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FRICTION AND WEAR STUDY OF DISPERSED PHASE INTERMETALLIC COMPOUNDS IN FERROUS MATRICES

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M.S. thesis

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TABLE OF CONTENTS

FRICTION AND WEAR STUDY
OF DISPERSED PHASE INTERMETALLIC COMPOUNDS
IN FERROUS MATRICES

By

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I. ABSTRACT

The development of materials with improved wear resistance, and the implementation of current wear-reducing technology in design procedures, are important steps in conserving energy and materials.

The basic wear process is defined as the degradation and removal of material surface, and is explained in terms of adhesion, abrasion, fracture and delamination, and corrosion.

The interaction of wear mechanisms, and design strategies for reducing wear, stress the importance of understanding the effect of the environmental and loading conditions on the material removal process.

Using powder metallurgy fabrication processes, wear test samples consisting of a ferrous binder and an intermetallic dispersed phase were made.

The results of the wear tests led to the conceptual design of an improved wear test apparatus, and demonstrated the effectiveness of the intermetallic compounds as a dispersed phase in reducing wear rates.

II. INTRODUCTION

Although wear represents a basic mode of material failure, it is generally not studied to the extent that other material failure mechanisms, such as static overstress, gross plastic yielding, buckling fracture or creep failures, are studied. This is probably due to two reasons. First, wear is characterized by complex, interactive material mechaisms for which it is difficult to develop analytical models. Secondly, wear problems are usually solved on a case-by-case basis, where empirical evidence and trial and error methods have sufficed to bring down wear rates to a tolerable level.

The impetus for further studies in wear control comes from the need for greater conservation in energy and materials, and the need for materials which will withstand the extreme operating conditions of new energy systems.

Allen G. Gray, publisher of "Metal Progress" and technical director of the American Society for Metals commented in a recent editorial that...

> ...the unnecessarily short life of a piece of equipment or a product represents both a waste of materials and of the energy required in their production and manufacture.
>
> Belief in this concept appears to be gaining momentum. And wear control is one of the important ways to increase "useful life" that is receiving increased attention.
>
> Billions of dollars in materials, energy, and time are wasted each year because industry does not have established techniques for wear control or does not utilize available wear technology, emphasizes an ASME Advisory Board in its proposal for a "Wear Control Handbook".

> The need for greater understanding and use of wear control technology to extend the life and efficiency of products and equipment was given added support at a recent "Workshop on Wear Reduction", sponsored by the Congressional Office of Technology Assessment (OTA).
>
> Another workshop observation was that while product durability can be impacted by wear control, research is still required to determine techniques to control damage resulting from contamination, vibration, misalignment, and other similar causes.
>
> ...In our opinion, we should go all out with technology to achieve a quantum jump in durability for conservation and reduced cost.[1]

In line with this call for greater understanding and better implementation of wear control techniques, the U. S. Energy Research and Development Administration is planning to put more emphasis on the more complex material phenomena which require attention in developing new energy systems. Specifically mentioned is basic research aimed at uncovering the fundamentals of friction and wear in engineering systems.[2]

With these objectives in mind, a continuing program has been developed at the Materials and Molecular Research Division of the Lawrence Berkeley Laboratory to study the applicability of powder metallurgy forming methods in creating new wear-resistant materials.

This part of the program is concerned with perliminary testing and development of the experimental wear test apparatus, as well as background research into the basis understanding of wear mechanisms.

The background research into wear mechanisms is briefly recounted in the hope that it may be helpful as a guide in future alloy design.

Given an original material, and its own unique wear mechanism and wear rates for given loading conditions, an understanding of the fundamentals of wear processes should help in answering the question as to what material property is most crucial in improving wear resistance.

Depending on whether fracture toughness, fatigue strength, tensile strength, hardness, ductility, or work-hardenability is chosen as the property most likely to improve wear-resistance, different alloying additions and fabrication processes may be considered. Because of this choice, the analysis of wear mechanisms will be centered around the properties of wear resistant sintered materials, particularly those with hard dispersed phases.

During the course of the project, it became apparent that modification of the existing wear test apparatus needed to be considered. This conclusion necessarily led to the consideration of different types of wear tests, and to a determination of the characteristics of an acceptable experimental setup. Included in this report will be the decision criteria for a modified wear test apparatus, and design plans for the new machine.

Finally, the results of the wear test sample fabrication and the wear test results will be considered, with some suggestion about future avenues for profitable research in developing wear-resistant materials.

III. WEAR MECHANISMS

A. Definition

Wear may be defined as the removal or degradation of a material surface due to contact with another material surface.

Under this broad, general definition of wear lies the important wear mechanisms of adhesion, abrasion, fracture and delamination, corrosion wear, erosion and fretting fatigue.

In any type of wear situation, the dominant wear mechanism will be dictated as much by the contact pressure, relative surface speeds, and operating environment, as by the material properties of the two surfaces.

The specific focus of this report will be to deal mainly with the unlubricated mechanical interaction of two surfaces at moderate loads, speeds, and ambient operating conditions.

To be able to reduce wear, an understanding of the basic wear mechanisms and the material's interaction with the wear environment is essential.

B. Adhesive Wear

Adhesive wear involves the transfer of material from one surface to another, as well as the formation of adhesive wear fragments.

Ernest Rabinowicz,[3] a leading proponent of the adhesive theory of wear and friction, describes the mechanism of adhesion as follows:

> The tendency of contacting surfaces to adhere arises from the attractive forces which exist between the surface atoms of the two materials. If two surfaces are brought together and then separated, either normally or tangentially, these attractive forces act in such a way as to attempt to pull material from one surface onto the other. Whenever material is removed from its original surface in this way, an adhesive wear fragment is created.

That adhesion between metals may exist is well substantiated. For example, M. E. Sikorski[4] has written a paper correlating the tendency of pure metals to adhere with each other with other physical properties such as hardness, frictional characteristics, modulus of elasticity, work-hardening properties, and crystal structure. By defining the adhesion coefficient as the ratio of the loading force (both tangential and normal) to the subsequent breaking force (tensile), Sikorski finds that metals with large elastic moduli, high hardness, resistance to plastic flow, and a hexagonal close-packed crystal structure have low coefficients of adhesion.

The adhesion theory of wear has also been used to investigate and explain the wear resistance of low carbon and tool steels. The Swedish investigators S. Hogmark and O. Vingsbo,[5,6] characterizing adhesive wear as the most severe material-wasting wear mechanism, have presented a detailed explanation of the material deformation and deterioration in adhesive wear.

They refer to this wear mechanism as prow formation, and it involves substantial plastic deformation and work-hardening in the surface layer affected by the wear process.

There is the expectation that parts made by powder metallurgy have unique properties in resisting adhesive wear. E. Rabinowicz[7] states that "sintered metal compacts are frequently used as bearing materials; not only do the pores act as reserviors for lubricant, but the fact that each junction is small tends to prevent adhesive wear".

The smallness of the adhesive wear junctions refers to the porous nature of the sintered materials. When a sintered material is brought into contact with another material surface, and normal and tangential forces are applied, the real area of contact between the sintered surface and the other surface is less than what it would be for a wrought material under the same conditions.

In terms of prow formation, one would expect that the way the wear surface of a sintered material plastically deforms and work-hardens is dependent on the material's porosity, and hence the behavior of sintered materials should differ from those of more conventional cast and wrought materials.

The expectation that parts made by powder metallurgy will have superior resistance to adhesive wear is substantiated in recent experimental findings, which relate to the wear of sintered iron parts.[8,9]

Quantitative laws for adhesive wear exist, and are used by both the theoretician and the practical designer to predict adhesive wear rates.[10,11]

The basic premises of the quantitative relation is that the wear rate of two unlubricated metal surfaces is directly proportional to the load and the length of surface contact, and inversely proportional to the surface's hardness.

In equation form, the volumetric wear is:

$$V = \frac{CLx}{P}$$

where C is a material constant which depends on the cleanliness of the material surfaces, and other properties of the material.[12]

P = hardness L = load
x = length (ft) of contact (linear)
C = volumetric wear

The equation for adhesive wear is perhaps deceptively simple, since there has been considerable effort to give a conclusive theoretical justification for both the form of the equation and the value of C, the wear coefficient. For example, Rabinowicz has related C to the surface energy of the material and the probability that any material wear junction will lead to a transferred fragment.

Nevertheless, it seems that in practice that C, the coefficient of wear, is treated like an empirical constant, and is estimated more on the basis of previous experience with a particular wear environment than theoretical considerations.

It is interesting to note that in this quantitative relation no mention is made of the relative speed between the two surfaces, their surface roughness, etc. While it is definitely known that these are factors in wear, their effect is considered to be of secondary importance.

C. Abrasive Wear

Abrasive wear is a very common wear mechanism, and is related to such familiar metal-finishing processes as grinding, polishing, and machining.

Abrasive wear occurs when a hard, rough surface comes into sliding contact with a softer surface. The asperities of the harder surface indent and plow into the softer surface forming a series of grooves or troughs.

The material removed to produce these wear tracks may also contribute to the abrasive process. The removed material becomes present between the two surfaces as a loose grit, and, due to friction and the relative motion of the surfaces, is lodged into the harder surface and plows into the softer surface.

Abrasive wear in this way is often closely related to adhesive wear. Adhesive wear fragments may become loose from either surface, or the adhering material may form a prow, and cut into the softer material.

The material from the wear tracks and the adhesive wear fragments are harder than the parent surfaces due to the work-hardening, and/or, the oxidation of the fragments, and are therefore able to produce the micromachining effect of abrasive wear.

A straightforward concept of abrasive wear is that there must be a difference in hardness between the abrasive and the abraded material. If the hardnesses of the two surfaces are the same, there is little abrasive wear. In deriving a quantitative relation for abrasive wear it is noted that the volume of penetration of the asperity into the abraded surface is directly proportional to the load and inversely proportional to the hardness. Therefore, the quantitative relation for abrasive wear is similar to that for adhesive wear, namely:

$$V = \frac{\text{kabr } Lx}{P}$$

Where V = wear volume
L = load
x = distance traversed in wear system
P = hardness of softer material

kabr, the coefficient of abrasion, is a geometry surface factor which relates to the surface roughness of the harder material.[13] This quantitative relation, though very simple, correlates well with the observed data for abrasive wear.[14,15,16]

There has been some discussion about the hardness value to be used in measuring the abrasion resistance of a material. That is, whether the hardness value used should be a microscopic or bulk hardness value, and if a bulk hardness value, whether it should be the bulk hardness of an annealed or a fully work-hardened material.

R. C. D. Richardson[17] has found that the abrasive resistance of a material correlates best with the maximum surface hardness, a condition obtained through severe cold working.

For sintered materials it is thought that a microhardness value gives the best indication of abrasion resistance, because, by considering the microhardness, the effect of the material porosity is excluded.[18]

In general, when abrasive wear is expected, it can usually either be controlled or accurately predicted because of this wear mechanism's long time period for large surface deformations.

One way to avoid abrasive wear is to eliminate contaminants between two year surfaces.

Another well-known method to control abrasive wear is to have a very hard surface coming into contact with a much softer surface. Any unavoidable system contaminants, which would act as a loose, wearing grit, are forced into the softer metal matrix, become buried there, and the abrasive action of the particle is greatly reduced. In such a system, the bearing material is usually one-third or one-fifth as soft as the harder journal surface.

A third way to reduce abrasive wear is to use very hard, very smooth surfaces when unlubricated contact occurs.

This third method is limited in two ways. As the two surfaces become very smooth, the real area of contact increases greatly; and adbesive wear or "frictional welding" destroys the metal surfaces. As the materials become very hard, they also become more brittle, and are more susceptible to fatigue cracking or spalling. The method of using harder materials to control abrasive wear arrives at a point of diminishing return -- where an increase in hardness produces no increase in wear resistance, as large fractured fragments become present between the two surfaces.

In composite materials, which are generally produced by powder metallurgy, and where a hard dispersed phase is bound by a softer matrix material, the wear process in an abrasive environment involves at least three steps;[19]

(1) removal of the binder phase by attrition (the wear mechanism described in this paper as micromachining);

(2) abrasion and impact on partly exposed hard dispersed phase grains, and

(3) fracture and removal of the dispersed phase.

The consideration of abrasive wear naturally leads into the study of the importance of the fracture toughness of the wear surface, and the fracture and delamination theory of wear.

D. Fracture and Fatigue Wear and the Delamination Theory Of Wear

Fracture and fatigue wear as surface phenomena are closely related to the failure mechanisms of fracture and fatigue in bulk materials.

Fracture wear, like fracture in bulk material, is caused by the propagation of a defect through a brittle material.[20] Figure 1 illustrates the general mechanism of material removal. Stress in the material builds up as a result of the applied tangential force until the material suddenly fractures starting from the initiating crack near the tip of the asperity. A sizeable wear fragment is formed, and the sudden drop in the force resisting the relative motion of the two surfaces causes the surfaces to jerk.

Surface fatigue wear is primarily associated with crack initiation, and like bulk fatigue, is dependent upon the number of cyclic stress loadings, the magnitude of the stress amplitude, and is subject to statistical variations.[21]

Although considerable effort has been expended in recent years to improve the ductility of parts made by powder metallurgy, brittleness is still a dominant property of sintered metals, and brittle materials are most susceptible to surface fracture and fatigue wear.

Both ductile metals and brittle ceramics have wear rates which are inversely proportional to the material's hardness. However, for a given hardness level, the wear rate due to fracture of the ceramic material might be twenty times that for a ductile metal.[22]

Sintered metals, intermediate in hardness between ductile metals and brittle ceramics, have fracture and fatigue properties which are influenced by the sintered metals' inherent porosity and brittleness.

Experimental evidence supports the idea that porosity reduces the fatigue strength of sintered metals, but that the pores do not act as internal crack nucleation sites. The effect of the porosity is to reduce the cross-section through which a fatigue crack must propagate, and fatigue strength decreases linearly with increasing porosity. Evidently the stress concentration in the material due to its porous structure is not as important a factor as the stress concentration of the fatigue crack. Therefore, as with wrought materials, the fatigue crack in sintered materials starts from the external free surface.[23]

As in fatigue properties, the effect of porosity on a sintered metal is to reduce its fracture strength, and the reduction in fracture strength is related to the materials reduced cross-section.

From experimental evidence, it has been determined that the micromechanism of fracture is related to the coalesence of the sintered material's pores or microvoids.

The fracture stress draws out the walls of the pre-existing pores until the internal necking exceeds the shear strength of the material, and the neck fails, linking the microvoids together. Barnby, Ghosh and Dinsdal[24] report that in every case the fracture micromechanism was the simple ductile type for a range of sintered steels with copper additions and that no evidence was found of cleavage along the material's ferrite grain boundaries.

This fracture micromechanism in sintered materials leads to the conclusion that increasing the yield strength of the material also increases the fracture toughness. As yet, there is no known limiting yield stress for which this is not true, and there is the hope that through the improvement of the sintered material's mechanical properties, a high strength, high toughness material may be developed.

It is likewise expected that tough, strong, sintered materials would have increased wear resistance.

Although the exact correlation between the strength and fracture toughness of a sintered material and its wear resistance is not completely known, it is clear that the effect of inherent porosity does not preclude sintered materials from being highly wear resistant.

Related to the fatigue and fracture wear of a material is the delamination theory of wear put forth by Professor Nam P. Suh[25,26,27] and others at the Massachusetts Institute of Technology. The delamination theory of wear postulates that there is a thin "non-work-hardening" soft surface layer which deforms continuously due to the instability of dislocations near the surface. Each time the surface is cyclically loaded, the plastic strain and hence the dislocations accumulate at a critical distance below the surface, where a crack nucleates. Upon further loading, the cracks extend and propagate parallel to the wear surface, due to the tensile state of loading existing below the surface in the region behind the asperities of the opposing wear surface.

As a design strategy to reduce wear, the delamination theory of wear would suggest a thin plating of a soft material, well bonded to the harder substrate. The effectiveness of this approach has been demonstrated in unlubricated low speed sliding wear of metals in inert atmospheres.

It is currently unclear as to whether the delamination theory of wear explains the dominant wear mechanisms, in opposition to the adhesive wear theory, and how the delamination theory of wear relates to sintered materials.

However, one particular aspect of the delamination theory of wear, the microstructural effect of inclusions and second phase particles, relates directly to the wear of sintered materials, since most wear-resistant sintered parts rely on the addition of second phase particles for high temperature and large load wear resistance.

The wear resistance of a softer metal matrix is improved with the addition of hard second phase dispersed particles because the resulting higher hardness of the combination reduces the amount of surface deformation, and hence the wear of the surface. On the other hand, large dispersed phase particles may act as crack nucleation sites. One would expect a minimum wear rate when the amount and size of the dispersed phase particles have contributed to the hardness such that any increase dispersed phase alloying percentage contributes more to crack nucleation than to increased hardness.

The interface between the hard dispersed phase particles and the matrix acts as a crack nucleation site because as the matrix is deformed, elastic stresses build up around the particles, which do not deform as easily. When the stresses exceed the strength of the interface, the matrix and the particle separate, and a void or cavity in the material is formed.

Clearly the adhesion between the particle and the matrix is critical in dispersion-hardened sintered materials. It has been demonstrated in some iron-based alloys that increasing the cohesion between the matrix and particles increases not only the strength but also the ductility of the material.[28] This is clearly the desired direction in the alloy design of two phase wear-resistant materials.

E. Corrosion Wear

Corrosion wear is not really a wear mechanism in the same sence that adhesion, abrasion, fracture and delamination are because corrosion has more to do with the changing of the wear surface's mechanical properties than material removal.

Corrosion is included as a wear process though because the chemical reaction of the wear surface with its environment is often one of the most critical factors in determining the wear rate.

As an example, the wear process of cylinders in internal combustion engines is greatly increased by the condensation of sulphuric and sulphorous acids on the cylinder wall when the engine is cold. When the engine is turned on, rubbing between the piston rings and the cylinder walls removes the corrosion products and degrades the cylinder walls.[29]

In the wear of sintered metals it has been noted that the chemical transition of the wear surface causes the dominant wear mechanism to change. Under metal to metal sliding condition, the frictional heating of the surfaces causes oxides to form, and the hard, stable oxides form a wear-resistant barrier. The formation of this hardened surface is instrumental in reducing wear rates of sintered metals.[30]

As an example of particular interest, it was found in the unlubriacted frictional behavior of sintered iron at low sliding velocities that wear actually decreased with increasing loads, over a moderate load range, because at the higher loads the higher friction-produced temperatures aided in the formation of iron-oxides, which provided a thin, adherent, wear-resistive surface.[31] This has led to the steam heat-treating of sintered iron parts to improve their wear and corrosion resistance.[32]

Adhesion, abrasion, fracture toughness, fatigue and delamination, and corrosion represent basic material removal mechanisms, and wear in a given environment for specific materials is primarily governed by the action of one of these mechanisms, or a combination thereof.

Other types of material removal mechanisms such as erosion, cavitation, and fretting fatigue are not considered in depth both to limit the scope of the paper, since there has been extensive effort expended in describing these phenomena, and because erosion, cavitation and fretting fatigue are often described as wear processes resulting as a combination of the previously mentioned basic wear mechanisms.

Erosion is similar to abrasion at low angles of particle impingement, where the particle acts as a small machining tool. At high angles of particle impingement the impact strength and fracture toughness of the material surface dominate.

Cavitation is described as being similar to high angle erosion and surface fatigue wear.

Fretting fatigue involves subsurface crack propagation in its initial stage, and hence may be described in terms of the delamination theory of wear. This suggestion is strengthened by the fact that fretting fatigue is reduced by the presence of a solid film lubricant.[33]

IV. MATERIALS AND DESIGN FOR WEAR

Now that the basic wear mechanisms have been described, particularly in connection to the wear of sintered materials, there will be some discussion as how to apply this understanding of fundamental material removal processes. Discussion of design strategies to reduce wear will include consideration of the possible interaction of wear mechanisms, strategies to reduce wear, and some specific materials which reflect the current alloy design procedures in sintered materials to reduce wear.

A. Design for Wear

The interaction of wear mechanisms influences greatly the way the material removal processes proceed. In the wear of martensitic steels with precipitated carbides it was found that suitable low temperature wear properties do not necessarily imply good high temperature wear properties.

For the test apparatus at room temperature and low sliding velocities, martensitic steels with 7 and 15 percent undissolved carbides were shown to have better wear properties than a similar steel without the carbide additions because the undissolved carbides have a considerable hardening effect on the overall steel surface. However, at higher sliding velocities, (60 m/min.), where surface contact temperatures may exceed 400°C, the steels with and without carbides have similar hardnesses, but the wear mechanisms are different.

For the martensitic steel without the undissolved carbides, the dominant wear mechanism at high sliding velocities was corrosive wear. That is, the metal surfaces oxidized, suppressing the adhesive wear mechanism, and work-hardened asperities on one surface plowed into the corrosion products on the other surface.

For the martensitic steels with the undissolved carbides the wear situation is much more severe.

It was found that the surfaces of the steels with the carbides had less tendency to corrode. At the high contact temperatures and sliding speeds, no time was available to create a protective oxide layer, and typically severe adhesive wear resulted.

Furthermore, the degradation of the cementing metal matrix allowed the carbide particles to separate and become present between the two materials as abrasive grains. Therefore, the harder material at room temperature - the martensitic steel with undissolved carbides - showed a larger wear rate, under severe operating condition than that of the softer material at room temperature.[34]

Clearly implied in this example is the idea that the operating conditions of the wear surfaces must be well defined, if one is to make a suitable judgment as to the proper materials to use in a wear-resistant design.

In the wear of sintered metals, a similar effect has been found, which causes a marked transition in the wear rate depending on the load and relative speed between the two surfaces.[35]

The transition in wear rates is associated with a change in the dominant wear mechanism, from abrasive to adhesive wear.

Particularly in sintered ferrous matrices, the wear process can be classified into three distinctive types.

1. For low loads, the initial or running-in wear for smooth surfaces is typically low, and an oxide film develops rapidly. As noted before, the oxide film acts as a wear resistant barrier.
2. At moderate loads, with friction produced surface temperatures, generally less than 250°C, the wear rates decrease to a fairly stable level. During the running-in process wear rates are high, with adhesive wear between the metal surfaces. Metallic debris is produced, and adherent particles are transferred from the softer to the harder surface. After a relatively short sliding distance, though, an oxide film begins to form in patches, and a lower wear rate characteristic of the abrasive and fracture wear of the oxide film exists.

3. At higher loads, frictional heating raises the surface temperature. However, since the wear rate is so large that an oxide film does not have time to form, severe degradation of the metallic surface occurs, and the wear rate does not reduce to a lower level.

Figure 2 shows experimental results substantiating transitional wear behavior for sintered iron. Curves A and B are for wear specimens fabricated with different powder compacting pressures and sintering temperatures, and therefore, with different resulting porosities and mechanical properties.

In designing for controlled wear rates the adhesive wear load region should be identified to avoid the material-wasting of the machine part.

Another example of the interaction of wear mechanisms relates to the presence of hard dispersed phase compounds in a metallic binder. In the wear of tungsten carbide-cobalt alloys it has been noted that the state of stress at the interface between the particle and the matrix brought about by the differential contraction of the materials in being cooled from sintering temperature influences the fracture toughness and hence the fracture wear of the alloy.

"It is thought that the surface stress state is a sensitive function of local composition so that the removal of a small amount of cobalt from between the first layer of tungsten carbide grains results in a lowering of the activation energy for the propagation of surface cracks".[36]

Indigenous to the sliding wear of metals is the oscillation or variation of the friction force, and hence the amount of frictional heating and surface temperature. Also, in applications where the sliding loads are periodically applied, the temperature change and thermal gradient in the material have an important influence on the fracture wear rate. In the wear of tungsten carbide-cobalt alloys thermal fatigue failure produces cracks that penetrate deeply into the material and accelerates wear rates.[37]

It is expected then, that in the wear of sintered materials with hard, dispersed phase particles, that the surface temperature and thermal gradients in the material, and the state of stress at the particle and matrix interface due to differntial thermal expansion and contraction, will be important factors in the resulting wear rate of the material.

Once the operating and load conditions of the wear environment are known, so that the dominant wear mechanism and the interaction of the wear mechanisms can be predicted with some confidence, valuable use may be made of published, tabulated design experience with similar wear situations.

An example of this type of information is the "Tribology Handbook", edited by M. J. Neale.[38] This book is valuable to the researcher interested in wear resistant alloys because it identifies the range of load and speed conditions for sliding surfaces which lie outside the range of the use of conventional lubricants to reduce wear and friction. It also makes suggestions as to commercial materials in use, and therefore, sets a standard of wear-resistance which the researcher hopes to improve upon. While much of the data in the handbook is of a qualitative nature, the book represents a comprehensive effort to contain something about every aspect of friction, wear, and lubrication between two covers.

An article by the ASM Committee on Wear Resistance on the selection of wear-resisting steels represents a similar undertaking but of more restricted scope.

A more quantitative approach is taken by the "Handbook of Analytical Design for Wear". This work is based on an engineering model of the wear process which states that the amount of surface wear can be controlled by limiting the maximum shear stress in the region of contact.[39] That is, for a certain length of sliding contact, the amount of material removal and surface deformation of the contacting surfaces will be negligible if the maximum shear stress does not exceed a certain fraction of the yield point in shear.

This model is of particular interest because it was extended to include the wear of sintered metals.[40] It contains some interesting theoretical sections on the effect of surface asperities and wear debris in inducing stress concentrations in the wear surfaces, but because the model concentrates more on describing particular coefficients to be used in given situations, rather than more general principles, it has limited use.

B. Materials for Wear

Many types of commercial materials are available which are made specifically to reduce wear. Sintered materials have several unique advantages which make them a logical choice for wear-resistant applications.

Powder metallurgy techniques are useful in producing parts made of complex alloys and composites or dispersion strengthened materials, and materials of high hardnesses and resistance to deformation.

The differences in the densities of the alloying elements of these materials, the wide variation of the alloying element's melting temperatures and lack of mutual solubility can lead to problems in attempting to make a homogeneous composite of these elements by conventional casting or fusion techniques. Therefore, sintered materials have found widespread use in making wear-resistant components for machining tools, internal combustion engines, dies, rock cutters, and high-speed computer printing and memory retrieval devices.

These wear-resisting sintered materials may be classified into three types by the kind of second phase inclusions present in the binder material. The three kinds of inclusions are soft, sometimes intermetallic, phases, very hard carbide or oxide particles, and hard intermetallic compound phases.

For wear resistance at high temperature and large contact pressures, the use of hard carbide or oxide phases has dominated, with the use of hard intermetallic compound phases becoming more important.

The use of carbides and oxides as second phase inclusions in metallic binders is widespread and well studied.

At the outset of this project it was decided to investigate the potential of using hard intermetallic compounds, which were similar in structure to a family of commercially available materials called "Tribaloy" (manufactured by the DuPont Company).

These alloys are cobalt or nickel based, and depend upon the unique properties of a Laves phase (type $MgZn_2$ hexagonal) intermetallic compound for high wear resistance in a temperature range from near 0°C to

1,000°C.[41] The materials are specifically designed for dry or poorly lubricated wear surface boundary conditions.

The Tribaloy powders may be mixed with steel or iron, or other suitably compactible powders, to compress parts with a useful green strength. However, when the blend contains greater than 25 percent Tribaloy powder, the powders are not generally compactible, and the parts must be produced using the extrusion methods.

The attainment of close to theoretical density in the material is critical in acquiring a part with the maximum mechanical properties.

The introduction of the Laves phase intermetallic compound into the metal matrix is comparable to the addition of dispersed phase tungsten or titanium carbides, in that the presence of these additions is to provide a hard, anti-adhesive wear surface.

At a hardness of 1,200 HV (hardness Vickers), the intermetallic dispersed phase is harder than the hardest tool steel, about 1,000 HV, but considerably softer than the common wear-resistant additions of tungsten and titanium carbide, and aluminum oxide at about 2,000 HV.

Because tungsten and titanium carbide and aluminum oxide are at least 100 percent harder than the metallic binder or mating surface they might rub against, any irregularity in the harder surface will wear against the softer surface, producing substantial abrasive action. To avoid this condition, carbide and oxide hardened wear parts must be carefully machined and finished at high cost to avoid wear.

Because the intermetallic compounds are also harder than any steel or binder phase they would probably contact, some initial abrasive wear against the opposing surface would occur. But since the intermetallic compound is not too much harder than the mating surface it tends more readily to have its sharp corners removed by plastic deformation, and eventually, the abrasive action will cease, and the two surfaces will move with no tendency to adhere.[42,43]

Also, the intermetallic compound particles retain well their contiguity with the metallic binder under severe plastic deformation.[44] This suggests that they will not become separated from the parent surface under arduous wear conditions.

Parts made from Tibaloy intermetallic materials have demonstrated low friction coefficients and high wear resistance in a variety of wear environments.[45]

Clearly, any new material must be better or cheaper than the already existing competition. Because Tribaloy materials have the same basic structure as the proposed

Nickel-Titanium-Silicon compounds, the Tribaloy materials become a standard against which the new material will be judged.

V. PREPARATION OF WEAR SPECIMENS

One of the initial decisions of the project was to begin the fabrication and testing process using Tribaloy 400 in various metallic matrices.

The reasoning was that the preparation and wear testing of these specimens would allow a verification of the accuracy and reproducibility of the results from the wear test apparatus, and would uncover any possible problems in the fabrication of the blended powder parts using standard powder metallurgy processes.

Table I shows the elemental composition to Tribaloy 400, and some of its basic mechanical properties.

Initially, metallic binder matrices of nickel, 304L stainless steel, 316L stainless steel, and iron were considered.

Because of the cost of nickel powders and the complexity of the microstructure in stainless steels, the iron matrix became the prime candidate for additional study.

There were several reasons for choosing iron as the binder phase in the wear test experiments. The reasons included:

(1) Iron powders are inexpensive and readily available.

(2) Sintered iron is widely used for machine components.

(3) Much information is available on sintering and compaction methods for iron.

(4) Because iron is a simpler matrix microstructurally, the presence of the intermetallic Laves phase, and the Tribaloy particle diffusion band will be easier to determine.

(5) Comparative background information is available on the wear of sintered iron alloys.

One disadvantage of choosing iron as the matrix material is that iron is not widely used in high temperature and corrosive environments, which are wear situations of particular interest.

But because using iron would fulfill the main goals of the initial phase research, iron was used most extensively as the wear test sample matrix.

Ten wear test samples were prepared. Three of the samples were wrought materials. They were used in the wear tests to give some comparison between the wrought and sintered materials. The other seven samples were fabricated, machined, and polished using accepted powder metallurgy techniques.

The fabrication process for the sintered wear test samples began with the selection and mixing of the commercially available powders. Table II gives the manufacturer and size characteristics of the metal powders used.

Table III gives the composition of the ten wear samples and some pertinent details about their fabrication.

The powder blend of the 316L stainless steel and 20% Tribaloy 400 was mixed in a ball mill for two hours to homogenize the mixture.

The powder blends of the iron and Tribaloy powders were mixed for approximately twelve hours.

The powders were compacted in the isostatic press at 100 ksi (Fig. 3), and sintered in a vacuum furnace at the temperature and time given in Table III (see Fig. 4 for a schematic of the furnace).

The powders were compacted in a cylindrical rubber mold with inside dimensions of approximately 0.5 inch by 2 inches long. At the completion of the sintering phase, the specimens were no less than 0.4 inch in diameter.

The as-sintered specimens were cut, removing about 0.25 inch at each end. These ends were used as the sample to be polished and for a determination of the density of the material.

The remaining section was machined to a cylindrical form of:

$$\text{diameter} = .375 \text{ inch } {}^{+\,.000}_{-\,.005}$$

$$\text{length} = .85 \text{ inch } {}^{+\,.01}_{-\,.03}$$

A 0.125 inch hole was drilled 0.5 inch into one end of the material to allow the insertion of a thermocouple near the wear surface.

Table IV gives a summary of the resulting densities and hardnesses of the wear samples.

The densities of the sintered parts were adequate, and the porosities of the iron and Tribaloy alloys were comparable to those shown in DuPont Tribaloy literature for similar materials.[46,47]

The wear specimens were polished on one end to a 600 grit finish, and then were degreased in trichloroethylene.

It has been experimentally determined that the resistance to wear may depend upon the initial surface preparation. In order of decreasing wear resistance surface preparations are as-sintered, mechanically polished, and mechanically polished and etched.

The 600 grit finish for the specimens was chosen to insure similarity with wear tests conducted for DuPont.[49]

The wear disk against which the test wear samples were rubbed was of AISI 52100 bearing steel. The disk was fully hardened to a Rockwell C hardness of 60. After each experiment was completed, the disk was reground to approximately a 500 or 600 grit finish using a large grinding wheel.

Figures 5 and 6 show what the resulting polished and ground surfaces look like prior to wear testing.

The sections of the wear samples that were polished for examination of the microstructure were viewed using both optical and scanning electron microscopes.

Figures 7 and 8 show microstructures typical of the 316L stainless steel plus Tribaloy 400 intermetallic addition. The lighter phase which appears to stand out in relief has been positively identified as containing the intermetallic Laves phase.

Figures 9, 10 and 11 show microstructures of the iron plus 20% Tribaloy 400 material. The unetched Tribaloy particles are somewhat difficult to observe at the lower magnification, but are seen more clearly as the lighter phase at the higher magnification.

Figure 11 shows an attempt to determine the amount of diffusion of the iron atoms into the Tribaloy particle.

The white line, which unfortunately should be displaced about 0.75 inch downward, shows that path of an electron beam focused on the material to analyze the microstructure's constituents. The output of the analysis is superimposed on the figure and shows the relative concentration of the iron in the microstructure. The concentration of the iron is high in the matrix on the far right, and much less in the lighter Tribaloy particle. This means that there is a region within the material where the Laves phase does exist. This was a matter of some concern, as there was the possibility that the diffusion of iron into the Tribaloy particle would eliminate the unique Laves phase structure.

With the successful fabrication of the wear test samples containing Tribaloy, a major goal of the research was completed.

VI. WEAR TEST PROCEDURES AND RESULTS

A series of wear tests were completed with two objectives.

The first objective was to determine how the addition of Tribaloy powders would improve the wear resistance of the stainless steel and iron materials.

The second objective was to see whether or not the existing wear test apparatus was adequate for further experimentation.

The project began with a wear test apparatus which needed some improvements to reduce the vibration of the rotating wear disk, and to instrument the relative speed and friction coefficient between the two wear surfaces. Figure 12 shows an assembly drawing of the wear test apparatus.

The wear test parameters were chosen so that the test results would be comparable to those reported in DuPont's "Tribaloy Intermetallic Materials" Bulletin No. 2, in which iron matrices and Tribaloy 400 powders were also used. Therefore, the relative speed between the two wear surfaces was set at 400 feet per minute with a normal load of approximately 10 lbs., and a test duration of one hour.

The speed between the sliding surfaces was monitored using a light emitting diode, a photovoltaic cell, and voltage impulse counter which gave a read-out in RPM.

The load was transmitted through the upper shaft assembly.

The friction coefficient between the two rubbing surfaces was estimated using the output of strain gages attached near the base of the horizontal arm of the upper shaft assembly. The output of the strain gages was viewed on an oscilloscope, and the friction coefficient was determined using a calibration factor extracted from knowing what output from the strain gages was present from a known static load, and assuming linear proportionality between the load and the strain in the bar.

The temperature of the wear surfaces was to be estimated using a thermocouple embedded in the wear pin. The tip of the thermocouple was initially about 0.35 inch from the wear surface.

The wear rate was measured from the weight loss or gain of the pin and the disk, and the dimensional changes in both. A microscopic examination of the wear surfaces and wear debris was conducted to help determine what the dominant wear mechanism might be, and gain other pertinent information about the wear process.

All the tests were conducted in air with the wear test apparatus and materials beginning at room temperature, and with no control of the ambient humidity.

Table V gives a summary of the wear tests conducted using the test apparatus of Fig. 12.

An immediate observation from the wear tests is that while the friction coefficient changed slightly, the wear rates varied by orders of magnitude.

This type of general situation has been noted by other experimenters, stating that for most metal combinations, the friction coefficient varies between 0.5 to 1.5 while the wear rates vary by an order of magnitude.[50]

The wear rate of 36.2 milligrams for the iron plus 10% Tribaloy 400 combination in test 10 compares well with the wear rate for the same metal combination published in DuPont's literature, where 6.9 milligrams of weight loss was noted for a similar wear environment except that the contacting pressure was one-third that in test 10.[51]

The coefficients of friction obtained seem gratifyingly small, but serious questions exist about the validity of the calibration for the friction coefficient. An effort to estimate the friction force in the upper shaft bearing gave a friction quantity greater than the friction force between the rubbing surfaces.

The misalignment of the upper shaft also raised serious questions about the amount of load transmitted normal to the wear disk surface.

Another undesirable aspect of the wear test was the lack of control in setting the RPM of the wear disk.

During the course of the experiments it was necessary to adjust the power input to the electric motor driving the wear disk every ten minutes or so to maintain the relative speed between the sliding surfaces at the desired level.

With no adjustment, the frictional force, RPM, and temperature in the wear pin would vary over fairly large ranges with a period of oscillation on the order of three or four minutes. The RPM of the wear disk would vary by as much as 100 RPM, the temperature would cycle in a range of 20°C and the frictional output would change by a factor of two. Other experimenters have noted oscillations in the temperature of the wear surfaces and friction force,[52] but poor speed control is a typical characteristic of the AC-DC series wound motor used as the drive.

The wide variations in the wear rates noted in these experiments are not without precedent, but serious questions about the validity of the wear tests would encourage one to repeat the tests several times to be sure of the conclusions. Theoretically there is no particular reason to expect an order of magnitude change in wear rates for wrought 316L stainless steel and the sintered 316L as shwon in tests 5 and 6.

Parts made using the Tribaloy 400 powders do show improved wear rates, but not as much as had been hoped for.

Figure 13 shows the worn 304L stainless steel wear pin. The leading edge is to the left in the picture. The fringes or the "flowered" effect of the wear pin is caused by adhering material which has plastically flowed form the wear surface.

Figures 14 and 15 show that the sintered 316L stainless steel material wore in similar fashion. The micrographs of the worn surfaces show evidence of severe plastic deformation of the surface layer, with wear debris being caused by the exceeding of the plastic strain limit and/or significant adhesion with the opposing surface.

In contrast, Figures 16 and 17 show micrographs of the worn 316L stainless steel plus Tribaloy 400 surfaces. Some evidence of plowing by the opposing hard asperities is seen, but the dominant wear mechanism seem to be the cracking and fracture of surface platelets. Figure 17 shows a closeup of a fracture surface extending into the matrix.

The micrographs of the iron and the iron plus Tribaloy 400 surfaces after wear are more difficult to interpret. Figures 18 through 22 show typical wear surfaces.

The evidently more wear resistant darker area increases with increasing Tribaloy content.

A closer examination of the lighter areas (Fig. 23) shows a granular material, which appears to be small oxide particles. No evidence of plowing or grooving due to hardened asperities is seen in either area.

Typical worn surfaces of the AISI 52100 wear disk are seen in Figs. 24 and 25. Some type of surface deformation or smearing has taken place, but for all tests, weight and dimensional changes due to wear were not measurable for the hardened disk.

VII. CONCLUSION AND SUMMARY

The most immediate conclusion to be drawm from the wear experiments was the need for an improved experimental apparatus.

In order to have accurate, reproducible experiments, the driving speed needs to be closely controlled. At a relative sliding speed of 400 feet per minute, an accuracy of plus or minus five percent would be considered adequate.

Also, the load applied should be able to be determined within small limits, with variations in the friction force causing little deflection in the pin holder. In general, vibration should be avoided by incorporating heavy rigid structural supports and good bearings in the improved wear machine design.

Also, some provision should be made to insure that the friction coefficient can be measured with confidence.

Figure 26 shows a schematic diagram of the proposed wear test apparatus.

While the final design details for the fabrication of the machine have not been completed, the conceptual design for the machine should afford several advantages over the current apparatus.

The DC motor with the tachometer feedback control system is rated to control the speed within one percent of the set value.

The frictional output to the strain gage can be effectively determined. The friction force between the pin and the wear disk will be proportional to the strain in the horizontal arm, and the effect of torsion in the arm can be ignored with the proper strain gage orientation.

A compromise will need to be made between flexibility of the horizontal arm to insure adequate sensivity to strain for the strain gage output, and rigidity which will reduce vibration and maintain the perpendicularity between the vertical axis of the wear pin and the wear disk surface.

The horizontal bearing in the left hand side of Fig. 26 will allow the weight of the horizontal arm, the wear pin, and the wear pin holder to be counter-balanced, so that the weights added above the wear pin will be the only normal load on the wear surfaces.

The pin and disk type concept for the wear machine will continue to be used. This type of wear-machine has found widespread application, and two specific practical reasons for continuing with this type of machine are:

(1) The size requirements for both the pin and the wear disk are such that parts of the required size may be fabricated using facilities at the University of California Laboratories. This is not true for pin on ring experimental assemblies.

(2) The resulting wear surfaces are such that they can be easily examined on optical and scanning electron microscopes.

Ideally, more tests on the given wear samples would have been performed to confirm the initial trends in the wear-resistance data. But because of the lack of confidence in the data produced on the existing apparatus, the development of an improved machine was given the higher priority.

After so much discussion on types of wear mechanisms and material design strategies to reduce wear, some conclussion has to be expected on this subject.

Unfortunately, this type of conclusion for these tests must be based on intuition and circumstantial evidence, rather than on precise scientific determination.

The addition of the Tribaloy powders to the stainless steel matrix evidently changed the dominant wear mechanism from simple abrasion to a combined wear mechanism involving both abrasion and fracture wear.

The adhesion between the hard dispersed particles and the matrix would seem to be the key in improving wear resistance in this case.

The order of magnitude decrease in wear rate between the sintered stainless steel, and the stainless steel plus Tribaloy, as compared to the wrought stainless steel, could be an important find, but bears further investigation.

The wear of the sintered iron, and iron plus Tribaloy materials, as evidenced by the micrographs of these surfaces, is consistent with other published data previously mentioned in this report concerning the wear of sintered iron parts.

That is, the ability to resist wear in these parts is based on the formation of a hard, tough material layer at the surface.

Also, evidently some material transfer took place from the wear pin to the wear disk (see Fig. 23). This would imply that the wear surfaces are able to achieve conforming surfaces through material transfer without large adhesive wear rates. This is a critical factor in reducing wear.

The material combination of the iron matrix and the hard, dispersed phase, intermetallic compound seems to be excellent in wear resistance when in sliding contact with a hardened metal surface.

The improvement of the wear resistance of the iron matrix and intermetallic dispersed phase material would include steps to increase the tensile strength of the material, and a determination of the optimum amount of the second phase compound.

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TABLE I

Tribaloy 400

Elemental composition - Wt.%				Laves Phase
Co	Mo	Si	Cr	Vol. %
62	28	2	8	50

The Laves phase is present as a close-packed hexagonal compound of cobalt, molybdenum, and silicon.

Melting temperature:	2250-2900°F (1230-1590°C)
Density:	0.325 lb/in^3 (9.0 x 10^3 kg.m^3)
Ultimate tensile strength:	
At 70°F (21°C)	100,000 psi (690 MN/M^2)
At 1500°F (816°C)	90,000 psi (620 MN/M^2)
Hardness:	Laves phase - 1000-1200 HV Overall - Rockwell C 50-60

TABLE II

Metal Powder	Manufacturer	Composition	Lot #	Size
Stainless Steel 304L	Hoeganaes Corp.		12-21179	-325 mesh
Stainless Steel 316L	Hoeganaes Corp.		12-27229	-325 mesh
Tribaloy 400	DuPont Company	(Table I)	30	-325 mesh
Iron	EMP	99.5% Fe <0.01 C	1455	Grade 300 M

TABLE III

Sample	Compaction Pressure	Sintering Temperature	Sintering Time	Vacuum mm of Hg	Cooling Time	Furnace
SS 316L + 20% T-400	100 ksi	1130°C	50 min.	6×10^{-5}	120 min.	ABAR
SS 316L	100 ksi	1130°C	50 min.	6×10^{-5}	70 min.	ABAR
SS 304L	100 ksi	1135°C	45 min.	5×10^{-5}	---	ABAR
Fe	100 ksi	1150°C	60 min.	3×10^{-5}	100 min.	ABAR
Fe + 5% T-400	100 ksi	1150°C	60 min.	3×10^{-5}	100 min.	ABAR
Fe + 10% T-400	100 ksi	1150°C	60 min.	2×10^{-5}	60 min.	BREW
Fe + 20% T-400	100 ksi	1150°C	60 min.	2×10^{-5}	60 min.	BREW

AISI 1018
SS 316L
SS 304L
} Wrought Materials

SS = Stainless Steel
T = Tribaloy
Cooling Time = Time form sintering temperature to removal from oven.

TABLE IV

DENSITIES AND HARDNESSES OF WEAR SAMPLES

Material	Density (g/cm^3) Method 1	Density (g/cm^3) Method 2	Theoretical Density	% Theoretical Density	Hardness Rockwell B
SS 316L + 20% T-400	7.05	7.076	8.136	87	63
SS 316L	7.245	7.261	7.92	91	63
SS 304L	7.07	7.14	7.84	90	65
Fe	7.92	7.46	7.86	94	15
Fe + 5% T-400	7.30	7.39	7.92	92	20
Fe + 10% T-400	7.28	7.39	7.97	92	32
Fe + 20% T-400	7.11	7.17	8.09	88	68

AISI 1018	7.71	Wrought Materials
SS 316L	7.92	
SS 304 L	7.84	

Method 1 = Volume measurement by micrometer of the machined specimen.

Method 2 = Volume measurement by water displacment from a material sample taken from an end of the machined specimen.

TABLE V

WEAR TEST SUMMARY

Test No.	Pin Material	Friction Coefficient	Maximum °C Temperature	Pin Weight (Grams) Loss	Pin Height (Inches) Loss	Comments
1	SS 304L, sintered	.14	---	1.933 g	.162 in.	---
2	AISI 1018	.15	---	.081 g	.009 in.	---
3	SS 304L, wrought	.14	165°C	1.3925g	---	Thermocouple installed
4	AISI 1018	---	---	---	---	Checkout test
5	SS 316L, wrought	.13	150°C	1.3533	---	Plastic flow of pin
6	SS 316L, sintered	.11	135°C	.1565	.0135	---
7	SS 316L + 20% T-400	.11	135°C	.1055	.009	---
8	Fe, sintered	.14	110°C	.0546	.005	---
9	Fe + 5% T-400	.10	82°C	.0514	.004	Load = 5 lbs.
10	Fe + 10% T-400	.13	130°C	.0362	.004	---
11	Fe + 5% T-400	.13	135°C	.1126	.008	---
12	Fe, sintered	.14	130°C	.0471	.004	---
13	Fe + 20% T-400	.12	125°C	.0271	.004	---

Speed = 400 ft/min on the average.

Load = 10 lbs. unless otherwise noted.

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FIGURE CAPTIONS

Fig. 1: Fracture wear mechanism.

Fig. 2: Transitional wear behavior for sintered iron. (From Eyre and Walker[30].)

Curve A: Compacting pressure 45,000 psi ($309\ N/mm^2$), sintered at 1050°C.

Curve B: Compacting pressure 67,000 psi ($464\ N/mm^2$), sintered at 1200°C.

Fig. 3: Isostatic press.

Fig. 4: ABAR furnace.

Fig. 5: Ground surface of AISI 52100 wear disk. 400x.

Fig. 6: Polished surface of 316L + 20% T-400 wear pin. 400x. (600 grit finish.)

Fig. 7: Polished surface of 316L stainless steel plus 20% T-400. 160x.

Fig. 8: Polished surface of 316L stainless steel plus 20% T-400. 2000x.

Fig. 9: Polished surface of Fe + 20% T-400. 400x.

Fig. 10: Polished surface of Fe + 20% T-400. 200x.

Fig. 11: Polished surface of Fe + 20% T-400 with EDAX trace showing concentration of iron. 10,000x.

Fig. 12: Weat test apparatus.

Fig. 13: Wear pin with the leading edge on the left. 1x.

Fig. 14: Worn surface of 316L stainless steel pin. 200x.

Fig. 15: Worn surface of 316L stainless steel pin. 2000x.

Fig. 16: Worn surface of 316L stainless steel + 20% T-400. 100x.

Fig. 17: Worn surface of 316 L stainless steel + 20% T-400. 1000x.

Fig. 18: Worn surface of iron pin. 100x.

Fig. 19: Worn surface of iron pin + 5% T-400. 100x.

Fig. 20: Worn surface of iron + 10% T-400. 100x.

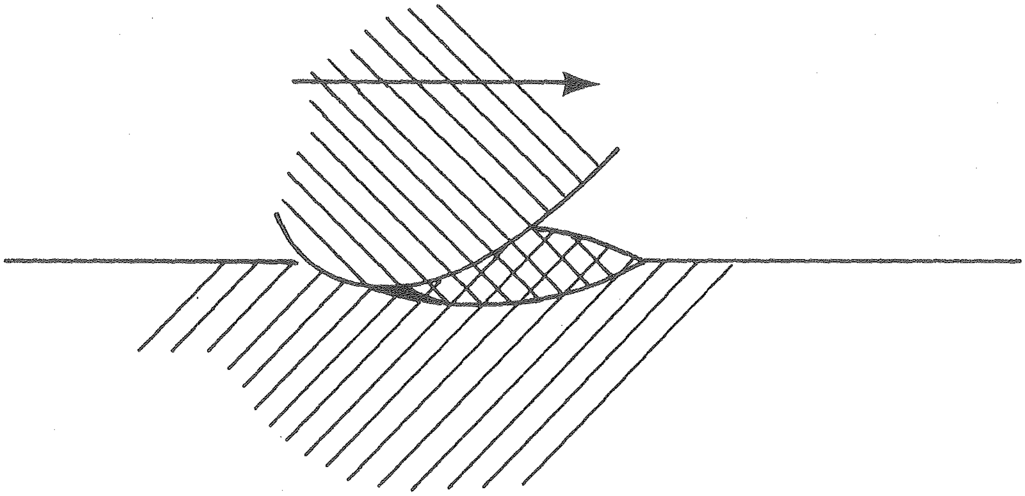
Fig. 21: Worn surface of iron + 20% T-400. 100x.

Fig. 22: Worn surface of Iron + 20% T-400. 100x.

Fig. 23: Worn surface of iron + 10% T-400. 1000x.

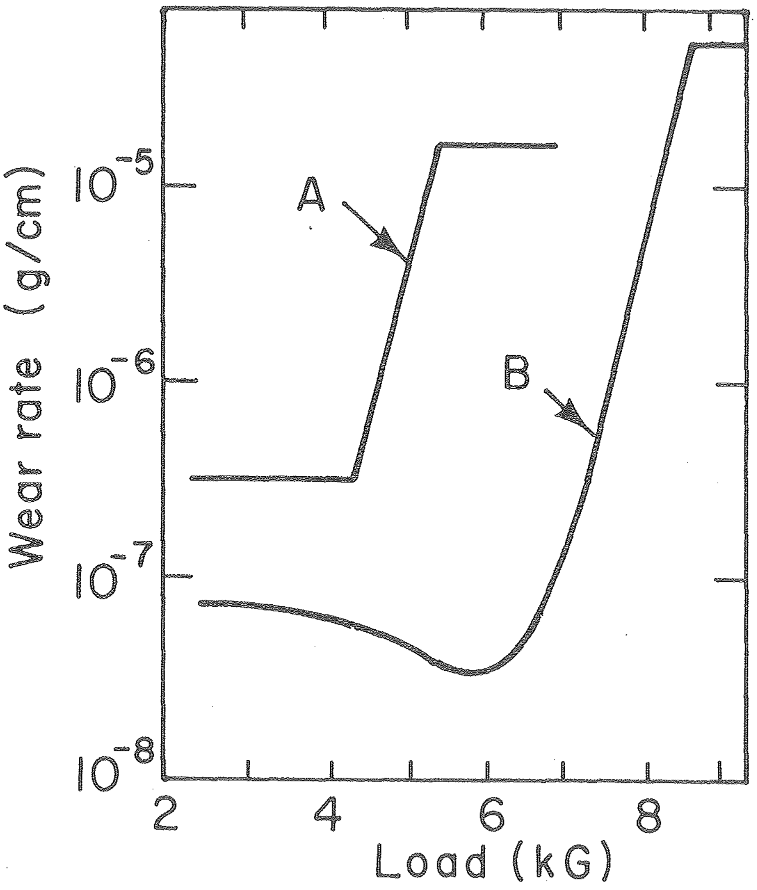
Fig. 24: Worn surface of AISI 52100 wear disk after test B. 100x.

Fig. 25: Worn surface of AISI 52100 wear disk after test 1. 100x.



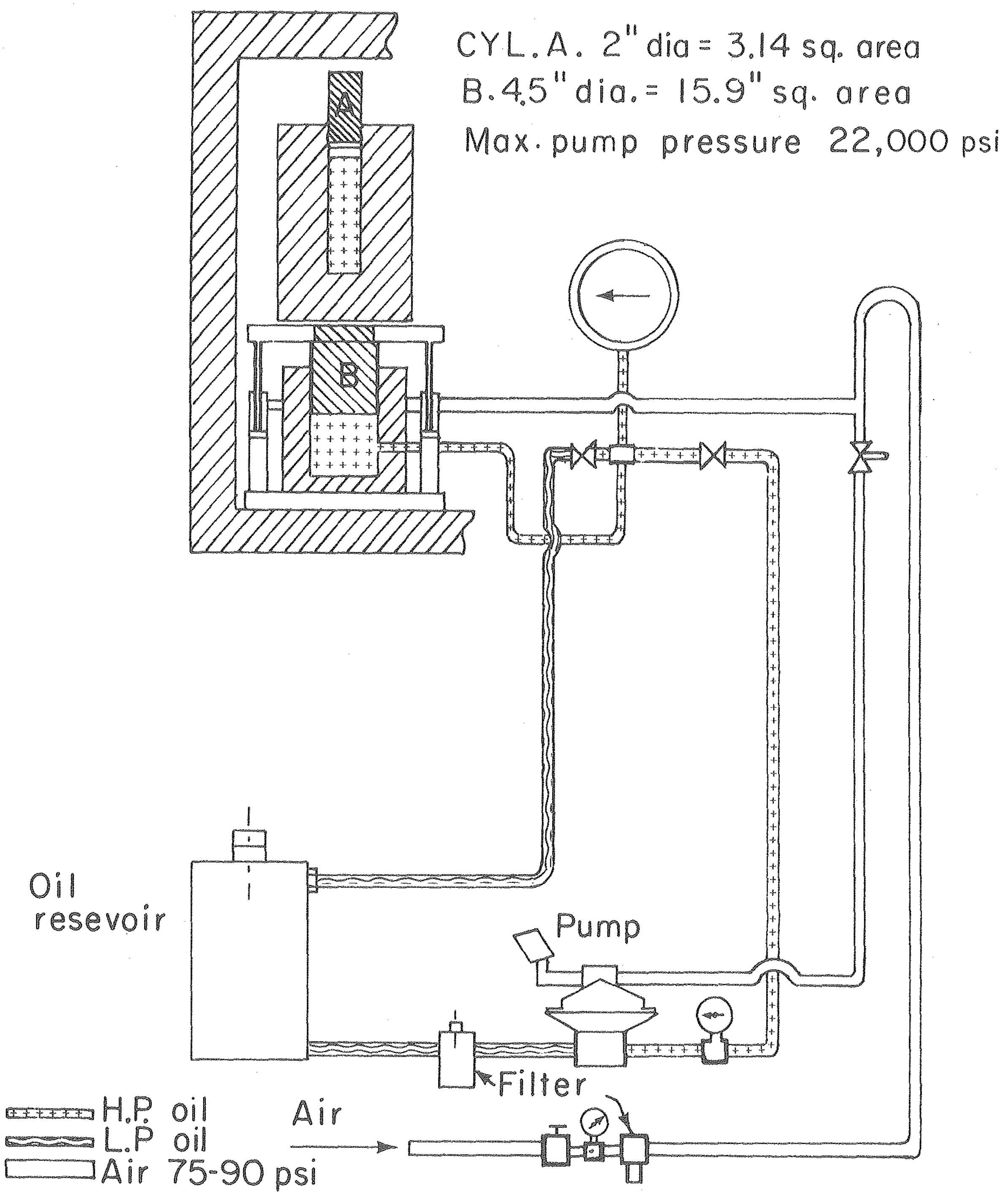
XBL772-286

Fig. 1



XBL 771-285

Fig. 2



Flow schematic isostatic press

XBL772-283

Fig. 3

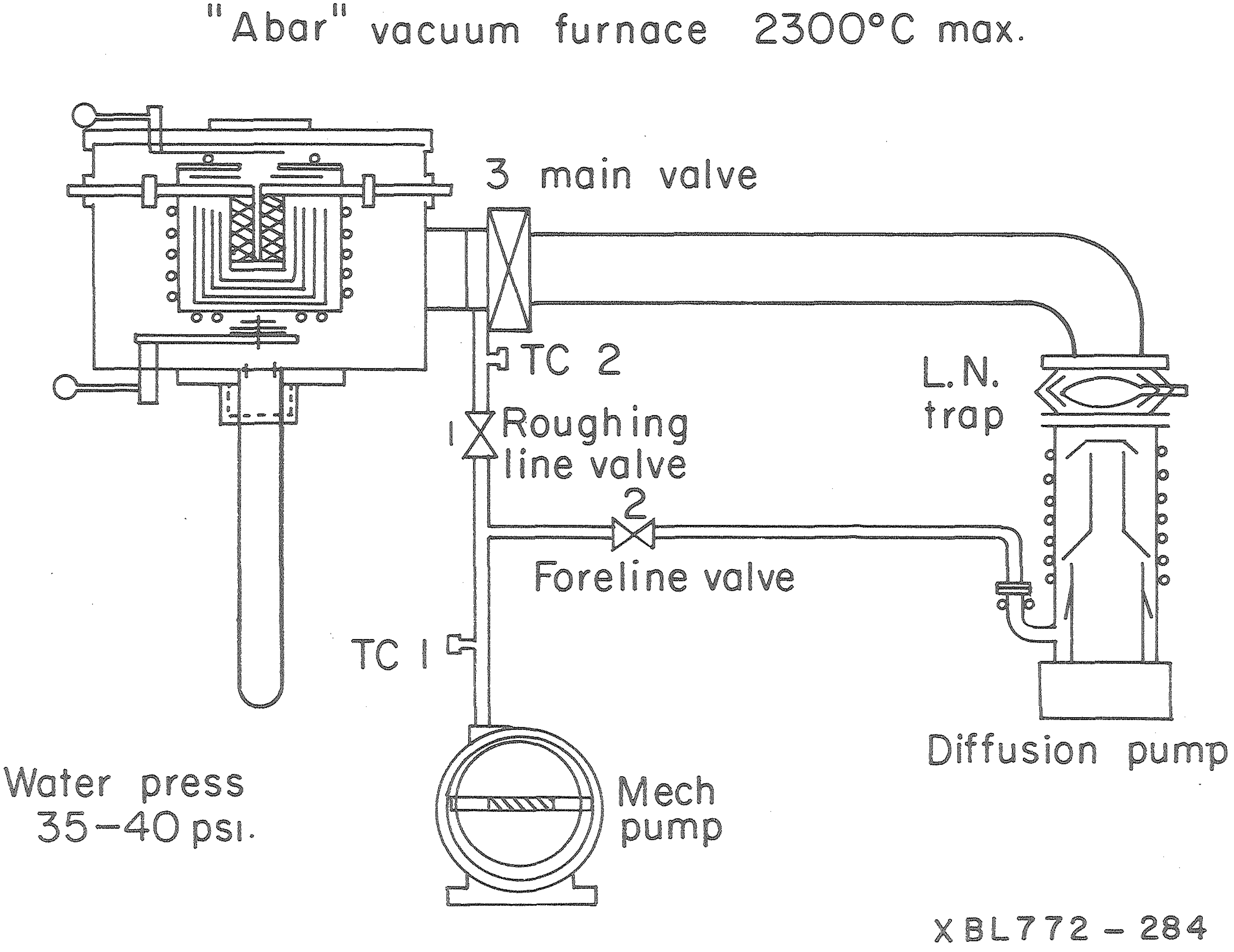

"Abar" vacuum furnace 2300°C max.
3 main valve
TC 2
1 Roughing line valve
2
Foreline valve
TC 1
L.N. trap
Diffusion pump
Water press 35–40 psi.
Mech pump
XBL772-284

Fig. 4

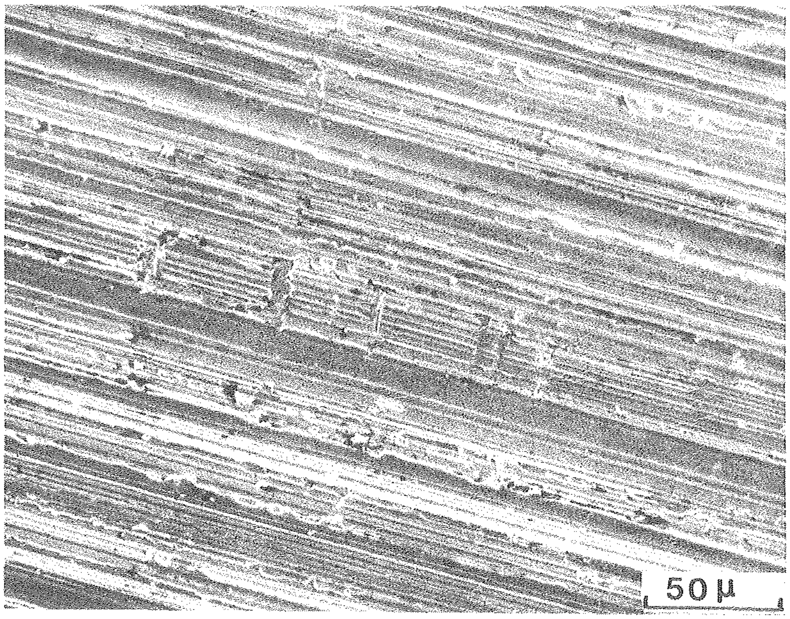
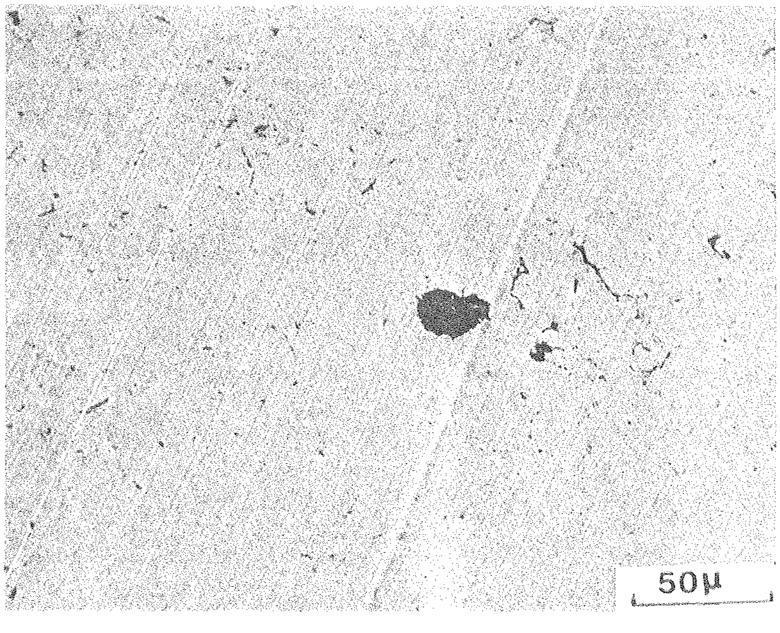


Fig. 5



XBB 773-1758

Fig. 6

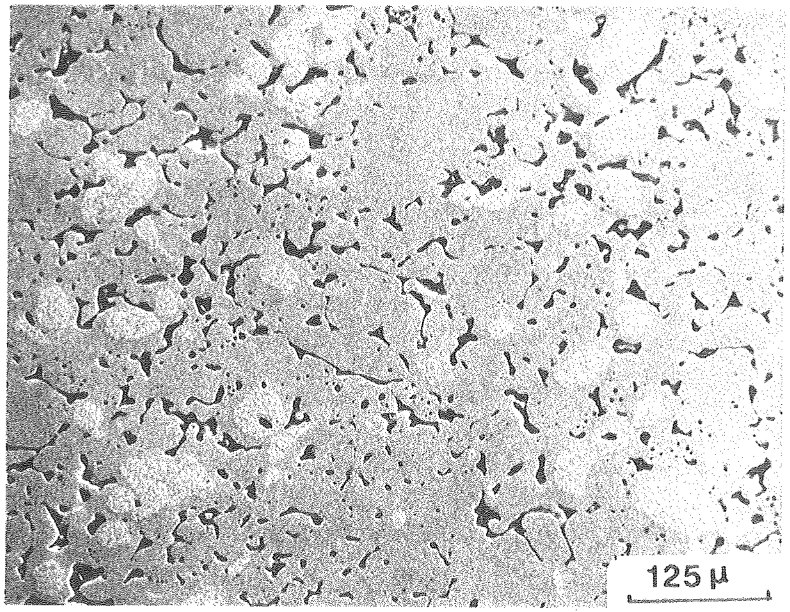
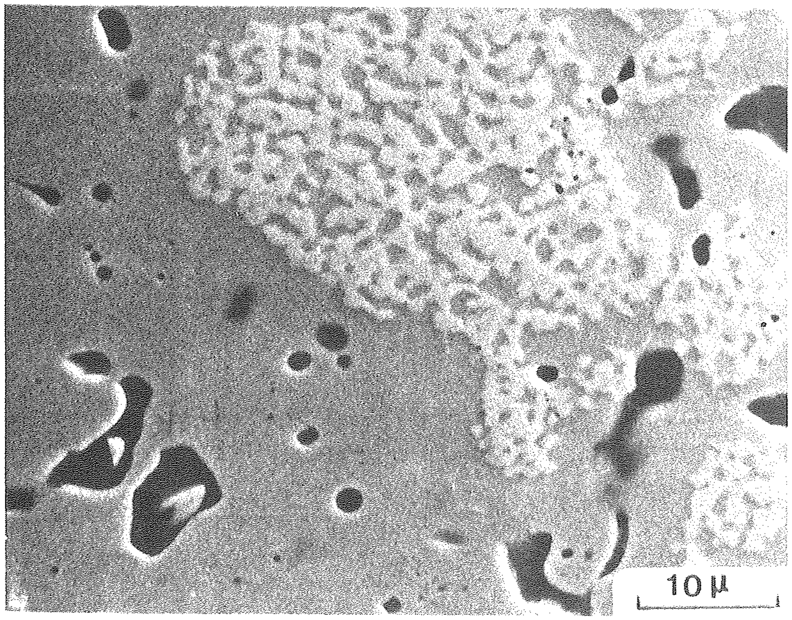


Fig. 7



XBB 773-1757

Fig. 8

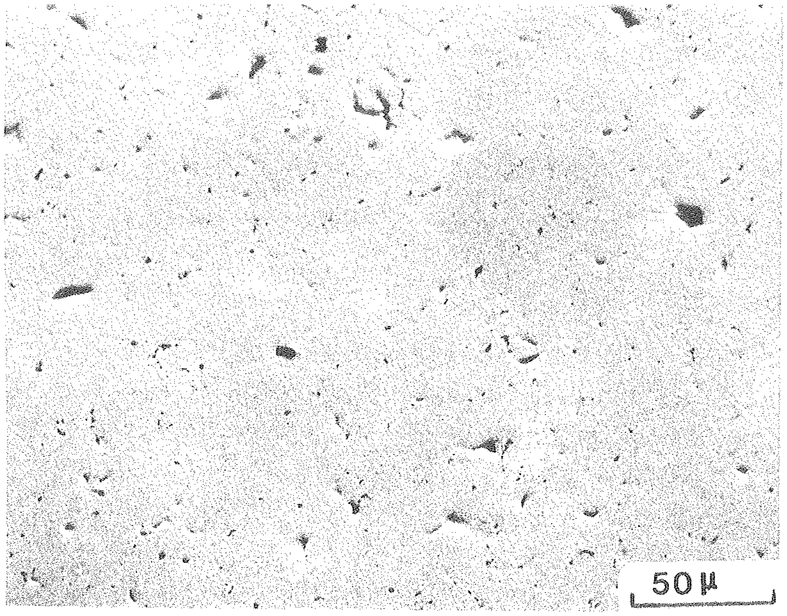
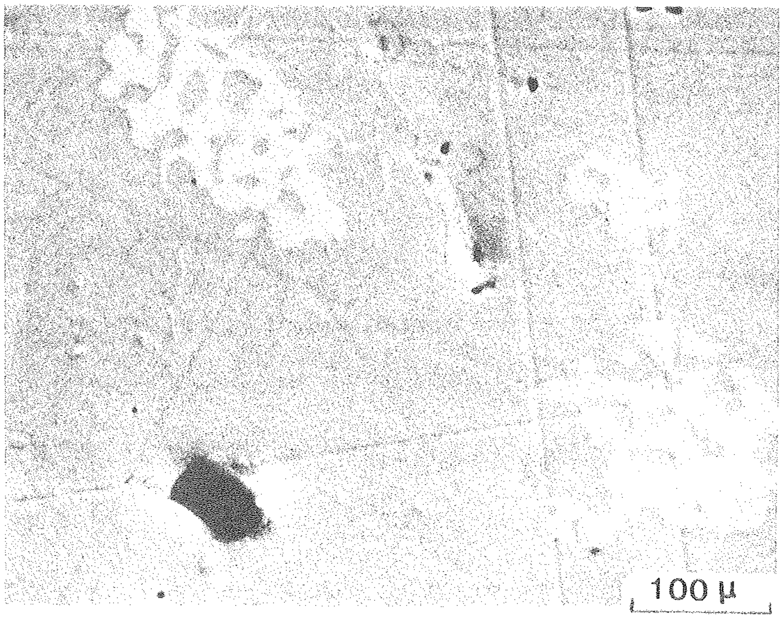
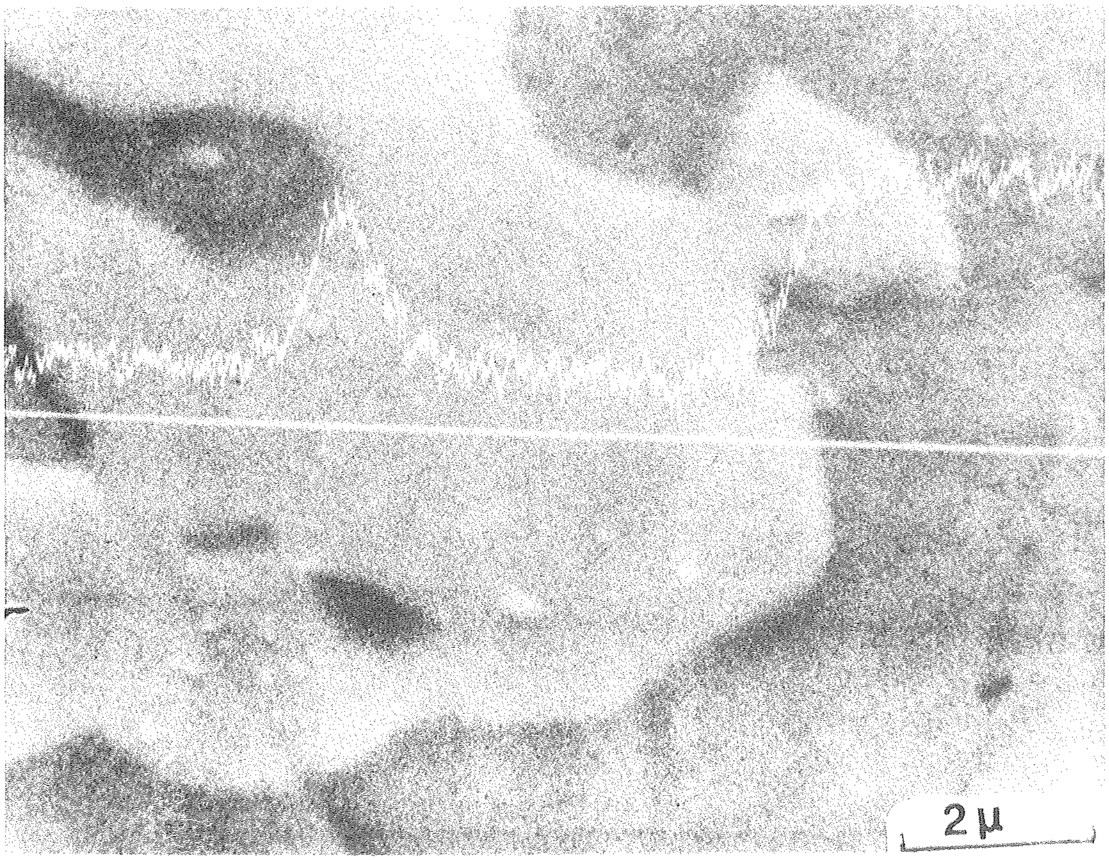


Fig. 9



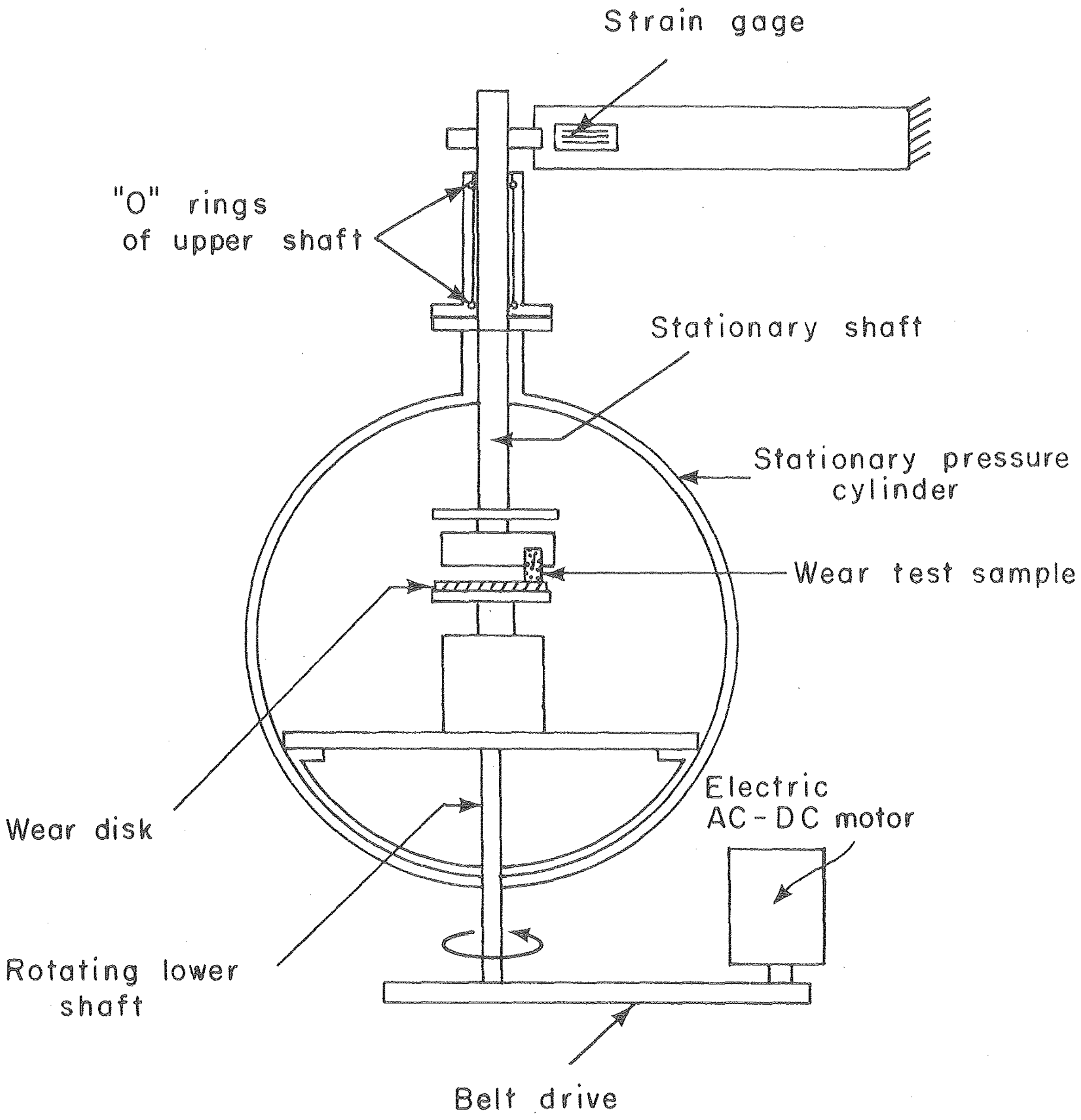
XBB 773-1756

Fig. 10



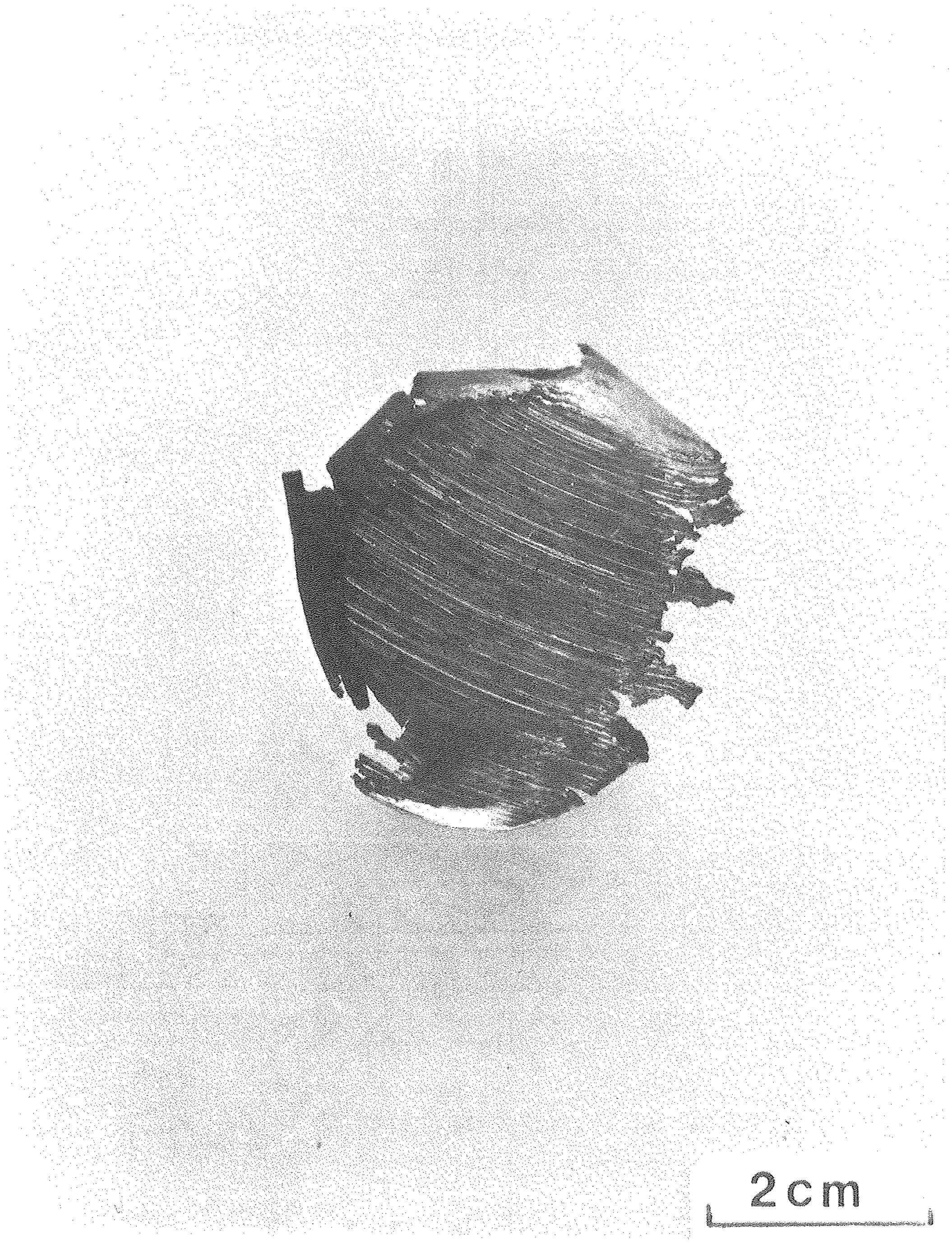
XBB 773-1759

Fig. 11



XBL 771-287

Fig. 12



XBB 773-1760

Fig. 13

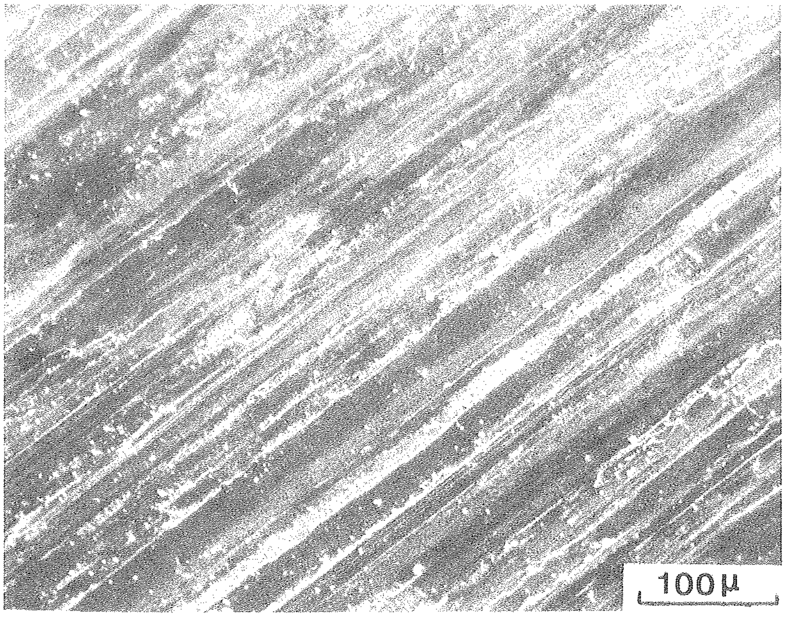
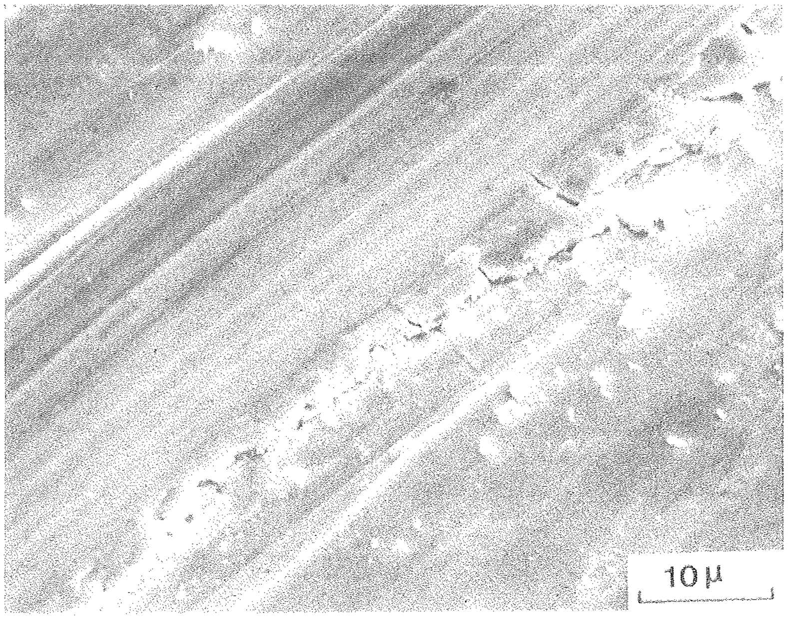


Fig. 14



XBB 773-1755

Fig. 15

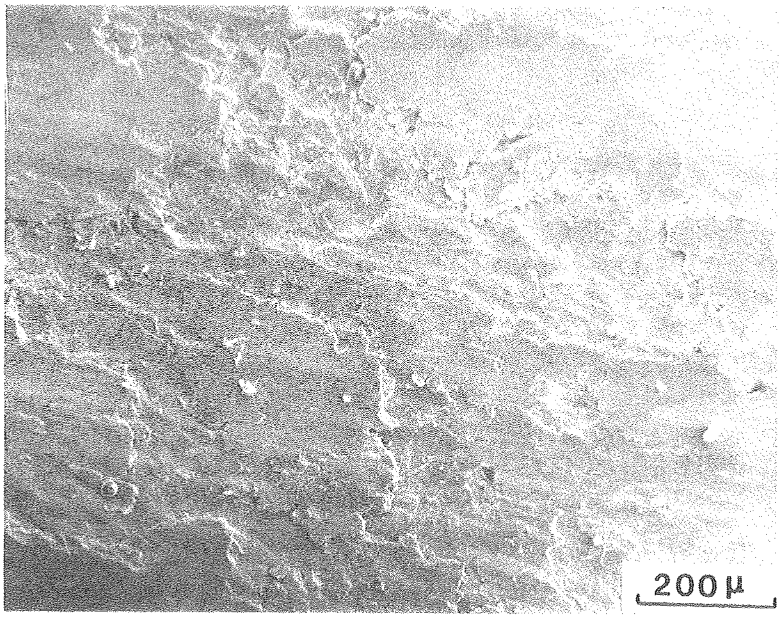
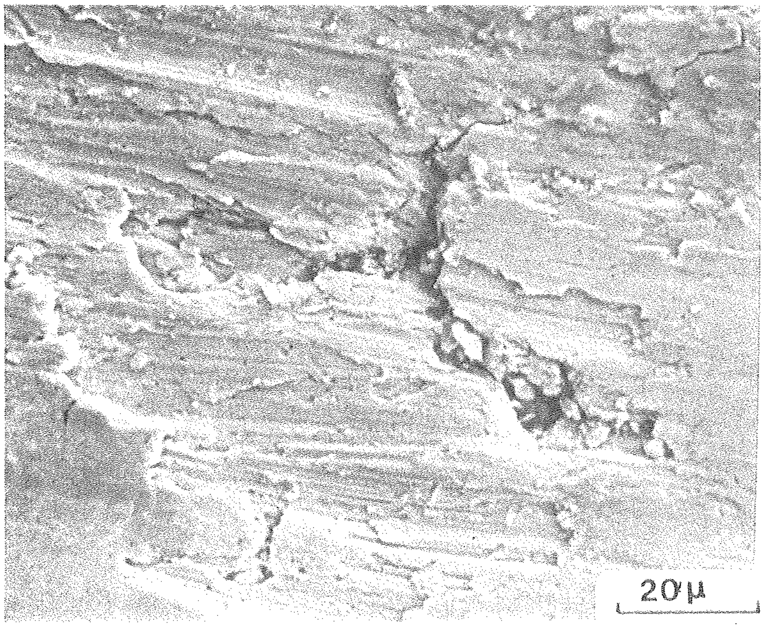


Fig. 16



XBB 773-1754

Fig. 17

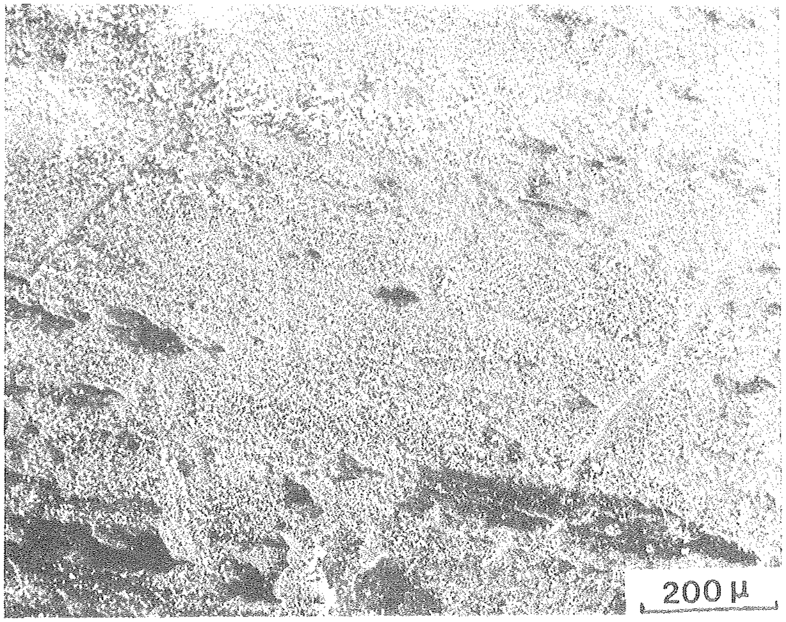
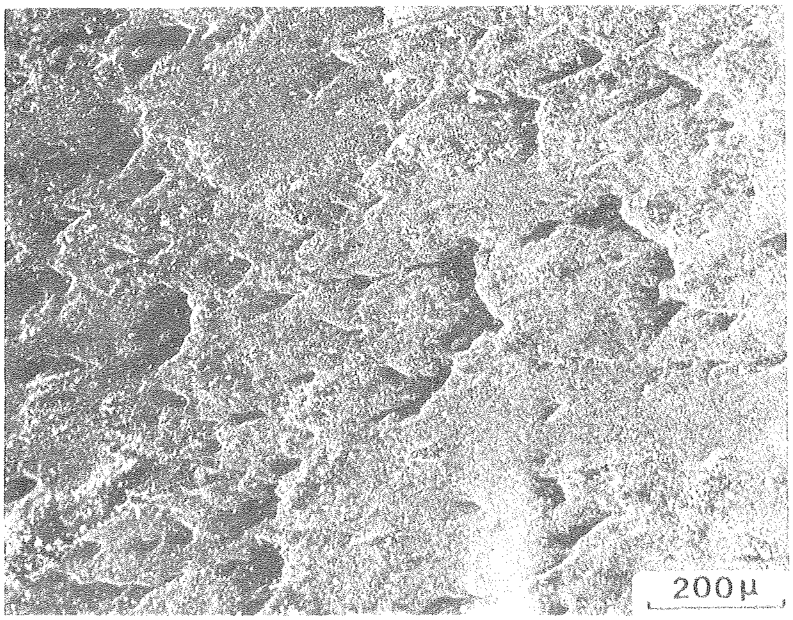


Fig. 18



XBB 773-1753

Fig. 19

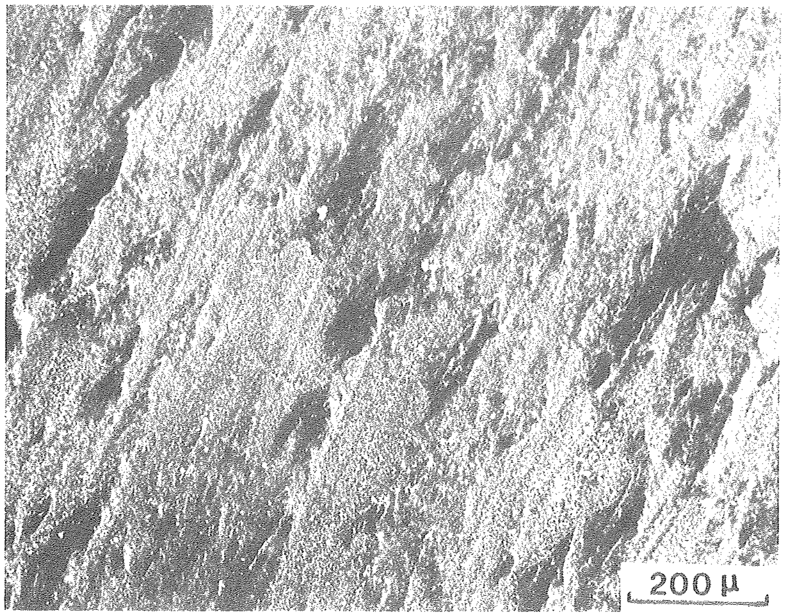
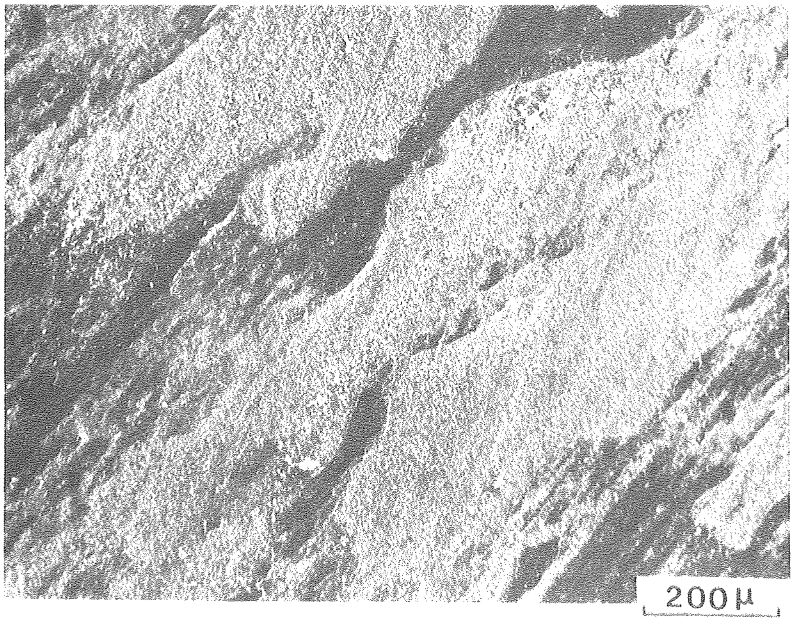


Fig. 20



XBB 773-1752

Fig. 21

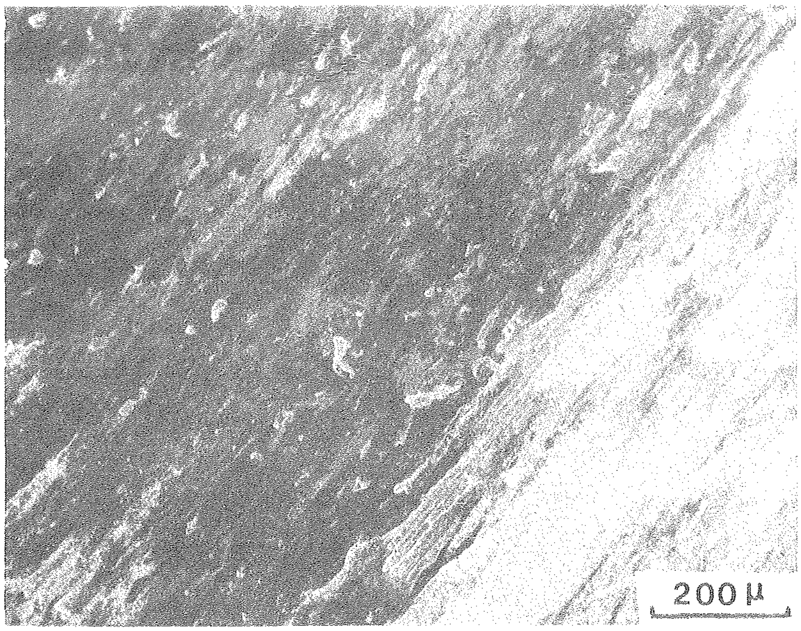
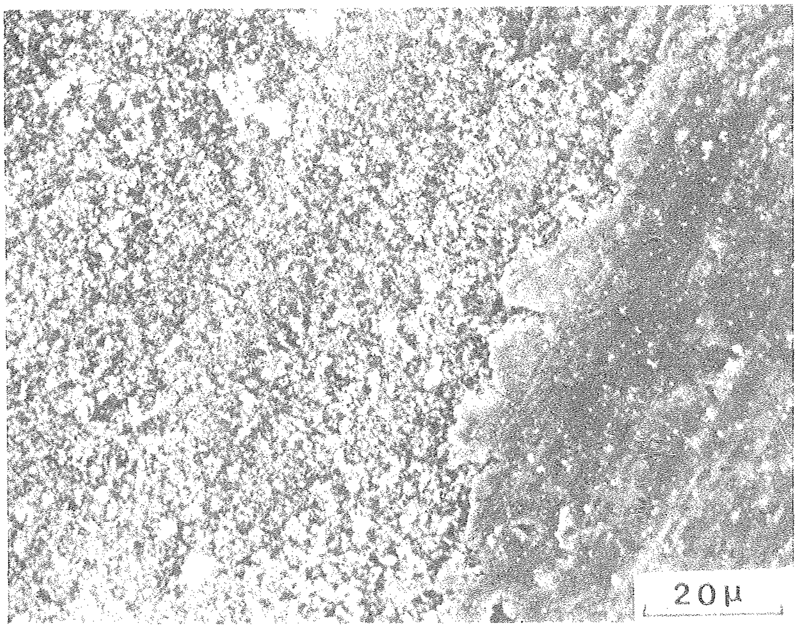


Fig. 22



XBB 773-4751

Fig. 23

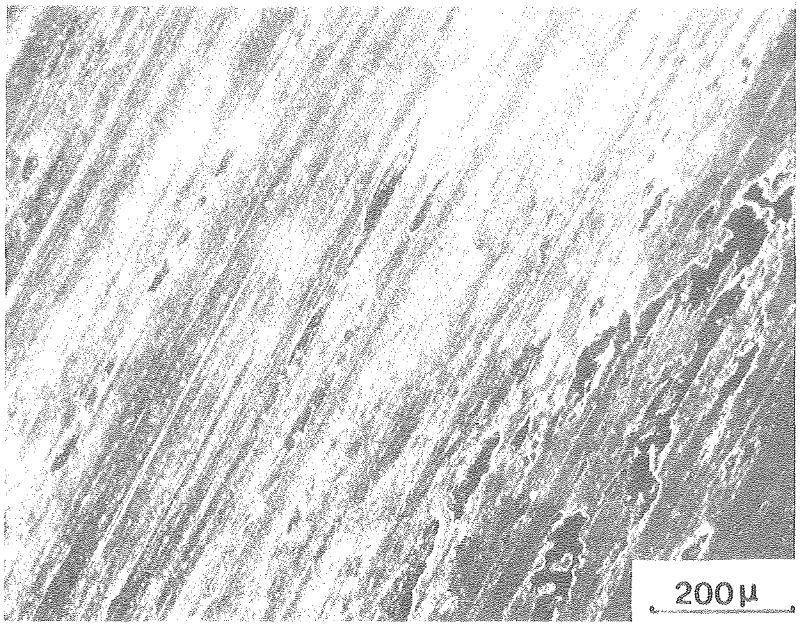
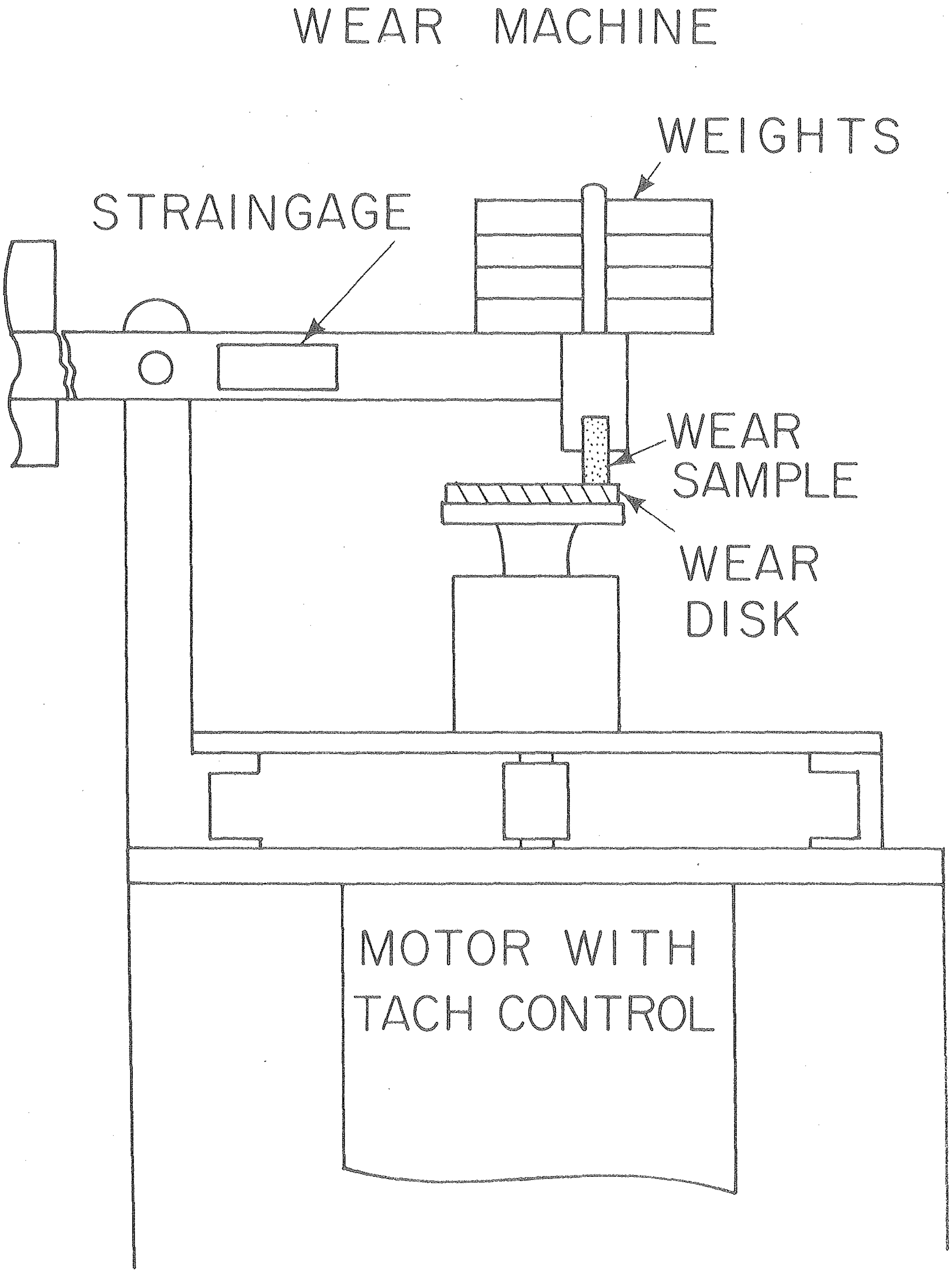


Fig. 24

XBB 773-1750

Fig. 25



XBL7612-4504

Fig. 26