 mm charge. Additionally, EDS detected transfer of aluminum to the steel surface.

Lim et al. compiled data from a number of researchers on steel sliding against steel over a wide range of sliding velocities (including high velocities) and contact conditions [79]. A plot of these data, as seen in Figure 2.14, shows significant scatter at velocities below 1 m/s. The coefficient of friction at the lower velocities was considered to be controlled by plasticity. The variation in the data at low velocities was considered to be the result of various surface properties and materials properties of the samples used by the different researchers. Above 1 m/s the coefficient of friction decreases with increasing sliding velocity for all of the data shown. It was proposed that frictional heating of the surfaces at higher velocities led to modifications of the surfaces which
resulted in a lower coefficient of friction. In particular, at higher velocities, friction was assumed to be controlled by the sliding of a layer of oxide or molten metal.

Nonequilibrium molecular dynamics simulations of sliding Cu/Cu interfaces were performed by Hammerberg et al. at sliding velocities ranging from meters per second to thousands of meters per second [3, 80]. The results suggest that the friction coefficient initially increases with increasing sliding velocity and later decreases after going through a maximum near 1500 m/s as can be seen in Figure 2.15 [3]. The sliding velocity in this plot was scaled by the transverse sound speed in copper. The authors scaled the friction force “so that the maxima are identical at the same scaled velocity” [3]. Similar behavior, including the location of the peak friction force, was found under various loads. In addition, the formation of nanoscale grains near the sliding interface and mechanical mixing of atoms across the interface were observed [80]. The increase in the coefficient of friction has been explained elsewhere by Hammerberg et al [81]. The decrease in the friction force at high velocities was proposed to be the result of structural transformations including localized plastic deformation. One important topic that was not discussed was the effect of temperature on the friction force. Experimental work on selected metals at much lower velocities has shown melting of surface material and a subsequent decrease in the coefficient of friction [4, 5, 76].

More recent work by Hammerberg et al. focused on nonequilibrium molecular dynamics simulations of the sliding of Al(111)/Al(110) surfaces from 50 m/s to 3 km/s [82]. Figure 2.16a shows a trend in the frictional force versus sliding velocity that is very similar to that shown in Figure 2.15 for Cu/Cu. In addition to calculating the friction force, the temperature at the interface was also calculated and is shown in Figure 2.16b
for various reservoir temperatures. The critical velocity, which was defined as the velocity at which the maximum friction force occurred, was found to decrease as the reservoir temperature was increased and occurred very close to the calculated melting point of 1130 °K. This result seems to suggest that the decrease in the friction force above the critical velocity may be related to thermal softening and/or melting of the material at the sliding interface.

Karthikeyan et al. used molecular dynamics to study the effect of sliding velocity on the coefficient of friction for both crystalline and amorphous materials and found a trend similar to that of Hammerberg for the crystalline case [83]. Karthikeyan attributed the decrease in the coefficient of friction to thermal softening of the material as a result of frictional heating. In addition, vorticity was found near the sliding interface and was proposed as a possible mechanism for nanostructure formation and mechanical mixing. However, at this point no experimental work has been performed that can verify vorticity as the mechanism behind nanostructure formation and mechanical mixing.

2.5 Nanocrystalline materials

Nanocrystalline materials are characterized as having grain sizes of less than 100 nm [84]. Nanocrystalline materials have shown the potential to exhibit superior mechanical properties to their coarse grained counterparts such as higher hardness and higher fracture toughness. Some authors have reported improved wear resistance of nanocrystalline materials [85]. Due to the possible benefits of nanocrystalline materials a large volume of work has been conducted on the development of nanocrystalline materials and the understanding of their properties.
Several techniques have been used to produce nanocrystalline metals and they can be broken down into the categories of solid state processing, chemical synthesis, thermal spray processing, powder consolidation methods, and electrodeposition methods [86]. The solid state processing methods include, but are not limited to, high energy ball milling, surface mechanical attrition (SMAT), equi-channel angular processing (ECAP), and various severe plastic deformation (SPD) techniques. In addition to these processing techniques it is widely known that the sliding friction of metals commonly results in the formation of a nanocrystalline subsurface region adjacent to the sliding surface [45, 56].

2.5.1 Yield strength of nanocrystalline metals

Nanocrystalline metals have been shown to have improved strength over their coarse grained counterparts [87]. The improved strength of nanocrystalline metals is based on the strengthening mechanism of grain refinement which is commonly used for strengthening materials. The strengthening is typically described by the Hall-Petch relationship, in which decreasing grain size results in an increase in strength governed by the following equation where, \( \sigma_y \) is the yield strength, \( d \) is the grain diameter, and \( K_d \) and \( \sigma_0 \) are constants for a given material.

\[
\sigma_y = \sigma_0 + K_d d^{-1/2}
\]

Dao et al. reviewed and compiled experimental data from a number of researchers which showed significant strengthening of copper at nanocrystalline grain sizes [87]. A plot of these data is shown in Figure 2.17. The smallest grain size of 10 nm corresponds to a yield stress of 1 GPa which is considerably higher than the yield stress for coarse grained copper of 50 MPa. An interesting aspect of Figure 2.17 is that several
of the data points exceed the strength predicted by the Hall-Petch relationship which is shown with the solid line. Dao et al. have proposed that this deviation from the Hall-Petch behavior may be the result of dislocation structures remaining from the severe plastic deformation processing techniques utilized in the experiments.

There is a large body of work in the literature reporting increased strength of a variety of nanocrystalline metals, a few of which will be briefly discussed here. Wei et al. studied the strength of nanocrystalline tungsten [88]. The W tested in the paper consisted of elongated grains with a width of about 100 nm. Based on TEM micrographs presented in their work the grains appear to be a few hundred nanometers in length. They performed both quasi-static and dynamic compression tests and found strengths of 3.0 and 4.0 GPa, respectively. These values were higher than those determined for ultrafine grained W with a 500 nm grain size which had reported flow stresses under quasi-static and dynamic compression of 2 and 3 GPa, respectively [89]. Coarse grained W was reported to have a flow stresses of 1 and 1.2 GPa under quasi-static and dynamic compression, respectively [90].

Nanocrystalline nickel has also been shown to exhibit improved strength. Li et al. performed tensile tests on nickel with a grain size of 44 nm and found a yield stress of 446 MPa [90]. Later work by Gu et al. reported a yield stress of 1200 MPa for nickel with a grain size of 40 nm [91]. These yield stresses are both considerably higher than the yield stress reported for coarse grained nickel.

There have also been reports of an inverse Hall-Petch effect at very small grain sizes where the strength begins to decrease with additional grain refinement [87, 92]. However, it is unclear whether this behavior does indeed occur. According to Dao et al.
early reports of the inverse Hall-Petch behavior for copper were due to flaws in the samples [87]. There are also arguments that at some critical grain size deformation is controlled by the grain boundaries. Molecular dynamics simulations by Van Swygenhoven et al. seem to agree with this line of thinking [93]. The simulations allowed for the study of the deformation mechanism for copper and nickel with grain sizes ranging from 5-12 nm during uniaxial loading. The authors found that the grain boundaries accommodated the deformation for the minimum grain sizes as opposed to dislocation motion which is a fundamental mechanism in the Hall-Petch relationship. Essentially, a change in the deformation mechanism seems to explain deviations from the Hall-Petch relationships for very small grain sizes. A large amount of additional work, both experimentally and computationally, has been performed on this subject, but lies outside the scope of this review.

2.5.2 Strain rate sensitivity of nanocrystalline metals

The strain rate sensitivity for nanocrystalline metals has been found to differ from that of their coarse grained counterparts [87]. Strain rate sensitivity, m, is defined as

\[ m = \frac{\partial \ln \tau}{\partial \ln \dot{\gamma}} \]

where \( \tau \) is the shear stress and \( \dot{\gamma} \) is the shear strain rate. Strain rate sensitivity ranges from zero for materials that are not strain rate sensitive to unity for amorphous materials [94]. Strain rate sensitivity is important for metals because it has a strong effect on the rate controlling deformation mechanisms. For example, Coble creep is considered to take place at \( m=1.0 \), and grain boundary sliding is considered to occur at \( m=0.5 \) [95]. High strain rate sensitivity has also been correlated with superplastic behavior and resistance to
localization of deformation. Another quantity that is commonly measured in conjunction with strain rate sensitivity is the activation volume ($v^*$) given by:

$$v^* = k_B T \left( \frac{\partial \ln \dot{\gamma}}{\partial \tau} \right)_T$$

where $k_B$ is Boltzmann’s constant and $T$ is temperature.

Dao et al. reviewed and summarized the work from several authors on the strain rate sensitivity of copper [87]. The general trend in the data was an increasing value of strain rate sensitivity with a decreasing grain size as shown in Figure 2.18. Values as high as 0.06 have been reported for nanocrystalline copper, which is much higher than reported values around 0.006 for coarse grained copper.

Wang et al. studied the effect of temperature on the strain rate sensitivity of nanocrystalline Ni and provided a comparison to coarse grained Ni [96]. They tested Ni with an average grain size of 30 nm at temperatures ranging from 77-373 K. The strain rate sensitivity reported for the nanocrystalline Ni was 3- to 6-times higher than that of coarse grained Ni over the entire temperature range tested. Furthermore, the strain rate sensitivity was found to increase significantly with temperature. For example, at 273 K the strain rate sensitivity of the nanocrystalline Ni was 0.019 compared to a coarse grained value of 0.0045. The strain rate sensitivities of the ultrafine and coarse grained samples at 363 K were found to be 0.034 and 0.0055, respectively.

May et al. performed similar work on ultrafine grained aluminum [97]. They determined the strain rate sensitivity of coarse grained and ultrafine grained 99.5% Al from 25-250 °C. The results reported follow a similar trend to those of Wang et al. in [96]. At 25 °C the strain rate sensitivity increased more than 3-fold from 0.004 for the
coarse grained Al to 0.014 for the ultrafine grained Al. At 250 °C a 10-fold increase in strain rate sensitivity was measured between the coarse (m=0.025) and ultrafine (m=0.25) grained Al. This work did not report the grain size of either the coarse or ultrafine grained Al that was used making it difficult to compare the results with others’ work.

This trend of increasing strain rate sensitivity with decreasing grain size holds for the cases of fcc metals that have been reported in the literature. However, quite the opposite effect has been reported for bcc metals. Wei et al. compared the strain rate sensitivity and activation volume of ultrafine grained copper with ultrafine grained iron and tantalaum, both bcc metals [95]. They used strain rate jump tests to determine the strain rate sensitivity for Cu with a 200 nm average grain size, Fe with average grain sizes of 200 and 300 nm, and Ta with average grain sizes of 150 and 250 nm. Their results for copper are part of the data reviewed by Dao et al. in [87] and will not be further discussed here. Wei et al. found a decrease in strain rate sensitivity with decreasing grain size for both Fe and Ta. The strain rate sensitivity of Fe with a 200 nm grain size was found to be 0.007 which is much lower than the coarse grained value of 0.045. Similar values were measured for Ta with a strain rate sensitivity of 0.007 for a 150 nm grain size compared to 0.046 for coarse grained Ta. Wei et al. has also reported similar behavior for vanadium [98]. The strain rate sensitivity was reported to decrease from 0.05 for coarse grained V to 0.014 for a grain size of 100 nm.

An explanation of the trends in the strain rate sensitivity for fcc and bcc metals was provided by Meyers et al. [99]. He related the increase in strain rate sensitivity to a change in the mechanism controlling the rate of deformation and the activation volume, $\nu^*$, which is defined in the previous equation. The activation volume measured for
nanocrystalline metals has been shown to be much smaller than that of coarse grained materials. According to Meyers et al. the activation volume observed for nanocrystalline materials can be attributed to thermally activated dislocation annihilation at grain boundaries.

The explanation for the decrease in strain rate sensitivity for nanocrystalline bcc metals is less clear. Meyers et al. suggest that bcc metals are not expected to undergo changes to the rate-controlling mechanism of deformation because small activation volumes are already present for coarse grained bcc metals [99]. This may explain why bcc metals don’t show increased strain rate sensitivity such as is seen with fcc metals, but it doesn’t explain why the strain rate sensitivity decreases with grain size.

### 2.6 Focused ion beam contrast mechanisms

In addition to being a powerful tool for TEM sample preparation the FIB can also be used for its imaging capabilities. The FIB may be equipped with a range of detectors including a secondary electron detector (SED) such as is used in SEMs. The advantage of using the FIB for imaging comes from its improved contrast over conventional SEMs when using the ion beam to produce secondary electrons which are known as ion-induced secondary electrons (ISE) [100, 101]. The observed contrast from ISEs is a result of the interaction between the ions and the surface being imaged. When ions impact a surface, electrons, ions, and electromagnetic radiation are emitted and can be detected [102].

The contrast resulting from ISEs is attributed to ion channeling. If an ion strikes a grain that is oriented such that the ion can channel more easily into the grain many of the secondary electrons that are produced are unable to escape from the material and reach the detector. Conversely, ions that channel very little produce secondary electrons much
closer to the surface, and these secondary electrons have a much better chance of escaping from the material and reaching the detector. When imaging polycrystalline materials, grains that are oriented such that channeling is prominent will appear darker, due to a lower secondary electron yield, than those that minimize channeling. The contrast produced by the channeling effect is much stronger than that from electron excited secondary electrons. An example of channeling contrast is given in Figure 2.19 which shows nickel foam that has been cross-sectioned and imaged with a FIB [102].

In addition to channeling effects, the secondary electron yield has been found to be a function of the atomic number for metals. Sakai et al. and Suzuki et al. compared the contrast of secondary electrons produced in SEM with those produced by a Ga+ ion beam for four different metals [103, 104]. They reported that the relationship between contrast and atomic number of metals was the opposite of that produced in SEM. That is, the lower atomic number metals appear bright in comparison to higher atomic number metals as shown in Figure 2.20 [104]. However, this conclusion does not agree with work by the author where a nanocrystalline layer of copper on a steel substrate was imaged in the FIB with ISE’s. Indeed, the author has found the opposite effect as is shown in Chapter 4.

Kudo et al. measured the secondary electron yield for 30 different metals under ultrahigh vacuum [105]. The secondary electrons produced by an electron beam and an argon ion beam were measured and compared. Additional comparisons were made between the secondary yields and the electron work functions of the different metals. The results from their work are shown in Figure 2.21. From this plot it can be seen that the ISE yield does not simply decrease with increasing atomic number as reported in [103] and [104], but instead shows a periodic behavior. The dependence of the ion-
induced secondary electron yield on the work function was found to be the opposite of that of secondary electron yield produced by an electron beam.

The results from Kudo et al. show very similar ISE yields for copper and iron, with copper having a slightly higher ISE yield. However, the author’s work has shown a significant difference between the relative ISE yield for copper and iron. The effect of grain size and defects such as dislocations were not discussed in [103-105] and may play some part in explaining the differences between the literature and current work.
Figure 2.1. Plot of the coefficient of friction vs. normal load for copper sliding against copper in air [18].
Figure 2.2 Plot showing the decrease in the coefficient of friction for steel sliding against copper in vacuum at high sliding speeds. The curves represent different initial sliding velocities [4].
Figure 2.3. Schematic showing subsurface zones found below wear surfaces [46].
Figure 2.4. TEM micrograph of a copper sample showing a nanocrystalline region, elongated subgrains, and a bent grain boundary produced by sliding against 440C stainless steel [47]. The arrow denotes the sliding direction. The dark layer below the arrow is the nanocrystalline layer.
Figure 2.5. Plot of the rotation of cells about the y-axis (transverse axis) vs. depth after sliding for (a) 1 cycle and (b) 100 cycles. The dashed line was fit to an exponential function [65].
Figure 2.6. TEM image of high purity copper after sliding against steel [45]. The hole around the scale marker is a result of the thinning process for TEM preparation. The scale marker corresponds to 2 µm.
Figure 2.7. Schematic of the high-speed tribometer developed by Philippon et al. [71]
Figure 2.8. Schematic of the modified torsional Kolsky bar used by Rajagopalan and Prakash [1].
Figure 2.9. Schematic of the modified torsional Kolsky bar used by Ogawa to study impact with friction [74].
Figure 2.10. Schematic of the pressure-shear plate-impact apparatus used by Rajagopalan et al. and Okada et al. [75].
Figure 2.11 Optical micrograph showing grain boundaries sheared in the direction of sliding in a pressure-shear plate-impact experiment [76].
Figure 2.12. Schematic of the FN6 apparatus used by Winter et al. to perform high velocity, explosively driven friction experiments [77].
Figure 2.13. (a) Optical micrograph and schematic showing the orientation of grain boundaries used as markers in the FN6 apparatus and (b) micrographs showing grain boundaries bent in the direction of sliding resulting from a test in the FN6 apparatus [77].
Figure 2.14. Plot of the coefficient of friction vs. sliding velocity for steel on steel contact under various contact conditions as compiled by Lim et al. [79].
Figure 2.15. Plot of the scaled friction force vs. scaled sliding velocity for self-mated copper calculated from nonequilibrium molecular dynamics simulations by Hammerberg et al. [3].
Figure 2.16 Plots of (a) the friction stress vs. sliding velocity at various reservoir temperatures, and (b) the temperature at the sliding interface vs. sliding velocity at various reservoir temperatures [82].
Figure 2.17. Plots of the hardness and tensile yield strength for copper with nanocrystalline grain sizes [87].
Figure 2.18. Plot of the strain rate sensitivity, $m$, vs. grain size showing a substantial increase in strain rate sensitivity for nanocrystalline grain sizes [87].
Figure 2.19. Ion-induced secondary electron image of polycrystalline nickel foam exhibiting ion channeling contrast [102].
Figure 2.20. Scanning electron microscope images and scanning ion images of various metals deposited on a Si substrate showing the opposite contrast dependence of the two methods [104].
Figure 2.21. Plots of (a) electron work function, (b) secondary electron yield produced by an electron beam, and (c) secondary electron yield produced by Ar$^+$ ions for metals covering a wide range of atomic numbers [105].
CHAPTER 3

EXPERIMENTAL DETAILS

3.1 Test equipment

3.1.1 Pin on disk tribometer

A new pin on disk tribometer was designed and built for this project. It was used for the low- and mid-range sliding velocity friction tests and it is shown in Figure 3.1. Disks were mounted on a vertical rotating shaft that was connected to a motor. Rotational velocity of the disk is varied with a motor controller and measured using a tachometer. The tribometer is capable of achieving sliding velocities from 0.02 to 1.5 m/s. Pins were mounted in a pin holder and secured with a set screw on the end of a lever arm. Load was applied through the lever arm with the use of a pulley system and a suspended weight with a maximum allowable load of 3 N. An electromagnet was used to load and unload the pin. The electromagnet was powered by a D.C. power supply and controlled by a timer to allow for precise loading times as short as 0.1 s. The system was housed inside a vacuum chamber allowing for control of the atmosphere. Vacuum is achieved through the use of a mechanical roughing pump and a turbomolecular pump and can reach pressures as low as 1x10^-6 torr (1.33x10^-4 Pa). A liquid nitrogen cold trap was used in the vacuum system to prevent backstreaming of the pump oil into the chamber.
Strain gauges with a resolution of better than one gram were used to measure the tangential and normal loads. The normal load was measured with a half bridge strain gauge configuration, and the tangential load was measured with a full bridge configuration. The coefficient of friction, $\mu$, was calculated by dividing the tangential load, $F_T$, by the normal load, $F_N$.

$$\mu = \frac{F_T}{F_N}$$

A high-speed data acquisition system was used to obtain dynamic friction data. Labview was used to control data acquisition and was capable of acquiring up to 400 points per second.

3.1.2 Rotating barrel gas gun

A rotating barrel gas gun was used to conduct short contact time impact with sliding tests and is shown in Figure 3.2a. The gun consists of a barrel mounted on bearings and driven by a motor. An annular projectile sits inside the barrel and spins with it. The back of the barrel is attached to a solenoid valve which is in turn connected to a plenum. The plenum is pressurized with nitrogen. When the solenoid valve is opened the pressure drives the projectile out of the barrel where it impacts an annular target. The projectile is both translating and rotating when it strikes the target. The contact time for the gas gun tests is less than 100 $\mu$s and depends on the length of the projectile and target rods. Upon impact elastic waves travel through the target and the projectile. When the elastic waves reach the far ends of the target and projectile they are reflected back as release waves. Contact is terminated once the first release wave returns
to the impact interface. This allows for control of the contact time by varying the length of the projectile and target rods.

The RBGG is capable of reaching axial velocities as high as 12 m/s and rotational velocities as high as 5.5 m/s. The rotational velocity was determined by measuring the rotational rate of the barrel with a tachometer. A high-speed digital camera was used to measure the axial velocity of the projectile. A laser and photodiode were placed on opposite sides of the barrel. Upon exiting the barrel, the projectile interrupts the laser, which in turn triggers the camera to take two pictures at a preset time interval. The axial velocity was calculated by measuring the distance the projectile traveled between the two images and dividing this distance by the time between the images.

The projectile was slip fit into a polyethylene sleeve which slides into the barrel. The target rod was mounted on a series of optical stages to aid in precise alignment with the projectile. The stages provided translation of the target rod in the y- and z- directions where the x-direction is parallel to the axis of the barrel. In addition to translation, the target rod could be tilted in the y- and z- directions. High-speed strain gauges were mounted on the target rod to measure the axial and torsional elastic waves produced from the impact and sliding, and they were connected in a Wheatstone bridge configuration. Two axial gauges were mounted 180° apart, and two torsional gauges were mounted 180° from each other and 90° from the axial gauges as shown in Figure 3.2.b. The gauges were all mounted at a distance of 38.1 mm from the impact surface. The strain gauge signals passed through a Kyowa strain amplifier before being collected by Labview at a
rate of 20 MHz. The friction coefficient is determined by dividing the measured torsional stress by the axial stress.

The RBGG was designed and built at Los Alamos National Laboratory (LANL). Initial tests were done there, but project reassignments at LANL stopped that testing program, so the equipment was shipped to OSU, where some of the tests reported in this dissertation were performed.

3.2 Sample preparation

Oxygen free high conductivity (OFHC) copper was selected as the disk material for the pin on disk tests. The copper disks were prepared first by cutting 6 mm thick disks from a 25.4 mm diameter rod of OFHC copper with a purity of 99.99%. The surfaces of the disks were then cut with a flycutting machining process to minimize subsurface deformation from the machining process. The disks were then mechanically polished with 400, 600, 800, and 1200 grit silicon carbide polishing papers using water as a coolant. The disks were then annealed in a vacuum furnace at 275 °C for one hour. The disks were allowed to furnace cool before the chamber was vented with dry argon. After annealing the grain size was about 100 µm with a small fraction of annealing twins. A secondary electron image of the disk surface is given in Figure 3.3. Finally the disks were chemically polished to remove an additional 50 µm of surface material. The chemical polishing solution was 55:20:25 by volume percent phosphoric acid: nitric acid: acetic acid. The disks were submerged in the solution for two minutes at 65-70 °C. Energy dispersive X-ray spectroscopy (EDS) of the disk surfaces, after all surface preparation steps, detected only copper.
The pins were machined from a 6.4 mm diameter rod of 440C stainless steel. The pin geometry was spherical with a 6.4 mm diameter. Machining marks were removed by polishing sequentially with 9, 3, and 1µm diamond paste. A rotating tool with a half-sphere geometry was used to apply the diamond paste while maintaining the geometry of the pin during polishing.

OFHC copper was selected as the projectile material for the rotating barrel gas gun, and Nitronic 40 stainless steel was selected as the target material. The targets and projectiles for the RBGG were machined from tubes with a 25.4 mm outer diameter and a 3.2 mm wall thickness. The projectiles were 7.62 cm in length, and the targets were initially 20.3 cm in length. Several of the projectiles were shorter because they were cut and re-used. The surface preparation of the copper projectiles followed the same procedure as the copper disks used in the pin on disk system. The Nitronic 40 stainless steel targets were flycut followed by polishing sequentially with 400, 600, 800, and 1200 grit SiC polishing papers to remove machining marks. After the polishing steps were completed strain gauges were mounted on the target rods in the positions described above.

3.3 Test procedures and conditions

The pin on disk tests were run in vacuum at a pressure of 5x10^-6 torr (6.7x10^-4 Pa) in the temperature range from 18-27 °C. The load was fixed at 1.5 N for all tests. Both sliding velocity and sliding time were varied between tests. Sliding velocity ranged from 0.05 to 1.5 m/s, and sliding time varied from 0.1 to 10^5 s. Prior to testing all samples were ultrasonically cleaned in methanol and dried with compressed air. The pin and disk were brought into contact with the disk rotating at the desired velocity so that the initial frictional transients could be measured. Data were collected at 300 points per
second for short term tests (less than ten minutes) and 10 points per second for long term tests.

The RBGG tests were performed in air with relative humidity ranging from 30–60% and temperatures from 18–22 °C. Tests were performed at axial velocities of 9.5 and 12 m/s which correspond to 40 and 60 psi plenum pressures. The rotational velocity was varied from 1-5.5 m/s. The target and projectile rods were ultrasonically cleaned with methanol and dried with compressed air before each test. The projectiles were then loaded into the barrel, and the target rod was secured in the target holder. The target rod was mounted such that the front flange of the projectile sleeve would stay within the barrel at impact. Once the projectile was loaded into the barrel, and the target mounted in the target holder, the pair was aligned.

Alignment was performed by first bringing the target and projectile into contact gently. Next a small LED flashlight was used to shine a light at the intersection of the target and projectile. A piece of paper was held on the opposite side of the samples from the light source to detect any light passing through the intersection. The stages were then adjusted until no light passed through the interface. The projectiles were then rotated by 90° to double-check the alignment. The alignment procedure was repeated if necessary.

Once alignment was finished the projectile was pushed completely inside the barrel so that the end of the projectile was 15 cm from the open end of the barrel. The barrel was brought up to the desired rotational velocity before firing the projectile, and the rotation was stopped immediately after impact by turning off the motor.
3.4 Characterization

After tests were performed, the structure and elemental composition of the surface and subsurface regions of the worn samples were characterized using a wide range of instruments and techniques including optical microscopy, scanning electron microscopy (SEM) with energy dispersive X-ray spectroscopy (EDS), transmission electron microscopy with EDS, and a focused ion beam (FIB). SEM with EDS was performed on a Philips XL-30 ESEM at an accelerating voltage of 15 kV. TEM was performed using a Philips CM200T operated at 200 kV. The FIB used was a Philips DB235 with a liquid gallium ion source operated at 30 kV for both imaging and milling.

SEM with EDS was used to characterize the wear track morphology and composition of the worn surfaces. The FIB was used to produce cross-sections from the wear tracks for analysis of subsurface regions. Ion-induced secondary electron (ISE) images, also known as ion channeling contrast images, were used to examine the grain size of subsurface regions. In addition to the imaging capabilities, the FIB was also used to prepare site specific TEM membranes. TEM membranes were plucked using an ex-situ micromanipulator with a fine-tipped glass needle and placed on gold grids that were coated with a 20-30 nm thick carbon film. TEM with EDS was used to characterize structural and compositional changes in the subsurface regions.
Figure 3.1. Schematic of the pin on disk tribometer.
Figure 3.2. Schematics of (a) the rotating barrel gas gun and (b) the strain gauge placement on the RBGG target rods.
Figure 3.3. Secondary electron image of the OFHC copper disks prior to testing and the corresponding EDS spectrum showing only copper.
CHAPTER 4

RESULTS AND DISCUSSION

4.1 Effect of sliding velocity for self-mated copper

4.1.1 Pin on disk tests

The effect of sliding velocity on the frictional behavior of self-mated copper was studied using both the pin on disk system and the rotating barrel gas gun. The pin on disk system was used to test OHFC copper disks sliding against copper pins in an air atmosphere. The tests were performed at room temperature which varied from 19-30 °C and relative humidity ranging from 30-60%. The load and sliding distance were fixed for all tests at 100 g and 30 m, respectively. The sliding velocity was varied from 0.05-0.5 m/s. The disks and pins were brought into contact with the disks already rotating at the desired sliding velocity.

4.1.1.1 Friction data

The coefficient of friction is plotted versus sliding distance for tests at six different sliding velocities in Figure 4.1. A steady state coefficient of friction was quickly reached at all sliding velocities with the transient periods occurring within the first 1.5 m of sliding. The steady state coefficient of friction was found to vary from a
minimum of 0.26 to a maximum of 0.42. No discernable trend was found between the sliding velocity and the steady state coefficient of friction.

4.1.1.2 Disk characterization

The disk surfaces were characterized with scanning electron microscopy (SEM) with X-ray energy dispersive spectroscopy (EDS). SEM revealed discontinuous wear track on the disk surfaces at all sliding velocities. Figures 4.2a and b show representative wear tracks for the disks after sliding at 0.2 m/s and 0.5 m/s, respectively. Some regions show grooves indicative of sliding wear, while other regions do not appear to have undergone contact. There was an increase in wear track width with increasing velocity. The width increased from around 250 µm for 0.05 m/s to 650 µm at 0.5 m/s. EDS was used to obtain compositional information both on and off the wear tracks. EDS results revealed a similar small amount of oxygen both on and off the wear tracks. An EDS spectrum is shown in Figure 4.2c for a region on the wear track of the disk tested at 0.2 m/s. Similar spectra were obtained at regions both on and off the wear track for disks tested at all sliding velocities. Quantification of the amount of oxygen was performed with the EDS software and yielded values ranging from about 2-5 atomic percent of oxygen both on and off the wear tracks for all of the disks.

The subsurface regions of the disks were characterized with ion channeling contrast imaging with the focused ion beam (FIB) and with transmission electron microscopy (TEM). Two transverse TEM foils were prepared with the FIB from the 0.05 m/s disk. The locations of these two sections are shown on the SEM image in Figure 4.3a. Figures 4.3b and 4.3c are ion channeling contrast images of these two sections and
show a refined grain size near the surfaces. The dark layer near the top of each image is a protective platinum layer that is deposited prior to FIB milling.

Additional TEM analysis of the transverse sections was used to obtain accurate grain size information. Figure 4.4 is a TEM bright field image taken just below the surface. The dark layer on the left edge of the image is the protective platinum layer from the FIB. A layer of nanocrystalline grains was observed just below the surface with grain sizes as small as 50 nm. The material below the nanocrystalline layer shows significant grain size refinement as well. Submicron sized grains were seen below the nanocrystalline region with grain sizes ranging from about 150-800 nm.

Transverse TEM foils were also produced with the FIB from the 0.5 m/s disk. A TEM bright field image from one section is shown in Figure 4.5. A fine grained region is observed adjacent to the sliding surface with larger grains further below the surface. In addition to grain refinement, bands of deformation can be seen running parallel to the surface. These bands run horizontally across the image. Higher magnification bright field and dark field images of the near surface region are shown in Figure 4.6a and 4.6b and more clearly show the grain sizes in this region. A few grains were observed in the refined near-surface region with grain sizes less than 100 nm, but a majority of the grains were a few hundred nanometers in diameter. Additional TEM analysis of disks tested at other sliding velocities yielded similar structures.

The overall tribological behavior of OFHC copper in this series of tests was largely unaffected by sliding velocity. Neither the coefficient of friction nor microstructural deformation changed significantly with changes in sliding velocity. It is
concluded that under the test conditions the mechanisms controlling the frictional behavior are essentially unchanged.

4.1.2 Rotating barrel gas gun tests

The rotating barrel gas gun (RBGG) apparatus was used to extend the sliding velocity for self-mated OFHC copper tests. The tests were all performed in air with relative humidity ranging from 20-60% and temperatures ranging from 18-24 °C. The impact velocity was fixed at 12 m/s which corresponded to an axial stress of about 180 MPa. The sliding velocity was varied from 0.5-5.2 m/s. Data were acquired at a rate of 20 MHz. Initial tests were performed at Los Alamos National Laboratory and covered sliding velocities from 1.7 m/s to 5.2 m/s. The RBGG was later sent to OSU where tests at sliding velocities of 0.58 m/s and 1.0 m/s were performed.

4.1.2.1 Friction data

Friction data from the highest sliding velocity test of 5.2 m/s are shown in Figures 4.7a and 4.7b. Figure 4.7a is a plot of the average axial and shear stresses as a function of time. The loading time occurs during the first peak on the plot. The remaining peaks and valleys in the data are the result of reflected waves from the opposite ends of the target rod and holder. The contact time was about 35 µs as measured from the time at which the axial stress was at a maximum. The average axial stress during contact was 173 MPa, and the average shear stress was 38 MPa. Figure 4.7b is a plot of the average shear stress and the coefficient of friction versus sliding time. The average coefficient of friction during contact was 0.22. The coefficient was found to decrease slightly with decreasing sliding velocity to values near 0.19 for sliding velocities of 1.7 m/s and 3.5 m/s. Similar contact times were observed for all of the tests.
4.1.2.2 Surface and subsurface characterization

Characterization of the target and projectile surfaces was performed with SEM and EDS. Figure 4.8a shows the surface of the projectile after sliding at 5.2 m/s. Wear scars were also observed on the projectiles tested at lower sliding velocities, but were fewer in number and appeared to be less well defined as can be seen in Figure 4.8b for a sliding velocity of 1.7 m/s. EDS analysis of all of the projectile surfaces showed primarily copper with a small percentage of oxygen (3-5% at.).

Subsurface characterization was performed with ion channeling contrast imaging and with TEM of foils prepared with the FIB. A TEM bright field image of a longitudinal section from the 5.2 m/s sliding velocity test is shown in Figure 4.9. The section was prepared from one of the well-defined wear scars located near the center of Figure 4.8a. Nanocrystalline grains were observed adjacent to the sliding surface with grain sizes as small as 30 nm. Below the nanocrystalline region deformed microstructures were found that were aligned parallel to the sliding direction. Subsurface characterization of wear scars from lower sliding velocity tests revealed grain refinement, but to a much lesser extent. TEM bright field and dark field images of a transverse section are shown Figures 4.10a and 4.10b and reveal the development of fine grained material near the surface of a projectile after sliding at 1.7 m/s. Some of the grains in this region have diameters of less than 200 nm. Bands of deformed material are observed running nearly parallel to the surface below the fine grained material.

Although differences were found in the deformation behavior at different sliding velocities, the differences did not seem to have a pronounced effect on friction coefficient data. A slight decrease in the coefficient from tests performed at 5.2 m/s to tests
performed at 3.5 m/s seems to correlate with a decrease in the extent of deformation of both the surface and subsurface regions. However, tests at lower velocities did not show noticeable changes in the coefficient of friction or in the observed deformation.

4.1.3 Summary of the effect of sliding velocity for self-mated copper

A plot of the average coefficients of friction from the pin on disk system and the RBGG is shown in Figure 4.11. The coefficient is essentially unaffected by sliding velocity for the pin on disk data and for the RBGG data, although there is an obvious difference between the data sets for two apparatuses. The source of the discontinuity in the data between the two apparatuses may be due to the large difference in contact times. The coefficient of friction values for the pin on disk tests represent average values at steady state sliding conditions. The values given for the RBGG on the other hand are from much earlier sliding times which very likely correspond to a part of the transient period.

4.2 The effect of sliding velocity for copper sliding against steel

4.2.1 Pin on disk tests

The effect of sliding velocity on the tribological behavior of copper sliding against steel was studied with the pin on disk system using OFHC copper disks and 440C stainless steel pins. The tests were all performed in vacuum at a pressure of 5x10⁻⁶ torr (6.7x10⁻⁴ Pa). The tests were performed at temperatures from 19-30 °C. The load and sliding distance were fixed at 150 g and 30 m, respectively. Tests were performed at sliding velocities ranging from 0.05 to 1.5 m/s. The disks and pins were brought into contact with the disks rotating at the desired sliding velocity.
4.2.1.1 Friction data

A plot of the coefficient of friction traces for the full range of sliding velocities is shown in Figure 4.12. The data in Figure 4.12 were smoothed using a 30-point average to reduce noise in the data. A steady state coefficient of friction was achieved at every sliding velocity, and transient periods occurred within the first 5 m of sliding at all velocities. The coefficient of friction was found to increase with increasing sliding velocity from 0.42 at 0.05 m/s to 0.75 at 1.5 m/s.

4.2.1.2 Surface and subsurface characterization

Characterization of surface and subsurface deformation was initially focused on the disks as they were the softer of the two materials and were expected to undergo most of the deformation. The disk surfaces were characterized with SEM and EDS. Subsurface regions were characterized with ion channeling contrast imaging in the FIB, and by TEM analysis of foils produced with the FIB. Transverse sections from several positions on the wear tracks were produced with the FIB.

The wear tracks produced at all sliding velocities showed evidence of plastic flow in the form of grooves parallel to the sliding direction. The appearance of the wear scars was uniform across the width of the wear tracks for sliding velocities up to 0.5 m/s. Figure 4.13 shows the wear track for the 0.25 m/s test which is representative of the wear tracks produced at sliding velocities up to 0.5 m/s. The wear tracks produced by sliding at 1.0 and 1.5 m/s showed grooves similar to those from the lower velocity tests, but also had regions consisting of much finer grooves along the wear track edges. These regions can be seen along the edges of the wear track in Figure 4.14 for the disk tested at 1.5 m/s. Additionally, the wear track produced from sliding at 1.5 m/s developed a somewhat
periodic build-up and drop-off of the wear track height which can also be seen in Figure 4.14. This behavior was reflected in the data as an increase in scatter of the data points. This can be seen in Figures 4.15a and 4.15b where the unprocessed raw data from the 1.5 m/s tests shows significantly more scatter than the data from the 0.05 m/s test. An increased vibration of the loading arm was also noticeable during the 1.5 m/s test.

The width of the wear tracks on the disks was found to increase with sliding velocity. The average wear track width for the 0.05 m/s test was 380 µm whereas the average wear track width for the 1.5 m/s test was over 700 µm. EDS analysis of the wear tracks revealed only copper at all sliding velocities. No transfer of components from the pin to the disk was detected. An EDS spectrum from the 0.05 m/s wear track is shown in Figure 4.16 and is representative of the spectra obtained from all of the wear tracks.

Subsurface characterization of the disks from the 0.05 m/s and 0.25 m/s tests yielded similar results. For each of these sliding velocities nanocrystalline grains with grain sizes as small as 30 nm were observed adjacent to the sliding surface. The grain size was found to increase gradually with depth below the surface as shown in the ion channeling contrast image in Figure 4.17a and the TEM bright field image of the same region in Figure 4.17b from the disk tested at 0.25 m/s.

The disks tested at sliding velocities of 0.5 m/s and above showed the same gradual increase in grain size in some of the regions studied. However, this behavior was not observed at all of the locations of the wear tracks that were studied. In some cases a thin nanocrystalline region was observed with a sharp transition to larger grains with grain sizes close to 1 µm. Such a region is shown in the ion channeling contrast image in Figure 4.18 for the 0.5 m/s disk. It is important to note that the fine grained region below
the nanocrystalline material shows significant grain size refinement over the initial grain size of 100 µm. This suggests that this region did indeed undergo deformation during sliding. The appearance of twins in this region, most likely annealing twins, suggests that annealing took place either during or after the test.

In addition to regions with gradual or sharp transitions in grain size, some regions were also found with larger grains extending to the surface. Such a region can be seen near the center of Figure 4.19 which is an ion channeling contrast image of a cross-section from the disk tested at 1.5 m/s. The grains near the center of this section have grain sizes of about 5-10 µm as well as twins, again suggesting that recrystallization is responsible for the observed microstructure. Dynamic recrystallization in copper during sliding has been reported by Dautzenberg et al. and others [44, 106]. Similarly, Degtyarev et al. have reported dynamic recrystallization of copper conditions of high-pressure torsion [107].

The microstructural characterization of the disk alone was insufficient to explain the trend of increasing coefficient of friction with sliding velocity which led to characterization of the pins. SEM and EDS were used to study the pin surfaces, while FIB and TEM were used to study the subsurface regions. Secondary electron images of the pin surfaces tested at all sliding velocities showed wear scars parallel to the sliding direction as seen in Figure 4.20a for the pin tested at 0.05 m/s. In addition to wear scars, apparent transfer material was observed on the pin surfaces at sliding velocities above 0.05 m/s. EDS was used to confirm the presence of copper on the pin surfaces. The amount of transferred copper appeared to increase with sliding velocity as shown in Figures 4.20a, 4.20b, and 4.20c.
SEM back scattered electron (BSE) images were used in conjunction with EDS elemental maps to determine the extent to which the contact areas were covered by copper. Due to atomic number contrast in BSE imaging the transferred copper (higher atomic number) appears brighter than the steel (lower atomic number) of the pin when imaging back-scattered electrons [108, 109]. This aids in the differentiation between the transferred copper and the steel pin. Figures 4.21a and 4.21b and 4.22a and 4.22b show BSE images and EDS elemental maps from pins tested at four different sliding velocities. The EDS maps are color coded by element. The red maps correspond to iron, and the green maps correspond to copper.

From Figure 4.21a it can be seen that copper transferred to the pin after sliding at 0.05 m/s is quite sparse. An increase in the amount of transfer is seen in Figure 4.21b for the 0.25 m/s test, but the contact area is still predominantly free of copper. Once the sliding velocity reaches 1.0 m/s a much greater amount of copper is detected on the contact area as shown in Figure 4.22a. The copper is distributed over most of the contact area, but does not form a continuous layer. At the highest sliding velocity of 1.5 m/s the copper is distributed over most of the contact area (Figure 4.22b). The individual transfer patches are the largest at this velocity and the transfer layer is more uniform.

Subsurface characterization of the transfer material was performed to obtain a detailed understanding of the thickness and microstructure of the material. A bright field TEM image of a transverse cross-section from the pin tested at 0.05 m/s is shown in Figure 4.23. Only two small regions of transferred copper were detected with EDS, and they are circled in Figure 4.23. This is in good agreement with the analysis of the pin surface. The thickness of these regions was less than 200 nm.
A longitudinal section from the 1.0 m/s pin is shown in Figure 4.24a and 4.24b with an ion channeling contrast image and a TEM bright field image, respectively. A strong contrast is seen in the ion channeling contrast image between the transferred copper and the steel pin giving a very clear indication of the boundary between the two materials. This contrast agrees with work by Kudo et al. in which the ion-induced secondary electron yield is dependent on the electron work function of metals [105]. The transfer layer varied in thickness, and it exceeded 2µm in some locations. TEM analysis of this cross-section revealed a nanocrystalline grain size in the transferred copper. The grain sizes were as small as 30 nm which was quite similar to the grain size in the nanocrystalline tribolayer regions of the corresponding disk. A SAD pattern from the transferred copper is shown in Figure 4.24b. This pattern is similar to what might be expected from a deformed single crystal (or large grain). However, given the history of the near surface material, it is also consistent with generation of a pronounced texture during sliding.

The overall trends that can be recognized from the friction data and characterization of the transfer material are that the coefficient of friction increases with sliding velocity and that the amount of copper transferred to the contact area of the pin increases with sliding velocity. These two trends can be related by using the commonly reported observation that self-mated metals typically exhibit a higher coefficient of friction than dissimilar metals under unlubricated sliding [13, 16]. Bhushan has listed a range of coefficients of friction that have been reported for self-mated copper of 0.8-1.2 and for copper sliding against mild steel of 0.6-0.7 [16]. At the lowest sliding velocity of 0.05 m/s the contact was found to be almost completely copper against steel which would
be expected to exhibit a relatively low coefficient of friction. The pins from the 0.25 m/s and 0.5 m/s tests showed an increase in the amount of transferred copper which led to a combination of copper-copper and copper-steel contact which would be expected to exhibit a higher coefficient of friction than the almost entirely copper-steel contact observed at 0.05 m/s. As the velocity increased to 1.0 m/s and 1.5 m/s the contact is shifted closer to a case of pure copper-copper contact which would be expected to show the highest coefficient of friction.

This does not, however, explain why the amount of transferred copper increases with sliding velocity. There are two possible explanations for the increase in transferred copper with sliding velocity. The first is related to strain rate effects. Higher strain rates at the higher sliding velocities could result in strain rate hardening of the near surface material. Extensive hardening of the near surface material could lead to a reduction in ductility and an increase in the likelihood of fracture and transfer. However, this seems unlikely as copper has a rather low strain rate sensitivity, m, even if one considers the increase reported for nanocrystalline copper [87]. Furthermore, no evidence of fracture was observed in any of the samples.

The more likely explanation is related to thermal effects. It is quite possible that thermal softening of the near surface material at higher sliding velocities increases the ease of material transfer. Significant temperature rises are common during the unlubricated sliding of metals and are affected by sliding velocity [48, 76]. Indeed, many of the models for calculating temperature at sliding interfaces assume a positive correlation between temperature and sliding velocity [15, 16, 110]. An increase in material transfer due to thermal softening is also supported by the microstructural
observations of annealed grains at higher sliding velocities. Annealed grains were first observed at a sliding velocity of 0.5 m/s which corresponds to the sliding velocity where significant transfer was first detected.

4.2.2 Rotating barrel gas gun tests

The rotating barrel gas gun was used to extend the sliding velocity of the copper/stainless steel tests above the range of the pin on disk system. OFHC copper projectile rods and Nitronic 40 stainless steel target rods were used for the RBGG tests. The tests were all performed at an impact velocity of 12 m/s. The sliding velocity was varied from 1.2-5.5 m/s. The tests were all performed in air at a relative humidity of 30-60% and temperatures of 18-24 °C. This series of tests was performed at OSU. Surface characterization was performed with SEM and EDS. Subsurface characterization was performed with ion channeling contrast imaging and TEM. TEM foils were prepared with the FIB.

4.2.2.1 RBGG Cu/SS friction data

Analysis of the data from this series of tests revealed a discrepancy in the axial and torsional stresses. The axial stresses varied from 46-60 MPa, but were much lower than values of 180 MPa obtained for self-mated copper tested at the same impact velocity. This discrepancy and additional difficulties with the RBGG will be discussed in Section 4.2.2.3. The coefficient of friction values for the OFHC Cu sliding against Nitronic 40 stainless steel RBGG tests were quite similar to those obtained for self-mated OFHC copper. At a sliding velocity of 5.5 m/s the average value of the coefficient of friction was 0.20. Plots of the axial and shear stresses and the coefficient of friction from this test are provided in Figures 4.25a and 4.25b. From these plots the contact time can be seen to
be about 70 µs. Similar contact times were observed for all tests. The coefficient of friction decreased slightly to 0.16 at a sliding velocity of 3.9 m/s. The coefficient of friction dropped by nearly 50% at lower sliding velocities giving values of 0.09 and 0.08 at sliding velocities of 2.5 m/s and 1.2 m/s, respectively.

4.2.2.2 Characterization

Wear scars were observed on the surfaces of the OFHC Cu projectiles tested at all sliding velocities. The wear scars were predominantly located near the inner radius, although some wear scars were observed at larger radii. The wear scars were not continuous around the entire circumferences suggesting that the samples were not perfectly aligned. The width of the wear scars was found to vary from less than 100 µm to greater than 500 µm at different locations on the surfaces. Wear scars located near the inner radii are shown in Figures 4.26a and 4.26b for sliding velocities of 2.5 m/s and 3.9 m/s, respectively. Transfer of components from the target rods was not detected by EDS analysis of the worn regions.

Characterization of the Nitronic 40 target surfaces revealed a few isolated wear scars. EDS analysis of the wear scars revealed the presence of copper on some of the scars. A BSE image of one such wear scar is shown in Figure 4.27a which is from the target tested at a sliding velocity of 5.5 m/s. Figure 4.27b is a copper EDS map of this region clearly showing the presence of copper along the wear scar.

Subsurface characterization was performed on transverse cross-sections taken from regions containing wear scars. Ion channeling contrast images of sections from projectiles tested at all sliding velocities revealed a thin region with apparent grain refinement adjacent to the sliding surface such as the section shown in Figure 4.28 for a
sliding velocity of 2.5 m/s. TEM analysis of the cross-sections was used to obtain more
detailed microstructural information.

A bright field image is shown in Figure 4.29 of a section from a projectile tested
at a sliding velocity of 2.5 m/s. Bands of deformation are observed running semi-parallel
to the sliding surface. Bright field and dark field images in Figures 4.30a and 4.31b,
respectively, illustrate the development of fine grained and nanocrystalline material
adjacent to the sliding surface. Some grains in this region are less than 50 nm in diameter
while others are on the order of 200 nanometers in diameter. Due to the small size of the
refined regions and the low number of refined grains it was not possible to obtain SAD
patterns with useful texture information.

A TEM image from the 5.2 m/s sliding velocity test is shown in Figure 4.31. In
addition to fine grained and nanocrystalline material near the surface, irregular
deformation patterns were observed. A dark field image of the region just below the
surface is provided in Figure 4.32a and shows that grain sizes vary from much less than
100 nm to more than 200 nm in the region. The region of refined grains does not extend
continuously across the section, but is more clearly defined than at lower sliding
velocities. The SAD pattern shown in Figure 4.32b is consistent with a structure that is
beginning to form a deformation texture.

The overall trend in the subsurface deformation is an increase in the extent of
grain refinement with an increase in sliding velocity. This result is not altogether
unexpected. The sliding time is nearly identical for all tests, but the sliding distance is
dictated by the sliding velocity. The higher sliding velocities give greater sliding
distances. The sliding distances were calculated from the contact times and sliding
velocities and varied from 84 µm to 385 µm. The higher sliding distances result in a higher input of plastic work into the samples which causes grain refinement to occur to a greater extent.

This result may also help explain the decrease in the coefficient with decreasing sliding velocity. If one looks at friction from an energy standpoint such as that proposed by Rigney and Hirth, where the plastic work was equated to the frictional work, the trend can be explained [24]. This model was developed for steady state sliding conditions, but the same basic principles should apply qualitatively during transient periods. Based on characterization of subsurface regions as discussed above it appears that the amount of plastic deformation, and work, increases with sliding velocity. If the plastic work is equated to the frictional work an increase in the coefficient of friction with an increase in sliding velocity would be expected.

4.2.2.3 RBGG experimental difficulties

It should be mentioned that various problems were encountered in the Cu/SS series of RBGG tests performed at OSU. The largest problem was sticking of the projectile and projectile sleeve inside the barrel either just prior to or upon impact with the target rod. This problem was not encountered in the self-mated copper tests performed at Los Alamos National Laboratory (LANL). Two RBGG tests of self-mated copper at OSU resulted in sticking of the projectile sleeve in the barrel. The self-mated copper tests performed at OSU yielded much lower axial stresses than those performed at LANL and were not used for comparison with the LANL tests.

The sticking of the projectile sleeve caused two noticeable differences in the data. The first was a variation in the measured axial stresses. The tests performed at LANL
and at OSU were all performed using an axial velocity of 12 m/s, as verified by the high speed camera, which should have produced similar axial stresses for all of the tests. However, this was not the case. The tests performed at LANL yielded axial stresses near 180 MPa, whereas the tests at OSU yielded axial stresses of 30-60 MPa. This suggests that the sticking of the projectiles led to a decrease in the impact velocity which was manifested as differences in the axial stress. Velocity measurements using the high speed camera corresponded to a time prior to impact, leaving open the possibility that the projectile decreased in velocity between the time of the second image from the camera and the time of impact. Additional images from the camera up to the time of impact would be necessary to determine if this was the case.

The second discrepancy was related to the contact time of the tests. The contact times for all of the copper/stainless steel RBGG tests were about 60 µs which is substantially longer than the contact time of 35 µs observed for the copper/copper RBGG tests. This is the opposite of what was expected. The stainless steel target rods have a higher elastic stiffness than the copper target rods which should have resulted in a higher velocity of the axial stress wave in the target and a shorter contact time. It is unclear as to why the contact time increased for the stainless steel targets, but it is possible that the sticking of the projectile in the barrel prevented the projectile from “bouncing” off of the target as easily.

The reason behind the sticking of the projectile sleeve in the barrel is not understood at this point. Identical testing procedures were used in tests at LANL and OSU. In the case of self-mated copper tests, identical materials were also used at both locations. The only known differences in test conditions were the temperature of the
laboratory and the relative humidity. Unfortunately these two variables were not recorded for the tests at LANL so comparisons with tests performed at OSU are not possible.

An additional difficulty with the RBGG comes from alignment. In all of the tests the wear tracks were found to extend only partially around the circumference of the projectile surfaces. This is a result of imperfect alignment between the targets and projectiles. Unfortunately, difficulties in alignment are an inherent problem with the flat-on-flat contact geometry of the gas gun and could not be entirely eliminated. The difficulties with obtaining perfect alignment may also affect the data collected from the RBGG. The axial and shear stresses were calculated as average values over the entire target surface. However, observation of incomplete wear scars on all of the surfaces revealed that only a small fraction of the surfaces was actually in contact, and it is only the contact at these regions that should have given rise to the measured strain gauge signals. Due to the method of calculating the stresses averaged over the entire surface it is likely that the calculated stresses are significantly lower than the actual stresses at the contact areas.

4.3 Tribolayer development

Two series of tests were performed to study the development and growth of the nanocrystalline tribolayer produced in copper. These tests were largely driven by results from analytical modeling performed by Karthikeyan et al. [111]. In particular, the results of the modeling suggested that at low sliding velocities the tribolayer would continue to grow into the sample at long times as opposed to high sliding velocities where the tribolayer would localize to the near surface regions. These hypotheses are drawn from a
plot of normalized velocity profiles versus distance from the sliding interface at three different sliding velocities as can be seen in Figure 4.33. The plot corresponds to the sliding of self-mated OFHC copper.

In order to test these hypotheses tests were performed on the pin on disk system at sliding velocities of 0.05 and 1.0 m/s. OFHC copper was used for disks while 440C stainless steel was used for the pins. The load was fixed at 150g. The tests were all performed in vacuum at a pressure of $5 \times 10^{-6}$ torr ($6.7 \times 10^{-4}$ Pa). The temperature of the tests varied from 19-30 °C. Several sliding times were tested at each velocity to allow for characterization of the tribolayer at different sliding times. SEM and EDS were used to characterize wear tracks on the disk surfaces. Characterization of the tribolayer was performed with ion channeling imaging with the FIB and with the TEM. Transverse cross-sections were used for analysis to avoid inconsistencies associated with varying wear track height due to peaks and ridges parallel to the sliding direction.

4.3.1 Low sliding velocity tests

4.3.1.1 Surface characterization

The first series of tests was performed at 0.05 m/s. The minimum and maximum sliding times were 1.0 s and 10 ks. The sliding time of 1.0 s corresponded to less than one revolution of the disk during sliding. Surface characterization was first performed on the wear tracks on the disks with SEM and EDS. The wear track was found to be discontinuous after a sliding time of 1.0 s as is shown in Figure 4.34. After sliding for 10 s the wear track was much more continuous, but still contained isolated regions that did not appear to have undergone contact. Sliding times greater than 10 s all produced continuous wear tracks as can be seen in Figure 4.35 which shows the wear track after
sliding for 10 ks. The width of the wear tracks was found to increase with sliding time from an average width of 160 µm after 1.0 s to 420 µm after 10 ks. EDS analysis of the worn samples revealed only small amounts of oxygen (1-2% at.) both on and off the wear tracks at all sliding times. Components from the pins were not detected on any of the wear tracks suggesting that transfer from the pin to the disk did not occur.

4.3.1.2 Subsurface characterization

The thickness of the nanocrystalline layer was quantified using TEM of foils produced with the FIB. After sliding for 1.0 s bands of deformation are observed running parallel to, and just below the surface as shown in a bright field TEM image in Figure 4.36. The left edge of Figure 4.36 does not show the bands of deformation because it comes from a region of the disk that did not undergo contact. The TEM foil shown in Figure 4.36 was milled from the rectangular region shown in Figure 4.37 where the left edge of the foil corresponded to a region that did not exhibit wear scars. The formation of nanocrystalline grains was observed near the surface and can be seen in a higher magnification image in Figure 4.38, but a continuous, measurable tribolayer was not developed.

After sliding for 10 s a continuous nanocrystalline layer with a uniform thickness was found to have developed as shown in Figures 4.39a and 4.39b. Minimum grain sizes of 20 nm were observed in the tribolayer. The grain structure after sliding for 100 s is shown in bright field and dark field TEM images in Figure 4.40a and 4.40b, respectively. The tribolayer was found to have increased in thickness to an average thickness of 320 nm. Minimum grain sizes of 20 nm were again observed. A region of well-defined submicron grains was observed below the nanocrystalline tribolayer.
Continued growth of a uniformly thick nanocrystalline tribolayer was observed after sliding for 1000 s as seen in Figure 4.41a. The average tribolayer thickness after 1000 s was 950 nm. Selected area diffraction (SAD) patterns of the nanocrystalline layer showed the development of texture. The non-uniform rings shown in Figure 4.41b are consistent with the development of texture. The submicron size grains below the tribolayer were found to contain nearly periodic arrays of closely spaced dislocations as shown in Figure 4.42.

The character of the tribolayer changed dramatically after sliding for 10 ks as shown in Figure 4.43. Figure 4.43 is an ion channeling contrast image of a relatively large cross-section from the wear track. A description of the method by which this section, and other similar sections, was prepared is provided in Appendix A. The dark strip near the top of the section is the protective platinum strip deposited during FIB milling. The variation in grain size with both depth and lateral position is easily discernible in this image. A somewhat periodic variation in the thickness of the tribolayer is quite obvious. A TEM image of a similar region is shown in Figure 4.44a revealing grain sizes as small as 30 nm. SAD patterns of the nanocrystalline regions, such as that shown in Figure 4.44b, were consistent with a structure that is beginning to develop texture. The spotty pattern is an indication that an insufficient number of grains were sampled to confirm the presence of texture. Very similar microstructural features were reported by Rigney et al. for the sliding of OFHC copper against steel in argon at the same sliding velocity, but at much larger loads and longer sliding times [112]. Although the nanocrystalline tribolayer showed large variations in thickness after 10 ks, the average thickness was found to increase from the previous time step.
Orientation imaging microscopy (OIM) was performed on a cross-section similar to that shown from the 10 ks test in Figure 4.43 to look for the development of texture. A step size of 0.1 µm was used for the scan. Figure 4.45 is an orientation map of the cross-section. It is color coded by orientation according to the [001] inverse pole figure key that is also provided. The first regions analyzed were the fine grained regions below and to the sides of the nanocrystalline regions. The [001] inverse pole figures for these two regions are shown in Figures 4.46 and 4.47.

Nanohardness measurements were performed on a transverse section of the wear track on the disk tested for 10 ks to determine the local hardesses of the nanocrystalline and fine grained regions. An ion channeling contrast image of the section is shown in Figure 4.48a. The measurements were performed by Hysitron, Inc. on a Hysitron TriboIndenter®. The measurements were performed using a diamond Berkovich probe in an automated displacement control mode. 55 nm indents were used with a five-second approach, two-second hold, and five-second withdrawal. A grid of indents was used with a 3 µm spacing between indents.

The hardness values are overlaid on the image in Figure 4.48b and are in units of GPa. The hardness values in the nanocrystalline regions showed some variation, but exceeded 3 GPa in most locations. The hardness values of the fine grained region were significantly lower and varied from 1.92 GPa to 2.58 GPa. The measured hardness values are considerably higher than those reported by Dao et al. (Figure 2.17) [87]. The nanocrystalline regions had minimum grain sizes of about 30 nm which, based on the work by Dao et al., would be expected to have hardness values around 2.5 GPa.
Likewise, the fine grained regions would be expected to have hardesses on the order of 1.5 GPa.

The reason behind this discrepancy is unclear. The hardness values compiled by Dao et al. [87] were measured with nanohardness [113] and microhardness [114-116] tests by a number of researchers so it seems unlikely that the testing technique is responsible for the difference. Many of the data points in the work compiled by Dao et al. were from copper specimens with similar purities to that used in the present work so it also seems unlikely that differences in sample purity are causing the discrepancy in hardness values.

The differences in hardness values for the nanocrystalline regions may be the result of a low number of data points. Several more data points would be needed from the nanocrystalline region to determine a reliable average hardness value for comparison with work by other authors. The increased hardness of the fine grained region is consistently higher than was expected. This may be the result of work hardening in the fine grained regions. TEM observations have revealed high numbers of dislocations in the fine grained regions.

4.3.1.3 Summary

To summarize the results from this series of tests, both the tribolayer thickness and wear track width were found to increase with sliding time for all sliding times tested. A plot of these two values versus the log of the sliding time is shown in Figure 4.49. Nanocrystalline grains were observed after as little as 1 s of sliding, and a continuous nanocrystalline layer formed after 10 s. The thickness of the nanocrystalline layer increased while remaining uniform at times up to 1000 s. After sliding for 10 ks the
tribolayer did continue to grow on average, but the thickness was non-uniform and showed variations of as much as 7 µm.

Texture was observed in the nanocrystalline regions, but not in the fine grained regions further below the surface. The absence of texture in the fine grained regions support the occurrence of recrystallization. Furthermore, the presence of high dislocation densities in the fine grained region indicates that the recrystallization occurred dynamically during the tests, and that the grains underwent additional deformation after recrystallization. If this region had recrystallized post-test the grains would be expected to have low dislocation densities.

The continued growth of the tribolayer at all times qualitatively supports the prediction based on the work of Karthikeyan et al. that the tribolayer would continue to grow in thickness indefinitely at low sliding velocities [111]. However, the development of a non-uniform tribolayer thickness after 10 ks is of some concern. It is unknown whether or not the average tribolayer thickness would continue to grow from such a microstructural state. Longer term tests would be necessary to make this determination.

4.3.2 High sliding velocity tests

4.3.2.1 Surface characterization

A second series of tests was performed to study the growth of the tribolayer at a sliding velocity of 1.0 m/s. Sliding times were varied from 0.1-3000s. The minimum sliding time of 0.1 s corresponded to 1.7 revolutions of the disk. EDS analysis of the wear tracks from all tests revealed only copper and a small amount of oxygen (1-2% at.) on the wear tracks. Transfer of components from the pins to the disks was not detected.
Transfer of copper to the pins was observed and was similar in character to the transfer discussed in Section 4.2.1.2.

SEM characterization of the wear track after sliding for 0.1 s revealed two distinct contact types. The first of these is shown in Figure 4.50a which shows three distinct contact areas separated by non-contacted areas. This behavior is a result of the method of applying the normal load. When the pin first comes into contact with the disk a small amount of bouncing occurs, resulting in isolated contact areas. The second type of contact region is shown in Figure 4.50b and is a continuous wear track representative of typical sliding wear of metals. The wear track produced after 1.0 s of sliding is shown in Figure 4.51. The wear track was continuous around the circumference, but was non-uniform in appearance. Two isolated contact areas are seen near the bottom edge of the wear track in Figure 4.51. These are most likely due to bouncing of the pin at early times as they are quite similar in appearance to the “bouncing” regions seen after 0.1 s.

The wear tracks from all tests of 100 s or longer contained ledges at various stages of development as shown in Figures 4.52a-c. Figure 4.52a shows ledges at a relatively early stage after sliding for 10 s. Figure 4.52b shows well-defined ledges after sliding for 100 s. The wear tracks at later times, such as the one shown for a 1000 s test in Figure 4.52c, were found to increase in width in the regions between ledges and decrease in width near the ledges. Similar results were found after sliding for 3000 s with a further increase in the width of the wear track. The formation of the ledges correlated with an increase in vibration of the loading arm and an increase in the scatter of the data.
4.3.2.2 Subsurface characterization

Characterization of the nanocrystalline tribolayer depth and the variation in grain size with depth was performed with the TEM and with ion channeling contrast imaging with the FIB. A consistent nanocrystalline layer was not formed after sliding for 0.1 s. An ion channeling contrast image of one transverse cross-section from the continuous region of the wear track is shown in Figure 4.53. Bright field and dark field TEM images of a similar transverse section in Figures 4.54a and 4.54b show that grain refinement does occur adjacent to the sliding surface with grain sizes in the submicron range. Bands of deformation were observed deeper into the sample and were oriented roughly parallel to the surface. Some of these bands have sharp walls reminiscent of dislocation cells reported by Heilmann et al., while others are only regions of increased dislocation density [56].

An ion channeling contrast image of a transverse cross-section of the wear track produced after sliding for 1 s is shown in Figure 4.55a. Grain refinement was observed across the top edge of the entire section, but a majority of the grains were above the nanocrystalline size scale. The one exception is the upper right hand corner shown at a higher magnification in Figure 4.55b. In addition to the nanocrystalline region twins can be seen in the refined grains below and to the left of the nanocrystalline region.

An additional transverse section was characterized with TEM. A bright field image of the section is shown in Figure 4.56a. A nanocrystalline region is clearly visible near the surface with a slight increase in size with depth. A SAD pattern of the nanocrystalline material in the upper middle area of the section is shown in Figure 4.56b which does not appear to show texture. The predominant feature in this micrograph is the
relatively large grain in the center of the section. In addition to being more than an order of magnitude larger than the surrounding grains, it also contained several twins. The appearance of the twins in grains such as in the central grain in Figure 4.56a is a strong indication that annealing occurred during or after the test.

A continuous nanocrystalline layer was observed after sliding for 10 s. Ion channeling contrast images from two transverse cross-sections of the wear track are shown in Figures 4.57a and 4.57b. In Figure 4.57a the thickness of the nanocrystalline layer is fairly uniform across the section with an average thickness of 1.63 µm. Annealed grains of a few microns in size were observed within the nanocrystalline layer in the upper left hand corner of Figure 4.57a. Figure 4.57b, also from the 10 s test, shows a variation in the thickness of the nanocrystalline layer due to a higher region of the wear track. The nanocrystalline layer was the thickest in the raised region. Recrystallized grains were again observed and have grain sizes of less than 10 µm. One other interesting feature is the appearance of a crack on the right edge of the raised region. This crack is potentially a precursor to material transfer. TEM analysis of a transverse cross-section from this test is shown in Figures 4.58a and 4.58b. Figure 4.58a is a dark field image showing an increase in grain size with depth. Figure 4.58b is a SAD pattern taken from the nanocrystalline region just below the surface which is consistent with the development of texture. One other notable feature was observed in Figures 4.57a and 4.57b which was a region of refined grains below the nanocrystalline region. Unfortunately, the grains in this region were not well defined in the ion channeling contrast images and were at a depth that was not easily accessible to preparation of TEM foils with the FIB.
A continuous nanocrystalline region was produced adjacent to the sliding surface after sliding for 100 s. The nanocrystalline grains can be seen in the top 2-3 µm of the transverse section shown in Figure 4.59. Well defined submicron scale grains were observed below the nanocrystalline material. A clear boundary could not be seen between the nanocrystalline and submicron grains. Indeed, a small number of submicron grains were found within the nanocrystalline regions and vise versa as can be seen in a dark field TEM image in Figure 4.60a. SAD patterns of the grains near the surface showed texture as can be seen in Figure 4.60b. Due to the existence of submicron grains within the nanocrystalline region, the SAD pattern in Figure 4.60b included spots from both the nanocrystalline and fine grained material. Therefore, the SAD pattern shown in Figure 4.60b could be due to either deformation texture or scattering from a limited number of larger grains.

The existence of ledges on the wear track led to significant variations in the thickness of the nanocrystalline layer for sliding times of 1000 s and greater. Shown in Figures 4.61 and 4.62 are transverse cross-sections taken from regions near the bottom and top of ledges, respectively. In Figure 4.61 the nanocrystalline material is isolated to within 3-4 µm of the sliding surface which is considerably different than in Figure 4.62 where the nanocrystalline grains extend to more than 30 µm below the surface. This large variation in nanocrystalline thickness between different locations on the wear track makes it difficult to determine the thickness of the nanocrystalline layer to any degree of accuracy. TEM analysis of a transverse cross-section from one of the low regions on the wear track was performed. A bright field image and corresponding SAD pattern from the section are shown in Figures 4.63a and 4.63b. As in Figure 4.60b, the SAD pattern
shown in Figure 4.63b could be due to either deformation texture or scattering from a limited number of larger grains.

TEM analysis of the samples showed texture in the near surface nanocrystalline material in all cases where it was present, but due to the limited depth of TEM foils similar information could not be obtained from the regions of submicron grains. Orientation imaging microscopy (OIM) was performed on the large cross-section prepared with the FIB shown in Figure 4.61 to determine whether any texture developed in these deeper regions. A color coded map of the region and the corresponding color coded [001] inverse pole figure key are shown in Figure 4.64.

The well-indexed region of submicron sized grains was cropped from the remainder of the data for analysis and is shown in Figure 4.65a. The data from this region is plotted on the [001] inverse pole figure shown in Figure 4.65b. Based on the inverse pole figure there does not appear to be any texture in this region. The orientation of the grains appears to be essentially random. The fact that this region does not show any texture, but the material near the surface does show texture is another indication that annealing did in fact take place.

4.3.2.3 Summary

In summary, a continuous nanocrystalline tribolayer was formed in as little as 10 s of sliding at 1.0 m/s. Shorter sliding times produced nanocrystalline grains only in isolated regions adjacent to the sliding surface. SAD patterns of the near surface nanocrystalline material revealed the development of texture at all sliding times where the nanocrystalline material was generated. OIM analysis of regions of submicron sized grains below the nanocrystalline grains did not reveal texture. The appearance of
submicron grains within the nanocrystalline regions and the results of the OIM analysis of the regions of submicron grains indicate that annealing occurs during sliding and may have a large influence in the final microstructure.

Ledges produced on the wear track surface strongly affected the tribolayer thickness making it difficult to obtain accurate values of the tribolayer thickness. Due to the large variations in the thickness of the nanocrystalline tribolayer it is difficult to prove or disprove the predictions from the work of Karthikeyan et al. [111]. Furthermore, the appearance of dynamically recrystallized submicron sized grains within the nanocrystalline region indicates that the tribolayer is constantly changing and cannot be defined simply as the region having a nanocrystalline grain size.
Figure 4.1. Plot of the coefficients of friction vs. sliding distance for six different sliding velocities from the sliding of self-mated copper in the pin on disk system. The tests were all performed in air with a fixed sliding distance of 30 m (526 revolutions) and normal force of 1.0 N.
Figure 4.2. SEM images of the disk wear tracks produced by copper sliding against copper at (a) 0.2 m/s and (b) 0.5 m/s and (c) a typical EDS spectrum from the wear track of the 0.2 m/s disk. The tests were both run for a sliding distance of 30 m (516 revolutions) with a normal force of 1.0 N. The tests were run in air with relative humidities of 30 % and 40%, respectively.
Figure 4.3. (a) SEM image showing the location of the two transverse TEM foils shown in ion channeling contrast images in (b) and (c). The foils were both milled from the wear track of a copper disk after sliding against a copper pin at 0.05 m/s for 30 m (516 revolutions) with a normal force of 1.0 N. The test was performed in air with a relative humidity of 40%.
Figure 4.4. TEM bright field image of a transverse section from a wear track on a copper disk. The foil location is shown in Figure 4.3 and the test conditions are the same as in the caption for Figure 4.3.
Figure 4.5. TEM bright field image of a transverse section of the wear track on a copper disk after sliding against a copper pin at 0.5 m/s for 30 m (516 revolutions) with a normal force of 1.0 N. The test was performed in air with a relative humidity of 40%.
Figure 4.6. (a) Bright field and (b) dark field TEM images of a transverse section from the wear track on a copper disk sliding against a copper pin at 0.05 m/s for 30 m (516 revolutions) with a normal force of 1.0 N. The test was performed in air with a relative humidity of 40%.
Figure 4.7. Plots of (a) axial and shear stresses and (b) the coefficient of friction and average shear stress vs. time for a RBGG test of self-mated OFHC Cu at a sliding velocity of 5.2 m/s and impact velocity of 12 m/s. The test was performed in air.
Figure 4.8. SEM images of OFHC Cu projectile surfaces after sliding against OFHC Cu targets at (a) 5.2 m/s and (b) 1.7 m/s. Both tests were run in air with an impact velocity of 12 m/s.
Figure 4.9. TEM bright field image of a longitudinal cross-section from a RBGG OFHC Cu projectile tested against an OFHC Cu target at a sliding velocity of 5.2 m/s and impact velocity of 12 m/s in air.
Figure 4.10. (a) Bright field and (b) dark field TEM images of a transverse section from a RBGG OFHC Cu projectile tested against an OFHC Cu target at a sliding velocity of 1.7 m/s and impact velocity of 12 m/s in air.
Figure 4.11. Plot of the coefficient of friction vs. sliding velocity for self-mated copper tested with the RBGG and the pin on disk systems in air.
Figure 4.12. Plot of the coefficient of friction vs. sliding distance for the sliding of OFHC Cu disks against 440C stainless steel pins at different sliding velocities. The tests were all performed in vacuum with a fixed sliding distance of 30 m (516 revolutions) and normal force of 1.5 N.
Figure 4.13. SEM image of the wear track on a copper disk after sliding against a 440C stainless steel pin in vacuum at 0.25 m/s with a fixed sliding distance of 30 m (516 revolutions) and normal force of 1.5 N.
Figure 4.14. SEM image of the wear track on a copper disk after sliding against a 440C stainless steel pin in vacuum at 1.5 m/s with a fixed sliding distance of 30 m (516 revolutions) and normal force of 1.5 N.
Figure 4.15. Plots of the coefficient of friction vs. sliding distance for OFHC Cu sliding against 440C stainless steel in vacuum after sliding against a 440C stainless steel pin in vacuum with a fixed sliding distance of 30 m (516 revolutions) and normal force of 1.5 N at (a) 0.05 m/s and (b) 1.5 m/s. The plots are both raw data that was not smoothed.
Figure 4.16. EDS spectrum from the wear track on an OFHC Cu disk after sliding against a 440C stainless steel pin in vacuum at 0.05 m/s with a fixed sliding distance of 30 m (516 revolutions) and normal force of 1.5 N.
Figure 4.17. Transverse section from the wear track on an OFHC Cu disk after sliding against 440C stainless steel in vacuum at 0.25 m/s with a fixed sliding distance of 30 m (516 revolutions) and normal force of 1.5 N showing increasing grain size with depth in (a) an ion channeling contrast image and (b) a TEM bright field image.
Figure 4.18. Ion channeling contrast image of a transverse section from the wear track on an OFHC Cu disk after sliding against 440C stainless steel at 0.5 m/s in vacuum with a fixed sliding distance of 30 m (516 revolutions) and normal force of 1.5 N.
Figure 4.19. Ion channeling contrast image of a transverse section from the wear track on an OFHC Cu disk after sliding against 440C stainless steel at 1.5 m/s in vacuum with a fixed sliding distance of 30 m (516 revolutions) and normal force of 1.5 N.
Figure 4.20. SEM images of 440C stainless steel pin surfaces after sliding against OFHC Cu disks in vacuum with a fixed sliding distance of 30 m (516 revolutions) and normal force of 1.5 N at sliding velocities of (a) 0.05 m/s, (b) 0.5 m/s, and (c) 1.5 m/s.
Figure 4.21. BSE and EDS maps of 440C stainless steel pin surfaces after sliding against OFHC Cu disks in vacuum with a fixed sliding distance of 30 m (516 revolutions) and normal force of 1.5 N at sliding velocities of (a) 0.05 m/s and (b) 0.25 m/s.
Figure 4.22. BSE and EDS maps of 440C stainless steel pin surfaces after sliding against OFHC Cu disks in vacuum with a fixed sliding distance of 30 m (516 revolutions) and normal force of 1.5 N at sliding velocities of (a) 1.0 m/s and (b) 1.5 m/s.
Figure 4.23. TEM bright field image of a transverse cross-section from a 440C stainless steel pin showing small localized regions of transferred copper as a result of sliding against a copper disk at 0.05 m/s in vacuum with a fixed sliding distance of 30 m (516 revolutions) and normal force of 1.5 N.
Figure 4.24. Longitudinal cross-section from a 440C stainless steel pin after sliding against OFHC Cu at 1.5m/s in vacuum with a fixed sliding distance of 30 m (516 revolutions) and normal force of 1.5 N shown in (a) an ion channeling contrast image and (b) a TEM bright field image with a SAD pattern from the transferred Cu.
Figure 4.25. Plots of (a) axial and shear stresses and (b) coefficient of friction from a RBGG test of an OFHC projectile sliding against a Nitronic 40 steel target with a sliding velocity of 5.5 m/s and impact velocity of 12 m/s. The test was performed in air at a temperature of 24°C and relative humidity of 60%.
Figure 4.26. SEM images of OFHC Cu projectiles after sliding against Nitronic 40 steel in air at (a) a sliding velocity of 2.5 m/s, an impact velocity of 12 m/s, a temperature of 27°C, and 44% relative humidity and (b) a sliding velocity of 3.9 m/s, an impact velocity of 12 m/s, a temperature of 23°C, and 59% relative humidity.
Figure 4.27. (a) BSE image and (b) corresponding copper EDS map showing Cu transferred to a Nitronic 40 target after sliding against an OFHC projectile with a sliding velocity of 5.5 m/s and impact velocity of 12 m/s. The test was performed in air at a temperature of 24°C and relative humidity of 60%.
Figure 4.28. Ion channeling contrast image of a transverse section from an OFHC Cu projectile after sliding against Nitronic 40 steel with a sliding velocity of 2.5 m/s and impact velocity of 12 m/s. The test was performed in air at a temperature of 27°C and relative humidity of 44%.
Figure 4.29. Bright field TEM image of a transverse section from an OFHC Cu projectile after sliding against Nitronic 40 steel under the same conditions described in the caption for Figure 4.28.
Figure 4.30. (a) Bright field and (b) dark field images showing refined grains near the surface of an OFHC Cu projectile after sliding against Nitronic 40 steel under the same conditions described in the caption for Figure 4.28.
Figure 4.31. Bright field TEM image of a transverse section of an OFHC Cu projectile after sliding against a Nitronic 40 steel target with a sliding velocity of 5.5 m/s and impact velocity of 12 m/s. The test was performed in air at a temperature of 24°C and relative humidity of 60%.
Figure 4.32. (a) Dark field TEM image showing grain refinement in a subsurface region of an OFHC Cu projectile after sliding against a Nitronic 40 target under the same conditions as described for Figure 4.31 and (b) a SAD pattern of the region of refined grains.
Figure 4.33. Plot of calculated normalized velocity profiles for self-mated OFHC Cu at different sliding velocities [39]. The unit of meters for the y-axis was the result of using an infinite system size for the model.
Figure 4.34. SEM image of a wear track on an OFHC Cu disk after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 0.05 m/s and normal force of 1.5 N for 1.0 s (<1 revolution).
Figure 4.35. SEM image of a wear track on an OFHC Cu disk after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 0.05 m/s and normal force of 1.5 N for 10 ks (8600 revolutions).
Figure 4.36. TEM bright field image of a transverse cross-section from a wear track on an OFHC Cu disk after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 0.05 m/s and normal force of 1.5 N for 1.0 s (<1 revolution).
Figure 4.37. Ion channeling contrast image showing the location of the TEM foil in Figure 4.36.
Figure 4.38. TEM bright field image of a transverse cross-section from a wear track on an OFHC Cu disk after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 0.05 m/s and normal force of 1.5 N for 1.0 s (<1 revolution).
Figure 4.39. (a) TEM bright field image of a transverse cross-section from a wear track on an OFHC Cu disk after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 0.05 m/s and normal force of 1.5 N for 10 s (8.6 revolutions) and (b) a higher magnification TEM bright field image of the near surface region.
Figure 4.40. TEM (a) bright field and (b) dark field images showing nanostructure formation in a transverse cross-section from an OFHC Cu disk after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 0.05 m/s and normal force of 1.5 N for 100 s (86 revolutions).
Figure 4.41. (a) TEM bright field image showing nanostructure formation in OFHC Cu after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 0.05 m/s and normal force of 1.5 N for 1000 s (860 revolutions) and (b) a SAD pattern from the nanocrystalline region.
Figure 4.42. TEM bright field image showing a dislocation array in a grain in OFHC copper resulting from sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 0.05 m/s and normal force of 1.5 N for 1000 s (860 revolutions).
Figure 4.43. Ion channeling contrast image of a transverse cross-section of an OFHC Cu disk after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 0.05 m/s and normal force of 1.5 N for 10 ks (8600 revolutions).
Figure 4.44. (a) TEM bright field image of a transverse cross-section from a copper wear track after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 0.05 m/s and normal force of 1.5 N for 10 ks (8600 revolutions) and (b) a SAD pattern taken from the nanocrystalline region.
Figure 4.45. OIM map and inverse pole figure key of a transverse cross-section from an OFHC Cu disk after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 0.05 m/s and normal force of 1.5 N for 10 ks (8600 revolutions).
Figure 4.46. Cropped OIM map of the fine-grained region on the left side of Figure 4.45 and the corresponding [001] inverse pole figure.
Figure 4.47. Cropped OIM map of the fine-grained region on the right side of Figure 4.45 and the corresponding [001] inverse pole figure.
Figure 4.48. (a) Ion channeling contrast image of a transverse section from an OFHC Cu wear track after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 0.05 m/s and normal force of 1.5 N for 10 ks (8600 revolutions), and (b) a higher magnification image of the section with overlaid nanohardness values in units of GPa.
Figure 4.49. Plot of the wear track width and tribolayer thickness vs. sliding time for OFHC Cu after sliding against 440C stainless steel pins in vacuum at 0.05 m/s with a normal force of 1.5 N.
Figure 4.50. SEM images from an OFHC Cu disk after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 1.0 m/s and normal force of 1.5 N for 0.1 s (1.7 revolutions) showing (a) a discontinuous region of the wear track and (b) a continuous region of the wear track.
Figure 4.51. SEM image of a wear track on an OFHC Cu disk after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 1.0 m/s and normal force of 1.5 N for 1 s (17 revolutions).
Figure 4.52. SEM images showing the development of ledges on the wear tracks of OFHC Cu after sliding against 440C stainless steel pins in vacuum at 1.0 m/s with a normal force of 1.5 N for (a) 10 s (170 revolutions), (b) 100 s (1700 revolutions), and (c) 1000 s (17,000 revolutions).
Figure 4.53. Ion channeling contrast image of a transverse cross-section from an OFHC Cu disk after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 1.0 m/s and normal force of 1.5 N for 0.1 s (1.7 revolutions).
Figure 4.54. (a) Bright field and (b) dark field TEM images of a transverse section of a wear track on OFHC Cu after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 1.0 m/s and normal force of 1.5 N for 0.1 s (1.7 revolutions).
Figure 4.55. (a) Ion channeling contrast image of a transverse cross-section from an OFHC Cu disk after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 1.0 m/s and normal force of 1.5 N for 1 s (17 revolutions) and (b) a higher magnification ion channeling contrast image.
Figure 4.56. (a) TEM bright field image and (b) SAD pattern from the near surface fine grained region of a transverse section of a wear track on OFHC Cu after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 1.0 m/s and normal force of 1.5 N for 1 s (17 revolutions).
Figure 4.57. Ion channeling contrast images of transverse cross-sections of OFHC Cu after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 1.0 m/s and normal force of 1.5 N for 10 s (170 revolutions).
Figure 4.58. (a) TEM dark field image and (b) SAD pattern from the near surface region of a transverse section of a wear track on OFHC Cu after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 1.0 m/s and normal force of 1.5 N for 10 s (170 revolutions).
Figure 4.59. Ion channeling contrast image of a transverse cross-section from OFHC Cu after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 1.0 m/s and normal force of 1.5 N for 100 s (1700 revolutions).
Figure 4.60. (a) TEM dark field image and (b) SAD pattern from the near surface fine grained region of a transverse section of a wear track on OFHC Cu after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 1.0 m/s and normal force of 1.5 N for 100 s (1700 revolutions).
Figure 4.61. Ion channeling contrast image of a transverse cross-section near the bottom of a ledge on the wear track of an OFHC Cu disk after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 1.0 m/s and normal force of 1.5 N for 1000 s (17,000 revolutions).
Figure 4.62. Ion channeling contrast image of a transverse cross-section near the top of a ledge on the wear track of an OFHC Cu disk after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 1.0 m/s and normal force of 1.5 N for 1000 s (17,000 revolutions).
Figure 4.63. (a) TEM bright field image and (b) SAD pattern from the near surface fine grained region of a transverse section of a wear track on OFHC Cu near the bottom of a ledge after sliding against a 440C stainless steel pin in vacuum with a sliding velocity of 1.0 m/s and normal force of 1.5 N for 1000 s (17,000 revolutions).
Figure 4.64. OIM map of the transverse cross-section shown in Figure 4.60.
Figure 4.65. OIM map of the upper portion of Figure 4.64 and corresponding [001] inverse pole figure.
Chapter 5

SUMMARY AND CONCLUSIONS

5.1 Summary of experimental findings

(1) The coefficient of friction was found to be relatively unaffected by sliding velocity for self-mated OFHC copper pin on disk tests. The coefficient of friction ranged from 0.26 to 0.42 and did not show a trend with respect to sliding velocity. The wear tracks were discontinuous at all sliding velocities with a small increase in width with increasing velocity. The tests produced refined grains adjacent to and just below the sliding surface. Minimum grain sizes of less than 50 nm were produced, but continuous regions of nanocrystalline grains were not. Similar subsurface microstructures were observed at all sliding velocities. The lack of changes in the coefficient of friction and observed deformation indicated that the mechanisms responsible for the frictional behavior were unaffected by sliding velocity under the conditions tested.

(2) The coefficient of friction for self-mated OFHC copper RBGG tests was slightly affected by sliding velocity. The coefficient of friction varied from 0.19 to 0.22 for sliding velocities of 1.7 m/s and 5.2 m/s, respectively. Contact times for all tests were about 35 µs. Wear scars were found on the sample surfaces tested at all sliding velocities and were more pronounced at the highest sliding velocity. Subsurface grain refinement
occurred at all sliding velocities. Refined grains extended further below the surface at higher sliding velocities. The increase in subsurface deformation at the highest sliding velocity was correlated with the increase in the coefficient of friction.

(3) Comparison of the data from the pin on disk and RBGG tests of self-mated copper showed a significant difference in the coefficient of friction between the two apparatuses. This difference is likely due, at least partly, to the difference in sliding times. The coefficients of friction reported for the pin on disk tests were for steady state sliding, but those for the RBGG tests correspond to the early transient period which could not be studied with the pin on disk system.

(4) Pin on disk tests for OFHC copper sliding against 440C stainless steel showed a strong dependence of frictional behavior on the sliding velocity. The coefficient of friction increased from 0.42 to 0.75 at sliding velocities of 0.05 m/s and 1.5 m/s, respectively. Grain refinement occurred in the disks at all sliding velocities with minimum grain sizes of 30 nm. Evidence of dynamic recrystallization was observed at sliding velocities of 0.5 m/s and higher. Transfer of copper to the pins was observed at all sliding velocities and increased with velocity. The increase in transfer was a result of thermal softening of the near surface material and led to predominantly copper on copper contact resulting in a higher coefficient of friction.

(5) An increase in the coefficient of friction with sliding velocity was found for OFHC copper sliding against Nitronic 40 steel in RBGG tests. The coefficient of friction increased from 0.08 to 0.2 at sliding velocities of 1.2 m/s and 5.5 m/s. Wear scars were produced predominantly along the inner radii of the copper projectiles. Grain refinement was present at all sliding velocities. The extent of grain refinement decreased with
decreasing sliding velocity and may be the result of shorter sliding distances at lower sliding velocities. The decrease in the coefficient of friction with decreasing sliding velocity appeared to be caused by the decrease in plastic deformation.

(6) The development of a nanocrystalline tribolayer in OFHC copper sliding against a 440C stainless steel pin started at very early times. Isolated nanocrystalline grains were formed after only one second while a continuous nanocrystalline layer formed in as little as 10 s of sliding. The nanocrystalline layer grew uniformly at times up to 1000 s, but showed large variations in depth at longer times. Fine grained regions with submicron sized grains developed below the nanocrystalline regions. Evidence of texture was observed in the nanocrystalline regions, but not in the fine grained regions. The nanocrystalline regions were found to have much higher hardness values (3.9 GPa) than the fine grained regions (~2.1 GPa).

(7) The growth of the nanocrystalline tribolayer in OFHC copper after sliding against a 440C stainless steel pin was strongly affected by ledges on the wear track, and by thermal effects. Ledges on the wear track developed at early times and resulted in large differences in the thickness of the nanocrystalline region. Dynamic recrystallization within and below the nanocrystalline region made it difficult to measure the thickness of nanocrystalline region. Evidence of texture was observed in the nanocrystalline regions, but not in the fine grained regions.

5.2 Conclusions

- Self-mated copper pin on disk and RBGG tests
  - Changes in the sliding velocity over the range tested were not significant enough to cause changes in the frictional behavior.
The relatively short sliding distances of the RBGG tests were responsible for the low coefficients of friction in comparison to the pin on disk tests. That is, all of the RBGG tests remained in the early part of the transient regime that preceded achievement of steady state conditions in the pin on disk tests.

- Copper sliding against stainless steel pin on disk tests
  - Transfer of copper from the disks to the pins controlled the coefficient of friction.
  - Thermal softening and dynamic recrystallization occurred during the tests.
- Copper sliding against stainless steel RBGG tests
  - An increase in plastic deformation caused an increase in the coefficient of friction with increasing sliding velocity.
- Low velocity tribolayer growth tests
  - The nanocrystalline tribolayer produced in copper grew continuously at sliding times up to 10 ks.
  - Continued growth of the tribolayer qualitatively supports predictions from Karthikeyan’s work.
- High velocity tribolayer growth tests
  - Dynamic recrystallization inhibits the development of a nanocrystalline tribolayer.
  - Dynamic recrystallization inhibits the development of deformation textures in the fine grained regions.
  - The definition of the tribolayer by grain size could not be used alone for measurements of the tribolayer thickness.
5.3 Future work

Before closing, ideas for future work on these topics will be discussed. One of the goals of this work was to obtain a solid understanding of the generation and behavior of the nanocrystalline tribolayer. However, there are some portions of the work that could be expanded upon. The development of texture in the nanocrystalline regions was noted, but an in depth analysis of the type of texture was not performed. High resolution OIM scans capable of resolving the nanocrystalline regions could be used not only to study the overall texture within the nanocrystalline regions, but also to investigate changes in texture with depth below the surface. The development of texture as a function of sliding time and sliding velocity could also be investigated.

The nanohardness data presented in this work showed very high hardness values for the nanocrystalline region. However, the data set consisted of only a small number of points from within the nanocrystalline region. Additional tests should be performed to determine if the hardness values measured here are representative of the nanocrystalline regions as a whole. It would also be of interest to determine whether the high hardness values follow the traditional Hall-Petch behavior.

There are also a couple of points that could be expanded upon for the fine grained regions produced below the nanocrystalline regions. Nearly parallel dislocation arrays were observed in many of the grains in these regions. Determination of the Burgers vector of these dislocations and their relationship to the applied stresses may shed some light on the deformation mechanisms of the fine grained regions.

Although dynamic recrystallization was proposed to have an effect on the fine-grained regions, no experimental technique was available to measure temperature
changes during sliding. The effect of sliding velocity on temperature changes could be studied from a somewhat qualitative standpoint by measuring temperatures near the disk-pin interface with the use of thermocouples. Estimates of temperatures may be obtained by using models of temperature changes in the literature.
APPENDIX

PREPARATION OF CROSS-SECTIONS WITH THE FIB

The preparation of large cross-sections for ion channeling contrast imaging, such as that shown in Figure 2.15 for a wear track on a copper disk, was achieved through the following steps. The disks were first sectioned using a low speed saw with a silicon carbide (SiC) wafering blade. Cuts were made normal to the disk surface and perpendicular to the sliding direction to produce transverse cross-sections of the wear tracks. The freshly cut transverse surfaces were then mechanically polished sequentially with 400, 600, 800, and 1200 grit SiC polishing papers. Next, the samples were mounted on 45° tilt SEM stubs using a colloidal silver paste. The sections were mounted such that the polished surface faced upwards. A SEM image of one such section is shown in Figure A.1. The section was rotated at an angle of 45° from the electron beam so that both the sliding surface and polished surface could be viewed simultaneously.

The remaining steps were performed with the FIB. Once the samples were loaded into the chamber the stage was tilted by 7° to bring the sliding surface perpendicular to the ion beam. A protective platinum layer was then deposited on the wear track over the region of interest as shown in Figure A.2. Next the FIB was used to remove material starting at the edge of the section and working in towards the region of interest. A beam
current of 20,000 pA was used for milling the material furthest from the region of interest. Decreasing beam currents were used as the region of interest was approached. Final milling was performed with a beam current of 1000 pA. The final milling step was positioned such that a portion of the platinum layer remained. This ensured that the region of interest remained protected during milling. A SEM image of a section after milling is shown in Figure A.3.

Once the milling steps were completed the sample was rotated by 180°, and then tilted to 7° such that the cross-section was perpendicular to the ion beam. Imaging with the FIB was then performed with a beam current of 50 pA. The secondary electron detector was used for imaging.
Figure A.1. SEM image showing the locations of the wear track and mechanically polished surface in the FIB preparation of cross-sections for ion channeling contrast imaging. The sample is tilted by 45° from the electron beam.
Figure A.2. FIB image showing the location of a protective platinum layer deposited prior to FIB milling of a transverse cross-section.
Figure A.3. SEM image of a transverse cross-section prepared with the FIB. The sample is tilted by 45° from the electron beam.


