

Surface Characterisation Techniques for Wear Surfaces

M G Gee

Centre of Materials Measurement and Technology
National Physical Laboratory
Queens Road
Teddington, Middlesex
TW11 0LW

Summary

This report summarises the different techniques which are available to perform characterisation of worn surfaces. A very wide variety of techniques are available which give information on the topography of the surface, its structure, and its composition.

As well as some comments on the applicability of the different techniques which range from conventional microscopy to scanning probe techniques, the report includes a table which gives a succinct summary of the capability of the different techniques considered.

© Crown Copyright, 1997
Reproduced by permission of the Controller of HMSO

ISSN 1361-4061

National Physical Laboratory Management Ltd
Teddington, Middlesex, TW11 0LW, UK

Extracts from this report may be reproduced provided the source is acknowledged.

Approved on behalf of the Managing Director, NPL, by
Dr C Lea, Head, Centre for Materials Measurement and Technology

TABLE OF CONTENTS

INTRODUCTION.....1

SUMMARY OF TECHNIQUES.....1

SAMPLE PREPARATION.....2

TOPOGRAPHICAL INFORMATION.....3

 MECHANICAL PROBE 4

 OPTICAL PROBE 4

 PROBE MICROSCOPES.....5

 ELECTRON PROBES 6

MECHANICAL PROPERTY 6

 INDENTATION 6

 SCRATCH TESTING 7

 PROBE MICROSCOPES.....7

STRUCTURAL INFORMATION 7

 VISUAL EXAMINATION.....7

 OPTICAL MICROSCOPY 8

 SECONDARY ELECTRON IMAGING 8

 TRANSMISSION ELECTRON MICROSCOPY 9

 ELECTRON BACK-SCATTERED PATTERN ANALYSIS.....10

 LOW VACUUM SECONDARY ELECTRON IMAGING 10

COMPOSITIONAL INFORMATION.....11

 BACKSCATTERED ELECTRON IMAGING.....11

 ENERGY DISPERSIVE ANALYSIS.....11

 ELECTRON ENERGY LOSS SPECTROMETRY 12

 RAMAN ANALYSIS.....12

 SECONDARY ION MASS SPECTROMETRY 12

 AUGER ELECTRON ANALYSIS 12

 GLOW DISCHARGE OPTICAL EMISSION SPECTROSCOPY.....13

 X-RAY PHOTOELECTRON SPECTROSCOPY 13

 X-RAY TOMOGRAPHY 13

 X-RAY DIFFRACTION.....14

 NEUTRON DIFFRACTION 14

 ATOM PROBE FIELD ION MICROSCOPY 15

ACKNOWLEDGEMENTS 15

REFERENCES.....15

INTRODUCTION

The evaluation of worn surfaces is a key element in the study of wear and friction. The information that is gained give important insights on the mechanisms of wear that have occurred. It is used to improve the understanding of wear and friction under given conditions, and also to ensure that the mechanisms of wear occurring in laboratory wear tests are similar to those encountered in the engineering application that is being simulated. It is important to achieve this goal in testing otherwise there is little hope that laboratory testing can effectively predict the service performance of components and materials.

A very wide range of different techniques have been utilised in the examination of worn surfaces, and the number of different techniques is increasing all the time. The information that can be gained includes mechanical properties, chemical composition, and topography or surface form. When evaluating the results, for worn surfaces, it is particularly important to consider the spatial extent of the source of the information, in both the lateral direction across the surface and the depth into the surface.

It is useful to compare and contrast the different techniques at a superficial level before looking at the individual techniques in a little more depth.

SUMMARY OF TECHNIQUES

Table 1 summarises a number of the common surface characterisation techniques. The techniques are grouped in the table according to the type of probe that is used to investigate the surface.

It is clear that most of the information about a surface, relevant to tribological performance can be determined using one technique or another, but that any one technique is often limited in its scope. It is therefore often useful to combine the information from a number of different techniques to form a more complete picture of what is occurring to the sample surface. In this process, it is important that the technique giving the widest view of the whole sample is used first, and then examination is narrowed down through higher magnification techniques which reveal more and more detail. An example of this is the use which should always be made of visual examination before any higher magnification optical or scanning electron microscopy examination.

Having said that, it may well be true that when a single specific technique is used definitive answers to key issues can be obtained.

Although in many ways it is easier to classify different techniques by probe type, it is often more useful to consider the different techniques in terms of the class of information that can be obtained.

SAMPLE PREPARATION

Samples obtained from laboratory wear tests have a clearly defined history, and the worn area on the sample is often very clearly defined. If the sample is properly stored, little special preparation can be needed. However, if there is a large amount of loose debris and other material on the surface, this may need to be removed to reveal underlying structure (although debris should always be retained for examination).

The preparation of worn samples and components which have been extracted from field trials or from real application environments is much more complex. The parts are often covered with oils, grease or other material from the working environment, which needs careful cleaning to reveal the worn surfaces. Often relevant samples have been left in a poor condition by inadequate storage, causing damage to the wear surfaces through corrosion and general wear and tear. It is always important to obtain as much information as possible about the situation of the worn part or component so that the best understanding of the mechanism of damage to the sample or component can be obtained.

Cleaning is normally carried out after removal of any loose material or wear debris by washing with organic solvents such as alcohols or acetone. For small samples, ultrasonic agitation can be beneficial.

In some cases, particularly for samples from field trials and real applications, samples of the actual worn component cannot be easily obtained. This can be either because the components are very large and sectioning them can be expensive and time consuming, or because the parts concerned cannot be removed from service. In these cases, replicas can be formed by applying cold setting moulding compounds to the worn surfaces of the components (after suitable cleaning). These can then be examined by a range of the techniques described in this report, although clearly compositional and mechanical property information cannot be acquired.

Sectioning of samples with subsequent preparation of the sectioned surface can reveal information about sub-surface structure and deformation. Care is needed to preserve the quality of the edge between the worn surface and the section surface. Taper sectioning (where the section surface is made at an acute angle to the worn surface) can be used to give an effective magnification to the features at the surface. However, achieving good taper sections can prove difficult, as it is often not easy to maintain the angle of the taper at the edge of the sample during the required polishing process.

A very effective method of producing effective taper sections is to use a ball cratering system. These systems were developed as a way of measuring the thickness of thin layers and coatings, and have more recently been suggested as a method for performing wear miniature abrasion tests. In this procedure a ball (often steel) is rotated against the sample under a normal load with an abrasive slurry introduced between the ball and the sample. When a shallow spherical depression has been produced, the ball is removed, the sample cleaned, and the edge of the spherical depression gives a taper section with a well defined geometry. In the example of the use of this technique shown in Figure 1, the technique reveals clearly the differences in the sub-surface damage that is caused by different preparation techniques.

TOPOGRAPHICAL INFORMATION

The shape of a worn surface gives clues about how it was formed. For instance, the presence of deep grooves on a surface is deeply suggestive that abrasion processes have been occurring; in fact, one definition of abrasion relies on this observation. Although considerable information on the shape of a surface can be given by visual examination, and optical and scanning electron microscopy, quantitative information relies on measuring the response of probes of different types as they move from position to position on the surface. A good introduction to these different measurement techniques is given by the introductory review written K J Stout [1].

Although surface topography measurement was initially developed as a 2D technique (ie the height variation along a single line defined on the sample), some techniques such as interference microscopy have always given 3D information, and even those techniques which traditionally gave only 2D information are now being used to give a full 3D view of the surface.

One of the problems with 3D topographical measurement is that the surface roughness parameters that have been developed, historically, for 2D profile measurements are not necessarily appropriate for 3D data. Indeed one of the deficiencies in 2D measurement has always been that sampling procedures need to be carefully selected so that the 2D measurement gives a good measure of the topography of the 3D surface.

New algorithms have recently been devised in an EC funded project which have the aim of providing a parameter set which can be used to effectively summarise the topography of a 3D surface [2].

It is also important to realise that the different types of probes that are used will give different numerical results for measurements on the same surface. This results from differences in the interaction that occurs between the different probes and the surface depending on the physical principles involved.

Mechanical probe

Mechanical probes have been used for many years in the determination of surface topography, and must be the most common technique used for the characterisation of surface topography. Normally, a diamond stylus with a well defined tip geometry (eg tip radius of a few micrometres) is used as the mechanical probe. Either the stylus is scanned over the sample, or the sample is scanned over the probe, and variations in the height of the surface from point to point result in a movement of the probe which is measured to give a measure of the sample height at that point. The geometry of the tip is well defined by national and international standards. One of the potential drawbacks with stylus instruments is that the movement of the stylus over the surface can cause damage to the surface when soft materials are being examined.

Optical probe

Optical probes are becoming increasingly popular. Their chief advantages is that they are non-contact devices which cannot damage the examined surface, and for full field instruments, can be much faster than scanned systems.

There are four main types of optical instruments.

The first is interference microscope based systems. The basic principle is that an interference pattern is formed by combination of the light reflected from the surface with a reference beam. Often the sample is scanned in height whilst several patterns are captured (sometimes

continuously) and analysed to calculate surface height information. These instruments have good spatial and height resolution, and have high measurement rates. There are some concerns that errors occur when rough surfaces are measured due to the difficulty in analysing the very complex interference patterns that result. These instruments also normally measure a single field of view at a time, limiting the area that can be examined.

The second type of instrument is the confocal microscope [3]. The utilisation of narrow apertures in the optical system gives optical sectioning where light is only captured by the microscope from the plane in focus intersecting the sample. The height of the sample is then scanned, and a set of optical sections is collected and analysed to give surface height data. The instrument gives good spatial and height resolution, but is again normally limited to single fields of view (Figure 2). A particular advantage of this technique is that images of the examined area are acquired simultaneously with the height information.

The third type of optical system is based on compact disc (CD) technology. This has been used as the basis of optical non-contact surface topography probes. They normally use infra-red lasers and gain the height information through an servo feedback mechanism which monitors the size of the spot focused from the reflected light. These systems have the advantage that large samples can be examined, but the large spot size limits the spatial resolution of the device.

The last type of instrument also uses laser probes, but relies on a triangulation system to calculate the surface height.

Probe microscopes

There has been considerable interest in the use of scanning probe instruments for the characterisation of surfaces. There are a wide number of different probe instruments available using a variety of different phenomena as the basis for the imaging. The two most common instruments are the STM (scanning tunnelling microscope), and AFM (atomic force microscope) (Figure 3). These instruments both employ stylii for their operation, with the stylus moved up and down by a feedback mechanism to maintain either the tunnelling current (for STM) or the force of contact (for AFM) constant. The size of the signal used to maintain the correct position of the stylus gives a measure of the surface height.

These instruments can have atomic resolution for relatively flat surfaces, but for rougher surfaces the resolution is limited by the finite dimensions of the stylus. There are also problems with the traceable calibration of these instruments.

Electron probes

Scanning electron beam instruments can be used to measure surface height. This is achieved by using multiple electron detectors to collect electrons scattered by the sample. The technique relies on the fact that the angular intensity of the electrons scattered by the sample will be dependent on the angle of the surface at the point struck by the beam. Proper combination of the signals gives an image where the intensity is proportional to the slope of the surface under the electron beam. This is integrated to give surface height information. The spatial resolution depends on the size of the electron probe, and the height information on the signal to noise characteristics, which are material dependent. However, for surfaces with a fine roughness where the scale of the roughness is of the same order as the size of the interaction zone of the sample with the electron beam, a straightforward interpretation of the intensity of the electron beam in terms of sample height is not possible.

MECHANICAL PROPERTY

Indentation

Indentation can be used to provide mechanical property information for worn surfaces. Microindentation, which normally uses indentation loads from about 0.1 N to a few N gives values of hardness with a spatial resolution of a few micrometres.

Nanoindentation, which typically uses indentation loads less than 0.1 N gives mechanical property information with a spatial resolution which can be as little as a few tens of nm.

In nanoindentation and with some microindentation instruments, the relative movement of the indenter into the sample is measured. This is often termed depth sensing indentation. The provision of depth information gives the ability to calculate additional mechanical properties of the surface such as the elastic modulus and time dependent deformation properties [4] (Figure 4).

The measurement of mechanical properties, particularly for small indentation sizes is subject to uncertainties which derive from an incomplete knowledge of the indenter geometry. There are also problems with some of the procedures for the calculation of mechanical properties

such as modulus, since these procedures rely on simplified models of the contact between indenter and sample and the deformation that occurs in that contact.

Scratch testing

Scratch testing was developed originally to test the adhesion of thin hard coatings. It is now extensively used for this purpose, but well defined scratches are made at light loads can also give important information on the way that surface layers interact with a moving point. Microscopic examination of the scratch shows the mechanisms of damage that have occurred. If the instrument is fitted with a load cell for measurement of the horizontal load that is generated between the indenter and the sample, then the friction properties of the surface layers can be measured.

Probe microscopes

This principle can be extended to give friction information with a very high spatial resolution by using an AFM in a lateral force mode (Figure 5). This can have atomic resolution for relatively flat surfaces, but a major problem with this type of instrument is that it is very difficult to design an instrument where the frictional information is isolated from the topographical information, and major problems remain with the interpretation of information obtained with type of imaging.

STRUCTURAL INFORMATION

Visual examination

It is always important to examine worn samples and components by eye before using any other technique. This visual examination gives information about the whole component that other higher resolution techniques can easily miss, such as appearance (smooth, shiny, rough etc), colour, and interrelationship between different features on the surface. The use of different lighting directions (vertical, oblique, glancing angle) should be explored.

Visual examination can be augmented by close-up photography (Figure 6). Modern digital cameras can be useful in this regard as they eliminate the need for photographic developing and printing, and allow for computer archiving and image processing.

Optical microscopy

Optical microscopy provides images of surface features with magnifications up to about 1500x. Images reveal information about the colour and placement of different features on the surface (Figure 7). One of the limitations of conventional optical microscopy is the small depth of focus (dependent on microscope objective and thus magnification). This limits the usefulness of conventional microscopes for the examination of rough surfaces.

By combining the best information from a number of optical slices obtained as the height of the sample is varied, the confocal microscope can give a field of view which is limited only by the working distance of the objective lens. It also give enhanced resolution (about 40% better) than conventional optical microscopes.

Although most optical microscopy is performed using brightfield imaging, additional information can often be obtained under other imaging conditions. Thus crossed polarising filters can give different contrast depending on the optical activity of the different phases being examined, and differential interference contrast gives contrast that depends sensitively on the topography of the surface being examined (Figure 8).

A useful introductory text on optical microscopy is given by Bousefield [5]

Secondary electron imaging

Secondary electron imaging in the scanning electron microscope uses secondary electrons that are emitted from the surface of a sample when the electron beam of the microscope hits the surface at the point being imaged [6]. Because the generation of secondary electrons is strongly dependent on the orientation of the surface at the point being examined, the image contains a significant contribution from topographical features on the surface of the sample. There is also some contrast that arises from the variation in the atomic number of the sample from point to point (Figure 9).

The scanning electron microscope can give very high spatial resolution, and has a high depth of focus. However, because the image is formed by an electron beam, sample charging problems can occur for non-conductive samples. These are normally solved by coating samples as required by thin layers of conductive material such as carbon or gold, but for surfaces such as powders or tribological layers where the contact between one part of the surface and another is not very good, charging can still occur.

For extreme cases this problem can only be overcome by the use of low vacuum scanning electron microscopy.

A problem with secondary electron imaging (and with all other forms of scanning electron microscopy) is that the minimum attainable magnification is often too high to get an overall feel for the damage to a component or sample. It also suffers from giving a much poorer detectability for some features on the surface of the sample such as pits. (A classic example of this is the imaging of Vickers hardness indentations in the SEM. Even when they are quite large and can be easily detected in by optical microscopy or even by eye, it can be very difficult to see the same indentations in the SEM.)

Transmission electron microscopy

Transmission electron microscopy is a useful technique where a very detailed analysis of the small scale structure of surface layers can be made. For some types of samples near atomic resolution can be achieved. Extra advantages of the technique are that simultaneous information on the degree of crystallinity and crystal type, and compositional information through EDS and EELS can be obtained.

There are two types of transmission electron microscope. In a conventional instrument, electromagnetic lenses are used to form an image from the electrons which penetrate the sample. Contrast is formed through a number of different scattering and absorption mechanisms whose effects can be interpreted to yield the information sought.

Scanning transmission instruments form images through similar interactions, but also give the ability to scan the beam across the sample giving flexibility for analysis and imaging.

The main drawback with transmission electron microscopy is that it requires the use of electron transparent samples. These need to be less than about 200 nm in thickness, and can be very difficult to prepare for solid samples. Normal techniques used for sample preparation are mechanical grinding followed by ion-beam thinning.

One area where sample preparation is extremely easy is for the examination of wear debris. The preparation of samples consists simply of scattering the wear debris onto carbon films and shaking off the loose powder. Sufficient particles adhere to the carbon film to allow examination and analysis at high magnification (Figure 11).

Electron back-scattered pattern analysis

The formation of back-scattered patterns from electron diffraction from the surface of a sample in the scanning electron microscopes can be used to define the crystallinity and orientation of crystalline regions. It can also be used for identification of phases, but this is not a straightforward process because of the complexity of the patterns. However, modern developments in software for pattern analysis have simplified this process.

A feature of this technique for the examination of wear surfaces is that it is very sensitive to the state of the surface so that very small changes in the surface can be observed, but also means that any significant damage or the presence of deformed material at the surface will prevent the formation of patterns.

Low vacuum secondary electron imaging

In low vacuum (high pressure) scanning electron microscopy a differential pressure is maintained between the electron column and the sample chamber so that the sample chamber contains a partial atmosphere. This gives several advantages at the expense of resolution. These are:

- The ability to examine non-conducting samples without special preparation techniques; the presence of a partial atmosphere in the sample chamber means that the charge on the sample formed by the incident electron beam is discharged through the gas to ground.
- The ability to examine wet samples.
- The ability to examine samples in the presence of lubricants.

Although these possibilities can be extremely useful, for samples where liquid is present on the surface, interpretation should be made carefully, particularly for operators used to images obtained with more conventional instruments.

Acoustic microscopy

In acoustic microscopy, acoustic waves are focussed by an acoustic lens onto the sample. The reflected acoustic wave is then analysed to yield information on the elastic response of the material under the probe, which can be used to form images of the sample structure (Figure 12). The major advantage of the technique is that it is possible to obtain subsurface images by focussing the lens below the surface of the sample. However, because of the complexity of the interaction of the acoustic waves with the sample, image interpretation is often difficult.

COMPOSITIONAL INFORMATION

Backscattered electron imaging

Back-scattered electron imaging is a scanning electron microscope technique which uses electrons elastically back-scattered (reflected) from the surface. The yield of back-scattered electrons is dependent both on the orientation of the surface and the atomic number of the phase being examined. Normally four quadrant detectors placed just below the polepiece of the objective lens of the microscope are used to form images. When the signal from all four quadrants is summed, a signal is generated with an intensity dependent on the atomic number of the material (compositional image); when the signal from two neighbouring quadrants is subtracted from the other two quadrants a signal is generated with an intensity dependent on the topography of the surface.

The compositional imaging can give very useful information on very small variations in composition from point to point on a sample, but suffers from the need for long acquisition times to reduce noise and a lower spatial resolution than with secondary electron imaging due to the size of the region in the sample where back-scattered electron generation occurs (Figure 10).

Energy dispersive analysis

Energy dispersive analysis is a technique that analyses the X-ray spectrum generated when an electron beam strikes a sample, and is, therefore, a common ancillary technique in electron microscopy. The X-ray spectra contain peaks which are characteristic of different elements at the point being examined. The relative intensity of these peaks gives a measure of the composition.

The main drawback of the technique is that it is not very accurate for light element (Atomic number $Z < 11$) analysis, although there have been major improvements in recent years, and that the intrinsic resolution of the analysis electronics often leads to problems due to peak overlap. The spatial and depth resolution of the technique are limited by the volume of the sample where X-rays are generated through the interaction of the sample with the incident beam.

Electron energy loss spectrometry

Electron energy loss spectrometry is an analysis technique ancillary to transmission electron microscopy. The energy spectrum of the electrons that is transmitted through the sample is analysed by an electromagnetic analyser to reveal the structure in the spectrum characteristic of the composition of the analysis point that has been produced by in-elastic scattering in the sample. Modern systems have a sensitivity for metals that approaches 3-4 atoms.

Raman analysis

In Raman analysis a laser beam is focussed on the sample surface and the light scattered from the surface is analysed. For Raman active materials a spectrum is produced which acts as a “fingerprint” for identification of different phases.

The major drawback with Raman analysis is that many materials do not produce Raman spectra, so that the technique is by no means universal. Also, Raman analysis is very sensitive to the surface condition, so that high degrees of deformation at the surface can reduce or eliminate Raman activity.

Secondary ion mass spectrometry

In secondary ion mass spectrometry, a primary ion beam is used to remove ions or clusters of atoms from the surface atom layer of the sample [7]. The secondary ions emitted are analysed by a mass spectrometer to form a spectrum characteristic of species with different atomic masses. Atoms in the deeper layers of the sample are sequentially analysed as the material is slowly removed by the ion beam. When combined with even the most basic compositional information, this gives unambiguous identification of the different chemical species that form the surface layers of the sample to depths of a few micrometres.

A drawback with the technique used to be that the spatial resolution of the technique was poor, but this has been improved to about 50 nm in recent years with the introduction of focussed ion beam systems.

Auger electron analysis

In Auger electron analysis [8], an incident electron beam is focussed onto the sample to be analysed. The Auger electrons that are emitted have an energy which is dependent on the energy gained in the transition of electrons from one energy level to another and are thus characteristic of different elements. These transitions complement X-ray emission as the

microprobe, but do not suffer from the disadvantage that intensity is lost for low atomic number elements. Thus all elements except H and He can be analysed with spatial resolutions down to 20 nm. Compositions through these layers to depths of a few micrometers can be analysed sequentially by the use of in-situ ion sputtering.

Glow discharge optical emission spectroscopy

In glow discharge emission spectroscopy [9], a glow discharge lamp is placed on the sample to be tested, and sealed against the sample by an O-ring. The lamp is evacuated and then back-filled with argon. The glow discharge is formed between the sample, which is the cathode, and the anode of the lamp. Atomic and molecular species are removed from the surface under analysis by sputtering from the surface by the application of a high voltage between the surface and the anode of the instrument. These species are excited and emit light characteristic of their composition. The light emitted from the glow discharge is analysed to give a spectra containing information on the composition of the sample being analysed. The technique is quick, and with proper calibration can yield accurate results with a high depth resolution. The major drawback of the technique is the limited lateral resolution which is defined by the geometry of the device.

X-ray photoelectron spectroscopy

X-ray photoelectron spectroscopy analyses the energy of electrons that are emitted when high energy X-rays bombard a sample [8]. The spectra shows features characteristic of the different elements in the sample in a similar manner to Auger electron spectroscopy except that the precise peak energies now contain chemical as well as elemental information. The analysis causes very little damage to even the most delicate samples and charging problems for insulators are rare.

X-ray tomography

In X-ray tomography a number of different X-ray transmission images are obtained by shining a bright beam of X-rays through a sample of material whilst the sample is moved relative to the source. Analysis of the intensity of the different images in relation to their orientation with the sample gives a 3D data set of X-ray opacity through the volume of the sample. This can then be related to electron density, and thus to composition.

The technique gives 3D information about the structure and composition of samples. However, the major problem with the technique is that the penetration of X-rays through engineering materials is small, restricting the use of the technique to small sample sizes and making the use of bright X-ray sources such as X-ray synchrotrons essential.

X-ray diffraction

In X-ray diffraction, an incident X-ray beam is reflected from the surface of the sample by diffraction within the atomic lattice of crystals at the sample surface. When an X-ray diffractometer is used to carry out these experiments, the angle of incidence of the X-ray beam is incremented and the intensity of X-rays reflected at the same angle is measured to give a X-ray spectrum characteristic of the crystal structure that is present (Figure 13).

Other types of X-ray diffraction are possible; for example in a Laue camera, a spot diffraction pattern is formed which is characteristic of the crystal structure at the X-ray beam. Both solid and powder samples can be examined.

X-ray diffraction techniques can be extended to obtain information on the size of crystallites present in the sample, the degree of deformation, and the presence of residual stresses in surface layers within the sample.

The major drawback with X-ray diffraction is the limited penetration of X-rays into the surface of samples. However, good knowledge of this penetration depth can usefully be used to interpret the size of the layers on a surface which lead to specific features in X-ray spectra. Since X-rays cannot easily be focused, another drawback is the limited resolution of X-ray beams which have to be formed by collimation.

Neutron diffraction

Neutron diffraction is very similar to X-ray diffraction but with the use of neutron beams instead of X-ray beams. The techniques for analysis of diffraction spectra are very similar.

The major difference is that the neutron beam techniques have very good penetration into and through samples by comparison with X-ray techniques. However, the techniques also require the use of bright neutron sources which are only found at a small number of very specialist establishments around the world, making this technique very expensive and difficult to use.

Atom probe field ion microscopy

In atom probe field ion microscopy, the samples are prepared into very sharp needles (with the point of the needle at the position of interest on the wear surface under examination). A high electric field is applied to the sample, and an image formed from the movement of light ions from points on the surface of the tip to a phosphor screen. The flux of ions is dependent on the position of atoms in the tip of the needle, so that an image of the position of atoms at the tip of the needle is formed. A particular area on the tip of the needle can then be selected, and by applying a sharp pulse at an even higher voltage, specific atoms can be removed from the surface of the sample. These are then subjected to mass spectrometry to identify their atomic mass. The end result is that a compositional profile of the sample can be built up which has atomic resolution.

The major drawback of the technique is that highly specialised sample preparation techniques need to be employed, with the preparation of even a single sample sometimes taking several weeks. Because the size of the sample is so small, there are also problems in ensuring that the sample is representative of the material which is being examined.

ACKNOWLEDGEMENTS

The author would like to acknowledge the support of DTI through its Materials Measurement Programme for this work.

REFERENCES

- 1) K J Stout, Three-Dimensional Surface Topography; Measurement, Interpretation and Applications
- 2) K J Stout, P J Sullivan, W P Dong, E Mainsah, N Luo, T Mathia, H Zahouani, The Development of Methods for the Characterisation of Roughness in Three Dimensions, EC Report EUR 15178 EN, Directorate General XII, Science, Research and Development, Programme for Applied Metrology and Chemical Analysis (BCR), EC Brussels, 1993
- 3) M G Gee and N J McCormick, The Application of Confocal Scanning Electron Microscopy to the Examination of Ceramic Wear, Journal of Physics D: Applied Physics, 25(1992)A230

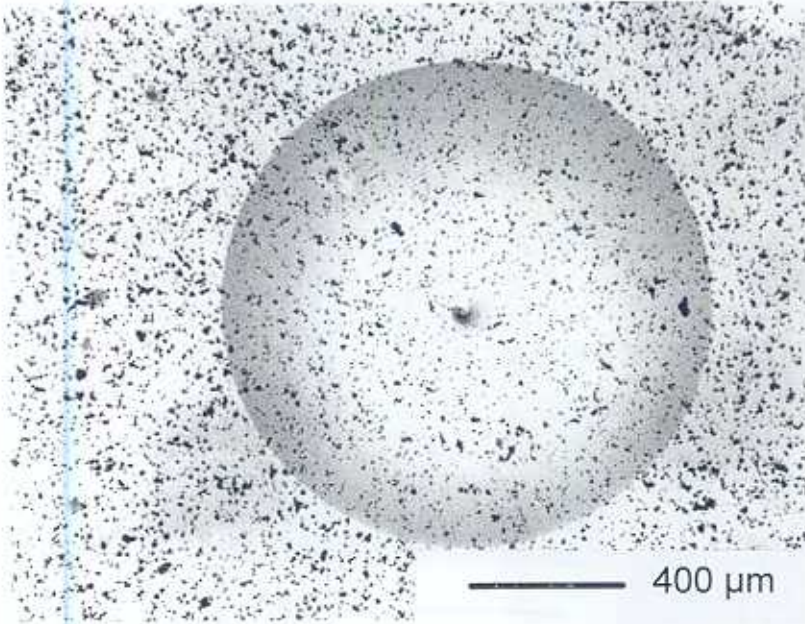
- 4) M G Gee and N M Jennett, High Resolution Characterisation of Tribochemical Films on Alumina, *Wear*, 193(1995)133-145
- 5) B Bousefield, *Surface Preparation and Microscopy of Materials*, Wiley, 1992
- 6) J I Goldstein, D E Newbury, P Echlin, D C Joy, C Fiori and E Lifshin, *Scanning Electron Microscopy and X-ray Microanalysis*, Plenum Press, 1981
- 7) D Briggs and M P Seah, *Practical Surface Analysis Volume 2: Ion and Neutral Spectroscopies*, Wiley, Chichester (1993)
- 8) D Briggs and M P Seah, *Practical Surface Analysis Volume 2: Auger and X-ray Photoelectron Spectroscopies*, Wiley, Chichester (1990)
- 9) T Bell and Y Sun, *Glow Discharge Depth Profiling - A Powerful Analytical Technique in Surface Engineering*, 5th International Seminar on Heat Treatment and Surface Engineering, IFHT, Isfahan, Iran, (1995)

Table 1, Techniques characterised by probe type; OM is optical microscopy, LP is laser profilometry, CM is confocal microscopy, RM is Raman microscopy, SEI is scanning electron imaging, BEI is back-scattered electron imaging, TEM is transmission electron microscopy, EDS is energy dispersive spectrometry, SEM is scanning electron microscopy, WDS is wavelength dispersive spectrometry, EELS is electron energy loss spectrometry, EBSP is electron back-scattered pattern, SAM is scanning Auger microscopy, XRF is X-ray fluorescence, XRD is X-ray diffraction, XPS is X-ray photoelectron spectrometry, XRT is X-ray tomography, SSIMS is static secondary ion mass spectrometry, DSIMS is dynamic secondary ion mass spectrometry, AM is acoustic microscopy, NI is nanoindenter, AFM is atomic force microscope, ND is neutron diffraction, APFIM is atom probe field ion microscope.

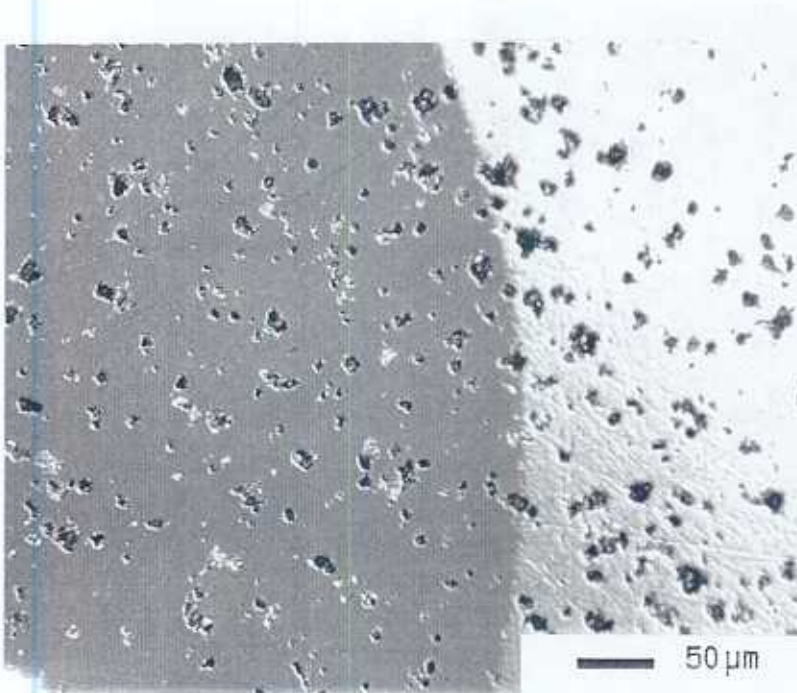
Technique	Spatial Resolution	Depth Resolution	Chemical Analysis	Applicability	Analytical Sensitivity
Optical (including Infra-Red) Probe:					
OM	0.2 µm	-	Phase by optical constant analysis (interference film, polarised light)	Imaging, structure	-
LP	1.5 µm	0.01 µm	-	Topography	-
CM	0.15 µm	0.03 µm	Phase as in OM	Imaging, topography	-
RM	2 µm	-	Identification and quantitative analysis for some phases	Chemical analysis	5 %
Electron Probe:					
SEI	1.5 nm	-	Some information from atomic number contrast	Imaging, topography by stereology or multiple detectors	-
BEI	50 nm	50 nm	Atomic number contrast	Composition with multiple detectors in addition, topography with detectors in subtraction.	0.1 in Z
TEM	0.1 nm	0.1 nm	Electron density	Imaging, crystal phase, orientation	-
EDS(SEM)	1 µm	1 µm	Characteristic energy X-ray generation	Chemical analysis	Z>4
WDS(SEM)	1 µm	1 µm	Characteristic energy X-ray generation	Chemical analysis	Z>2
EDS(TEM)	10 nm	20 nm	Characteristic energy X-ray generation	Chemical analysis	Z>4

EELS(TEM)	10 nm	20 nm	Characteristic energy loss	electron	Chemical analysis, structural information	All Z
EBSP	100 nm	50 nm	Phase by diffraction	electron	Crystal phase, orientation	-
SAM	5 nm	3 monolayers	Identification of elements by Auger electron energy	of	Chemical analysis	0.3 %
GDOES	1 mm	10 nm	Glow discharge spectroscopy	discharge	Chemical analysis	All Z
X-ray Probe:						
XRF	10 mm	10 µm	Identification of elements by fluorescence	X-ray	Chemical analysis	1-10 ppm
XRD	10 mm	10 µm	Phase by diffraction	X-ray	Crystal phase, structure information	5 %
XPS	5 µm	3 monolayers	Characteristic energy ray generation	X-ray	Chemical analysis	0.3 %
XRT	1 µm	1 µm	-	-	Structure	-
Ion Beam Probe:						
SSIMS	1 µm	2 monolayers	Analysis of ions emitted from target surface	emitted	Chemical analysis	0.01 %
DSIMS	20 nm	10 monolayers	Analysis of ions emitted from target surface	emitted	Chemical analysis	<1 ppm
Acoustic Probe:						
AM	1 µm	1 µm	Some information through variation in elastic constants	information	Elastic Properties	-
Mechanical Probe:						
NI	10 nm	10 nm	-	-	Mechanical property information	-
AFM	0.1 nm	0.1 nm	-	-	Topography, mechanical property information	-
Neutron Probe:						
ND	1 mm	1 mm	Phase identification	by	Crystal phase, structure information	

				neutron diffraction		
Electrostatic Field:						
APFIM	1 nm	1 monolayer	Identification of individual constituent ions and atoms from surface	Chemical information	analysis, structure	0.1-1 %



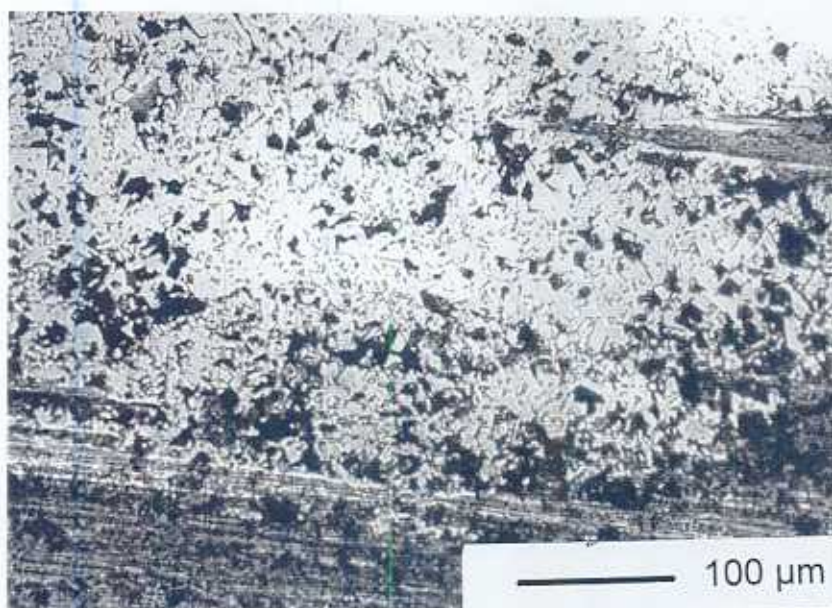
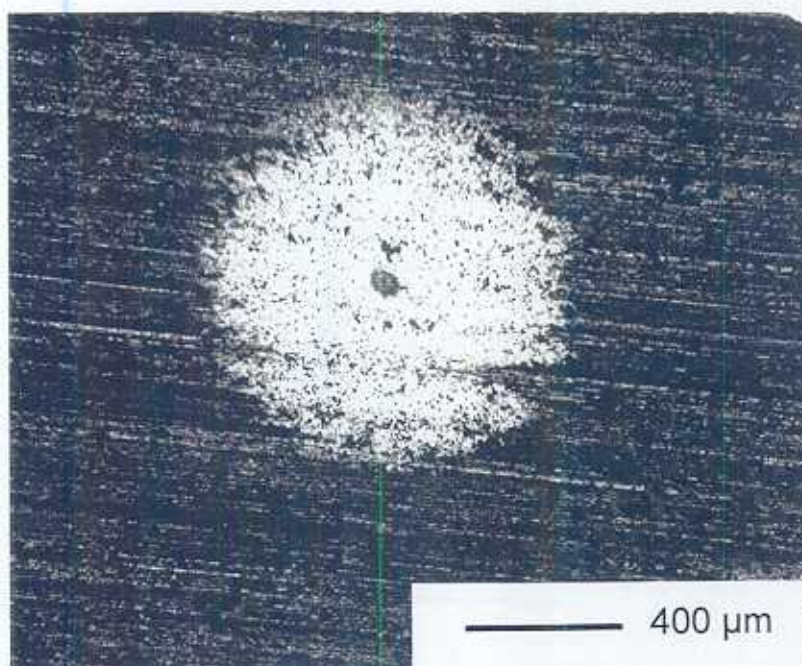
a)



b)

Figure 1, The structure of ground and polished alumina samples revealed by ball crater sectioning, a) and b) lapped and polished sample, c) and d) fine ground surface, e) and f) coarse ground surface.

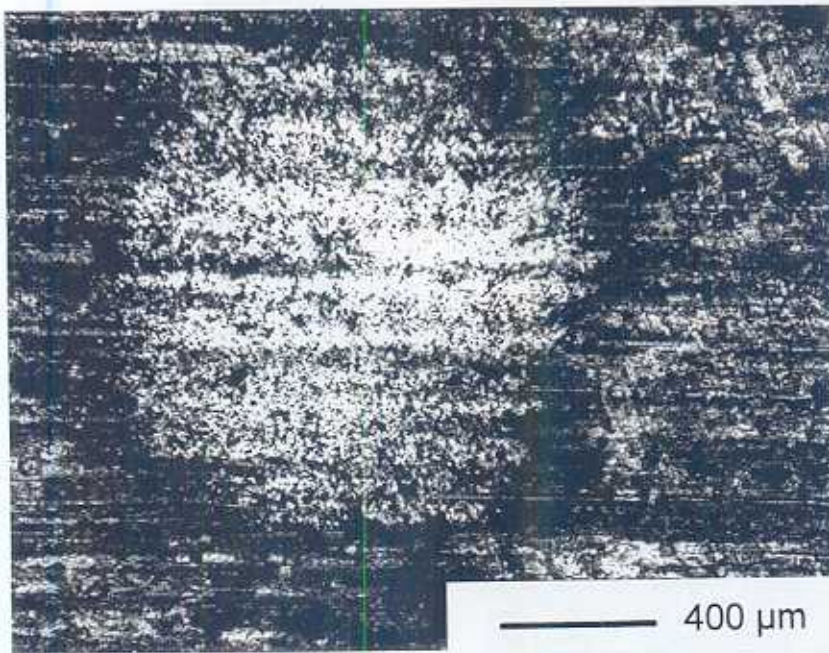
c)



d)

Figure 1, The structure of ground and polished alumina samples revealed by ball crater sectioning, a) and b) lapped and polished sample, c) and d) fine ground surface, e) and f) coarse ground surface.

e)



f)

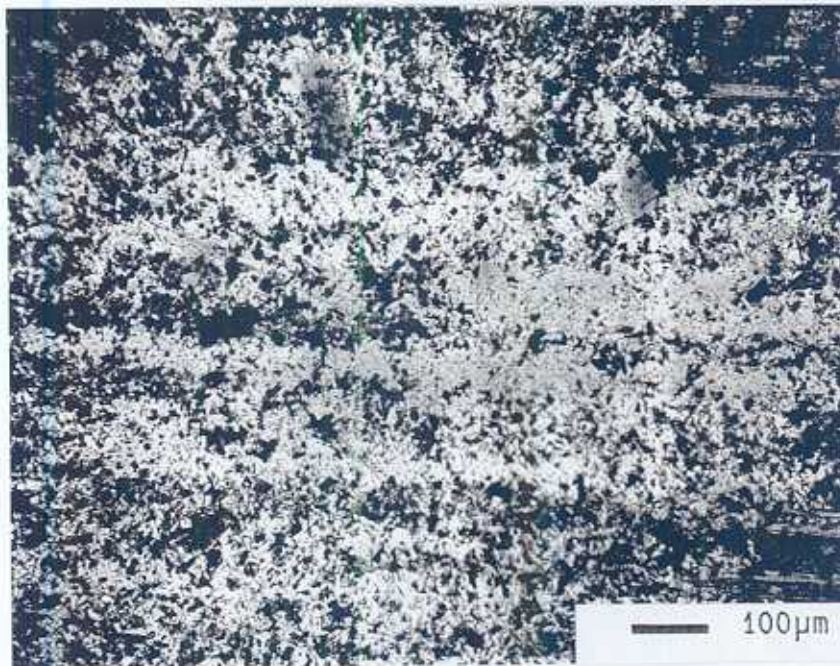


Figure 1, The structure of ground and polished alumina samples revealed by ball crater sectioning, a) and b) lapped and polished sample, c) and d) fine ground surface, e) and f) coarse ground surface

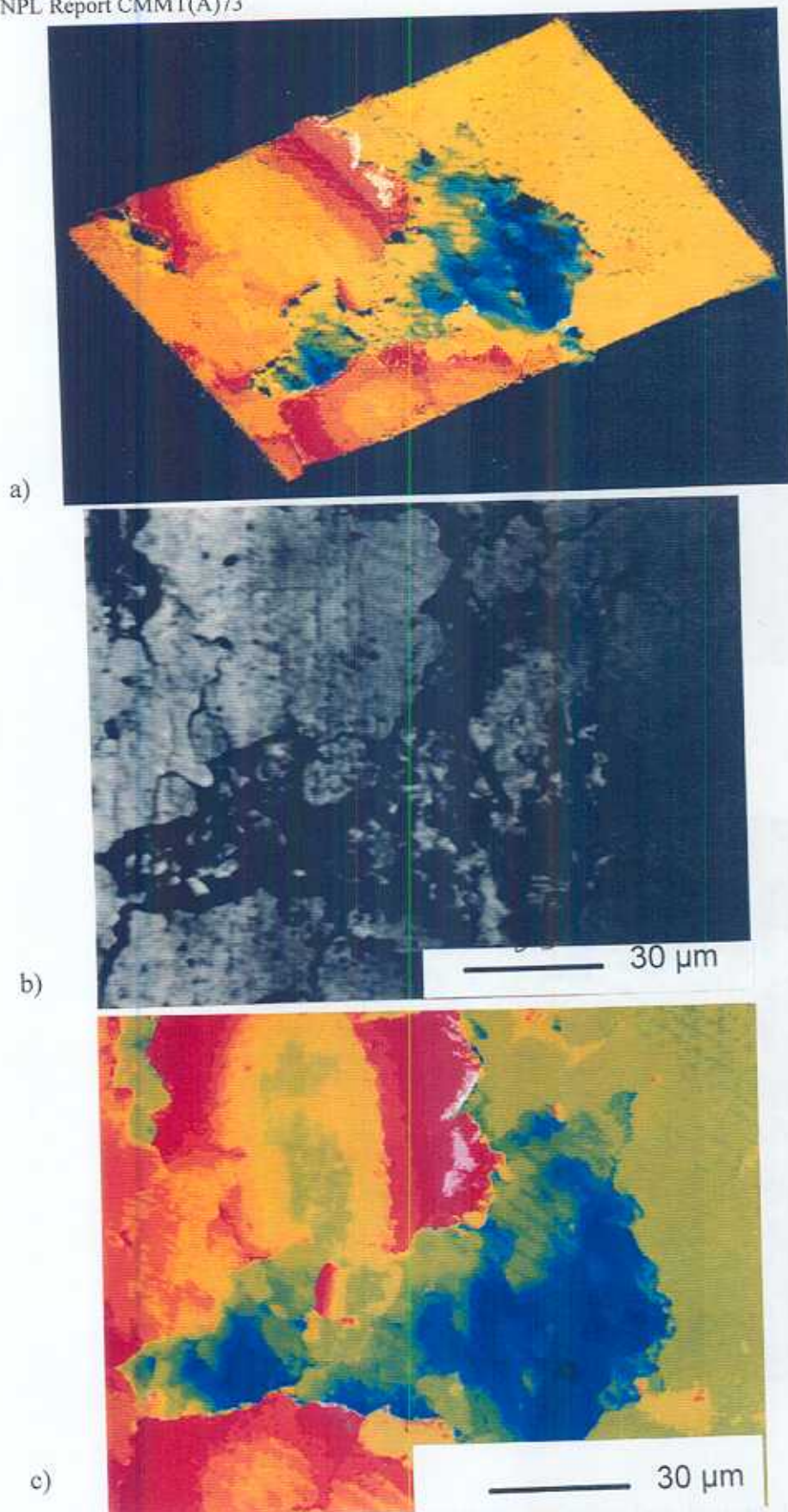


Figure 2, Height map and corresponding image of worn area on alumina ceramic produced by confocal optical microscope, a) isometric view of surface topography, b) image captured simultaneously with height information, c) contour map of height information.

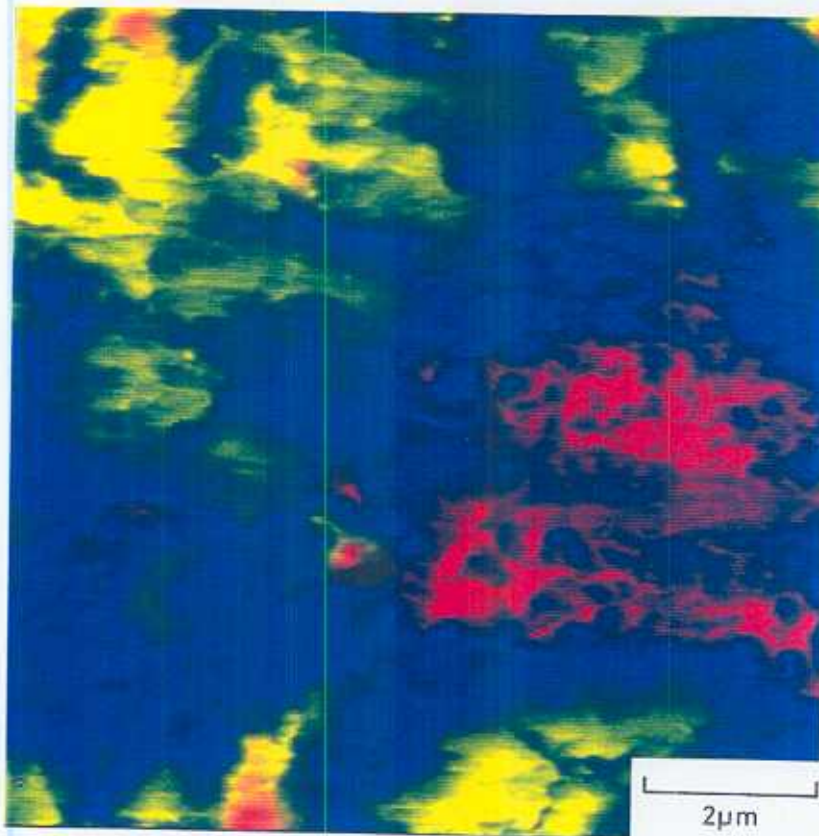
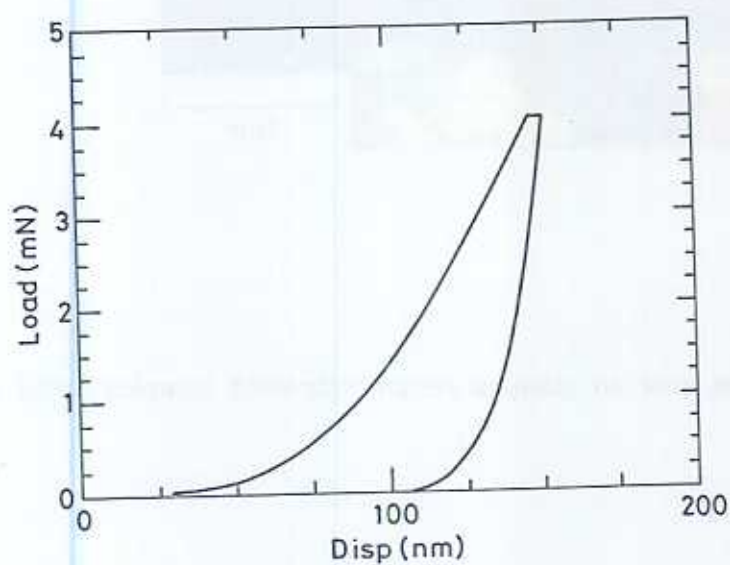


Figure 3, AFM image of worn area on alumina ceramic showing complex small scale topography.

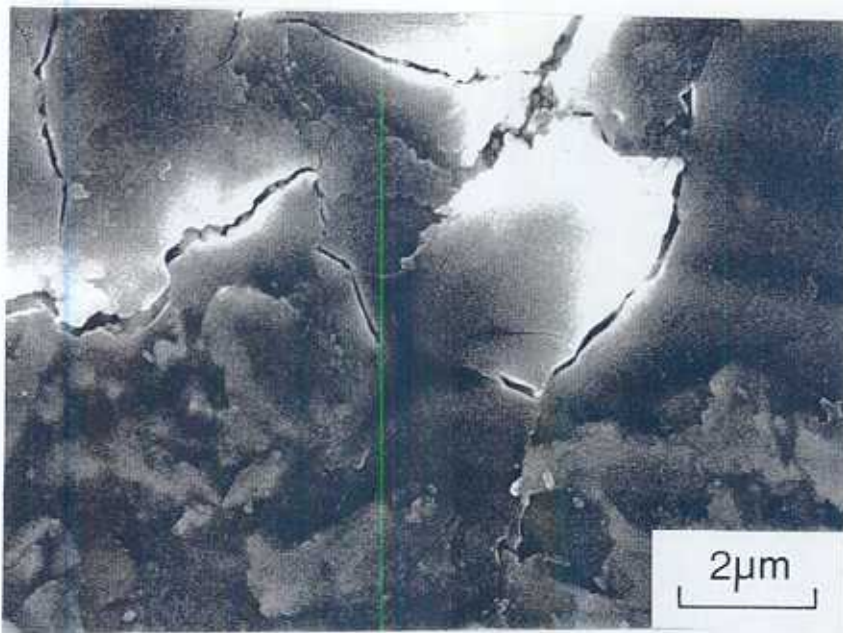


a)

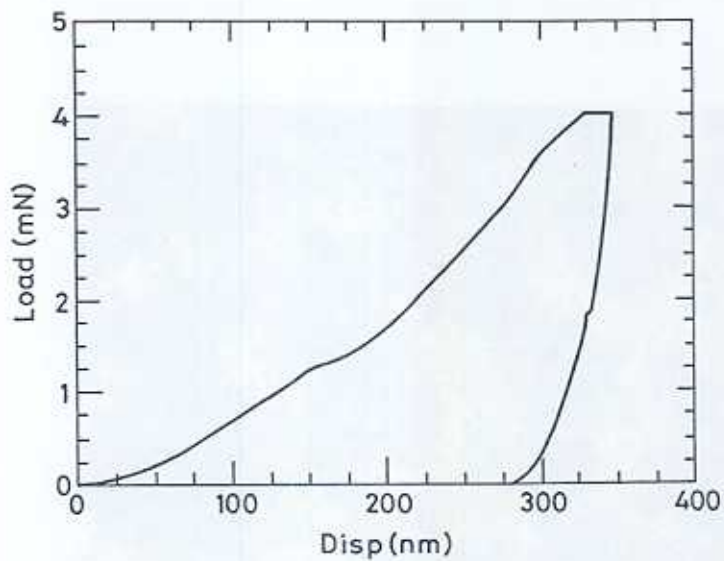


b)

Figure 4, Images of indentations produced by nanoindenter on worn alumina ceramic with corresponding load-displacement curves, a) indentation on alumina area covered with thin layer of aluminium oxide produced by tribochemical reaction, b) load-displacement curve corresponding to a), c) indentation on area of thick aluminium hydroxide film produced by tribochemical reaction, d) load-indentation curve corresponding to c).

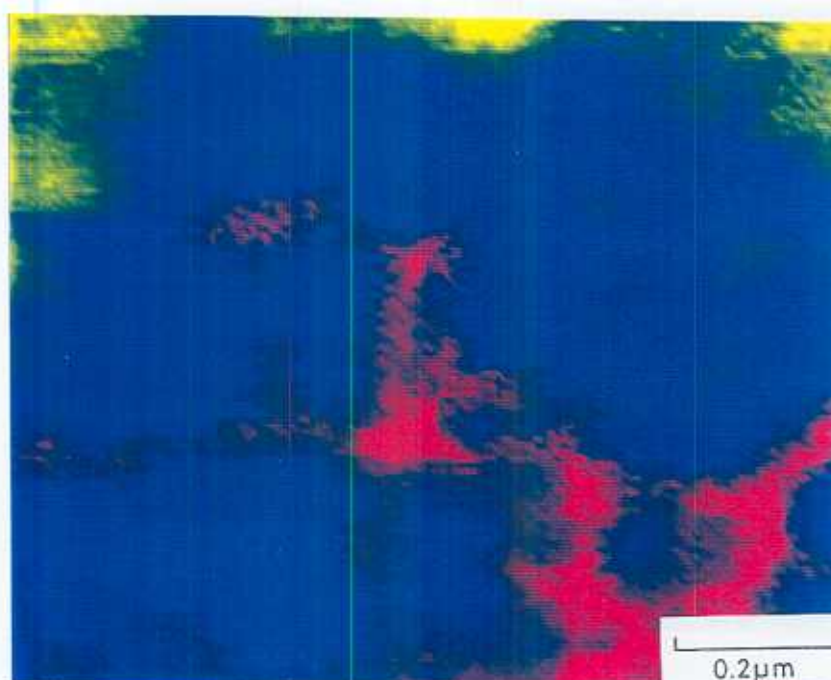


c)

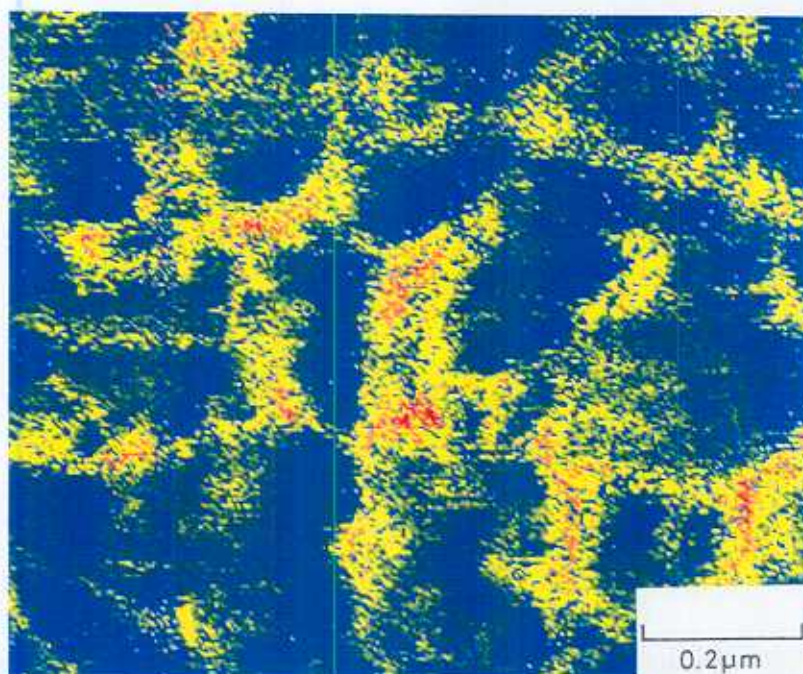


d)

Figure 4, Images of indentations produced by nanoindenter on worn alumina ceramic with corresponding load-displacement curves, a) indentation on alumina area covered with thin layer of aluminium oxide produced by tribochemical reaction, b) load-displacement curve corresponding to a), c) indentation on area of thick aluminium hydroxide film produced by tribochemical reaction, d) load-indentation curve corresponding to c).



a)



b)

Figure 5, Lateral force image and AFM image of worn area on alumina ceramic, a) AFM image, b) LFM image.

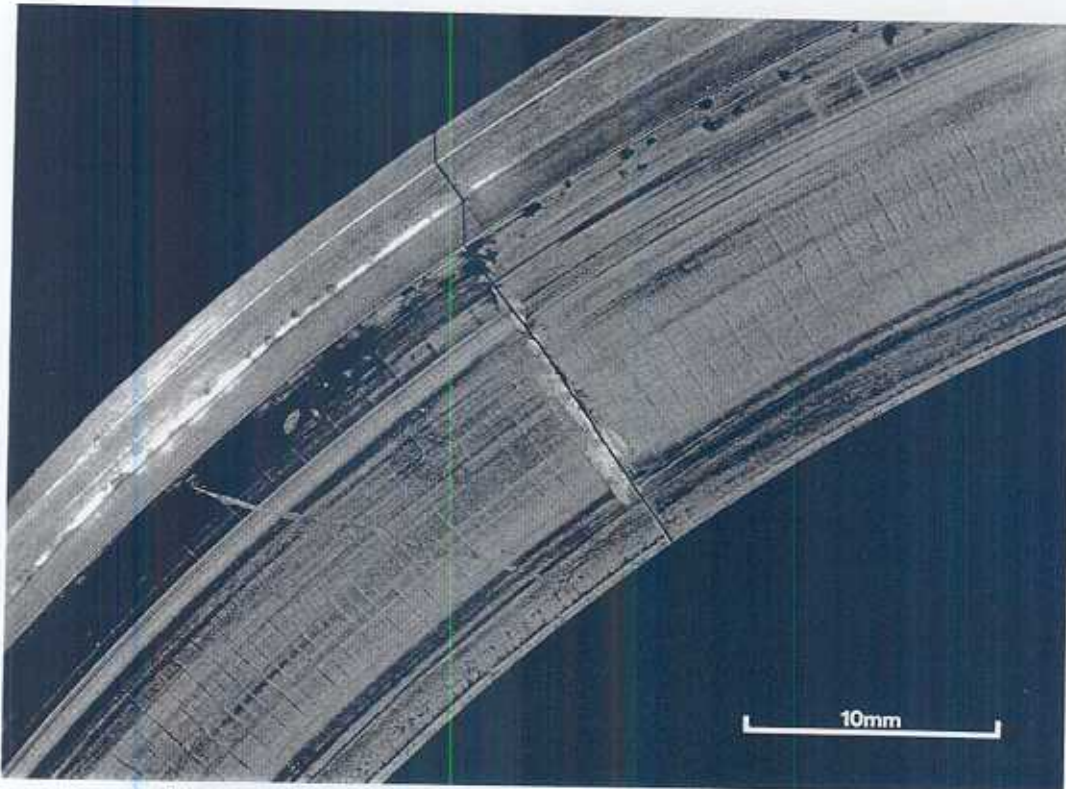
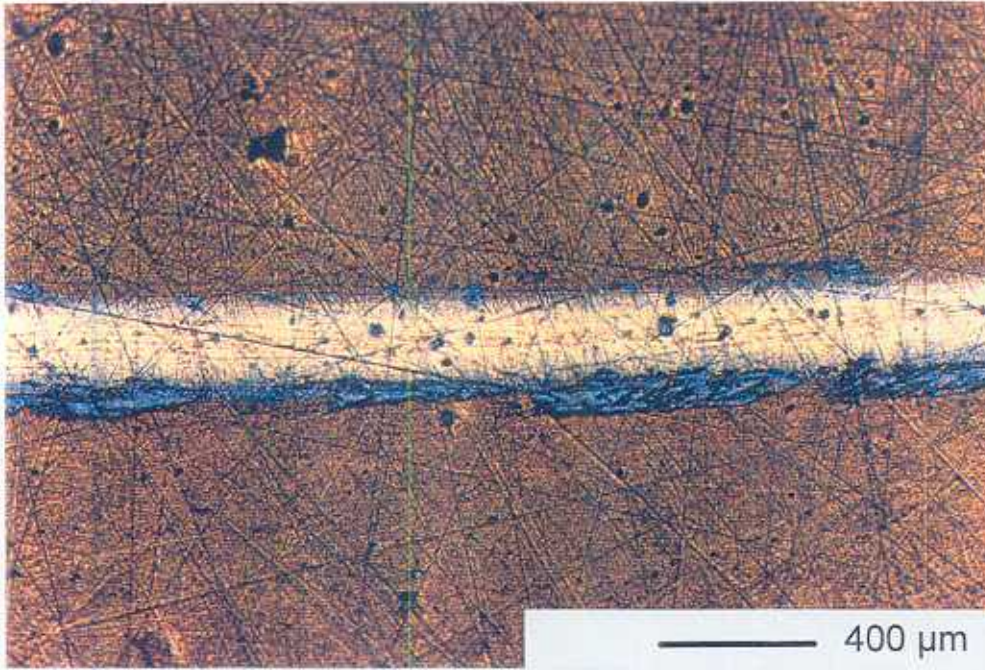
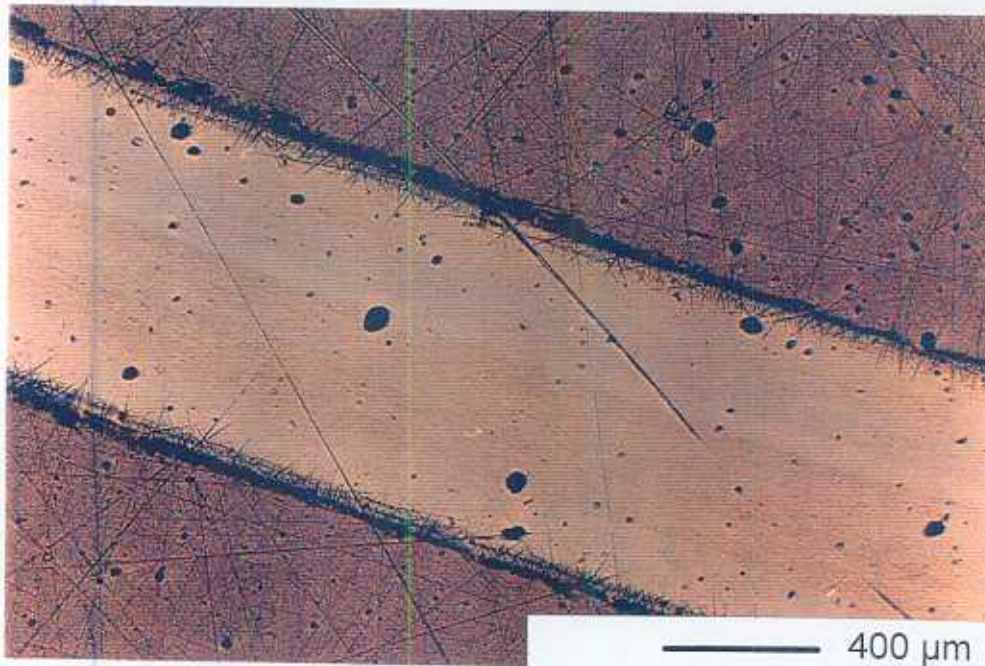


Figure 6, WC/Co mechanical sealing ring which has failed by thermal fatigue.

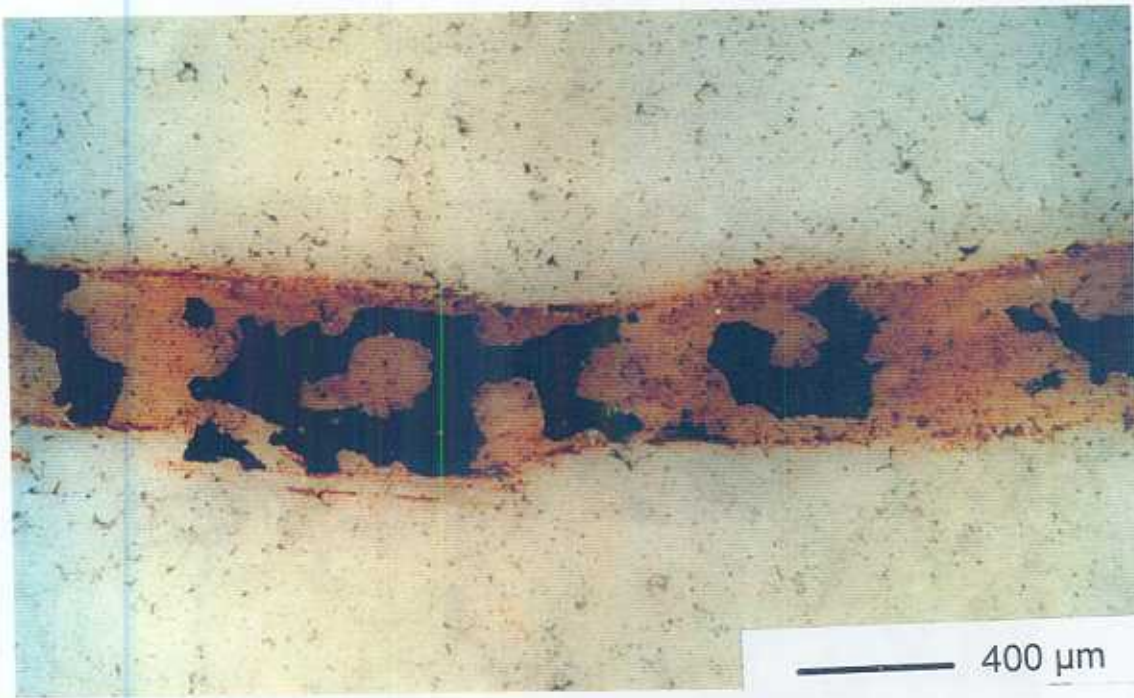


a)

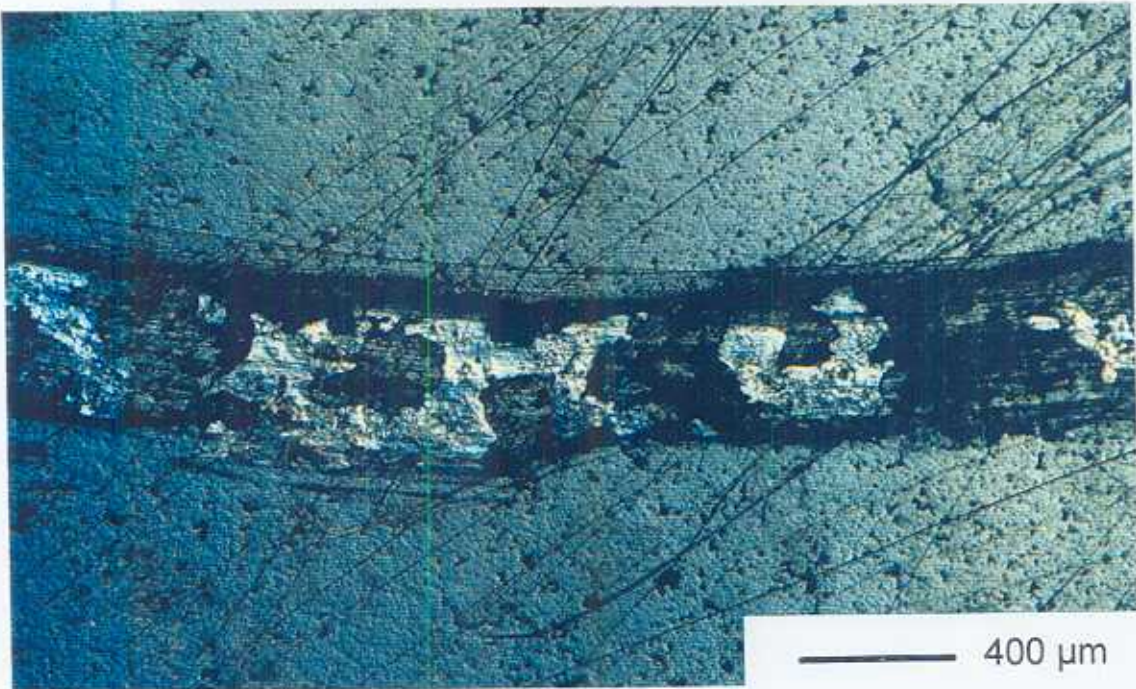


b)

Figure 7, Wear tracks on TiN coated samples worn in pin-on-disc configuration a) light wear showing build up of titanium oxide at boundaries of wear track, b) more heavily worn wear track with removal of scratches from centre of wear track by wear process.



a)



b)

Figure 8, Iron rich transfer films on alumina sample formed during wear of steel pin against alumina sample, a) is imaged with near crossed polariser illumination and emphasises the iron oxide reaction layers on the surface of the wear track, b) is the same area imaged with differential interference contrast emphasising the transferred layers of steel on the wear track.

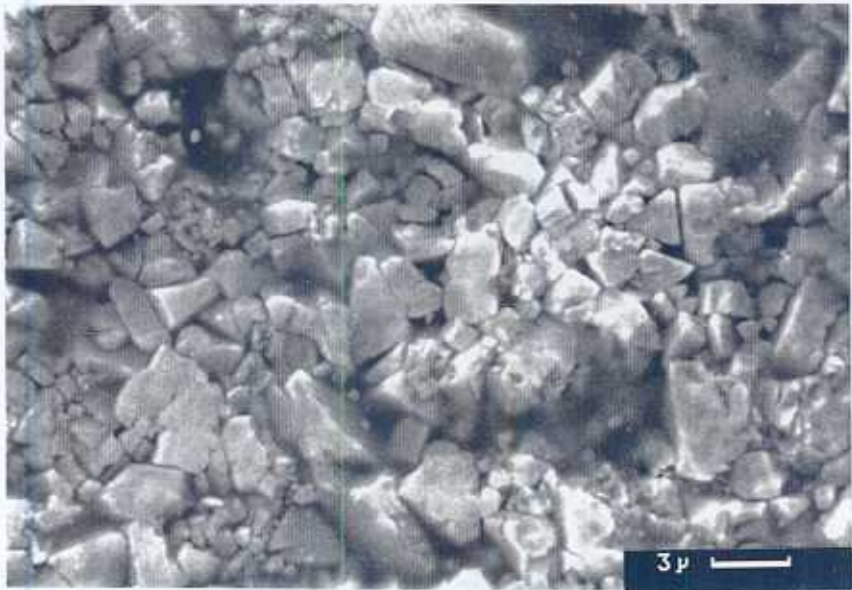


Figure 9, Worn WC/Co hardmetal surface.

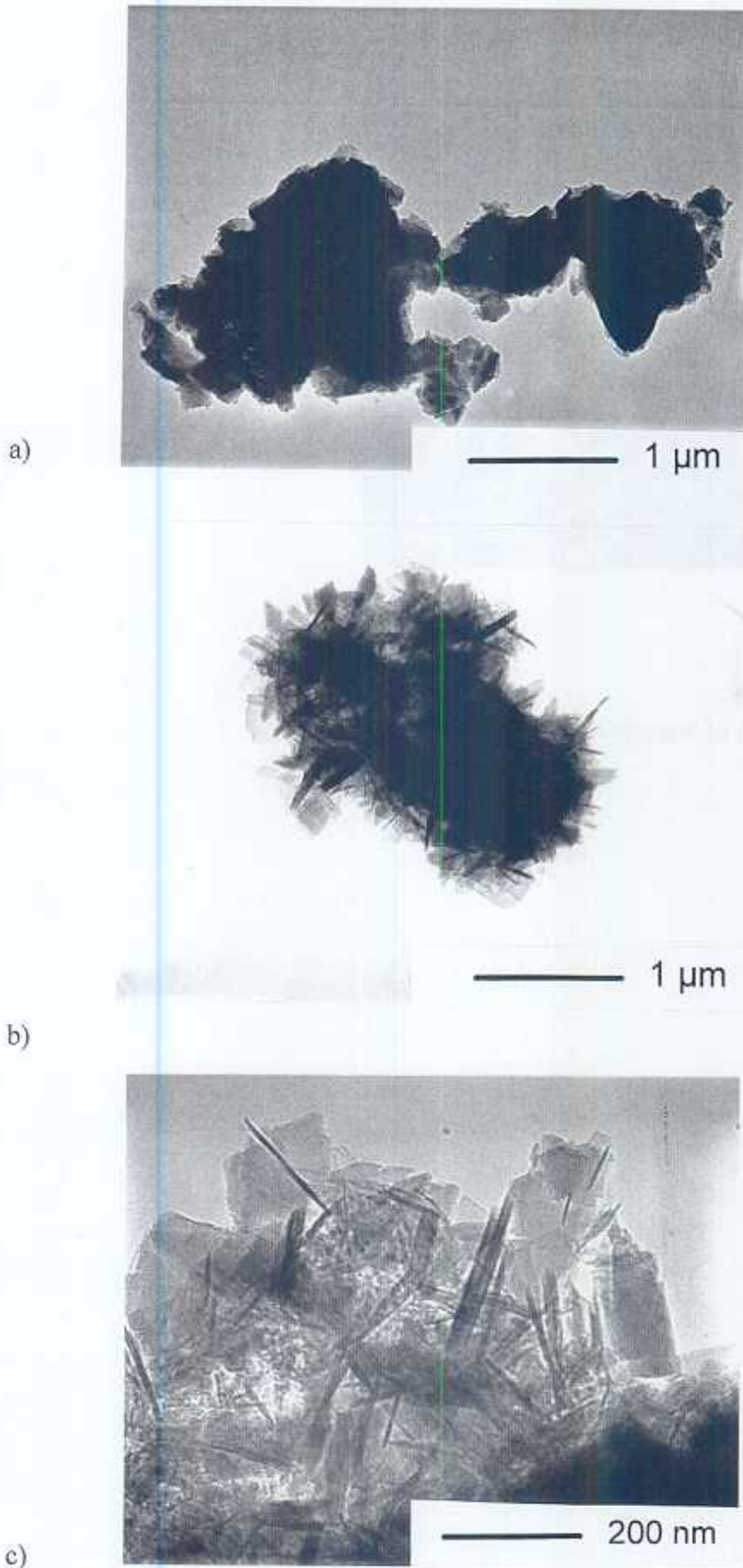


Figure 10, Alumina wear debris a) produced in wear test, b) reacted with water at high temperature and pressure to demonstrate formation of aluminium hydroxide, and c) close-up of edge of particle shown in b).

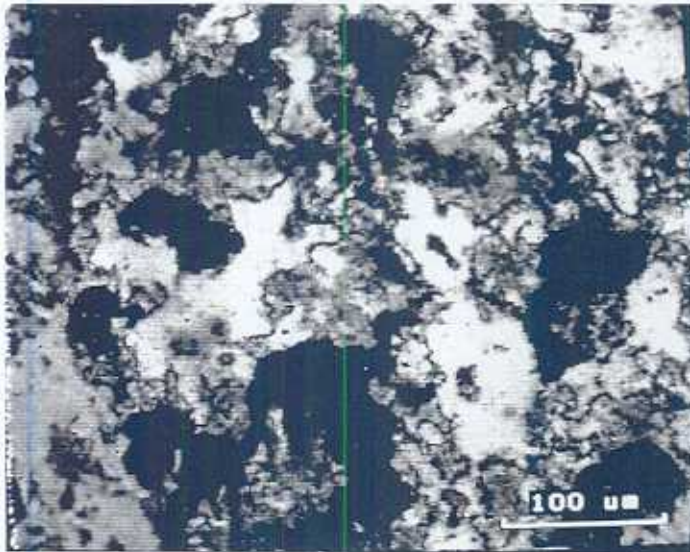
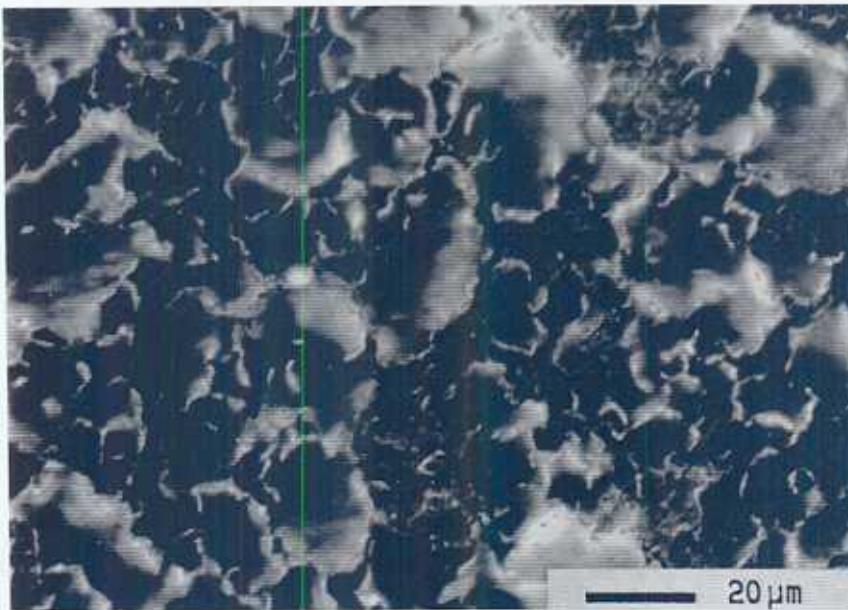


Figure 11, Acoustic micrograph of worn alumina surface.



a)



b)

Figure 12, Backscattered electron image a) of worn alumina surface compared with secondary electron image b) of same area showing aluminium hydroxide areas giving darker contrast.

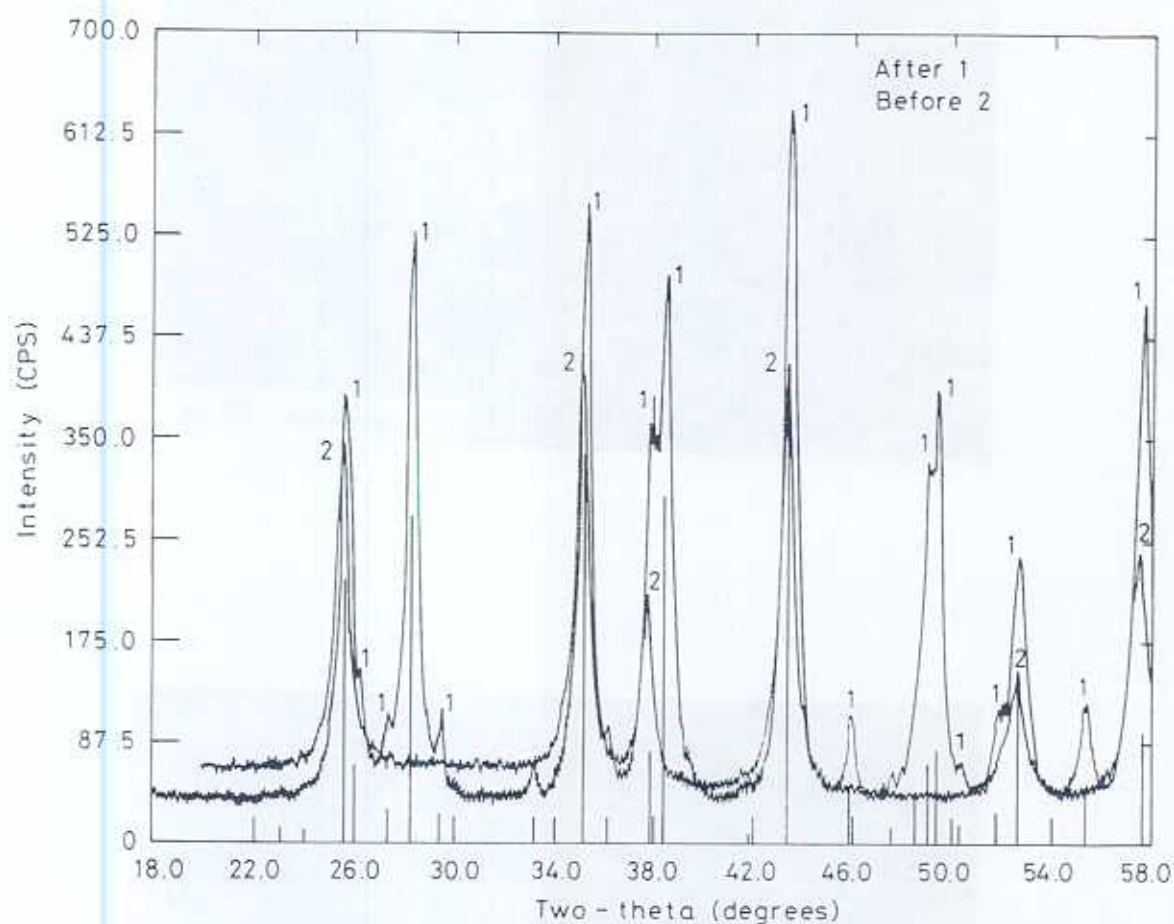


Figure 13, X-ray diffraction spectra showing formation of aluminium hydroxide in wear debris from sliding wear test on alumina. Peaks marked 2 are from a sample of debris taken directly from the test. The X-ray peaks are attributable to alumina. The peaks marked 1 are from a sample of debris which has been subjected to treatment by water at a high temperature and pressure; the additional peaks are attributable to aluminium hydroxide (boehmite).