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**Sliding friction and wear of plain carbon steels in air and
vacuum**

Venkatesan, Sreekanth, Ph.D.

The Ohio State University, 1991

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**300 N. Zeeb Rd.
Ann Arbor, MI 48106**

**SLIDING FRICTION AND WEAR OF PLAIN CARBON STEELS
IN AIR AND VACUUM**

DISSERTATION

**Presented in Partial Fulfillment of the Requirements for
the degree Doctor of Philosophy in the Graduate
School of The Ohio State University**

By

Sreekanth Venkatesan, B.Tech., M.S.

*** * * * ***

The Ohio State University

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ACKNOWLEDGEMENTS

I would like to express my gratitude to Prof. David. A. Rigney for his encouragement and advice through the course of this project.

I would like to express my appreciation to Michelle Kaitchuck and Joe Kalen of the Van De Graff Laboratory at The Ohio State University for their help with the radioactive tracer experiments.

I would like to thank Ms. Clare McDonald, Mr. Henk Colijn, Mr. Jay Ellis, Mr. Bud Farrar and Mr. Jack Kocka for their help with the SEM, TEM, machining, photography and metallography during the project.

Finally, I would like to thank the members of the wear group, N.V. Chandrasekhar , Lihua Zhang, Meiji Sato, Tomoharu Akagaki and Nigel Trilk for helpful discussions and assistance with different parts of this project. Special thanks are due to Srinivasan Ganapathi and Masaaki Sawa for their help with the TEM work and the design of the test equipment.

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CHAPTER I

INTRODUCTION

1.1. Background:

The sliding wear behaviour of steels has been the subject of numerous investigations since the early part of this century. The early work revealed the complex nature of the wear behaviour of steels and its dependence on a number of factors such as the load, sliding speed, the carbon content of the steel and the environment. Further work established the fact that the steady state wear mode for steels, depending on the load and sliding speed, is either mild or severe. Mild wear has been associated with low wear rates, smooth worn surfaces and fine, apparently oxidized debris and severe wear with high wear rates, rough worn surfaces and coarse, metallic debris. The role of the environment, in terms of oxidation effects was also recognized to be significant in determining the steady state wear behaviour of steels. Besides the load and speed dependent transitions, steels were found to exhibit a sliding distance-dependent severe-mild wear transition within a certain load range.

1.2. Aim:

The present study was designed to identify and understand the processes involved in the sliding wear of carbon steels. Although past

work has revealed a broad pattern involved in the wear of steels in terms of severe and mild behaviour, much of the work was done with inadequate control of experimental variables. Also, due to the lack of modern analytical instruments such as the scanning electron microscope (SEM) and transmission electron microscope (TEM) information on a fine scale was not be obtained.

Data from existing literature have been used to define the range of variables for the present investigation. The aim of the present investigation has been to determine the roles of deformation, transfer and oxidation during the unlubricated sliding of carbon steels. The work has focussed on the load dependent mild-severe and distance dependent severe-mild wear transitions.

1.3. Approach:

Wear tests have been conducted under a well defined set of variables. To understand the role of the environment better, experiments have been conducted in air as well as in vacuum (0.2×10^{-3} Pa). In addition to collecting wear rate and friction data from the tests, the worn surfaces of the test specimens, the wear debris and wear track cross-sections have been characterized both structurally and chemically using optical microscopy, scanning and transmission electron microscopy, profilometry and microhardness testing. Transfer effects have been investigated by making one of the samples radioactive. The experimental observations have been interpreted in light of existing theories on the wear behaviour of steels.

Chapter 2 is an overview of the literature on the sliding wear behaviour of steels. Chapter 3 describes the experimental arrangement and defines the range of variables used in the present investigation. A brief description of the various techniques used in the analysis of samples is also presented in chapter 3. Chapter 4 contains results of the experiments performed in the present study. Chapter 5 contains a discussion of the results presented in chapter 4. Chapter 6 contains the conclusions.

CHAPTER II

LITERATURE REVIEW

2.1. General:

Wear may be defined as the gradual and unwanted removal of material from surfaces in contact and in relative motion. The resulting material loss is usually in the form of fine particles referred to as wear debris. The present work focusses on the unlubricated wear behavior of steels under conditions of sliding,

Sliding wear depends on a number of variables which can be classified as: 1) material properties of the interacting surfaces, 2) the stress distribution at the sliding interface, 3) the sliding velocity, 4) the nature of the surrounding environment and 5) the total distance of sliding. In a sense, these variables may be considered fundamental variables as other factors either modify one or more of these variables or may be derived from them. For example, the sliding geometry influences the interface stress distribution, whereas the interface temperature is a complex function of all the above mentioned variables.

As wear depends on a number of variables and their complex interactions it should not be a surprise to find the absence of a universally accepted mechanism or a quantitative relation which can account for wear under different operating conditions with different materials. It is generally recognized today that there are many different mechanisms of

wear. Burwell (1) has proposed adhesion , abrasion, corrosion and surface fatigue to be the dominant mechanisms affecting wear. Vingsbo (2) lists more than forty different mechanisms responsible for wear under different conditions. Under a given set of conditions wear may occur with more than one type of mechanism operating simultaneously. In such cases, using a single term or mechanism to describe wear would not be appropriate.

Archard and Hirst (3) have classified wear as mild and severe. Mild wear is characterized by low wear rates and severe wear by high wear rates. Wear rate is conventionally measured as the volume or weight of material removed per unit distance slid. In mild wear the wear rate is of the order of 10^{-6} - 10^{-8} mm³/mm and in severe wear it is of the order of 10^{-4} - 10^{-5} mm³/mm. Alternatively, wear can be described by the dimensionless wear coefficient, k , where k is the proportionality constant between wear rate and load. The wear coefficient k lies is greater than 10^{-6} for severe wear and between 10^{-7} and 10^{-9} for mild wear under unlubricated conditions. The mild and severe wear classification of Archard and Hirst is a good starting point for analysing and classifying wear as these terms do not implicitly indicate particular mechanisms.

2.2. Some early work on the wear of steels :

Rosenberg and Jordan (4) investigated the wear of three steels (0.43, 0.81 and 1.26 wt %C) in the form of disks rotated in line contact. Testing in air yielded low wear rates and the worn surfaces appeared smooth and oxidized. Tests in nitrogen or hydrogen atmospheres yielded different results. Steels hardened and tempered at low temperatures yielded low

wear rates and smooth, oxide covered surfaces, whereas those hardened and tempered at high temperatures yielded high wear rates and rough bright surfaces. It was proposed that an oxide film forms on the steel surfaces in air and prevents severe wear for steels of any hardness, while in nominally oxygen free atmospheres, a thin oxide layer forms and prevents severe wear only when the surface hardness is sufficiently high.

Mailander and Dies (5) while studying the wear of soft steel pins on hard steel chrome disks reported that wear decreased as load increased for intermediate loads, as shown in Fig.1. This decrease was associated with the formation of hard patches on the pin surface. Kauzlerich et al. (6) detected similar behaviour with fully hardened 0.4 %C steel pins rubbed in air on rings of like material, although a significant change in surface hardness was not observed in this case. They postulated that the transient temperatures at the sliding interface induce temper brittleness in the steel and this brittleness allows welds between the points of contact to break at or closer to the junction than they normally would, causing a lower wear rate.

Kehl and Siebel (7) studied high carbon steels and cast irons in the form of hollow cylinders rubbed end on against like cylinders. They detected a variation in the wear rate when the sliding speed was changed (Fig.2). Kragelskii et al.(8) also found a critical speed for a 0.8%C steel below which wear was severe and above it mild. Mild wear was associated with fine oxidized debris and severe wear with coarse metallic debris. Critical sliding speeds were also detected for certain non- ferrous metals. The authors suggested that that the critical sliding speed is associated with frictional heating which caused softening of the points of contact and that this softening localized the damage and reduced the overall wear rate.

Fig. 1. Wear rate plotted against load for soft iron pins on hard steel disks.
(Ref.5).

Fig. 2. Plot showing the influence of speed on the wear rate for a 0.64% C steel.

Load 30 kg. Hardness (V.p.n.): x, 160; o, 178; Δ, 348; +, 445; □, 690. (Ref. 7).

The early work on the wear of steels was carried out in the absence of modern analytical instruments like the SEM and TEM, so detailed chemical and structural characterization of worn surfaces and debris was seldom attempted. Widely differing hypotheses such as surface hardening, surface softening due to frictional heating, temper embrittlement and oxidation were presented to account for the experimental observations with very little concrete experimental evidence. This early work however revealed that the sliding wear behaviour of steels is a sensitive and complex function of the applied load, the sliding speed, the hardness of the steel and the environment and that, depending on these variables, the wear of steels can be either mild or severe.

2.3. Archard's and Hirst's studies of the mild wear of steels:

Archard and Hirst studied the wear behaviour of a wide variety of material combinations with loads and speeds ranging from 50gms-10kg and 2-660 cm/sec, respectively (3). On the basis of these experiments they concluded that wear of materials is either mild or severe. Furthermore, they concluded that wear rate is independent of the apparent area of contact and proportional to the applied load. These conclusions could only be drawn by assuming that the wearing surfaces remained unchanged during the wear process. It is now known that sliding causes changes in the surface conditions via the formation of a highly deformed layer adjacent to the sliding interface (9). Moreover, changes in load, speed and sliding distance will affect the interface temperature and hence the kinetics of chemical interactions with the environment. Thus sliding surfaces change in the course of sliding.

Archard and Hirst (10) followed their study of the general wear behaviour of materials with a detailed analysis of the mild wear process for a hardened tool steel pin rubbing on a ring of like material. By making the pin radioactive they were able to monitor transfer of material from the pin to the ring. Fig.3 represents the quantity of transfer as a function of time for different loads used in the experiments. It can be seen from the graph that the amount of steady state transfer and the time needed to form it is independent of the load. The ease of removal of transfer material was indicated by a loss of activity on the ring surface. The ring was subjected to various mechanical and chemical treatments and the results indicated that most of the transferred material was loosely adherent to the ring and was either partially or wholly oxidized. The equilibrium amount of transferred material was found to be independent of load and the thickness was estimated to be 5-10 nanometers. The transferred material was visible as grey lumps under the optical microscope. However, experiments also suggested that these lumps are not present at light loads.

Archard and Hirst concluded that during the early stages of rubbing most of the pin wear occurs via transfer to the ring. They reasoned that, during the early stages of rubbing, because of the lack of conformity of the surfaces, the load is borne by a relatively small number of contacts, leading to the formation of welded junctions and subsequent transfer. As sliding continues and the surfaces wear to achieve conformity, the load becomes supported over a larger contact area. Transfer and removal of metallic particles continue but on a smaller size scale. When the surfaces reach a high degree of conformity and approach their equilibrium condition transfer ceases to be the rate determining step.

Fig. 3. Plot showing transfer as a function of sliding cycles for various loads.

A, 18 kg; B, 10 kg; C, 6.7 kg; D, 3.5 kg. (Ref. 10).

Archard and Hirst used a replica technique to observe worn surfaces in an electron microscope. Based on the observation that the heights of the loose wear particles are markedly greater than the asperity heights on the underlying surfaces, they postulated that steady state wear was due to abrasion by these loose particles.

2.4. Kerridge and Lancaster's analysis:

Kerridge and Lancaster (11) performed experiments in which 60/40 brass pins (hardness 150 VHN) were slid against hard rings made of tool steel or Stellite (hardness 750-850 VHN). Under the conditions of their experiments the wear was severe and the debris produced was metallic brass. Kerridge and Lancaster suggested that wear occurs via a multi-stage process involving transfer of the soft brass to the hard ring surface as the first step, followed by the production of loose wear debris from the transferred layer. They found both the equilibrium wear rate and transfer to be proportional to the load. They also found an increase in the size of transfer fragments with load. This size increase was associated with an area increase as the thickness was found to be load independent. The mean thickness of the transfer fragments in Lancaster and Kerridge's analysis was about 10^{-3} cms. At a given load the wear particles were found to be considerably larger than the transfer fragments both in terms of area and thickness. Therefore they suggested that incipient wear particles grow on the surface of the ring by successive transfer and are finally detached when the the bond between them and the underlying material weakens due to the alternating compressive and tensile stresses to which they are subjected during the sliding cycles.

In a different series of experiments Lancaster (12) detected a transition from mild to severe wear for a 60/40 brass pin sliding against a hardened steel ring. The main conclusions of this work were that mild wear occurs at light loads, low speeds and low ambient temperatures when there is sufficient time to establish a protective film by oxidation, and at heavy loads, high speeds and high temperatures due to an increase in the rate of oxidation. As the variations of the rates of mild wear with load and speed were found to be complex it was acknowledged that there was probably no unique mechanism for mild wear. Lancaster concluded that the mild wear of brass occurs due to 'intermittent metal to metal contact and abrasion by oxide particles'.

Kerridge has made a detailed study of the role of metal transfer in the wear of an annealed tool steel pin (270 VHN) sliding against a hardened tool steel ring (860 VHN) (13). By making the pin radioactive, Kerridge was able to demonstrate the build up of an steady state transfer layer on the hard ring. Like Archard and Hirst (10), Kerridge determined that the steady state amount of transfer was independent of load but the time taken to form this layer was inversely proportional to the load. In contrast to Archard's and Hirst's results, Kerridge found no back transfer from the pin to the ring. X-ray powder photographs showed that the wear debris consisted of Fe_2O_3 or $\text{Fe}_2\text{O}_3 \cdot n\text{H}_2\text{O}$. Experiments performed at reduced pressures (10^{-1} Pa) yielded low wear rates compared to experiments at atmospheric pressure. This phenomenon was more pronounced at the heavier loads where the reduction in wear rate was by more than a factor of ten. The wear debris produced was still oxide. Kerridge concluded that, in the case of a soft pin sliding against a hard

ring, wear of the pin occurred by transfer of pin material to the ring surface followed by oxidation of the transferred layer. The oxide layer, not being strongly adherent to the base material, ' was rubbed off ' by the repeated sliding ' to generate loose wear debris.

2.5. Welsh's analysis:

Welsh's early experiments (14) were performed in air with a stationary flat-ended pin sliding against a rotating disk at a speed of 2 m/sec. The carbon content of the steels used in the experiments varied from 0.12 - 0.78 %C and the loads ranged from 1-3 kg. In all the experiments, like steels rubbed against each other. At the lowest loads used , the 0.12 and 0.34 %C steels exhibited severe wear, whereas the wear-time curves for the higher carbon steels exhibited mild wear after an initial short period of severe wear (1-10 minutes). At a load of 3 kg all steels showed a transition to mild wear after an initial severe wear period. The debris was not analysed during these experiments. Microhardness measurements on the wear track yielded values in the range 700-900 V.p.n. compared to 130-300 V.p.n., depending on the carbon content, for the original unworn steel. Based on this observation, coupled with an estimation of the temperature at the sliding interface, Welsh suggested that the severe to mild wear transition occurs due to the formation of martensite on the wear track. To account for the abnormally high hardness values on the 0.12 %C steel, he suggested that nitrogen from the atmosphere increased the hardness of martensite. It should be noted however that no conclusive evidence for the presence of martensite on the wearing surface was provided in this work.

Welsh recognized that the facility with which the system changes from severe to mild wear depends on at least three variables, i.e., load, speed and carbon content, and that the two stage wear behaviour may appear over a limited range of rubbing conditions. Welsh followed up this initial set of experiments with a more detailed investigation using a broad range of loads (10 gms-40 kg) and speeds (1.7-266 cm/sec). Experiments were performed with a steel ring rubbing against a pin. Details of Welsh's experimental arrangement can be found in references 16 & 17.

The most important result of Welsh's analysis is that, for steels the wear process at equilibrium, depending on the load/ speed combination, is either of the mild or severe type. The difference in wear rates for the two modes is more than two orders of magnitude. Fig.4 represents the typical wear rate- load graph obtained by Welsh. Welsh designated the low load mild to severe transition as the T1 transition, and the high load severe to mild transition as the T2 transition. It should be noted that T1 and T2 refer to loads where the transition occurs for a fixed sliding speed. Welsh also found that for a steel of fixed carbon content, a decrease in speed resulted in a shift of both T1 and T2 to lower loads and that an increase in speed shifted T1 and T2 to higher loads. For a fixed sliding speed, an increase in the carbon content of the steel resulted in a shift of T1 to higher loads and T2 to lower loads, whereas a decrease in carbon content had the opposite effect.

According to Welsh, mild wear at loads lower than T1 occurs primarily by the combined effects of strain hardening and oxidation. The effects of strain hardening and oxidation were investigated by performing annealing and etching experiments on worn samples. The explanation

Fig. 4. Plot showing the variation of wear rate with load for a 0.52% C steel. Sliding speed 100 cm/s. A, Pin; B, Ring. (Ref. 16).

proposed for the T2 transition involves surface hardening resulting from a phase change caused by frictional heating. This was based on measurements of high surface hardness values and an estimation of interface temperatures based on a graphical method devised by Archard (18). It should be mentioned that for the higher carbon steels at loads above T2, a third transition T3 was discovered. This transition was not accompanied by a drastic change in wear rates, but was characterized by an increasing wear rate for the pin and a decreasing wear rate for the ring. By etching (to remove traces of oxidation on the wear track) and annealing (to study the effect of hardening) above T2, it was established that both hardening and oxidation were necessary to preserve the mild wear state between T2 and T3. T3 was defined as the load and speed combination corresponding to a significant surface hardening responsible for arresting severe wear without the aid of oxidation. The increase in the wear of the pin compared to the ring was thought to be due to the thermal asymmetry of the system. The pin, being smaller than the ring, was expected to attain much higher temperatures, resulting in thermal softening of the pin compared to the ring. This effect was thought to be significant only for load/speed combinations causing significant frictional heating.

Welsh's work established the detailed pattern characterizing the sliding wear behaviour of steels. Surface hardening and oxidation were reported to be the important factors governing the wear mode. The absence of the T3 transition for the low carbon steels was rationalized to be due to the merging of the T2 and T3 transition points.

The estimation of interfacial temperatures by Archard's method (18) is based on assumptions which are not fully realistic. To account for

the high temperatures necessary for phase hardening it was assumed that all the load was borne by a single asperity contact, which may not be the case. Moreover the temperature estimated is the interface temperature and not that of a finite volume of metal. In the absence of conclusive evidence for the presence of martensite the term " phase hardening " has been used by Welsh and due to the uncertainties in the estimation of the interface temperature the cause of hardening above T_2 is uncertain.

2.6. Ashby's wear transition map for the unlubricated sliding of steel:

The work described in the preceding sections has identified transitions in the wear rate of steels due to variations in load, sliding speed and distance of sliding. Surface hardening and oxidation have been identified as important factors for load and speed dependent transitions. These transitions have been graphically summarized on a wear transition map (20). The details underlying the construction and interpretation of the map will be discussed next.

Wear mechanism maps, analogous to deformation mechanism maps used in creep studies, summarize data and models for wear by a suitable set of axes. Ashby has constructed a wear map for the unlubricated sliding of steel (22) based on data obtained from a wide variety of sources, i.e., from sliding wear tests performed under different conditions using different sliding geometries. To correlate these data, the load, speed and wear rate data have been normalized. The normalized variables are defined next:

Fig. 5. Wear transition map showing regions of mild and severe wear. (Ref. 22). The shaded areas correspond to transitions in wear rates.

$$1) W' = W/A_n$$

$$2) F' = F/A_n.H_0$$

$$3) v' = v.r_0/a$$

where A_n = apparent contact area of the wearing surfaces

H_0 = room temperature hardness of the sliding member

a = thermal diffusivity of the sliding member

r_0 = radius of the contact area.

W is the volume of material worn per unit distance slid, which, as mentioned before, is the conventional way of representing wear rate. W' is the wear rate normalized with respect to the apparent contact area. F and v are the load and sliding speed. F' and v' represent the normalized load and velocity; v' is the sliding velocity divided by the velocity of heat flow.

The wear transition map shown in fig.5. summarizes the work of different researchers and classifies mild and severe wear regions corresponding to different loads and speeds. As the range of the axes is large, the map incorporates data from most of the published work on the wear of steels.

As closing remarks to this overview of early work on the wear of steels the author would like to say that any detailed wear mechanism theory should be capable of describing the generation of wear debris. Ideally the model must be both quantitative and have a physical basis. Developments in the theory of plastic deformation, fracture and adhesion of metals, combined with chemical and structural characterization of worn surfaces and debris on a fine scale have provided means to gain a better understanding of the mechanisms underlying debris formation during wear. Oxidation, though undoubtedly important in the wear of steels, cannot by itself account for wear debris generation. Wear debris generation

is a mechanical process which can be modified by oxidation, resulting in an overall change of the wear process.

The next section examines some of the more recent work on the wear of steels. The first part focusses on environmental effects in the wear of steels with a special emphasis on the role of oxidation. The second part is on the mechanical aspects of wear.

2.7. Environmental effects in the wear of steels:

2.7.1. The role of oxidation in the wear of steels:

Numerous researchers (23-32) have investigated the role of oxidation to try to explain the mechanisms underlying the mild wear of steels. A review of such work may be found in reference 33.

In his initial work Quinn attempted to relate classical models of oxidation to the wear of steels. The work focussed on steady state mild wear under high load/speed conditions when temperatures at the sliding interface may become appreciable. The basic idea underlying Quinn's model is the formation of an oxide film followed by its removal after it has attained a critical thickness. By assuming that the steady state mild wear rate is controlled by the rate of oxidation, Quinn derived an equation for the rate of oxidation of a wearing surface. The details of the derivation can be found in reference 23. The assumption of parabolic kinetics used in the derivation cannot be justified, as oxidation during sliding involves fresh areas of the wearing surface which are continually exposed to the environment.

The idea that an oxide film develops to a critical thickness before being removed as debris has also not been addressed in detail. Moreover

the derived equation contains some parameters which cannot be precisely determined, such as the distance over which a wearing contact is made, the temperature of oxidation and the real area of sliding contact. Adjusting the values of these parameters to experimentally obtained wear rate data led to discrepancies in the values of the activation energy and the Arrhenius rate constants used in the derivation of oxidation kinetics compared with the values of these constants determined by Kubaschewski and Hopkins (34) for static oxidation conditions.

Quinn's original theory was later revised (24) to account for the discrepancies mentioned above. By assuming that the rate constant for oxidation will be proportional to the diffusion coefficients of the various ionic species in the relevant oxide, Quinn has postulated that the values of the Arrhenius constant and the activation energy for oxide growth will depend on both D_0 and Q according to the familiar expression $D = D_0 e^{-Q/RT}$ for the diffusion coefficient. Q depends on potential barrier heights which are not expected to differ in static and sliding conditions, so the differences must be due to differences in the value of the Arrhenius constant. Kubaschewski and Hopkins (33) have discussed the effect of surface conditioning, voids and dislocations on the value of the Arrhenius constant.

Quinn's model has some uncertainties in that it involves only rough estimates of interface temperature and sliding contact distance. As mentioned earlier Welsh had found that oxidation helps to sustain the mild wear state at high loads and speeds once the surface had hardened. Quinn has not addressed the issue of surface hardness. The mild wear state at high loads and speeds is preceded by a period of severe wear (16,17) . Incorporation of this aspect might shed additional light on the issues of

oxidation kinetics and surface hardening. Quinn has analysed the wear debris using X-ray powder diffraction techniques and detected both iron and iron oxide. There is some debate as to the type of oxide produced, i.e., whether the oxide produced is $\alpha\text{Fe}_2\text{O}_3$, Fe_3O_4 , $\gamma\text{Fe}_2\text{O}_3$ or some mixture. Analysis of the powder patterns indicating the presence of both iron as well as oxide does not clarify whether the debris is a mixture of oxide particles and metal particles or partially oxidized metal particles. Examination of individual debris particles in the TEM, as done in the present work sheds light on this aspect. Quinn's theory also fails to specify clearly whether oxidation occurs at the contacting asperities or over the whole wearing surface, as the analysis starts with an estimate of temperatures at asperity contacts and proceeds to model a thick oxide layer on the wearing surface.

Sullivan and Hodgson (26) have studied the effects of oxidation under conditions of low load and speed, i.e., below Welsh's T1 transition. At low loads and speeds heating effects are negligible (20) and Sullivan et al. have proposed that out-of- contact oxidation is primarily responsible for oxidation under these conditions. Assuming logarithmic oxidation kinetics (as oxidation occurs over a relatively short time when the wearing surface is out of contact), they derived an equation which indicated an inverse relationship between the volume of oxide produced and the speed of sliding. This was used to explain the inverse relationship between the wear rate and sliding velocity observed in their experiments. The existence of a critical speed above which severe wear ensues could also be explained, as this speed could be associated with the minimum amount of oxidation necessary to sustain mild wear. Their analysis, however, could not account for the significant amount of metal debris detected. According to

them, the "production of agglomerate oxide-metal plateaux" and "subsequent removal of the plateaux once critical thickness has been attained" accounts for the mixed metal/ metal-oxide on the wearing surface and debris. Sullivan and Hodgson used a vertical pin sliding on a horizontal disc in their experiments. Under these circumstances one can visualize the debris as trapped at the sliding interface and undergoing an agglomeration process. This would be consistent with the different mechanism proposed by Granville (27) (diffusion controlled growth). He used a vertical disc in his experiments , so most of the debris fell from the sliding interface. Thus, the possibility of the sliding geometry having an influence on the mechanism of mild wear must be considered.

Stott et al. (28) have investigated the role of oxidation by conducting experiments with a flat disc specimen sliding against a hemispherical upper specimen in a horizontal reciprocating mode. They have proposed that asperity oxidation is significant when the temperature rise at the contacting regions is high. Under conditions of negligible interfacial heating (low load/speed conditions) they propose that metal debris is generated at the start of sliding. The debris particles subsequently fracture and refracture with further sliding, until they become so small that they oxidize spontaneously at room temperature. These oxide particles then agglomerate in the wear track and reduce metal-metal contact, which results in a considerable reduction in wear. The sliding geometry used by Stott et al. should be borne in mind while considering this explanation.

A novel approach towards understanding the role of oxygen in mild wear can be found in the work of Sasada et al. (29). By measuring the "consumption" of various gases during the course of pure iron sliding on

itself under conditions of low load and speed, they observed that oxygen consumption was high during mild wear. The severe to mild transition depended on the partial pressure of oxygen and was also accelerated by the presence of a magnetic field. Besides iron, various other sliding systems were investigated and oxygen consumption during wear was found to depend on the position of the elements in the periodic table. These facts, coupled with the study of published chemisorption data (34,35) led to the conclusion that mild wear occurs due to the increased chemisorption of oxygen. The variation of chemisorption activity for different elements was thought to be due to differences in their electronic structure, with iron exhibiting a high activity because of its d-electron vacancies. The observation of increased chemisorption of oxygen in the presence of a magnetic field with all other factors held constant has been used as evidence to support this view. Sasada has used the results of this work to account for his observations from a different series of experiments (30) in which he found that mild wear is only encountered when one of the sliding members is a transition metal or rare earth metal.

The work described above cannot by itself account for the oxides observed in the debris produced during the mild wear of steels. However, incorporation of these data might help remove some of the uncertainties associated with the mechanisms underlying the formation of oxides, especially at low load and low speed conditions.

To understand better the role of oxidation in mild wear, both the mechanism of oxidation under conditions of sliding, as well as the mechanical effects of the oxidation process must be considered. In the

following sections some aspects of the mechanical effects of oxidation are briefly discussed.

Critical thickness theory : This theory involves the growth of an oxide layer to a critical thickness followed by its removal, the reasons for which are not immediately obvious. Aranov (36) has suggested that the oxide layer is removed after a certain number of rubs as in a fatigue process. Another approach is that of Yoshida et al. (37) in which the stresses at the interface of a hard layer and a soft substrate have been analysed for sliding contacts. The analysis predicts that high shear stress concentrations develop at the interface of the hard layer and soft substrate at a certain thickness of the hard layer relative to the contact width. This could lead to fracture and debris generation. The applicability of this analysis is however dependent on the type of oxide film as Fe_3O_4 is harder than steel whereas FeO is not. Moreover, effects of subsurface hardening should be taken into account. This theory is applicable only for high load/speed conditions when surface heating effects are appreciable and the formation of thick oxide layers is feasible.

Compacted layer theory : Stott and Wood (31) have investigated the role of oxides in the mild wear of Fe and Fe-Cr alloys at low load/speed conditions. They concluded that mild wear is due to the formation of smooth oxide glazes consisting of fine crystalline oxide particles, typically 10-50 nm in diameter. The first stage in glaze formation is the generation of oxide or partially oxidized debris. Next, the debris particles fragment and are compacted under the pressures existing at the sliding interface. Recompaction of debris from the glaze is thought to be the cause for the low wear rate.

Iwabuchi et al. (32) have investigated the role of oxides in mild wear by supplying oxide particles at the interface during the sliding of 0.45 wt% C steel under low load/speed conditions. They concluded that the transition from a severe running-in stage to a steady state mild wear stage occurred when a compacted layer with sufficient load bearing capacity was formed at the sliding interface.

2.7.2. Effects of the environment on adhesion:

Adhesion plays an important role in the friction and wear of metals. Buckley (38) has shown that even a monolayer of oxygen adsorbed on a clean steel surface reduces the adhesivity (maximum tensile load of the contact) of that surface by a factor of 4. Hartweck and Grabke (39) have found that the maximum tensile or adhesive strength of iron-iron contacts rose with oxygen coverage until 0.15 of a monolayer was formed, after which the maximum adhesive strength sharply decreased with an increase in oxygen coverage. Pethica and Tabor (40) have shown that , for a clean tungsten tip on a clean nickel flat, a layer of oxide about 5 nm thick greatly reduces the amount of plastic deformation of the underlying metal. Also, as the size of the deformed zone is reduced, the plastic yield stress increases. For deformed zones of the order of 100 nm in diameter, the hardness may be three or four times the bulk hardness. These results on adhesion after normal contacts have not yet been incorporated into models of wear.

2.8. Mechanical aspects of the wear of steels :

2.8.1. Adhesion and plastic deformation in friction and wear :

In the early stages of development, attempts to explain the friction and wear behaviour of metals concentrated on the formation of adhesive junctions at the sliding interface. Friction and wear were attributed to the shear and failure of these junctions (41,42). Transfer of material from one component to the other was also explained on the basis of the formation and failure of these junctions. Friction values predicted from traditional adhesion theories were too low when compared with experimental data. A review of the development of adhesion theories of friction can be found in reference 43. A serious drawback of adhesive theories of wear, as pointed out by Rabinowicz (44), is that they neglect plastic deformation. Observations of wear scars, wear track cross sections and wear debris have indicated the importance of plastic deformation in the wear process. Reports of very high plastic strains adjacent to the wear tracks can be found in references 45-47.

Friction models have been developed which take into account the large amount of plastic deformation adjacent to the sliding interface. (48-50). Hirth and Rigney (50) have proposed an energy based model of friction which is based on the assumption that frictional work equals the plastic work in the steady state, with most of the work being dissipated as heat. This model has been extended by Heilmann and Rigney (51) to take into account observed plastic displacements.

Sliding causes large plastic strains which commonly exceed 1000 % near the interface. Thus the surface layer has near-surface structure and

properties different from those of the base material (52-54). The surface is frequently either partially or wholly covered with a layer of ultra-fine grained material with the grain size ranging from 2-30 nm (52). Don et al. (53) have shown that the smoothness of sliding and the type of debris generated depend on the relative hardness of the fine grained layer and the adjacent base material. Chen (55) has investigated the development of the fine grained material for a number of simple metal systems sliding in vacuum. His experiments indicated a complex sequence of events where local plastic deformation occurs with and without transfer of fragments of material between the sliding components. Chen (55) has shown further that the transfer material is gradually mixed with the substrate to form a mechanical mixture with nanocrystal grains. Debris particles are generated from this mixed material. Chen and Rigney (56) have found a good correlation between the direction and amounts of transfer with simple estimates of the cohesive and adhesive strengths of sliding components. In general, transfer of material predominantly occurs from the cohesively weaker to the cohesively stronger sliding member. Heilmann (52) has studied the near surface layer for a Cu block sliding against a 440C ring. The dislocation structure was shown to consist of dislocation cells and subgrains. Salesky et al. (57) have observed a similar near-surface structure for a 52100 steel pin sliding against a 52100 steel disc.

Kato and Akagaki (58,59) have studied the predominant wear mechanisms of a 0.45 wt. % carbon (222 VHN) steel disk sliding against a high carbon steel bearing ball (863 VHN) under lubricated conditions. Based on wear rate data and SEM observations of wear scars and wear debris they have classified the wear of the steel disk as flow wear,

ploughing or abrasive wear and adhesive wear, as shown on a wear mode diagram which uses contact pressure and sliding velocity as axes (Fig.6). According to Kato et al. flow wear occurs at low contact pressures when 'filmy wear layers' are extruded perpendicular to the sliding direction, leading to the formation of 'filmy wear debris'. Ploughing or abrasive wear has been associated with high contact pressures and large wear debris particles.

Soda (60) has observed an increase in debris particle size with load for a number of different systems. Kato postulates that large wear debris particles plough the disk wear surface causing it to become rough. Work hardening of the asperities on the rough wear surface and the loose debris trapped at the interface promote the ploughing action and increase the wear rate. Adhesive wear is associated with still higher contact pressures with large plate/block type wear debris. Ploughing wear has been considered to be a precursor to the adhesive wear process. Kato has proposed that the large wear debris formed during ploughing either transfers to the opposing sliding member or is trapped at the interface and forms a wedge. Mutual transfer and adhesion play an important role in the growth of debris particles leading to the formation of a wedge at the interface. Kato also proposes that adhesive wear leads to seizure when the driving torque is not sufficient to overcome the resistance to sliding offered by the wedge formed at the interface. This work was performed under lubricated conditions and neglects other environmental effects.

Both adhesion and plastic deformation play important roles in sliding wear processes. However, it is difficult to separate these two effects.

Fig. 6. Wear mode diagram for a 0.45 wt %C steel in lubricated sliding. o, flow wear; Δ , adhesive wear; $\blacktriangle$, abrasive wear. (Ref. 58).

2.8.2. Mechanisms of debris generation :

Debris particles generated during sliding wear have a wide variety of shapes and sizes (61). Sometimes, the debris type might change during the course of sliding. The particle size ranges from tens of nanometers to a few centimeters. The particle shapes include cutting chips, spheres, flakes, plates, ribbons , etc.

Suh (62) has used a phenomenological approach to describe qualitatively the generation of flake type debris formed during the sliding wear of steels and a number of other materials. The basic assumption of his original delamination theory, is that dislocation image forces create a soft surface layer and cracks nucleate below that layer. Under the effects of the applied stress, the cracks join and propagate parallel to the wear scar, eventually turning to the surface to form flake type debris. However, as Rigney and Hirth (63) have pointed out, the surface layer affected by image forces is only about 10 nanometers, which is much thinner than flake type debris found experimentally. Suh (64) later revised his theory, placing a greater emphasis on cracks nucleated below the surface. He suggested that crack nucleation very close to the surface is not favoured due to the presence of a triaxial state of high compressive stress just below local contacts.

Suh's assumption of a soft surface layer is debatable. Rigney and Hirth (63) have discussed reports of both surface hardening and softening. Suh has also neglected the effects of a transfer layer at the sliding interface. Rigney and Glaeser (65) have suggested that a cell type structure adjacent to the sliding interface can present suitable ways for subsurface crack generation and propagation. However, experimental support for

delamination is provided by reports of subsurface cracks (57,62). The detailed mechanisms underlying the origin and propagation of these cracks continue to receive much attention.

An analytical description of wear based on fracture mechanics concepts is quite difficult. According to Rosenfield (66) the difficulties arise because of the fact that during sliding wear, debris generation occurs by crack nucleation in material which is highly deformed and for which the mechanical properties are poorly understood. The local stresses at the contacting regions are also difficult to specify accurately. The assumption of continuum mechanics, on which classical fracture mechanics is based, is also questionable as the crack size can be of the same order of magnitude as the microstructural features.

Despite these problems, Fleming and Suh (67) have tried a linear elastic fracture mechanics analysis for delamination wear. The predicted crack growth rates based on this analysis were low compared with experiments. Some drawbacks in the assumptions of Fleming's and Suh's analysis responsible for the slow crack propagation rates have been discussed by Hills and Ashelby (68,69). Rosenfield (66) has modified the analysis of Fleming and Suh (67) by taking into account the friction between the opposing faces of the crack. However, the stress intensity value calculated turned out to be too low to cause crack growth.

The analyses described in the preceding paragraph were based on considering the material to be perfectly elastic. Rosenfield (70) has also used elastic-plastic fracture mechanics to deal with semi-ductile materials. This approach takes into account any significant amount of plastic deformation which may occur ahead of the crack tip. The dislocation shear

crack model developed by Bilby et al. (71) was used by Rosenfield to calculate the crack-tip displacement. His analysis led to the following predictions: 1) delamination crack growth is possible provided that the friction between the opposing faces of the crack is not too great and 2) there is a critical stress above which any crack that is formed will grow catastrophically. Welsh's T1 transition was used as experimental evidence to support this model.

2.9. Summary and aim of the present work:

As discussed in section 2.5, Welsh has demonstrated that large changes in wear rate can result from small changes in applied load and/or sliding speed. Welsh produced a series of curves from which three transitions, T1, T2 and T3, were defined. Since Welsh's investigation there have been extensive studies of the wear of carbon steels under unlubricated sliding conditions. Most of these studies have concentrated on the region above the T2 transition. The present study is aimed at the region just below and above the T1 transition where the loads and/or speeds are low and frictional heating negligible.

As discussed in section 2.8.1, transfer has been determined to be an important factor in the wear process for a number of sliding systems, but the role of transfer in the wear of carbon steels has not been investigated. One of the primary aims of this investigation is to analyse the role of material transfer in the wear of steels under the different conditions tested.

In the present investigation, tests have been conducted both in air and in vacuum so that the role of the environment can be understood better. Characterization of debris generated in different environments should reveal the relative importance of different mechanisms, e.g., oxidation (discussed in section 2.7.1) or delamination (discussed in section 2.8.2).

CHAPTER III

EXPERIMENTAL PROCEDURE

3.1. Wear testing apparatus :

To allow direct comparison with the results of Welsh (16, 17) all tests were performed using the crossed cylinders geometry with the axes of the cylinders at right angles to each other (Fig.7). A schematic drawing of the arrangement is shown in Fig.8.

The wear testing equipment consists of a rotating shaft which carries the larger (1" diameter) cylinder and a loading arm on which the smaller (0.25") stationary cylinder is mounted. The loading arm is pivoted at one end with the load being applied at the other end. Consequently the load at the point of contact is multiplied by a factor of 3.6 (Fig.8). The rotating cylinder is held in the shaft by means of a collet. The set up shown in Fig.8 and the shaft carrying the large cylinder are housed in a bell jar which can be evacuated by means of rotary and diffusion pumps. The shaft carrying the rotating test piece is driven by a 0.5 H.P. motor. The rotational speed can be varied continuously from 30-1800 r.p.m. with the help of a speed controller. This corresponds to sliding speeds ranging from 4 cm/sec to 240 cm/sec. A strain gauge is attached to the loading arm assembly so that it experiences a tensile force due to the frictional force at the sliding interface (Fig.8). The strain gauge is connected to an X-Y recorder via a strain

Fig. 7. Schematic of the crossed cylinders arrangement.
S = Stationary cylinder. R= Rotating cylinder.
Arrow indicates direction of rotation.

Fig. 8. Schematic of the experimental arrangement.

indicator and this arrangement provided a trace of friction force versus time.

The crossed cylinders geometry has the advantage that the wear rates at very low loads may be computed from the dimensions of the wear scar when weight changes are negligible (17). The sample dimensions were chosen to be similar to those used by Welsh (17) .

The curved surface of the wear scar provides some difficulty in optical microscopic examination and microhardness testing. This aspect is dealt with later. One special aspect of the crossed cylinders arrangement is that at the start of a test the cylinders touch each other only over a small area, but as wear occurs the surfaces begin to conform to each other and the contact area increases. As a result, the stress decreases

3.2. Test materials :

The materials used in this investigation were AISI 1045 and AISI 1020 steels obtained from Art Iron Co. (Columbus, OH) and Brown Steel Co (Columbus, OH), respectively. The elemental composition and bulk hardness values for both steels are shown in the Table 1.

Table 1.

Composition and bulk hardness values of the test materials:

Material	wt % C	wt % Mn	wt % S	wt % P	VHN
AISI 1020	0.21	0.81	0.038	0.022	220
AISI 1045	0.45	0.7	0.05	0.04	250

Fig. 9. Microstructure of the AISI 1045 steel used. 2% Nital etch.

Fig. 10. a) Schematic of the samples.

Fig. 10. b) Schematic of the wear scars (indicated by shaded areas).

After establishing that the general pattern of behaviour for both steels was the same (i.e., both showed mild and severe wear), it was decided to analyse the wear of AISI 1045 steel in detail. Fig. 9 shows the microstructure of the AISI 1045 steel. The materials were obtained in the form of rounds and samples of the desired dimensions (see Fig. 10) were machined on a lathe. The machined samples were then polished with 120, 240 and 400 grit SiC papers in sequence, to provide a standard finish for the test samples.

3.3. Wear testing procedure :

The standard procedure followed for performing the wear tests is summarised below:

- 1) Before starting the test the machined and polished samples were cleaned ultrasonically, first with acetone and then with methanol. Once cleaned, the samples were always handled with gloves. The samples were then weighed in a chemical balance capable of weighing to 0.1 mg. Prior to the start of the test the testing chamber and the samples were cleaned with a jet of Freon gas.
- 2) The small cylinder was carefully mounted in the the pin holder assembly, making sure that the axis of the cylinder was parallel to the length of the loading arm. With the small cylinder in position the loading arm was balanced by a counterweight so that it was horizontal in the test chamber. This was checked with a level.
- 3) The large cylinder was then mounted on the shaft and the loading arm positioned such that the small cylinder just touched it.

- 4) A fresh piece of lens tissue to collect the wear debris was placed in a receptacle just below the samples.
- 5) The desired load was then applied.
- 6) The strain gauge was connected to the recording device.
- 7) The bell jar was lowered. At this point tests in air could to be started by switching on the motor driving the shaft. Vacuum tests were started after the chamber was pumped down to a level of $2 * 10^{-5}$ Torr ($2.6 * 10^{-3}$ Pa). The relative humidity and ambient temperature were monitored by placing a hygrometer and temperature indicator inside the chamber.
- 8) After the test the wear tracks were brushed to remove any debris sticking to them. The debris was stored in glass vials. Following a test, the samples and the vials containing the debris were stored in a desiccator pending further analysis.

3.4. Wear testing conditions:

Tests were conducted in both air and vacuum ($2 * 10^{-5}$ Torr, $2.6 * 10^{-3}$ Pa) at different loads, speeds and numbers of sliding cycles. The loads ranged from 180 gms to 1.65 kg and the speeds used ranged from 2-24 cm/sec. The number of sliding cycles varied from 50 cycles to 30,000 cycles. These conditions were chosen based on Welsh's data from experiments in air. This allowed studies of both mild and severe wear behaviour.

3.5. Wear rate measurements :

The wear rates of both the samples were computed by measuring the weight changes of the samples. The loss in weight divided by the sliding

distance and the density of steel yielded the volume of material worn per unit distance slid. Wear rates were also determined by measuring the area of the wear scars obtained from profilometer traces .

3.6. Optical microscopy and microhardness testing :

The wear tracks and their cross sections were examined with an Olympus BO61 optical microscope. The curved surface of the wear track made it difficult to obtain detailed topographical information as the optical microscope does not have a very good depth of focus. However the size and shape of the scar were noted carefully.

Longitudinal cross-sections from the wear track (i.e., sections taken parallel to the sliding direction and perpendicular to the wear track) can provide useful information regarding substructural deformation caused by sliding. Longitudinal cross-sections from the centre of the wear track were prepared from test samples using a diamond wheel cutter. To ensure that the cutting process itself did not cause microstructural changes, a slow cutting speed (4 ms/min) and a continuous supply of coolant (ethylene glycol) were used. The resulting sections were mounted using standard 1" (2.54 cm) mounts in Bakelite at a temperature of 150⁰ C. The mounted samples were then polished and etched using a 2% Nital solution and viewed in an optical microscope. Photomicrographs were obtained with a Nikon Epiphot metallograph.

Microhardness values, both on and off the wear tracks, were measured using a Buehler Micromet II Digital Microhardness Tester. The load used was 50 gms and the time of application of the load was 13 secs.

The curvature, roughness and the oxidized nature of the wear tracks presented problems with some samples. A large number of trials was then necessary to obtain well defined indentations. An average of five indentations was used to obtain a single microhardness value.

3.7. Scanning Electron Microscopy :

The wear debris, wear tracks and cross-sections of the worn samples were examined in a Hitachi S510 scanning electron microscope (SEM).

The shapes and range of sizes of the debris were studied in the SEM. The debris was mounted on a graphite disc using a graphite glue which ensured good adhesion between the disc and the debris. The glue also provided a good conducting surface. The SEM, by virtue of it's excellent depth of field, high resolution and high magnification capabilities, was used to study the topography of the wear tracks in detail. The longitudinal cross sections (described in Sec 3.6) were also observed in the SEM. Records of these observations were obtained in the form of Polaroid photographs.

3.8. Profilometry :

Profilometry is a technique used to analyse the topography of surfaces. The technique basically involves the traversal of a fine diamond stylus over the surface of interest. Variations (ups and downs) on the surface cause a movement of the stylus and this motion is used to generate an electrical signal. The output from the profilometer can be either graphical (usually referred to as a profilometer trace) or numerical. The

latter is achieved by converting the analog electrical signals generated by the stylus motion to a digital form.

Profilometer traces from some representative samples were obtained from a Talysurf 6 computer controlled profilometer at Battelle Laboratories in Columbus . This instrument had the special attachments necessary to obtain traces from curved surfaces. Besides topographical information the traces provided accurate dimensions of the wear scar which were used to compute the volume of material worn. This allowed verification of the wear rate estimates obtained from weight loss measurements .

3.9. X-ray Diffraction analysis :

The powder diffraction method was used to identify the phases present in the debris produced from sliding wear tests. Unfiltered Co radiation was used. The technique posed some problems for the larger debris sizes as these could not fit in the capillary tubes available. The technique is not capable of providing information regarding the phases present in individual debris particles, but this was achieved with the Transmission Electron Microscope (TEM).

3.10. Transmission electron microscopy of the wear debris :

The TEM provided a means of obtaining diffraction information from individual debris particles so that their structure could be identified. Debris samples for TEM observation were prepared using methods documented by R. Antoniou (72). The debris collected from the wear tests was mixed with acetone and ultrasonically treated to break up the agglomerates and obtain

a suspension of individual particles in acetone. This suspension was then sprayed on to a carbon film with a syringe. The carbon film was then floated off in water and deposited on a TEM copper grid. This technique only requires very small amounts of debris for analysis and a number of samples could be prepared using the debris from one wear test. The samples were first examined in an optical microscope at 400X, so that the sample with the largest number of debris particles could be used for examination in the TEM. Due to the very small size of the debris particles, they had some naturally thin areas which could be used for observation in the TEM.

3.11. Analysis of transfer material using radioactive tracers :

Experiments in this investigation were performed with like materials rubbing against each other. In the case of dissimilar metals sliding against each other, transfer of material from one component of the sliding system to the other can be studied using techniques such as EDS (energy dispersive spectroscopy) or WDS (wavelength dispersive spectroscopy). For identical metal combinations, the radioactive tracer technique at present seems to be the only way to monitor transfer. Other researchers have used this technique to study the role of transfer in wear (9, 10, 73-75). The technique involves the activation of one of the sliding members followed by a check for activity on the unactivated sample after a sliding wear experiment.

Before using this technique Joe Kalen evaluated the feasibility of producing a relatively safe but detectable (in terms of radiation levels)

radioisotope in steel with the facilities available at the Van De Graaff Laboratory at The Ohio State University. This was done by determining the energy necessary to produce Co-56 by activation of steel. Examination of the nuclear data indicated that Co-56 is the dominant radioisotope produced when steel is bombarded by protons (H^+) accelerated to an energy between 6 and 6.5 MeV. Radioactive Co-56 is produced from Fe-56 and has a half life of 77.7 days. This allows sufficient time to carry out the desired sliding experiments and analysis. Co-56 decays by emitting 846 keV γ -rays. These can be detected using a Ge-Li detector which has a good efficiency and resolution in terms of detecting γ -rays of a specific energy.

Past research (55) has shown that transfer occurs mainly from the cohesively weaker material to the cohesively stronger. In the present work both components of the sliding pair are identical. For transfer analysis most of the experiments were performed with the large cylinder activated. With one activated ring three runs could be achieved, provided the wear scars were not too large. Figure 11 is a schematic of the activated area on the samples. Several experiments were also performed with the pin activated to see if the geometry of the sliding member has any effect on transfer.

The experimental procedure involved the following steps in sequence :

- 1) The sample to be activated was placed in the target chamber and a part of the sample was activated by exposing it to protons which passed through a 1 cm² slit before impinging on the target area.

- 2) The activation was performed by impinging protons accelerated to 6-6.5 MeV on to the target area. The activation time was between 1-2 hours depending on the beam current which could be achieved.
- 3) Following activation, the sample was allowed to remain in the target chamber for a period of ten days. This allowed the radiation levels to decrease.
- 4) After the holding period the sample was removed from the target chamber and placed in front of a Ge-Li detector to count the 846 keV γ -rays. A standard geometry was used for all counts so that the results from different counts could be compared. Before each count the detector was calibrated with a standard Na-22 source. Data from the counts were stored in a computer. The radiation associated with a freshly activated sample was of the order of 0.01 micro Curie.
- 5) The activated sample was then slid against an unactivated sample and a check for transfer was carried out by placing the unactivated sample in front of the detector. Debris from activated tests was also counted by placing the vials containing the debris in front of the detector.
- 6) When not in use the samples and the debris were stored in lead containers which in turn were kept in a separate desiccator.

Fig. 11. Activated area on the samples shown by shaded regions.

CHAPTER IV

EXPERIMENTAL RESULTS

The present chapter deals with the results obtained from the wear tests and subsequent analysis. The chapter has been divided into a number of sections with each section dealing with a specific type of information obtained from the wear tests , e.g., wear rate results, SEM observations, etc. The results are presented and the general trends indicated. A detailed discussion of the results will be presented in the next chapter. For the sake of brevity the large cylinder will be referred to as the ring and the small cylinder as the pin. The results presented in this chapter are from experiments performed with AISI 1045 steels.

4.1. Wear rate results :

Air Tests : The experimental conditions and the wear rates associated with some of the severe wear tests in air are summarised in Table 2. The loads and speeds were chosen on the basis of Welsh's results to produce severe wear conditions in air. Intended values for the speeds were 5, 10, 15 and 25 cm/sec. However, the speed controller had a wide range, so it could not be set precisely in the low speed range. The exact

Table 2. Wear rate data from severe wear tests in air

Load (gms)	Sliding Velocity (cm/sec)	Number of cycles	Sliding distance (cm)	Wear Rate Ring (cm ³ /cm)	Wear Rate Pin (cm ³ /cm)
648	12	4500	35908.4	$3.5 \cdot 10^{-7}$	$1.4 \cdot 10^{-7}$
648	5.3	1500	11969.5	$1.4 \cdot 10^{-7}$	$9.2 \cdot 10^{-8}$
648	10	20,200	161188.9	$1.4 \cdot 10^{-7}$	$1.0 \cdot 10^{-7}$
648	12	36,120	288224.7	$1.5 \cdot 10^{-7}$	$1.0 \cdot 10^{-7}$
648	5	3000	23938.9	$6.6 \cdot 10^{-8}$	$5.9 \cdot 10^{-8}$
648 *	8	32,525	259522	$1.7 \cdot 10^{-9}$	$2.2 \cdot 10^{-9}$
648	12	32,000	255348.6	$6.0 \cdot 10^{-8}$	$5.3 \cdot 10^{-8}$
1100	24	3000	23938.9	$4.3 \cdot 10^{-7}$	$2.9 \cdot 10^{-7}$
1100	24	1075	8578.1	$8.3 \cdot 10^{-7}$	$2.8 \cdot 10^{-7}$

Table 3. Wear rate data for mild wear tests in air

Load gms	Sliding Velocity (cm/sec)	Number of cycles	Sliding Distance (cm)	Wear Rate Ring (cm ³ /cm)	Wear Rate Pin (cm ³ /cm)
360	13.3	35,000	279,287.6	2.8×10^{-10}	6×10^{-10}
540	12	37,900	302,428.5	7.6×10^{-10}	1.2×10^{-9}
360	4.65	3500	27,928.7	1.8×10^{-9}	5×10^{-9}
180	6	1800	14,363.4	3.6×10^{-9}	2.67×10^{-9}

Table 4. Wear rate data from tests conducted in vacuum

Load gms	Sliding Velocity (cm/sec)	Number of cycles	Sliding Distance (cm)	Wear Rate Ring (cm ³ /cm)	Wear Rate Pin (cm ³ /cm)
648	5	7000	55,857.5	8×10^{-8}	9.1×10^{-10}
360	5	2500	19,949.1	9×10^{-8}	no change
648	10	8000	63,837.2	4.8×10^{-8}	1.8×10^{-9}
648	6.4	5250	41,893.1	4.7×10^{-7}	4.8×10^{-8}

sliding speed was determined at the end of each test by dividing the total number of cycles slid by the duration of the test. These values are listed in the wear rate Tables.

In air at loads of 648 gms and more the wear rates for both the pin and the ring were of the order of 10^{-7} cm³/cm, which corresponds to the severe wear regime of Welsh (16,17). In general the wear rate at 648 gms and above increased with increasing load and speed in the load-speed range tested. The low wear rate (10^{-9} cm³/cm) for a 648 gms test, which is indicated by an asterisk next to the load value in table 2, was associated with a change in the nature of the debris formed during the course of the test. This aspect will be discussed in the next chapter.

The wear rates associated with tests performed at loads less than 648 gms are presented in Table 3. It can be seen from the table that at these lower loads the wear rates are of the order of 10^{-9} - 10^{-10} cm³/cm, which corresponds to the mild wear regime of Welsh (16,17).

Vacuum tests : Table 4 is a compilation of the experimental conditions and the wear rates for some of the tests performed under vacuum. The wear rates are in the range of 10^{-7} - 10^{-8} cm³/cm, which is between the mild and severe wear rates in air. Another difference to be noted is that in vacuum, the wear rate of the pin is an order of magnitude less than that of the ring.

4.2 . Friction measurements :

Figures 12-15 are reproductions of the friction traces obtained from some of the wear tests. The wear testing system was not completely free of

Fig.12. Friction trace from a mild wear test in air. Load 360 gms. Speed 4.65 cm/sec.

Fig. 13. Friction trace from a wear test in air with distance dependent transition. Load 648 gms. Speed 8 cm/sec.

Fig. 14. Friction trace from a severe wear test in air. Load 648 gms. Speed 12 cm/ sec.

Fig. 15. Friction trace from a wear test in vacuum. Load 648 gms. Speed 7 cm/sec.

vibrational effects which might have influenced the friction measurements by contributing to the electrical signal generated by the strain gauge. Therefore the friction coefficients reported should be considered as approximate values. However, the traces serve a useful purpose by revealing differences between mild and severe behaviour in air and the wear behaviour in vacuum.

Figure 12 is a reproduction of the friction trace obtained from a wear test conducted at a load of 360 gms and at a sliding speed of 4.65 cms/sec in air. This type of trace is typical of all air tests conducted at loads of less than 648 gms and is associated with mild wear behaviour. At the beginning of a test the trace is noisy as indicated by the spikes and after a certain number of cycles (which varied from 200-600 cycles) it becomes smooth and remains so until the test is terminated. In mild wear the friction coefficient is estimated to lie between 0.2 and 0.3.

Figure 13 is a reproduction of the trace obtained from an air test conducted at 648 gms and at a sliding speed of 8 cms/sec. This type of trace was not typical of all air tests conducted at a load of 648 gms or more, but was obtained only when there was a distance dependent transition. It was associated with a change in the nature of the debris being produced. In these cases the traces became smooth only after 18,000-25,000 cycles.

Figures 14 and 15 correspond to traces obtained from an air test and a vacuum test, respectively. The air test trace is typical of all severe wear tests and the vacuum trace is typical of all vacuum tests. Both traces are noisy throughout but the spikes are bigger and more irregularly spaced for vacuum. The average value of the friction coefficient for the severe wear

and vacuum traces is estimated to lie between 0.4-0.6. In severe wear the largest spikes correspond to a friction coefficient of 1.2 and in vacuum this value is 1.8.

4.3. Profilometry:

Figures 16a and 16b are reproductions of the profilometer traces obtained from the wear scar on a pin after a test in vacuum conducted at a load of 648 gms and a sliding speed of 5cm/sec. Figures 17a and 17b are reproductions of traces from the wear scar on a pin after a test in air at the same load and speed. Figures 16a and 17a correspond to traces taken perpendicular to the sliding direction and figures 16b and 17b to traces taken in the sliding direction. Figure 17a indicates grooving whereas the corresponding Figure for the vacuum test (Fig. 16a) is smoother and rather featureless. Figures 16b and 17b indicate that the surface of the wear scar is extremely smooth in the sliding direction after sliding in air and quite rough after sliding in vacuum.

Figures 18a and 18b are reproductions of the traces taken perpendicular to the sliding direction from the wear tracks of the rings tested in air and vacuum, respectively. The air track again indicates grooving and is much smoother locally compared to the vacuum track.

4.4. Microhardness measurements :

Microhardness values measured on and outside the wear tracks for the pin and ring specimens tested in air and vacuum have been compiled in Tables 5 and 6. The hardness values obtained from severe wear air tests

Fig. 16. a) Profilometer trace on pin wear track perpendicular to the sliding direction. Vacuum test.

Fig. 16. b) Profilometer trace on pin wear track in the sliding direction. Vacuum test.

Fig. 17. a) Profilometer trace on pin wear track perpendicular to the sliding direction. Air test.

Fig. 17. b) Profilometer trace on pin wear track in the sliding direction. Air test.

Fig. 18. a) Profilometer trace on ring wear track perpendicular to the sliding direction. Air test. Load 648 gms. Speed 5cm/sec.

Fig.18. Contd.

Fig. 18. b) Profilometer trace on ring wear track perpendicular to the sliding direction. Vacuum test. Load 648 gms. Speed 5 cm/sec.

Table 5. Microhardness data from tests in air

Testing Load gms	Off Ring Track (VHN)	On Ring Track (VHN)	Off Pin Track (VHN)	On Pin Track (VHN)
648	260	441	240	360
648	260	482	235	310
648	270	424	240	340
360	270	341	240	310
1100	265	510	248	380

Table 6. Microhardness data from tests in vacuum

Testing Load gms	Off Ring Track (VHN)	On Ring Track (VHN)	Off Pin Track (VHN)	On Pin Track (VHN)
648	260	1020	248	875
648	275	900	230	900
648	270	815	225	650
360	280	750	215	650

were around 450 VHN for the ring and around 350 VHN for the pin. The hardness values obtained from mild wear air tests were around 350 VHN for the ring and around 300 VHN for the ring. In vacuum however both the pin and ring surfaces had regions where very high hardness values were measured. The values recorded in Table 6 are not indicative of the whole surface but represent averages of the highest values recorded. In other words there were regions on the vacuum tracks where the hardness values measured were comparable to those obtained from air tests. For both the air and vacuum tracks the hardness values measured were higher when the test load was higher. The sliding speed did not seem to have any significant effect on hardness in the speed range tested.

4.5. Scanning Electron Microscopy :

Topographical information from the worn surfaces of the pin and the ring as well as information regarding the size and shape of the wear debris were obtained using the SEM. In the micrographs shown the sliding direction is indicated by an arrow.

Ring wear tracks : Figures 19 and 20 are micrographs of the ring wear track from tests done at loads of 360 gms and 560 gms, respectively, and at a sliding speed of 12 cm/sec. The wear rates associated with these tests were in the mild regime. The micrographs are quite similar and the wear surface is grooved in the sliding direction with the grooves themselves being quite smooth. Figure 21 is a micrograph of a ring wear track from a test done at a load of 648 gms and a speed of 12 cm/sec. The associated wear rate was in the severe range. In contrast to figures 19 and 20 the wear scar

**Fig.19. SEM micrograph of ring wear surface . Mild wear. Load 360 gms.
Speed 12cm/sec. Air.**

**Fig. 20. SEM micrograph of ring wear surface. Mild wear. Load 560 gms.
Speed 12 cm/sec. Air.**

Fig.21. SEM micrograph of ring wear surface . Severe wear.
Load 648 gms. Speed 12 cm/sec. Air.

Fig. 22. SEM micrograph of ring wear surface. Load 648 gms. Speed 7
cm/sec. Vacuum.

in Figure 21 is rougher and covered with material formed by lateral flow. Figures 22 and 23 are micrographs of typical wear scars formed on the ring after a test in vacuum at a load of 648 gms and a speed of 7cm/sec. The surface is rougher compared to the surfaces slid in air and is covered by plate-like layers which are sheared in the direction of sliding.

Pin wear tracks : Figures 24-26 show different magnifications of a pin wear track. The corresponding ring wear track is shown by Figure 20. At a low magnification the wear track appears rather smooth and featureless. Higher magnification micrographs reveal the surface to be covered by a porous and thin film which is probably oxide as the corresponding debris diffraction pattern contained only oxide lines.

Figures 27-29 are micrographs of the pin wear track at different magnifications with the corresponding ring track shown in Figure 21. The surface is quite similar to the ring wear surface . One notable exception is the presence of fine particles all over the surface. Figures 30 and 31 are micrographs at two different magnifications of typical wear tracks on the pin after sliding in vacuum. Figure 22 is the corresponding ring track micrograph. The surface is much rougher compared to the surfaces worn in air and there is an appreciable amount of shearing in the sliding direction.

Wear debris: Figures 32-34 are micrographs of tests conducted in air at loads of 648 gms, 1kg and 1.65 kg respectively. The sliding speed associated with the 648 gms test was 12 cm/sec and the speed for the other two loads was 24 cm/sec. In Figure 32 the debris particles can be seen to exhibit a wide size range with most of the larger particles being 10-20

Fig.23. SEM micrograph of ring wear surface . Load 648 gms. Speed 7 cm/sec. Vacuum.

Fig. 24. SEM micrograph of pin wear surface. Mild Wear.
Load 560 gms. Speed 12 cm/sec. Air.

Figs. 25 . Higher magnification of region shown in Fig. 24.

Fig. 26. Higher magnification of region shown in Fig. 25.

Fig.27. SEM micrograph of pin wear surface. Severe wear.
Load 648 gms. Air.

Fig. 28. Higher magnification of region shown in Fig.27.

Fig.29. Same region as shown in Fig.27. Higher magnification.

Fig. 30. SEM micrograph of pin wear track after sliding in vacuum. Load 648 gms. Speed 7 cm/sec.

Fig.31. Higher magnification of region shown in Fig.30.

Fig. 32. SEM micrograph of wear debris from a severe wear test.
Load 648 gms. Air.

Fig.33. SEM micrograph of wear debris from a severe wear test . Load 1.1 kg. Speed 24 cm/sec. Air.

Fig. 34. SEM micrograph of wear debris from a severe wear test. Load. 1.65 kg. Speed 24 cm/sec. Air.

Fig.35. SEM micrograph of wear debris from a wear test in vacuum. Load 648 gms. Speed 7 cm/sec.

Fig. 36. SEM micrograph of wear debris from a wear test in vacuum . Load 648 gms. Speed 7 cm/sec.

Fig.37. SEM micrograph of wear debris from a mild wear test.
Load 560 gms. Speed 12 cm/sec. Air.

microns (longest dimension). Charging effects associated with the smallest particles may be caused by either partial or complete oxidation. Inspection of Figures 33 and 34 shows an increase in the average size of the debris with load and a decrease in charging effects. Most of the particles possess a flake-like shape. Figures 35 and 36 are micrographs of typical debris particles from a test conducted in vacuum. For the vacuum tests no obvious variation in debris particle size with load was observed. The particles are flake-like with most of the particles being 100-150 microns in size (longest dimension). Figure 37 is a micrograph of the debris obtained from a mild wear test in air done at a load of 560 gms and a speed of 12 cms/sec. The debris size is extremely fine compared to the micrographs shown so far and charging effects indicate substantial oxidation. Examination of the debris in the transmission electron microscope provided more detailed information regarding the nature of the debris. This is presented in the next section.

4.6. Transmission Electron Microscopy :

Figure 38 is a diffraction pattern obtained from a debris particle produced during a severe wear test conducted in air at a load of 648 gms and a sliding speed of 12 cm/sec. The wear rate associated with the test was 1.5×10^{-7} cm³/cm for the ring and 1×10^{-7} cm³/cm for the pin. Figures 39 and 40 are the corresponding bright field and dark field micrographs, respectively. Indexing of the pattern reveals the debris particle to be composed of both α -Fe as well as an oxide which is either γ -Fe₂O₃ or Fe₃O₄. The dark field micrograph was taken with part of the (110) ring of

Fig. 38. Diffraction pattern of debris from a severe wear air test. Load 648 gms. Speed 12 cm/sec.

Fig. 39. Bright field micrograph corresponding to Fig.38.

Fig. 40. Dark field micrograph corresponding to Fig. 38.

Fig. 41. Bright field micrograph from debris produced during severe wear in air. Load 648 gms. Speed 12 cm/sec.

Fig. 42. Diffraction pattern of debris from a wear test in vacuum. Load 648 gms. Speed 7 cm/sec.

α -Fe. Based on the fact that only the grains diffracting into that part of the ring will appear bright in the micrograph, the grain size was estimated to be in the range of 10-50 nanometers. Figure 41 is a bright field micrograph of two typical debris particles with a particle size of 1-2 microns. The oxide rings have a lower intensity than the α -Fe rings.

Figures 42-44 are the diffraction pattern and the corresponding bright field and dark field micrographs obtained from a debris particle produced during a test in vacuum. Indexing of the particle reveals it to be composed of the same phases as those obtained from a severe wear experiment in air. However, the rings in Fig.42 are more spotty.

Figures 45-47 are the diffraction pattern and corresponding bright field and dark field micrographs obtained from a debris particle produced during a mild wear run in air. Indexing of the pattern reveals the particles to be composed entirely of α -Fe₂O₃. From the dark field micrograph the grain size is estimated to be 10 nm. Figure 48 is a bright field micrograph of a number of particles from which the particle size was estimated to be in the range of 0.05-0.3 microns.

4.7. Longitudinal cross-sectional analysis :

Figures 49 and 50 are optical micrographs of two ring specimens which were tested in air and vacuum, respectively, at a load of 648 gms and a speed of 12cm /sec. The top part of each micrograph (black) is the Bakelite in which the sample was mounted. The interface between the Bakelite and the microstructure is the sliding interface. The micrograph of the sample slid in vacuum shows extensive shearing of the grains in the

Fig. 43. Bright field micrograph corresponding to Fig. 42.

Fig. 44. Dark field micrograph corresponding to Fig. 42.

Fig. 45. Diffraction pattern of debris from a mild wear experiment in air.
Load 560 gms. Speed 12 cm/sec.

Fig. 46. Bright field micrograph corresponding to Fig.45.

Fig. 47. Dark field micrograph corresponding to Fig. 45.

**Fig. 48. Bright field micrograph of typical debris particles from mild wear.
Load 560 gms. Speed 12 cm/sec.**

Fig. 49. Optical micrograph of a longitudinal cross section through the wear track on a ring specimen after a severe wear test in air. Load 648 gms. Speed 12 cm/sec.

Fig. 50. Optical micrograph of a longitudinal cross section through the wear track on a ring specimen after a wear test in vacuum. Load 648 gms. Speed 7 cm/sec.

sliding direction whereas the sample slid in air does not show this feature. The thickness of the deformed region in Fig. 50 is 10-20 microns. In Fig.49 the thickness of the deformed layer is less than 2 microns.

4.8. Results from radioactive tracer experiments to detect transfer :

A number of experiments were performed with activated samples at different load-speed combinations and different sliding distances in both air and vacuum. Typical results from two tracer experiments, one performed in air and the other in vacuum are presented in the form of plots in Figures 52a-53d. These plots are representations of the gamma ray spectra recorded by the detector. The ordinate on the plots is the measure of the number of counts associated with gamma rays of a specific energy. The abscissa on the plots contains the channel numbers, with each channel number corresponding to a specific gamma ray energy value. The correspondence is established by obtaining a spectrum using a standard radioactive source which produces two peaks whose energies are known. As the relationship between channel number and energy is linear, two peaks of known energy are adequate to determine the energy value associated with each channel number. Figure 51 represents a calibration spectrum obtained by using a Na-22 standard gamma ray source. The two prominent peaks on the spectra correspond to 0.511 MeV and 1.274 MeV energy gamma rays.

Figure 52 is the spectrum obtained by placing an activated ring in front of the detector before a wear experiment. Gamma rays having an energy of 0.846 MeV coming from radioactive Co-56 give rise to the peak

indicated by an arrow in the figure. After having established the presence of the desired tracer isotope in the ring, the activated ring was used in a wear experiment in vacuum at a load of 648 gms and a sliding speed of 18 cm/sec for 2025 cycles. Figures 52b-52d are the spectra obtained by placing the worn ring, the wear debris and the unactivated worn pin, respectively, in front of the detector. As can be seen in the plots, the 0.846 MeV peak (indicated by an arrow) is present in Figures 52b and 52c but absent in 52d. The counting times associated with the spectra are indicated on the plots. The worn pin was counted considerably longer (20,000 secs compared to 1000 secs for the ring and the debris) to enable the detection of even a small amount of transferred radioactive material. Radioactive decay calculations associated with the wear experiment (presented in Appendix A) showed the total activity on the unworn activated ring to be accounted for in the worn activated ring and the wear debris.

Figures 53a-53d are the spectra associated with the activated unworn ring, the activated worn ring, the wear debris and the unactivated worn pin after a wear experiment in air. The test was performed at a load of 1100 gms and a speed of 10.6 cm/sec for 250 cycles. It can be seen from the plots that the 0.846 MeV peak (indicated by an arrow) is absent in the spectrum obtained from the pin but present in the debris and ring spectra.

Figures 54a-54b are the spectra associated with the activated ring and the unactivated pin after a wear test in air at a load of 648 gms and a sliding speed of 6 cm/sec for 200 cycles. This was the only case in which transfer was detected in the present work. Loosely adhering activated debris on the pin from the ring is a possible source of activity in this case. Figures 55a

and 55b are the spectra obtained from an activated pin and an unactivated ring after a wear test performed in air at a load of 648 gms and a sliding speed of 8 cm/sec for 500 cycles. No transfer was detected in this case.

Fig. 51. Calibration spectrum obtained using a Na-22 standard.

Fig. 52. Spectrum from the activated ring before wear testing.

Fig. 53. Spectrum from the activated ring after the wear test

Fig. 54. Spectrum from the debris after the wear test.

Fig. 55. Spectrum from the pin after the wear test.

Fig. 56. Spectrum from the activated ring before wear testing.

Fig. 57. Spectrum from the activated ring after the wear test.

Fig. 58. Spectrum from the debris after the wear test.

Fig. 59. Spectrum from the pin after the wear test.

Fig. 60. Spectrum from the ring before the wear test.

Fig. 61. Spectrum from the pin after the wear test.

Fig. 62. Spectrum from the pin before the wear test.

Fig. 63. Spectrum from the ring after the wear test.

CHAPTER V

DISCUSSION

The wear rate and friction data presented in chapter 4 reveal clear differences in the wear behaviour of the steels tested in air and in vacuum. The present chapter deals initially with a detailed discussion of the results presented in chapter 4 followed by a discussion of the mechanisms governing the wear behaviour of these steels under the different test conditions.

5.1. Topography of the worn surfaces :

Topographical information about the wear scars on the pin and the ring was obtained in the form of SEM micrographs (Figs. 19-32) and profilometer traces (Figs. 16-18). The scars on the ring from mild wear tests are grooved in the sliding direction, the grooves probably being formed by the ploughing of asperities on the pin. (Figs 19,20). The scar on the pin is covered with a thin porous layer (Figs. 25,26); it will be discussed later in greater detail. The severe wear scars on the pin and the ring are much rougher and are covered with layers of material formed by plastic flow in a direction lateral to that of sliding (Figs. 21,29). The pin wear scar in severe wear has many particles distributed over the surface. This observation is significant as it has an important bearing on the sliding distance dependent transition encountered during some of the severe wear tests. The

increased roughness and plastic deformation observed on the severe wear scars compared to the mild wear scars are consistent with the higher loads associated with the severe wear tests.

In contrast to the worn surfaces obtained from experiments performed in air the surfaces obtained from vacuum tests are much rougher and are characterized by plate-like layers which are sheared in the direction of sliding. The surface also has some crack-like features running perpendicular to the sliding direction (Figs. 22,31). The surface is similar to the worn surfaces obtained by Kato and Akagaki (58) in their experiments performed in the regime labeled ' adhesive wear ' by them. Adhesion is expected to play a much more significant role in experiments conducted in vacuum compared to those in air. The spikes associated with the friction traces obtained from vacuum experiments are larger compared to those obtained from air tests. This is an indication of higher instantaneous friction force. The increased friction should result in significant subsurface plastic deformation assuming, as was described in the literature review, that the frictional work equals the plastic work. This is consistent with the increased levels of subsurface plastic deformation visible in the longitudinal cross sections of the samples slid in vacuum compared to those in air (Figs.49,50). The surface topography of specimens slid in vacuum can thus be understood in terms of subsurface plastic flow resulting in the generation of plate-like layers .

5.2. Shape and size of debris:

SEM examination of debris particles produced during severe wear experiments in air indicates an increase in debris particle size with load. The size difference is more dramatic for tests conducted under the same load and speed conditions but in different environments (air and vacuum, Figs. 32 & 35). The wear rate associated with the vacuum test was lower. Also larger but fewer particles are produced in vacuum. A comparison of Figures 32-36 shows that increasing the load in air has an effect similar to that of conducting a test in vacuum but at a lower load. That is, the proportion of particles having a size and shape similar to those obtained from vacuum tests increases as the load is increased for air tests. The high-load tests were not conducted for the same duration as the low-load air tests or vacuum tests as the wear was quite severe and the testing apparatus was unstable because of vibration effects. Mild wear debris was too fine and charged too easily to be analysed in the SEM.

5.3. Transmission electron microscopy of debris:

The main aim of analysing the debris using TEM was to identify the phases present in the debris obtained from tests performed under different conditions. The debris particles for tests in air and from tests in vacuum are composed of α -Fe and Fe_3O_4 and/or γ - Fe_2O_3 . As indicated in Table 7 (76) this uncertainty in the identification of the oxide exists because the 'd' values associated with different oxides are similar. The absence of α - Fe_2O_3 in the debris from severe wear air tests and vacuum tests helps to simplify the identification.

TABLE 7.

Table showing crystallographic and diffraction data
for iron and some iron oxides.

<i>d</i> (Å)	Relative intensity	Possible identity	<i>d</i> (Å)	<i>hkl</i>	Relative intensity
4.68	46	$\left\{ \begin{array}{l} \text{Fe}_3\text{O}_4 \\ \gamma\text{-Fe}_2\text{O}_3 \end{array} \right.$	4.85	111	10
			4.82	110	5
3.68	86	$\alpha\text{-Fe}_2\text{O}_3$	3.68	110	70
2.95	49	$\left\{ \begin{array}{l} \text{Fe}_3\text{O}_4 \\ \gamma\text{-Fe}_2\text{O}_3 \end{array} \right.$	2.97	220	34
			2.95	220	42
2.69	100	$\alpha\text{-Fe}_2\text{O}_3$	2.69	211	100
2.52	214	$\left\{ \begin{array}{l} \alpha\text{-Fe}_2\text{O}_3 \\ \text{Fe}_3\text{O}_4 \\ \gamma\text{-Fe}_2\text{O}_3 \end{array} \right.$	2.52	110	80
			2.53	311	100
			2.52	311	100
2.19	32	$\alpha\text{-Fe}_2\text{O}_3$	2.20	210	70
2.13	69	FeO	2.15	200	100
2.13 } 10 } 2.02 }	*	$\left\{ \begin{array}{l} \text{Fe}_3\text{O}_4 \\ \gamma\text{-Fe}_2\text{O}_3 \\ \alpha\text{-Fe}_2\text{O}_3 \end{array} \right.$	2.10	400	34
			2.09	400	33
			2.07	200	10
2.02	81	$\alpha\text{-Fe}$	2.03	110	100
1.84	34	$\alpha\text{-Fe}_2\text{O}_3$	1.84	220	70
1.75	21	?	?	?	?
1.70	89	$\left\{ \begin{array}{l} \alpha\text{-Fe}_2\text{O}_3 \\ \gamma\text{-Fe}_2\text{O}_3 \\ \text{Fe}_3\text{O}_4 \end{array} \right.$	1.69	321	80
			1.70	422	16
			1.71	422	11
1.60	87	$\left\{ \begin{array}{l} \alpha\text{-Fe}_2\text{O}_3 \\ \gamma\text{-Fe}_2\text{O}_3 \\ \text{Fe}_3\text{O}_4 \end{array} \right.$	1.60	332	40
			1.61	511, 333	45
			1.61	511, 333	41
1.52	36	FeO	1.52	220	60
1.48	84	$\left\{ \begin{array}{l} \text{Fe}_3\text{O}_4 \\ \alpha\text{-Fe}_2\text{O}_3 \\ \gamma\text{-Fe}_2\text{O}_3 \end{array} \right.$	1.48	440	62
			1.48	310	70
			1.48	440	65
1.45	49	$\left\{ \begin{array}{l} \alpha\text{-Fe} \\ \alpha\text{-Fe}_2\text{O}_3 \end{array} \right.$	1.43	200	19
			1.45	331, 211	80

Time and temperature are two important factors to be considered in understanding oxidation phenomena. Ashby (22) has constructed a 'temperature map' (Fig. 64) which can be used to provide a rough estimate of the 'flash temperature' (instantaneous temperature of contacting asperities) and the average temperature at the sliding interface for the case of plain carbon steels sliding against each other. The map was constructed by assuming that all the frictional work is converted into heat which is then partitioned between the two samples. When the map is used to estimate the temperatures associated with the experiments discussed in the present work, the temperatures (both flash as well as surface) are well below the hundred degrees Celsius contour even for the highest load and speed combination. The map has superimposed on it an envelope of points corresponding to the load-speed combinations used in the present work. Thus, temperature effects due to frictional heating are not a significant factor in the present investigation.

The phases constituting the debris obtained from severe wear air experiments and vacuum tests can now be interpreted in light of the above discussion. By examining the intensities of the rings in the diffraction patterns (Figs. 38 and 42) it is clear that significant amounts of both α -Fe and Fe_3O_4 are present. Figures 38 and 42 correspond to diffraction patterns obtained from air and vacuum debris, respectively. Based on the TEM analysis it cannot be said with certainty whether the debris was produced in a partially oxidized state (i.e., from a partially oxidized surface) or whether it was oxidized after being generated in a metallic state. The steel samples used in the experiments had a thin layer of oxide on their surface

Fig.64. Plot showing the flash and bulk temperature rise for carbon steels during sliding as a function of the load and sliding speed. (Ref.22). Shaded area shows region studied in the present investigation.

to begin with since even at very low temperatures (boiling point of nitrogen) an iron surface oxidizes very quickly. When the oxide film reaches 2 nanometers oxidation virtually ceases (77). This thin film is expected to be removed during the first few cycles of sliding, allowing metal to metal contact, but the surfaces can re-oxidize even in the vacuum tests. The temperatures at the sliding interface are close to room temperature and the partial pressure of oxygen in the vacuum chamber is quite low (0.4×10^{-5} Torr, 2.6×10^{-3} Pa). However, both the air and vacuum debris particles yield similar diffraction patterns. Moreover the oxide is not $\alpha\text{-Fe}_2\text{O}_3$, which is the thermodynamically favoured oxide at room temperature. Hence the debris particles may oxidize after their generation and prior to their analysis in the TEM. Boggs et al. (78) have investigated the oxidation of iron at pressures ranging from 0.01-100 Torr and at temperatures from 200-350 degrees Celsius and reported that hematite forms at room temperature by the oxidation of Fe_3O_4 , although at the lowest temperature studied Fe_3O_4 was the dominant oxide . In the absence of favourable kinetics, a coating of Fe_3O_4 on the debris particles obtained from severe wear air tests and from vacuum tests can thus be understood.

In contrast, the debris from mild wear experiments consists entirely of $\alpha\text{-Fe}_2\text{O}_3$, as indicated by the diffraction pattern (Fig.45). The temperature argument remains unchanged for the mild wear tests. The only difference is that the mild wear debris particles are a few tenths of a micron in size, in contrast to the severe wear and vacuum debris particles which are on the average more than ten microns in size, with some of them being as large as 100 microns. The small size associated with mild wear

debris allows complete oxidation to $\alpha\text{-Fe}_2\text{O}_3$. The generation of this small debris and its influence on the mild wear process is via the formation of the thin and porous layer which will be discussed in section 5.6.

In addition to providing information regarding the constituent phases of the debris particles, TEM observations also indicated the debris particles to be composed of nanocrystals. As reported in the previous chapter the crystallite size is extremely fine (1-2 nm) in the case of mild wear debris. It is much larger (50-100nm) in the case of vacuum debris which is consistent with the spottiness of the rings in the diffraction pattern obtained from a vacuum debris particle (Fig.42). However, some fine grains (< 10nm) can be detected in the bright field micrographs (Figs. 39, 43). The presence of nanocrystals on the wear surface and wear debris has been detected for a variety of sliding systems and a detailed account regarding their generation and properties can be found in ref. 79.

5.4. Transfer analysis:

The aim of analysing transfer in the present work was to study its role in wear rate transition behaviour. Sawa's work on dual phase steels (80) sliding against a Mo counterface had indicated correlations between transitions in friction coefficient and transfer (Fig. 65). At the start of the test the friction was low but after a few cycles of sliding the friction coefficient increased. This transition was associated with the cumulative effects of localized transfer events. Longer sliding distances were associated with a gradual decrease in friction with most of the surface

Fig.65. Plot showing the transitions in friction coefficient during sliding as a function of sliding distance. Vertical bars indicate range of friction fluctuations. (Ref.80).

being covered with a well-mixed layer of transfer material and base material.

As reported in the previous chapter, transfer did not play a significant role in the present work. Either no transfer occurred or transfer occurred but the amount was too small to be detected with the technique used. To detect transfer a γ -ray peak corresponding to Co-56 should be visible in the spectrum with significant counts above background. Counting times to obtain a significant peak with activated material were known experimentally. That is, the activated material was placed in front of the detector and within 1000 seconds a Co-56 peak of sufficient intensity was obtained. Based on this, the counting times necessary to detect 0.01% of the activated material as transfer material was estimated. The counting efficiency was further improved by placing the target closer to the detector. Moreover, the total activity was accounted for by the activity of the activated sample and the debris (see Appendix A). Therefore it is unlikely that a significant amount of transfer occurred under the test conditions.

5.5. Mechanics of contact:

Before proceeding to describe the possible mechanisms governing the wear of steels under the conditions tested, a brief review of the mechanics of contact between two sliding surfaces in the crossed cylinders geometry is provided.

In the case of the crossed cylinders geometry the initial contact for ideally rigid materials is at a point. However real materials deform elastically and contact occurs over a finite area which is given by:

A_n = apparent or nominal contact area = πab ; where; 'a' and 'b' are the major and minor axes of the ellipse of contact and their magnitudes depend on the radii, the Young's modulus, the Poisson's ratio of the cylinders and the applied load. Details regarding the derivation of the contact area can be found in reference 81. The nominal pressure over the contact area is then given by P/A_n , where P is the applied load. These expressions were used to locate the position associated with the testing conditions on Ashby's temperature and wear mechanism maps (see appendix B).

The expressions presented above would be valid if surfaces were perfectly smooth on a microscopic level. In reality, engineering surfaces consist of ' hills and valleys ', i.e., they are characterized by asperities, the shape and size of which depend on surface finishing processes. The real area of contact is then the sum of the areas over which the asperities on the two surfaces contact. Archard (82) has further shown that for metallic surfaces plastic deformation of the contacting asperities almost always occurs (unless the load is vanishingly small). The effect of increasing the load on a contact is to increase both the number as well as the area of contact associated with individual asperities, although there is some debate regarding which of the two effects dominates. Table 8 from reference 41 is reproduced below to illustrate the effect of increasing the load on asperity contacts between two steel flats. This Table takes into account only the effects of normal loads. Tangential forces will also have an effect on the number and area of contacting asperities by influencing the formation and growth of contacting junctions.

Table 8.

Effect of load on the number and area of contacting asperities. (Ref. 41).

Load (kg)	a(microns)=average radius of individual contact areas	N=number of contacts
2	50	3
5	60	5
20	90	9
100	120	22
500	210	35

The next point to be considered in a discussion of the mechanics of contact between surfaces is the state of stress associated with contacting asperities. Under stationary conditions a loaded asperity contact is associated with compressive and shear stresses which vary in magnitude with increasing distance from the point of contact into the subsurface. The compressive stress peaks at the surface and the shearing stress at some distance below the surface (81). Under conditions of sliding, frictional forces influence the stress distribution. The addition of a frictional force to a normal force on a contact causes the location of the point associated with the maximum value of the shearing stress to move closer to the contacting surface and also increases its magnitude. For values of the friction coefficient larger than 0.3 the maximum shearing stress is at the surface (81). The presence of

the frictional force also increases the magnitude of the maximum value of the largest principal compressive stress and changes the nature of the other two principal stresses from compressive to tensile in the region immediately behind the contacting region.

A final point to be made with respect to the mechanics of contact between surfaces, and one which bears special significance to the present work, is that as sliding commences and wear occurs the surfaces conform better and the area of contact between them increases. This area increase can reduce the stress borne by each asperity as the load is kept constant for a given run.

5.6. Severe and mild wear behaviour in air:

As mentioned at the start of this thesis, the term severe wear is used when the wear rates are in the range of 10^{-7} - 10^{-6} cm³/cm (10^{-5} - 10^{-4} mm³/mm) and mild wear to denote wear rates in the range 10^{-8} - 10^{-10} cm³/cm (10^{-6} - 10^{-8} mm³/mm). This definition is precise in contrast to a qualitative description involving the characterization of severe and mild wear in terms of the worn surface roughness and the *apparent* state of oxidation of the debris. This definition is particularly significant when one takes into account the results of the wear tests performed in vacuum in this work. The size and shiny appearance of the debris together with the rough worn surfaces produced in vacuum indicate severe behaviour in terms of the qualitative definition quite commonly used in wear literature. However, the wear rates associated with these tests do not warrant the label severe

wear. Consequently the wear behaviour in vacuum will be discussed separately.

Severe wear was observed only in air at loads of 648 gms and above. In some of the tests conducted at 648 gms the wear behaviour changed from severe to mild after a considerable distance of sliding. At the beginning of the experiment the cylinders come into contact over a small area. The effect of oxygen is interesting. Under similar load-speed conditions in vacuum only large debris particles (but fewer in number) are generated. Rao and Rigney (83) have observed increased wear rates and finer debris in wear tests performed with Ni₃Al samples in air compared to vacuum tests. Sawa (80) also observed increased wear rates in air compared to the rates in vacuum for AISI 1018 steels (Type B microstructure: ferrite islands encapsulated in a matrix of martensite). In both cases oxygen enhances fracture of either the surface material or the debris.

Mild wear was observed at loads of 180, 360 and 540 gms. TEM analysis indicated that the debris is composed of α -Fe₂O₃ particles which are about 0.1 microns in size. A severe to mild wear transition was also observed in some of the tests at 648 gms but only after 20,000 cycles of sliding. Even at the lower loads the initial sliding was rough as indicated by the friction trace. The beginning stage of mild wear is similar to severe wear as the contact area is small and contact pressures quite high even at the lower loads.

In view of the low flash temperature rise associated with the experimental conditions used in this work, surface oxidation of the wear tracks and debris particles should be more significant than the oxidation at

contacts due to frictional heating. Stott et al. (28) have estimated the ratio of the mass of oxide generated at asperity contacts to that generated over the entire apparent area of contact to be quite low (0.001-0.01) under low speed sliding conditions for some iron base alloys sliding against each other. A simple quantitative analysis to demonstrate the effect of particle size on the extent of oxidation (28) is presented below.

If t is the thickness of oxide on a metal debris particle of radius R then the volume of oxide on the particle is (to a first approximation) given by $4\pi R^2 t$. If V is the volume ratio of the metal-oxide system (the ratio of the volume of the oxide to that of the metal converted to oxide) then the ratio of the volume of metal consumed to that of the original metal is: $3 \cdot 4\pi R^2 t / 4\pi R^3 V$ or $3t/RV$. From this expression it can be deduced that as the debris particle size decreases the proportion of oxide increases. For iron or dilute iron alloys a limiting oxide thickness of 2 nm is achieved within 0.1s at room temperature (77). Thus debris particles 0.1-0.01 microns in size will have 6-60% of their mass converted to oxide within a fraction of a second at room temperature.

A plausible mechanism for the mild wear process will now be discussed. During the early stages of sliding, because of the small apparent contact area and consequent high stresses, relatively large debris particles are generated irrespective of the magnitude of the applied load. Some of the debris particles are trapped at the sliding interface or on the grooves formed on the pin. These particles undergo subsequent fracture and refracture. Figures 66 and 67 are SEM micrographs of the pin wear surface at two different magnifications after a test performed for approximately 8000

cycles at 648 gms in air . The micrographs show that the grooves on the track are filled with fine debris. Stott and Wood (31) have reported that development of compacted oxides on sliding metal and alloy surfaces can result in significant decreases in friction and wear rate. The development of the compact involves the formation, agglomeration and compaction of the oxide debris. In this present work the extremely fine size of the debris facilitates oxidation. This, combined with the high local pressures, can lead to the development of the oxide layer from debris trapped on the pin wear track. As the ring rotates the chances of debris falling off its surface is quite high. Moreover each portion of the ring wear track experiences the contact pressures only periodically as opposed to the pin wear track which is in continuous contact. Thus the thin and porous layer (Figs.25 & 26) is observable only on the pin surface. Both the layer and the friction trace associated with sliding on it are smooth. The wear rate is low because some of the fine debris can be recompacted into the layer.

During the early stages of sliding at low loads, the wear surface deformation and debris produced are on a smaller scale compared to higher loads. Thus, formation of extremely fine debris which can be completely oxidized can occur more readily. Moreover the grooves on the pin track are shallower and can be more easily filled leading to an earlier establishment of a compacted oxide layer. When the load is increased the debris particles are larger and so is the wear track roughness (deeper grooves). The wear scar size is also larger. These factors combine to delay the onset of mild wear.

Fig.66. SEM micrograph of pin wear surface. Severe wear.
Load 648 gms. Speed 8 cm/ sec. Air.

Fig. 67. Higher magnification of region shown in Fig.58.

In some of the tests performed at 648 gms mild wear was never observed even after 35,000 cycles of sliding. This might be either because not enough oxide debris is generated to cover the entire wear track or because the surface topography is not conducive to the formation of a compacted oxide. Iwabuchi et al.(32) have performed experiments in which $\alpha\text{Fe}_2\text{O}_3$ particles of different sizes were supplied to the sliding interface of steels of varying surface roughness. They observed that the most effective condition in terms of low mild wear rates corresponded to the smallest particle size of the oxide together with an initial surface roughness of twenty microns.

5.7. Wear behaviour of steels in vacuum :

The main features of the vacuum tests performed in this work were the increased debris particle size, surface roughness and subsurface deformation. The wear rates obtained were intermediate compared with the mild and severe wear rates in air. Larger debris particle size and a lower wear rate compared to severe wear in air suggest that the rate of production of debris is lower in vacuum. The wear surface topography in vacuum is characterized by plate-like layers and the surface does not readily accommodate the trapping and compaction of debris. Oxidation is also less significant in the vacuum tests than in air tests.

Debris formation in vacuum probably occurs by fracture of the highly deformed near-surface material. The origin of cracks is not clear. One possibility is that cracks initiate at the surface at sites prepared by local plastic flow. Another possibility is the formation of a shear crack by

dislocation pile up at an obstacle (such as an inclusion or grain boundary) following plastic flow. This crack could then propagate under combined shear and compression. The relevant fracture toughness parameter is K_{IIc} under superimposed compression and data for these conditions is scarce. The role of fatigue cannot be discounted. In vacuum tests the wear rate of the pin was lower than that of the ring (Tables 3 & 4). The ring specimen is subjected to cyclic stressing, so fatigue effects may be important. However, fatigue, if important, would occur under combined shear and compression, and relevant data for a quantitative analysis are not available.

5.8. Summary :

To summarise, the wear behaviour of the steels studied in the present work is strongly influenced by the environment. In vacuum there is higher friction and significant subsurface deformation. Wear is postulated to occur by crack formation and propagation in the highly deformed subsurface region.

In air, two types of behaviour were observed. At low loads the wear was mild and the debris was oxidized. The pin wear surface was covered with a smooth layer which is probably compacted oxide and the friction trace was extremely smooth. At high loads material removal occurs at a high rate. Ploughing by work hardened asperities and abrasion by loose debris particles trapped at the interface, combined with some type of embrittling effect by the oxygen from the environment may be responsible for this efficient process of material removal. The severe wear behaviour

observed in this work is similar to the ploughing wear described by Kato and Akagaki (59) in their investigation of the lubricated sliding of 0.45% C steel. In some cases the wear behaviour changed from severe to mild after a long period of sliding. Mild wear was associated with oxidized debris and a smooth friction trace.

CHAPTER VI

CONCLUSIONS

The main conclusions of this investigation are outlined below.

I. Radioactive tracer analysis did not indicate significant transfer in the load-speed range tested for either air or vacuum. Therefore, transfer is not important for the unlubricated sliding of carbon steels under the conditions used in these experiments.

II. Air tests:

1. Wear rate increased by about two orders of magnitude as the load was increased. Low wear rates and a smooth friction trace were obtained at low loads after a short (200-500 cycles) period of rough sliding. In some cases, low wear rates and smooth sliding were obtained at a high load after 20,000-25,000 cycles of sliding.

2. Wear debris associated with low wear rates was α -Fe₂O₃ and was a few tenths of a micron in size. The high wear rate debris was partially oxidized (Fe and Fe₃O₄) and the size was of the order of tens of microns. Severe wear debris exhibited a significant variation in size and the average size increased with load. TEM analysis of the debris from both severe and mild wear tests indicated a grain size in the nanocrystalline range.

3. The high wear rate wear tracks on the pin and ring are grooved in the direction of sliding and the mild wear track on the pin is covered with a compacted layer. The mild wear rate ring-track is grooved in the sliding direction. These grooves are shallower than the severe wear-track grooves on the ring.

4. Longitudinal cross sections from specimens tested in air did not indicate significant subsurface deformation.

III. Vacuum tests:

1. The wear rate in vacuum was between the high and low wear rates obtained in air. The friction trace in vacuum was noisy and the spikes were larger indicating higher instantaneous friction in vacuum. The wear rate of the pin was lower than that of the ring in vacuum. In contrast, the wear rates for the pin and the ring were similar in air.

2. The debris was substantially larger than those obtained from air tests. TEM diffraction patterns were similar to those obtained from severe wear air debris. Debris particles had a grain size in the nanocrystalline range, but the grain sizes were larger (40-50 nm) compared to those obtained in air (10-20 nm).

3. The wear tracks were rough compared to the air tracks and microhardness values in the range 800-1000 VHN were measured on parts of the wear tracks of both the pin and the ring.

4. Longitudinal cross sections of the samples tested in vacuum indicated extensive subsurface deformation.

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APPENDIX A

Decay calculations for radioactive tracer experiments :

Radioactive decay is governed by the exponential law : $N = N_0 e^{-\lambda t / \tau}$

where N_0 is the initial number of counts measured, and N is the counts measured after a time interval equal to t . λ is a constant and is equal to 0.693. τ is the half life of the radioactive isotope. Co- 56 has a half life of 77.3 days.

In the present work the peaks were counted using a computer program. The program evaluated the counts associated with each peak and the error involved with each count.

Sample calculation :

Counts measured on the 0.846 MeV peak in figure 52 was 7433 counts for a counting time of 1000 secs with an error of +/- 147 counts. This corresponds to the counts on the activated sample before wear testing. By the exponential law the counts measured on the sample after testing should be 6100 counts for 't' equal to 11 days, which was the time period between the two counts.

Counts measured on the 0.846 MeV peak (indicated by an arrow) in figure 53 was 4882. The counting time was 1000 secs and the error associated with the counts was +/- 112 counts. The amount of radioactivity missing on the ring is 1362 counts.

Counts measured on the 0.846 MeV peak in figure 54 (debris) was 1417 for a counting time of 1000 secs with an error of +/- 45 counts and this accounts for the residual activity.

APPENDIX B

Calculation of the nominal contact stress for the case of two cylinders made of the same material, and loaded normally in the crossed cylinders arrangement with their longitudinal axes perpendicular to each other (Ref.81) :

$$\text{Nominal contact stress} = \frac{P}{\pi ab} ; \dots\dots\dots A.1$$

where P is the applied load and 'a' and 'b' are the semi-major axis and semi-minor axis of the ellipse of contact respectively.

$$'b' = C_b (P \Delta)^{1/3} \dots\dots\dots A.2$$

$$\Delta = 2 (1 - \mu^2) / (A + B) E \dots\dots\dots A.3$$

$$\mu = 0.3 \text{ (Poisson's ratio)}$$

$$E = 30 * 10^6 \text{ .psi (Elastic modulus of steel)} \dots\dots\dots A.4$$

$$A = 0.5 [(1/R'_1 + 1/R_2)] \dots\dots\dots A.5$$

$$B = 0.5 [(1/R_1 + 1/R'_2)] \dots\dots\dots A.6$$

where R1, R'1, R2, R'2 are the radii of the curvatures of the two cylinders.

C_b is a constant and it is plotted as a function of B/A (81). For the dimensions of the cylinders used in the present work $C_b = 0.6$.

$'a' = b / k$; where 'k' is plotted as a function of B/A (81). For the dimensions of the cylinders used in the present work $'k' = 0.4$.