Abstract

Throughout the history & development of materials engineering the microscope has been used to reveal & understand the microstructure of a material. The microstructure is the fundamental characteristic that gives a material its properties. Along with this development the procedure for revealing this microstructure has developed as well. This procedure is metallography. Over time many publications by notable scientists and engineers have enabled the metallographer & materials scientist to understand how to prepare their materials in the correct manner. Samuels - (Samuels, L. E. 1967) - Bousfield (Bousfield, B. 1992) and VanderVort (Vander Voort, G. 1999) for example

Whilst theses publications are very detailed & contain huge amounts of information on metallographic preparation, they barely touch on what can be accomplished and understood by metallographers in a typical materials laboratory just using microscopy. Not only is it possible to gain great insight into a material using assorted contrast techniques on a correctly prepared sample but microscopy also enables the metallographer to examine in detail the metallographic consumables employed in their materials preparation.

When the consumables and their interaction with the sample are understood by regular microscopic examination during the preparation procedure, it is possible to create preparation procedures that are consistent & repeatable.

In this project it will be shown what information can be obtained through a wide range of microscopical techniques, how different techniques provide different information and how even the simplest of techniques can give great insight into the consumables to be used. In addition, it will show how microscopical examination during preparation aids in generating consistent preparation procedures, and also how the use of assorted microscopical contrast techniques post preparation can greatly increase the information that can be obtained from a prepared sample.

Introduction

Metallography is often erroneously defined as the study of metals when in fact Metallurgy is the correct term for the study of metals. Metallography can be defined as the study of the physical structure and components of metals typically examined and studied by microscopy (Diez. D - 2013)

More current studies of a wide range of materials by similar techniques and equipment are occasionally recognised by the term Materialography. This reflects the same processes in the study of non-metallic materials.

The principle aim in Metallography is to reveal the microstructure of a material and thus a greater understanding of a material's properties and behaviour through the use of microscopical techniques.

This can be achieved in many ways but all require the systematic removal of material to reveal the underlying structure. This systematic removal is the critical path to revealing the true microstructure of the material.

The operations that are used in metallographic preparation require the use of a range of assorted abrasive surfaces, abrasive compounds and lubricants to aid in the removal of all traces of structural damage. This is undertaken using stages of progressively finer abrasives and progressively less aggressive surfaces until all previous induced damage has been removed. The result is a true microstructure ready for examination and evaluation.

The early history of metallographic preparation

Metallography was first introduced to the world by Henry Clifton Sorby (1826 – 1908). (Clinging, V - 2009). Sorby was a pioneering amateur scientist with interests in marine biology, chemistry & geology. During his life he prepared numerous rock thin sections (Raith M.M, Raase,P, Reinhardt.J 2012) and examined them using an optical microscope to determine their microstructure & properties. Reminiscing in his latter years he recalled:

"In those early days people laughed at me. They quoted Saussure who had said that it was not a proper thing to examine mountains with microscopes, and ridiculed my action in every way. Most luckily I took no notice of them."

However in 1863 Sorbys attention was turned to the study of metals and thus established the science of microscopical metallurgy and metallographic preparation. Commenting towards the end of his life Sorby noted.

"In those early days, if railway accident had occurred and I had suggested that the company should take up a rail and have it examined with the microscope, I should have been looked upon as a fit man to send to an asylum. But that is what is now being done..."

Sorby through the use of etching metallographically prepared samples noticed that the microstructure of metals could be revealed & observed using an optical microscope.

It was by using an etchant to create contrast on a polished block that Sorby was able to discover and assess the fundamental characteristics of iron and steel.

Sorby undertook numerous chemical experiments and determined that with the addition of small quantities of carbon to iron, the strength of steel was increased significantly. This pioneering work enabled the engineer Henry Bessemer (1813 - 1898) and businessman Robert Forester Musher (1811 - 1891) to develop methods for the mass production of steel (Hammond, 1989), and the industrialisation of the present day. Sorby's pioneering microscopical work in metallurgy resulted in the invention of the spectrum microscope, an instrument that initiated yet another new branch of scientific study "Microspectroscopy" (Hardwick and Williams, 1980).

The continued development of metals & alloys necessitated a higher degree of detailed microscopical examination and the employment of certain preparation techniques. It was not until the 1930's that any further advances occurred. The development of the metallurgical microscope by Rosenhain (Rosenhain, 1920 Gifkins, 2001) was a significant milestone while in 1938 Vilella (Vilella, J. R. 1938) published methods detailing the preparation and examination of steels for microstructural evaluation,

During the mid-nineteenth century it could take 5 weeks to prepare a sample for evaluation and even in the 1930's it could take many hours. (Gifkins, 2001)

The next significant development occurred during the 1940's – 1950's where Samuels (Samuels, L. E. 1967) carried out work into the understanding of metallographic preparation of steels. The developments in optical & electron microscopy during the mid 20th century provided the tools to further the investigation of metallographic preparation. Initially samples were prepared manually and concentrated primarily on metals such as steels, cast irons, aluminium, bronzes and brasses; typically the ferrous & non-ferrous metals.

For this simple range of materials, a combination of grinding with silicon carbide abrasives of various grades and 'polishing' with hard and soft cloths with diamond paste were often all that was needed to provide a satisfactory surface finish. These items are typically called metallographic consumables.

In the late 20th century a wide range of engineering materials such as advanced composites and ceramics were developed and further metallographic consumables and techniques were introduced enabling the preparation of damage free sample for microstructural evaluation. With these advances in both the understanding of the consumables and the progression towards using semi-automatic machines replacing labour intensive and variable manual preparation, Metallography can be considered to have developed from an art to a science. Vander Voort (Vander Voort, G. 1999) & Bousfield (Bousfield, B. 1992) In particular Bousfield's work towards creating traceable metallographic standards and auditing allows for continuous monitoring and standardising procedures in the metallurgical laboratory. With control of parameters such as platen speed, head speed and specimen load combined with consistent quality consumables, repeatable and standardised preparation procedures can now be generated.

Such machines as illustrated above combine a programmable head, twin platens, and a sample holder to allow individual samples to be prepared ensure controlled & repeatable metallographic sample preparation. Controlled preparation means operator independent results, faster throughput and consistent microstructures revealed.

With the global development of the science of Metallography, several companies have formed to produce dedicated machines and consumables to enable the preparation of damage free micro-sections – a common term used to describe samples prepared for microstructural examination. Companies including MetPrep, Buehler & Struers to name but three have become household names for those in the field of metallography. Such companies offer high quality products with proven preparation procedures to match.

Getting back to first principles

What metallographic preparation procedures should be employed to produce these damage free prepared samples and how can this be achieved? More importantly how can it be decided from first principles what consumables should be employed and when?

As the aim is to prepare materials to reveal a true microstructure for examination, then as well as understanding the properties of the material to be prepared it is necessary to understand the metallographic consumables. The material to be prepared will have particular mechanical properties that of course need to be known. For example, is the material brittle or ductile? Is it hard or is it soft? Is it tough or friable etc? We need to know such information before we start any thoughts of preparation.

Just as materials have been developed to possess different properties and characteristics, so the metallographic consumables needed to prepare them have developed accordingly. Understanding the properties of both the material to be prepared and the metallographic consumables to be used is the key to getting a true microstructure in an efficient manner and at a cost effective price. There has been a huge investment in developing a wide range of engineering materials possessing many varied properties; therefore, it is understandable that not all engineering materials can be prepared metallographically in the same manner.

Fortunately, in the materials laboratory there is the technique of microscopy. As a microscope is usually used to examine the metallographically prepared materials, then it makes sense to use microscopy to examine the metallographic consumables as well. In addition, when microscopy is employed in its wide variety of forms it is possible to evaluate how the consumables interact with the materials being prepared. It is then possible to start to understand what is actually happening when metallographic preparation is taking place.

Those carrying out materials preparation in a laboratory will most likely have access to some type of microscope; some will even have access to some of the more sophisticated microscopy techniques at their disposal. With the assorted techniques available it is possible to evaluate consumables microscopically and determine how they will behave and interact with engineering materials during metallographic preparation.

Materials & Methods

There is a wide range of microscopical techniques that exist in metallographic laboratories. It is the intention to examine these techniques to see what can be accomplished in our aim of evaluating our consumables with a view to preparation.

Specimen preparation requires an abrasive, a surface and a lubricant. As mentioned in the introduction, there is a wide range of metallographic consumables available. These include abrasive papers, diamond grinding discs, polishing cloths and diamond suspensions.

It is the intention within this investigation to determine what microstructural techniques can be employed and consequently what information can be gleaned from them to assess the metallographic consumables and their behaviour.

It is also intended to determine how microscopy can be used to assess our sample preparation both during and after completion and also to evaluate any artefacts that may occur during the preparation process.

Finally, we will study the prepared damage free specimens using various light microscopy contrast techniques using a metallurgical microscope and determine what each individual technique can reveal. We can then determine what extra information can be gleaned by the use of different contrast techniques on a selection of materials.

The first area of investigation will be to look at grinding surfaces using fixed abrasives. This includes Silicon Carbide paper, Zirconia paper & fixed diamond discs

The next area to be investigated is the surfaces where the abrasive is usually dispensed on to the surface. Typically these are called polishing cloths. Polishing cloths is actually a misnomer as we will be using these surfaces to remove structural damage by grinding as well as polishing but this will be addressed later.

Cloths should really be considered to be of two types. The harder cloths that are used to remove damage by progressively finer grinding to produce damage free microstructures and the polishing cloths, usually with a nap of some description that can be used to remove any final scratches. In addition to these various surfaces we will also look at the abrasives used on or embedded in these surfaces.

The equipment available to carry out the examination includes;

Olympus LEXT 3100 - Laser Scannng Confocal Microscope - (LSCM)
Joel Scannng Electron Microscope - (SEM)
Leica DM2500 Metallurgical Microscope - DM2500
Letca S6d Stereo Microscope - S6d
Leica DFC 295 camera combined with Leica Application Suite imaging package will be employed on both S6d and the DM2500 optical microscopes.

The characteristics of these instruments for investigation purposes are as follows;

LSCM - No sample preparation required, large samples can be examined, high resolution in the Z axis using 408nm laser and white light for colour imaging. Options to carry out measurements, magnifications up to 1000x. Drawback, limited illumination from above

SEM - Great depth of field by using secondary electrons, options for elemental analyse, High resolution and greater magnifications to 10000x or more. Drawbacks, monochrome imaging, time consuming, requires prior preparation for non conductive materials and sample size is restricted

DM2500 - Uses white light in the reflective mode to create observations up to nominally 1000x. No sample preparation required unless looking at cross sections, a range of contrast techniques available, large samples can be examined, colour imaging and measurement possible. Drawbacks, limited depth of field but z-axis software available to allow imaging stacking, designed for polished and prepared samples therefore limited use on surfaces with topography.

S6d – Allows examination from 6 – 40x and creates '3D' stereo views using two independent optical axes though not usually imaged in stereo it is possible to do so. Ideal for studying surface texture and morphology at low magnifications, can examine large surfaces, no additional preparation required. Drawbacks, limited resolution with a limited magnification range.

The subject examination and the microscopical techniques to be employed will be as follows and the associated figures generated are indicated.

• Silcon Carbide paper	S6d, DM 2500, LSCM, SEM	*Figures* 1 - 17
• Zirconia paper	S6d, DM 2500, LSCM, SEM	*Figures* 18 - 24
• Fixed diamond grinding discs	S6d, DM 2500, LSCM, SEM	*Figures* 25 - 26
• Grinding & Polishing cloths	S6d, DM 2500, LSCM, SEM	*Figures* 27 - 42
• Cloths in Preparation	S6d, DM 2500, LSCM, SEM	*Figures* 43 - 51
• Loose diamond abrasives	SEM	*Figures* 52 - 53
• Assessing preparation progress	DM2500, LSCM, SEM	*Figures* 54 - 63
• Preparation of Cast Iron & CFC	DM2500	*Figures* 64 - 66
• Assessing Artefacts	DM2500, LSCM, SEM	*Figures* 67 - 75
• Evaluating prepared samples	DM 2500	*Figures* 76 - 84

Lapping, Grinding & Polishing

Before starting our investigation, it is necessary to define three of the most commonly used words in Metallography. Whilst it is possibly to argue about the below definitions, if this approach is chosen it gives a concise way of getting to grips with the principles of what is trying to be achieved.

Lapping. Lapping is typically the use of a hard surface covered with a loose rolling abrasive that is not fixed at the point of contact with the sample. It is used to provide a flat and relatively dull surface and is primarily used in the petrographic examination of rocks. Its primary goal is to prepare a flat surface with low damage to view constituent grains in transmitted light. Here on most occasions the sample observed still has relatively large amounts of remaining structural damage but when it is of the correct thickness it can be cover slipped and be studied for mineral content and type identification. For more serious work the sample will require further preparation to remove this damage.

Grinding. Grinding should be thought of as the condition where the abrasive is fixed at the point of contact with the sample. An obvious example is Silicon Carbide paper where the abrasive is clearly fixed to the surface when it comes in contact with the sample. What is not often considered is a similar situation can occur when using abrasives on a cloth. Looking at the cloths without a nap, again the abrasive is actually fixed when in contact with the sample. That is why straight continuous scratches are produced and significant stock and damage removal is achieved.

Polishing. In Metallography, particularly with materials not considered ceramics we ought to think of polishing taking place when a cloth with a nap is employed to remove the final stage scratches. Here the abrasive is moving within the nap as it moves across the surfaces of the sample and is not as fixed as in the grinding mode.

The procedure of metallographic preparation involves taking a sample of material and progressively removing damage by grinding and then removing any final scratches by polishing. Polishing can only be employed when all the damage in the sample is removed and the true structure is capable of being revealed. The grinding will be accomplished in successive stages steadily reducing the aggressive nature of the cloth and additionally reducing the size of the abrasive employed. This should result in a damage free surface for microscopical examination. This procedure will be carried out with consumables particularly suited to the properties of the material being prepared.

In the preparation of ceramics and other hard materials a final polishing stage with a napped cloth is often not required as the damage free surface is already scratch free.

Results and Discussion

Surfaces & abrasives

Silicon Carbide paper has for many years been the most popular grinding abrasive in the metallographic preparation laboratory. Silicon Carbide as well as being a relatively inexpensive product is very sharp and efficient in the removal stock from metallic and softer materials. It is ideal for some composite materials where it can cut multiple hardness materials with little problem. Silicon Carbide paper does however degrade and loses its cutting ability very quickly. This means that the life of a single silicon carbide paper under usual semi automatic preparation conditions is in the region of 60 – 90 seconds. Silicon Carbide is typically available in grades ranging from P80g – P4000g – The equivalent micrometer sizes being 197um & 6.5um respectively.

(a) Examination of Silicon Carbide paper:

To understand this popular metallographic consumable, we can carry out a simple range of examinations using a range of microscopical techniques.

Examinations with a typical laboratory Stereo microscope a Leica Sd6 (*figs1&2*), something that most laboratories possess reveals the presence of particles in a regular arrangement across the paper. In the larger grades a highly reflective surface is also seen. It is however difficult to resolve any real detail at the finer grade papers.

Examinations using a typical metallurgical microscope, (Leica DM2500M) reveals much greater detail. The higher magnification and increased resolution aids in resolving the silicon carbide particles. It also highlights the nature of a highly reflective coating (*figs3&4*).

Examination on the metallurgical microscope was kept to the lower objective magnifications due to the reduced depth of field as the magnification of the objective increases. It does however indicate a slightly different surface morphology in the P4000g paper (*fig 4c*).

Examination with a Laser Scanning Confocal Microscope was carried out using the Olympus LEXT 3100. Examination was carried out at a range of magnifications but only the observations with a 20x (*fig5&6*) and 50x (*fig7*) objective have been reported. Using both a colour camera & a 408 nm blue laser the LSCM allows the collection of data from various Z axis positions. This data can then be combined into 2D or 3D reconstructions to give great detail of a surface as well as numerical measurement data (*fig8*).

Comparing the images revealed by the LSCM with the same magnification images from the metallurgical microscope it can be observed that much greater detail is revealed by the LSCM. This is due to both the resolution of the laser and the resultant 3D image generated. Even at the finer grades more detail is revealed by the LSCM than by using the standard metallurgical microscope. The nature of the Silicon carbides paper surface can be clearly distinguished as can the reflective coating. Additionally we can use the Zaxis data to determine the size of these abrasive particles in 3 dimensions, an operation that is not easily done with a standard metallurgical microscope.

Examination by a Scanning Electron Microscope or SEM is now an option in many laboratories, more so now than in the past. The drawback with the SEM is the time taken to

pump the instrument down and often the requirement of coating the sample with gold to enable non-conductive samples to be examined correctly. Coating is an additional process and incurs more equipment costs. The benefit SEM however is the dramatic increase in depth of field that can be achieved. Observing at the same 200x magnification as used in the metallurgical microscope and LSCM examination allows comparison of the technique rather than the magnification. With the SEM it is now possible to see individual particle details at the coarse particle sizes and considerably more details of the varied surface morphology at the finer sizes. (*figs 9&10*).

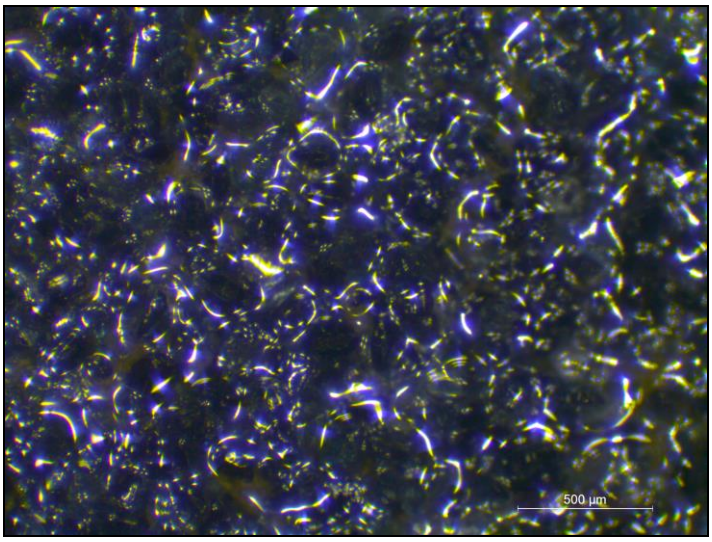
Observing the silicon carbide grades at 1000x in the SEM, the higher grades such as P120g show great detail of the abrasive cutting faces as well as cracking in what appeared to be the earlier highly reflected layer (*figs11&12*). Examination of the finer grade of P4000g it is possible to see that the morphology of the P4000g is completely different to the coarser grades (*fig13&14*).

Further investigations made after this microscopical discovery revealed that silicon carbide paper can be manufactured in one of two ways. The coarser grades tend to be manufactured using the Electro Coating process that produces high angular particles and in turn more aggressive stock removal. The lower sizes, usually only the P2500g & P4000g, are often made using a Slurry Coating process. This is a less aggressive grinding media but consequently a finer finish is left on the sample. (Townsend.N 2012). The latter is always helpful when one is trying to keep damage to a minimum.

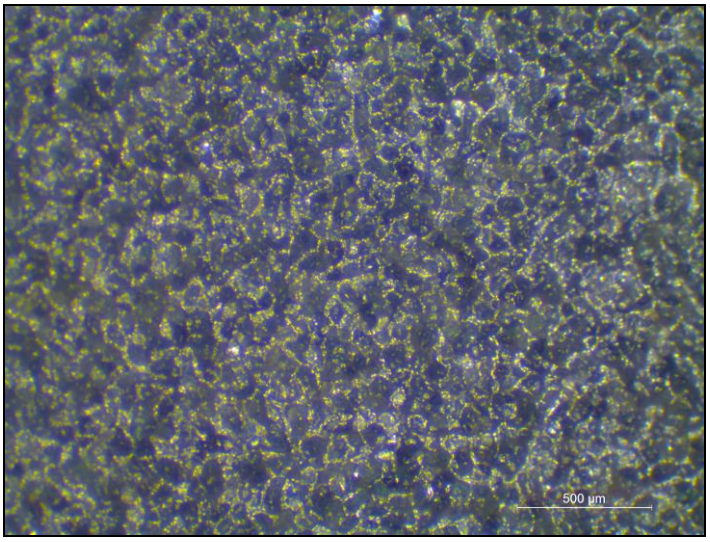
Comparing the above microscopical techniques (*fig15*) it is clear that all the techniques give a good insight in to the nature of the abrasive though the traditional light microscopy techniques can only show limited data of the particles and morphology. The use of the LSCM gives excellent information regarding the nature of particles and to some extent of the coating. In addition, the opportunity to measure the sizes of these particles is a useful advantage particularly if you are trying to compare suppliers for instance. The SEM generated the most detailed information in this examination and managed to resolve the difference between the two different types of Silicon Carbide paper. Information that could have a bearing on the surface produced during preparation.

Unfortunately, little information could be gleaned about the highly reflective coating using the above the techniques therefore a metallographic section was prepared to see what other information could be obtained. Encapsulated in an Epoxide resin and prepared using standard metallographic techniques it was now possible to view a cross section of the Silicon Carbide paper using the metallurgical microscope. (*figs 16&17*). It can now be seen how the Electro Coating process generates the high pointed angle abrasives on the coarser grades when compared to the P4000g paper made by the process of Slurry Coating. Examination in Brightfield and Darkfield illumination using a metallurgical microscope shows a slightly different view but not one that generates further information. There is an indication of a thin bond coat over the abrasives but it is not easily resolved in the photomicrographs. The thin bond coat would indicate the silicon carbide paper would be more aggressive but not last very long. If the bond coat were much thicker it would grind away more slowly but not remove as much material in a given time. For the coarser grades and in particularly on a semi automatic machine it would be preferable to have the increased stock removal.

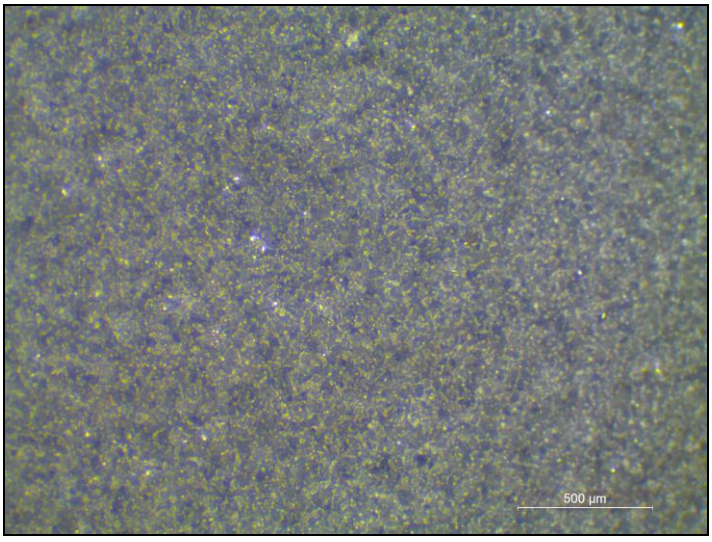
Fig 1. Silicon Carbide Paper - Stereo Microscope Examination



P120g Stereo at 40x

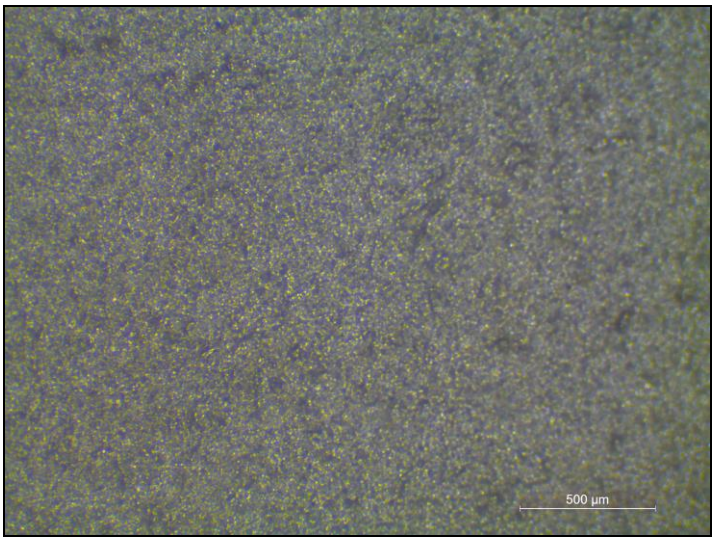


P240g Stereo at 40x

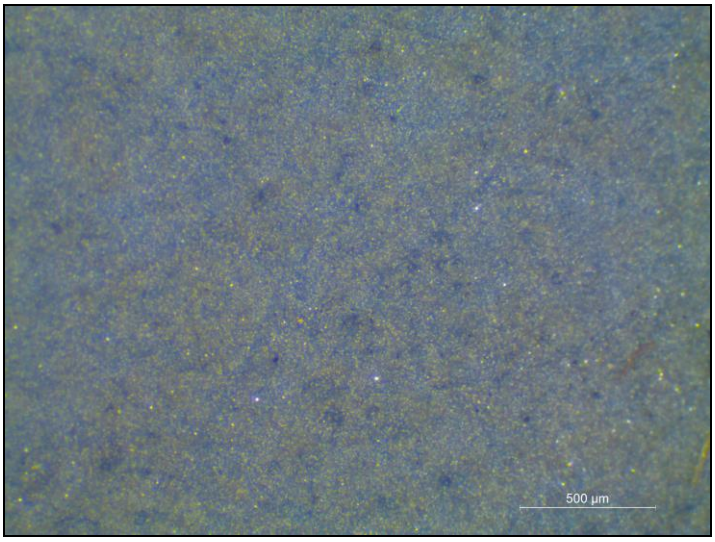


P600g Stereo at 40x

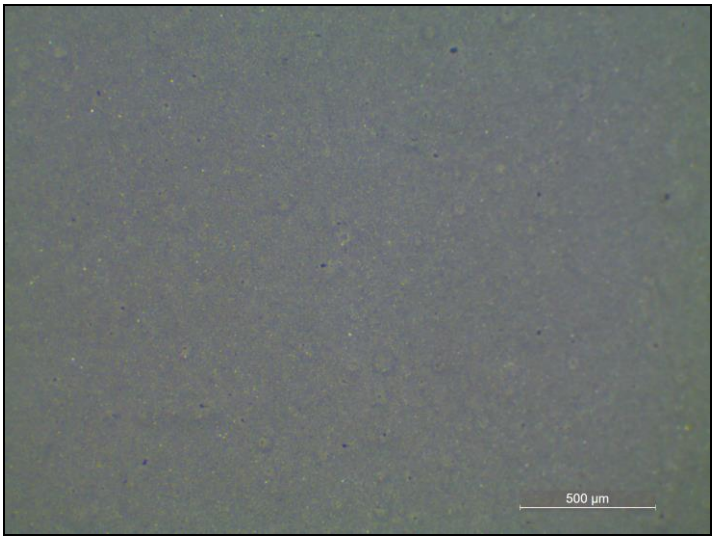
Fig 2. Silicon Carbide Paper - Stereo Microscope Examination



P1200g Stereo at 40x

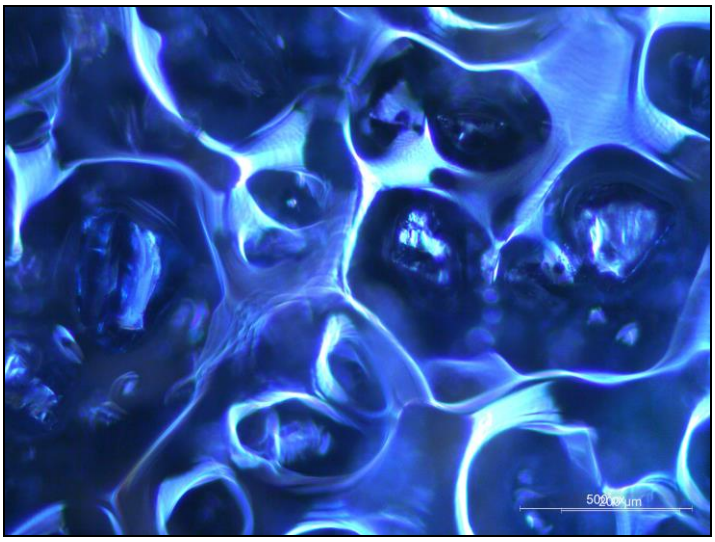


P2500g Stereo at 40x

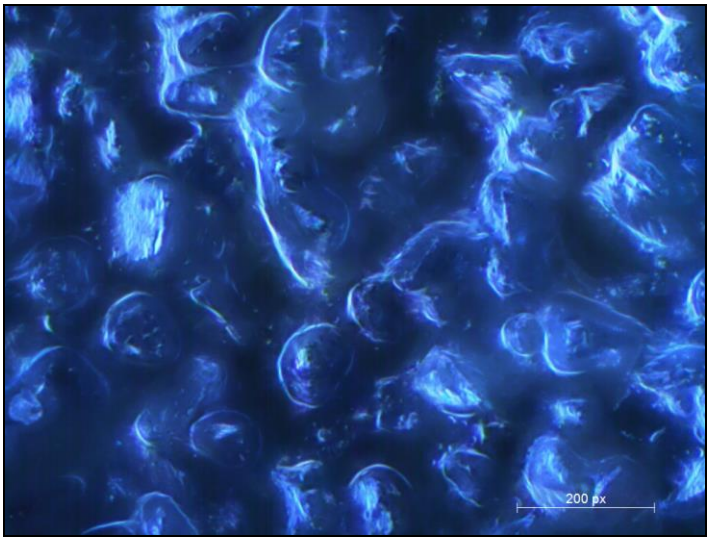


P4000g Stereo at 40x

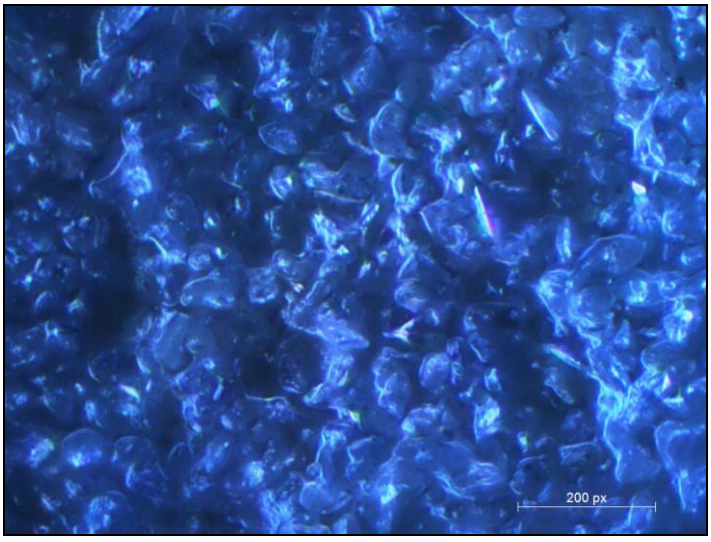
Fig 3. Silicon Carbide Paper - Metallurgical Microscope Examination



P120g 20x objective - Brightfield

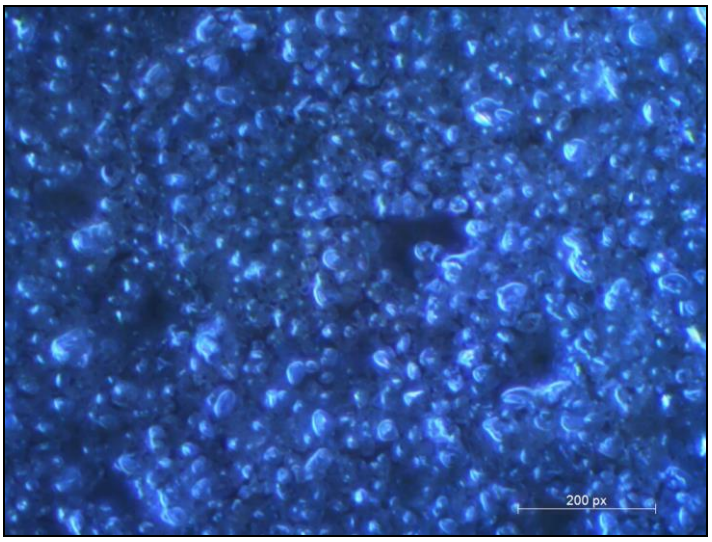


P240g 20x Objective - Brightfield

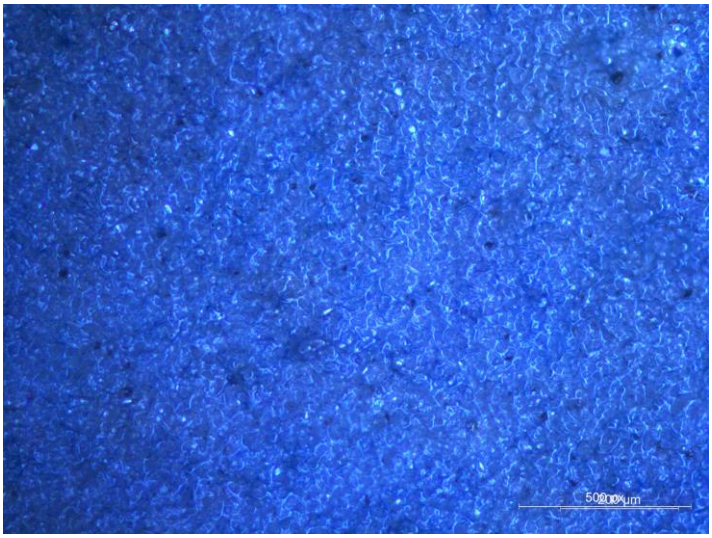


P600g 20x Objective - Brightfield

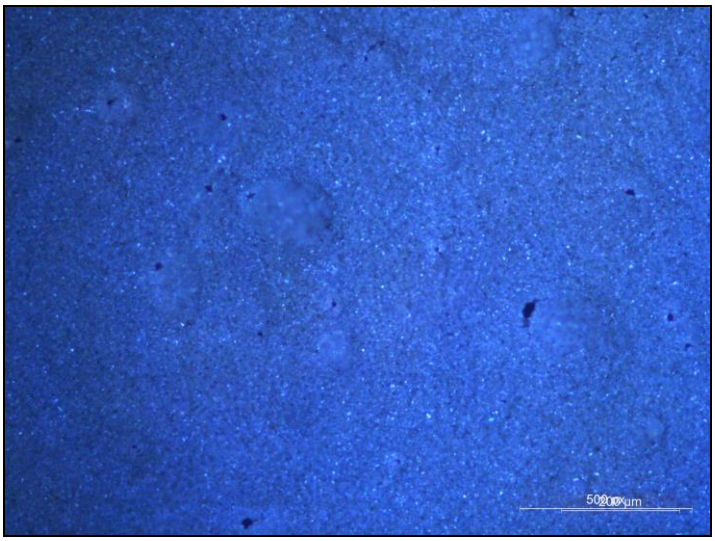
Fig 4. Silicon Carbide Paper - Metallurgical Microscope Examination



P1200g 20x objective - Brightfield

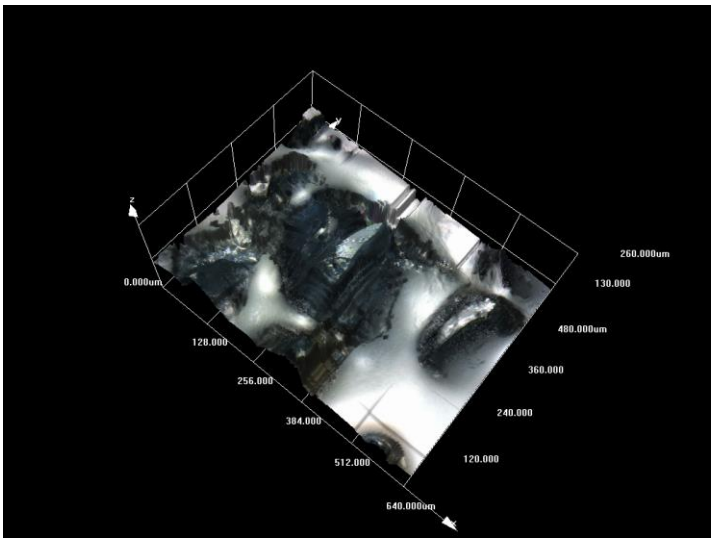


P2500g 20x objective - Brightfield

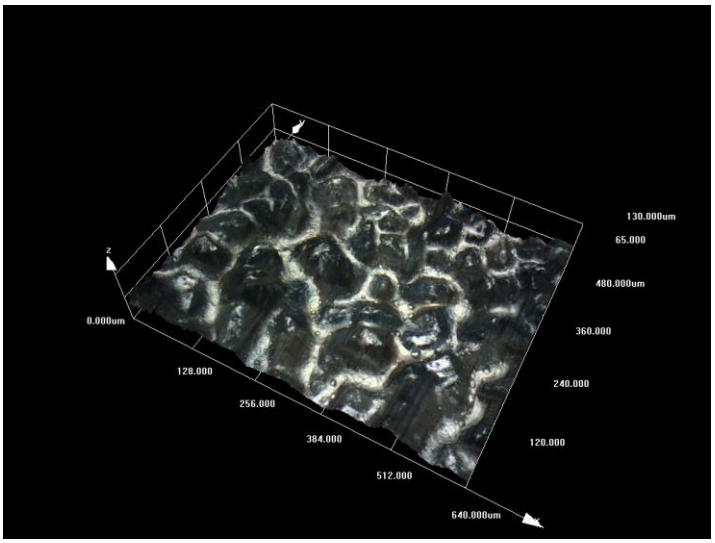


P4000g 20x objective - Brightfield

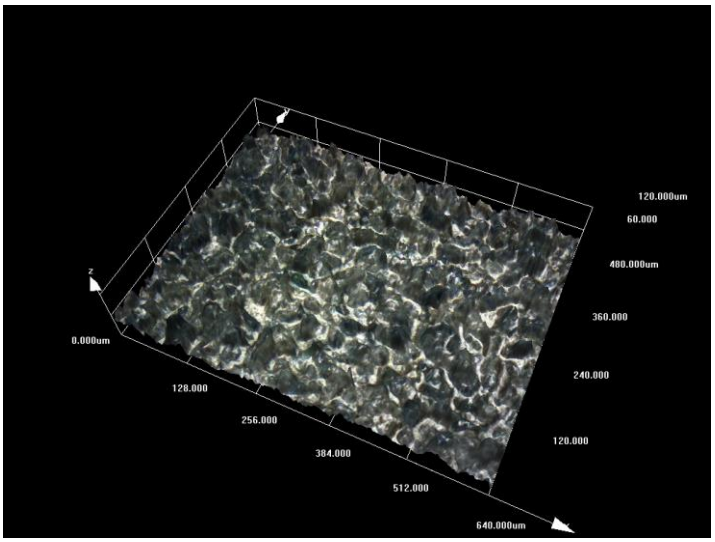
Fig 5. Silicon Carbide Paper - Laser Scanning Confocal Microscope Examination



P120g 20x objective

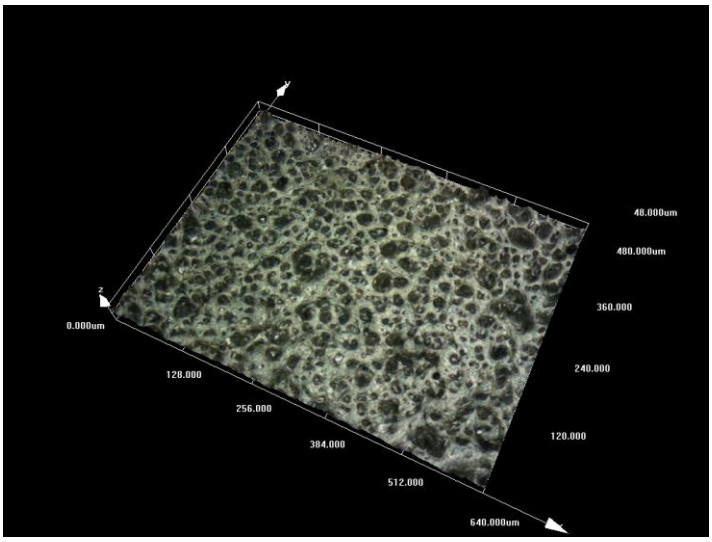


P240g 20x Objective

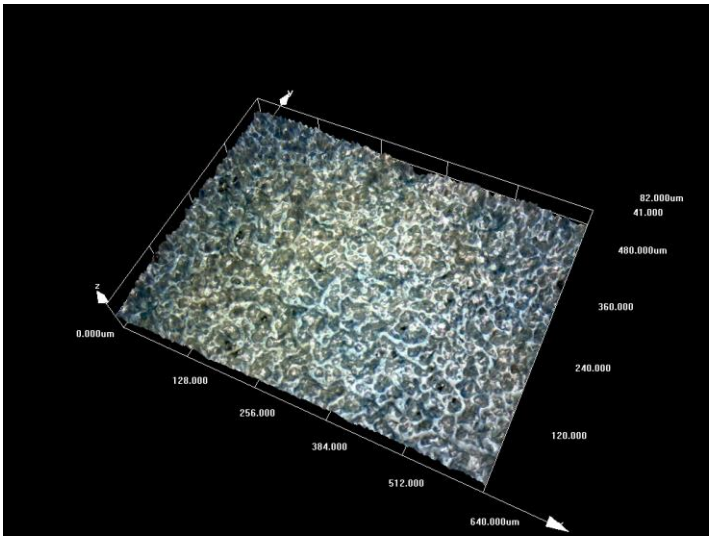


P600g 20x Objective

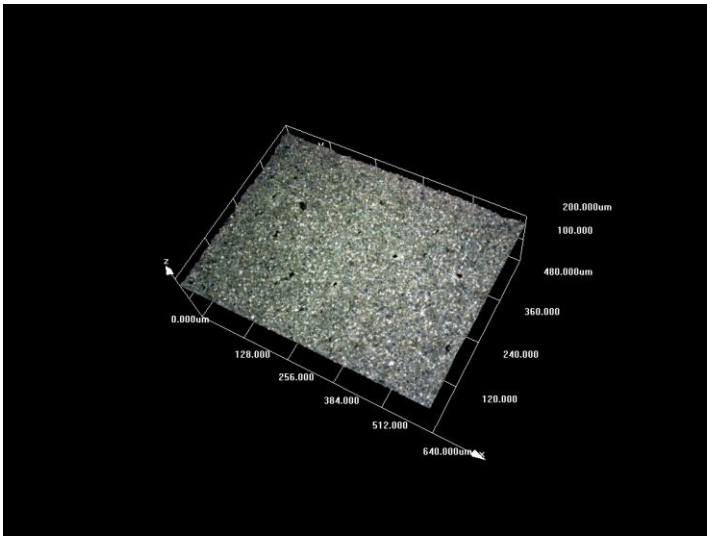
Fig 6. Silicon Carbide Paper - Laser Scanning Confocal Microscope Examination



P1200g 20x objective

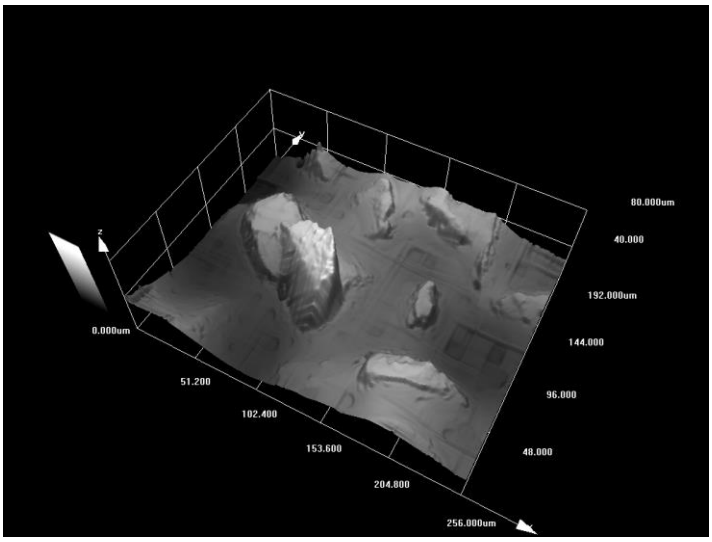


P2500g 20x objective

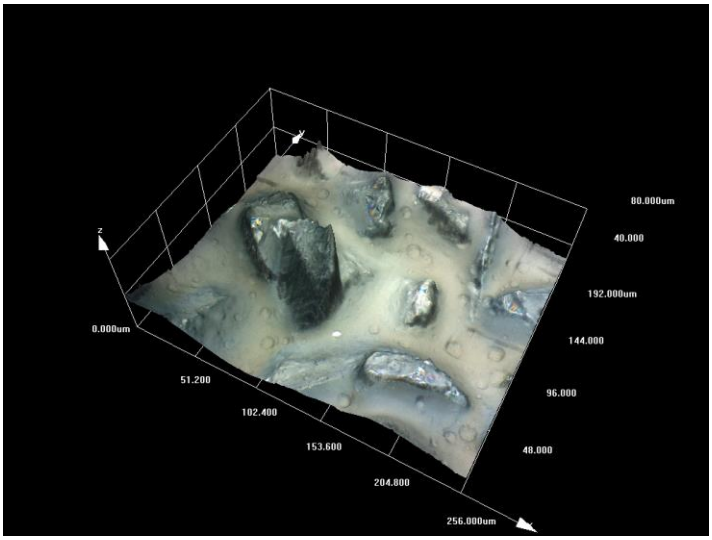


P4000g 20x objective

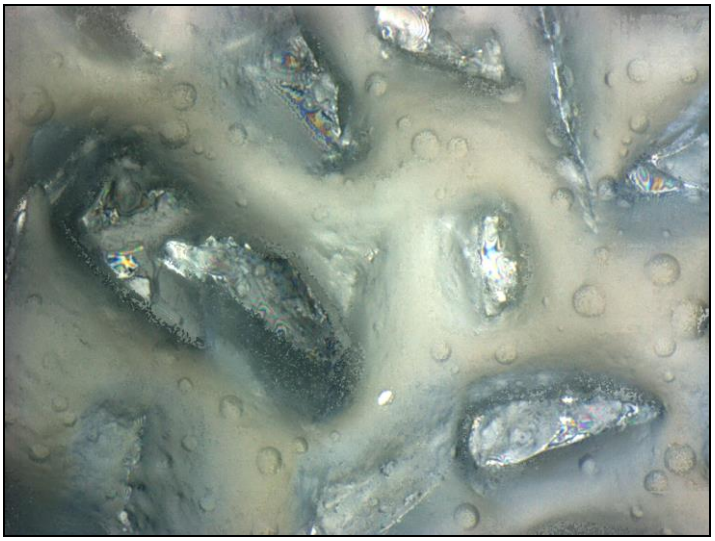
Fig 7. Silicon Carbide Paper - Using Laser Scanning Confocal Microscope techniques



P240g 50x Obj – 3D B & W image

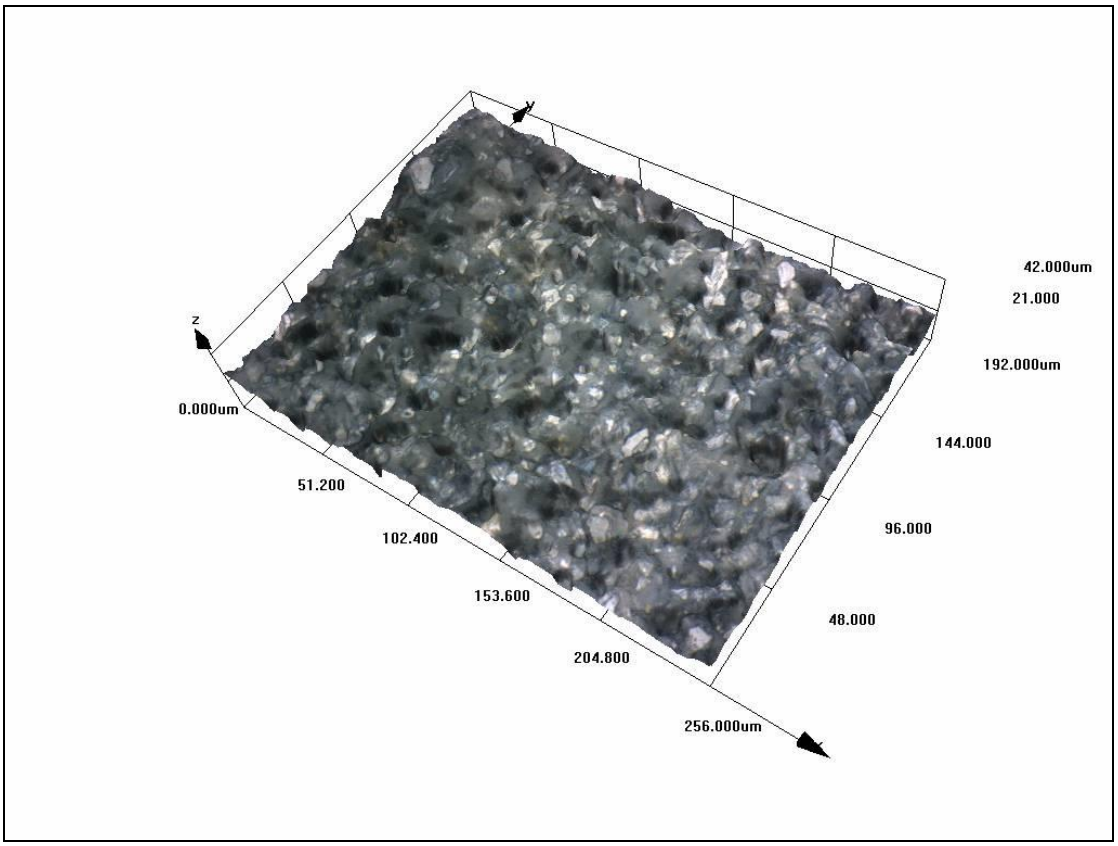


P240g 50x Obj – 3D colour image

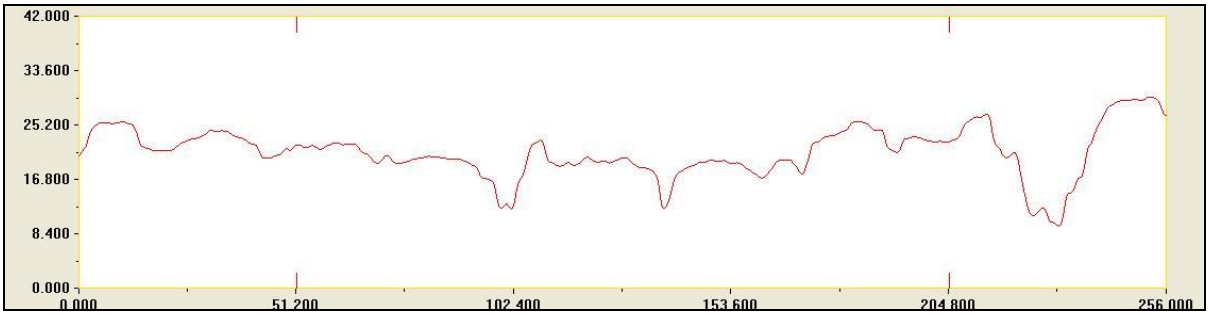


P240g 50x Obj – 2D colour image

Fig 8. Silicon Carbide - Using Laser Scanning Confocal Microscopy techniques



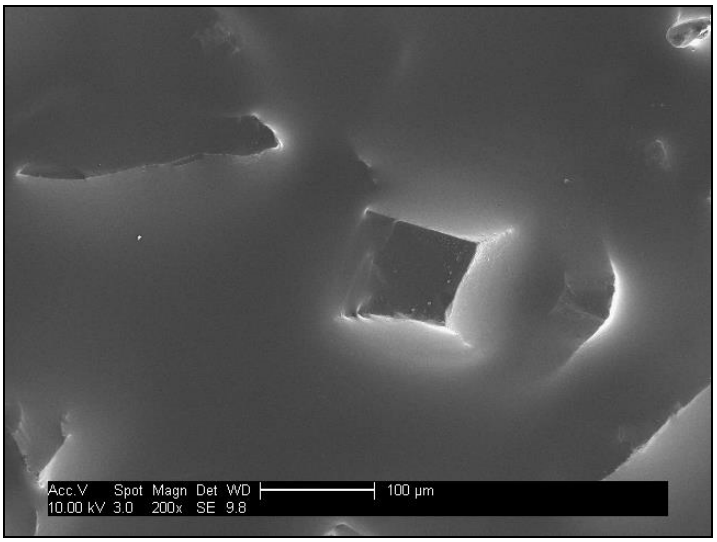
Laser Scanning Confocal 3D Image showing morphology of P2400g in real colour - 100x Objective



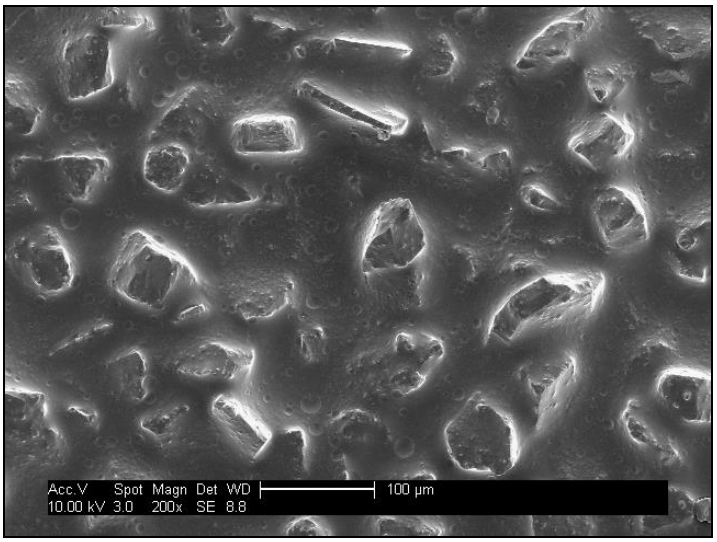
Profile along length of region showing surface topography data. – 100x Objective

The Laser Scanning Confocal Microscope is an ideal instrument for determining true data of a range of surfaces. In addition, samples of a range of surface sizes can be examined quickly and with next to no sample preparation.

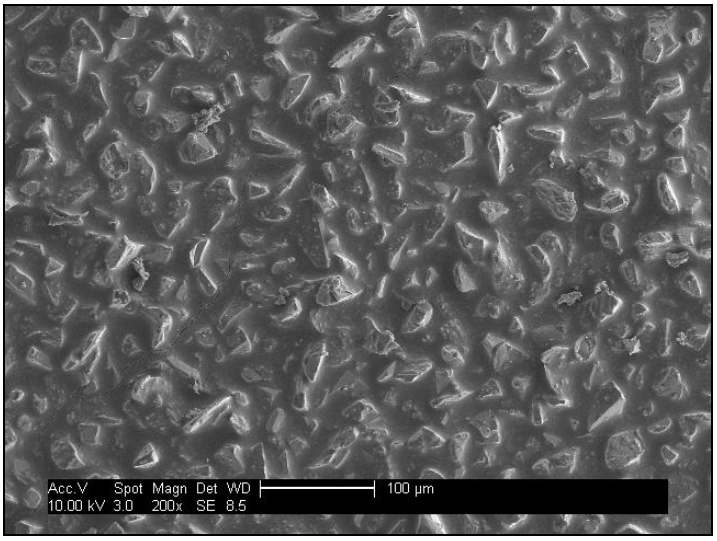
Fig 9. Silicon Carbide Paper - SEM Examination 200x



P120g 200x

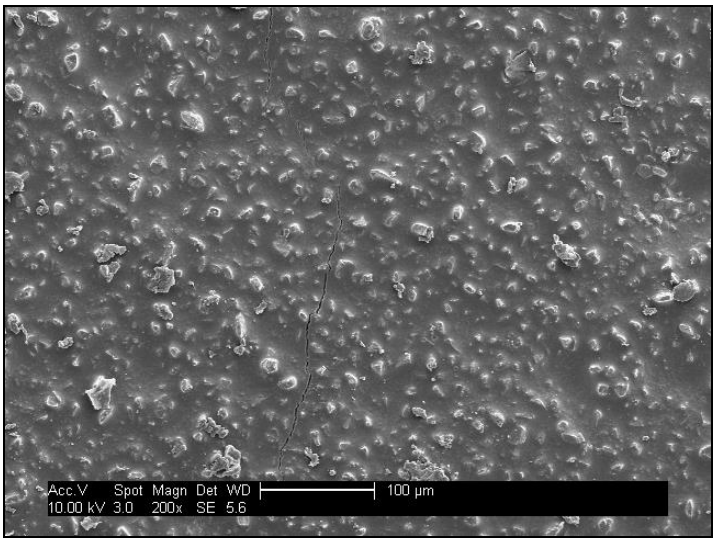


P240g 200x

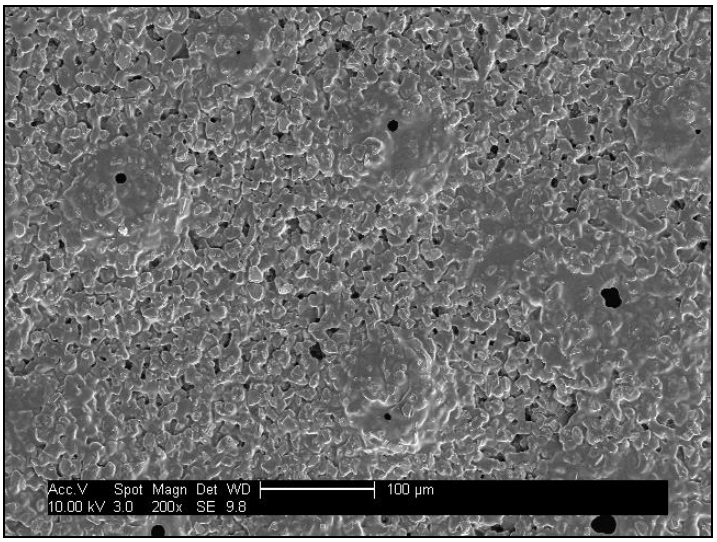


P600g 200x

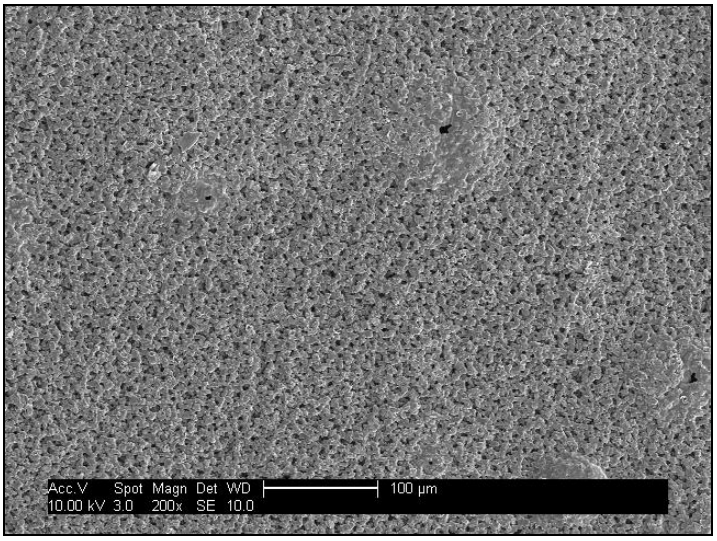
Fig 10. Silicon Carbide Paper - SEM Examination 200x



P1200g 200x

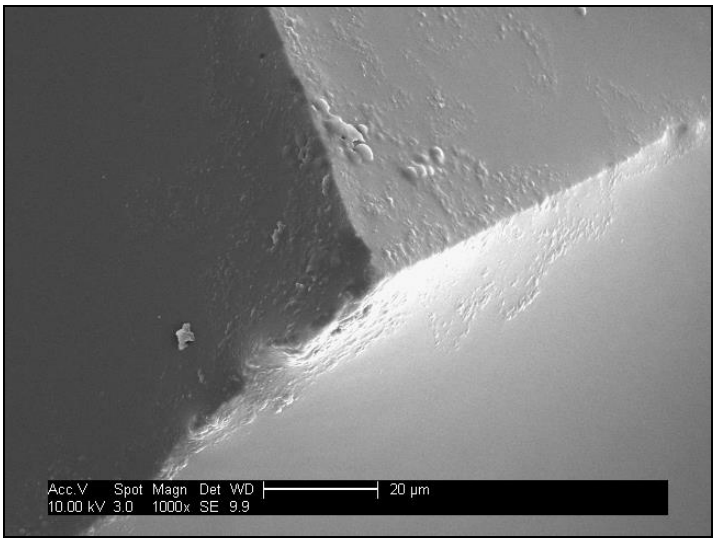


P2500g 200x

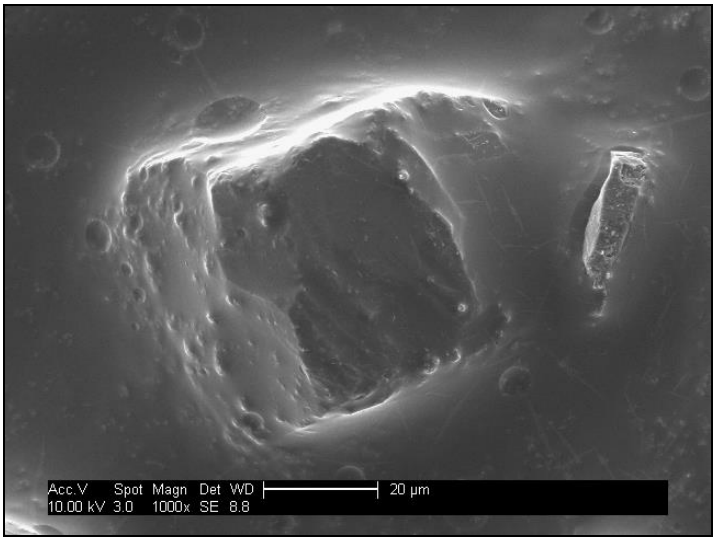


P4000g 200x

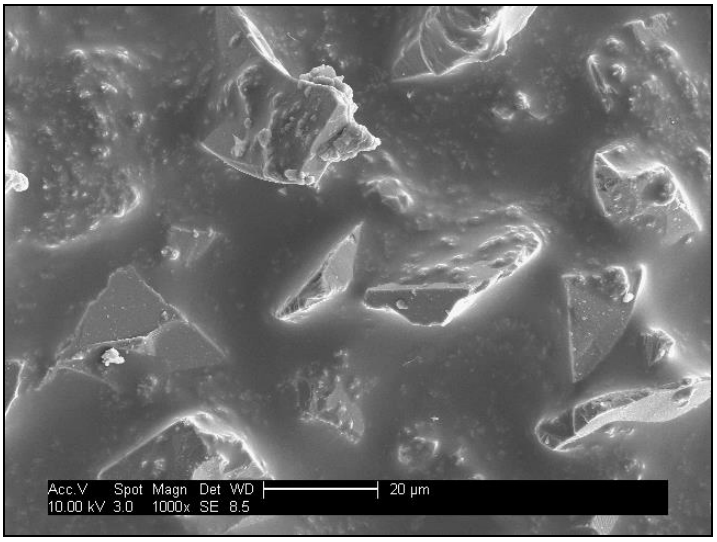
Fig 11. Silicon Carbide Paper - SEM Examination 1000x



P120g 1000x

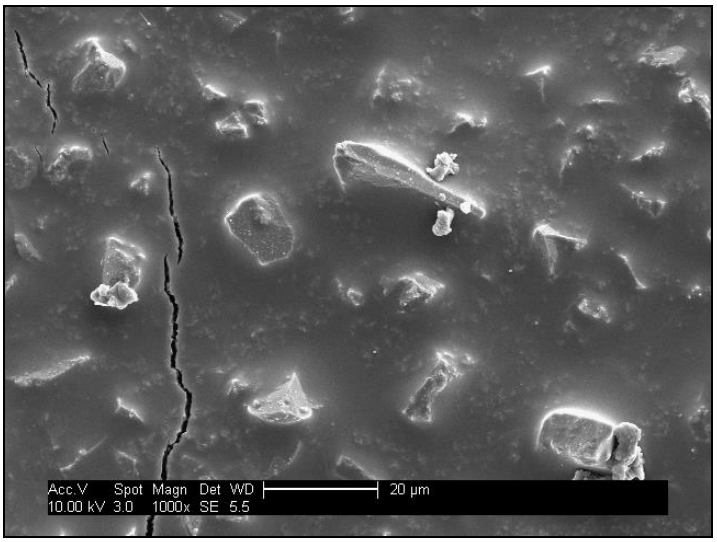


P240g 1000x

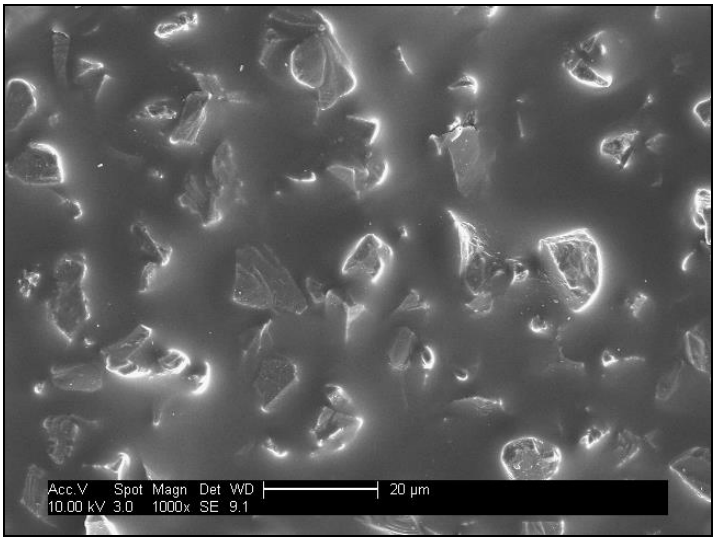


P600g 1000x

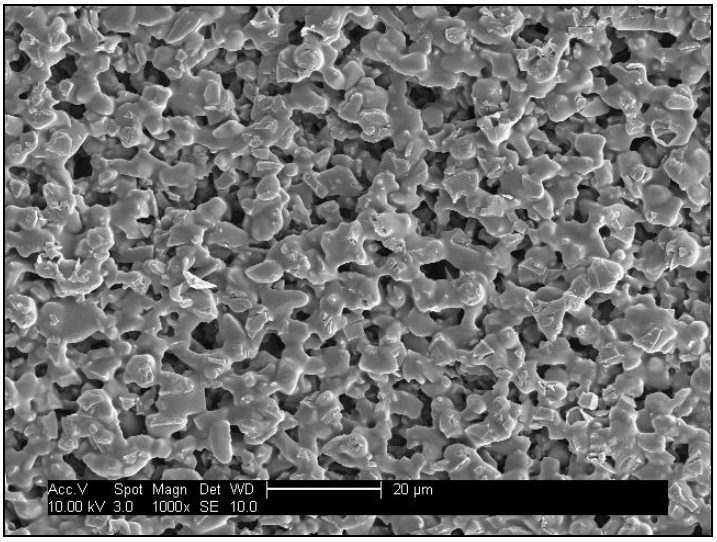
Fig 12. Silicon Carbide Paper - SEM Examination 1000x



P1200g 1000x

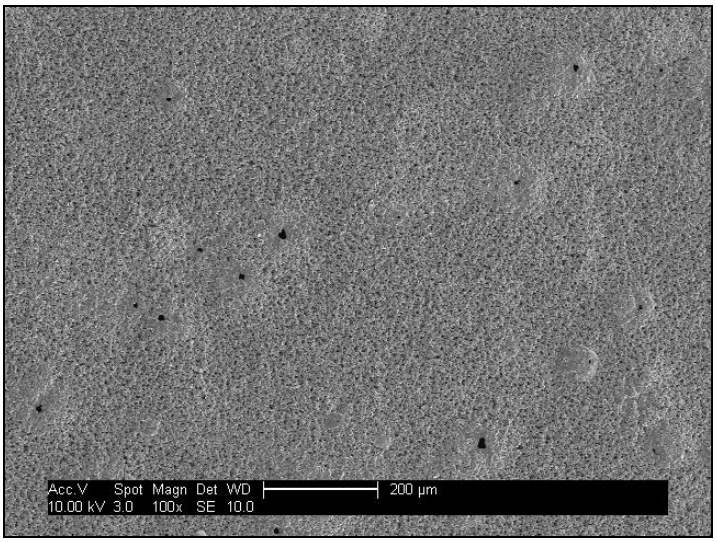


P2500g 1000x

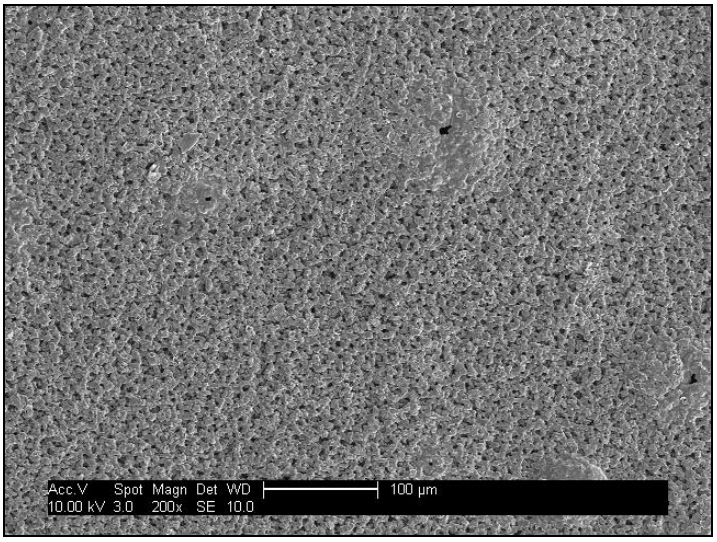


P4000g 1000x

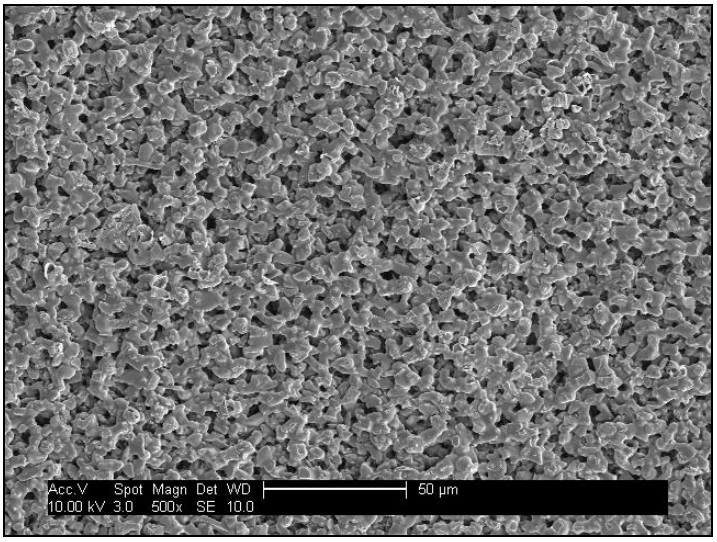
Fig 13. Silicon Carbide Paper - P4000g SEM Examination



P4000g 100x

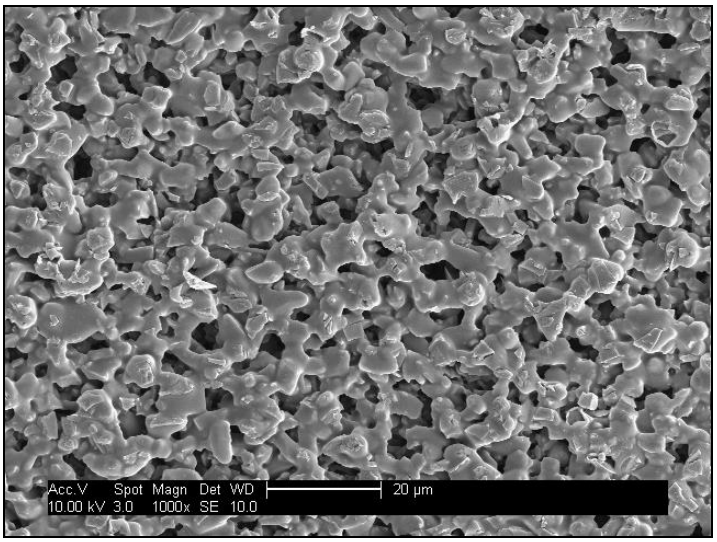


P4000g 200x

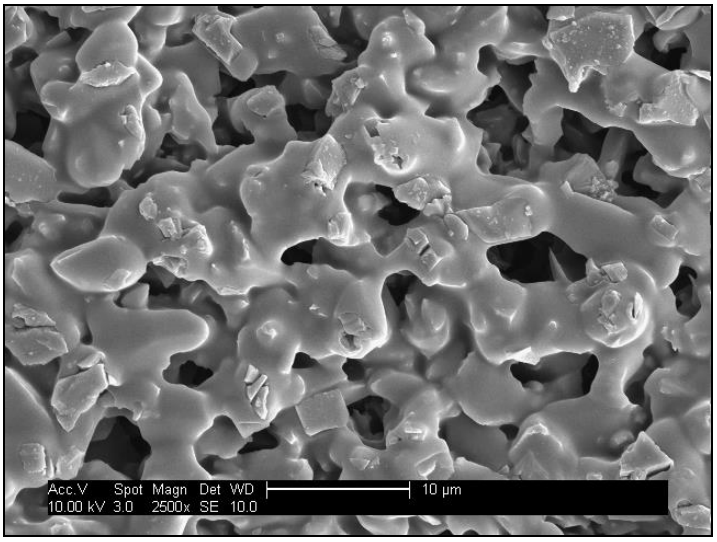


P4000g 500x

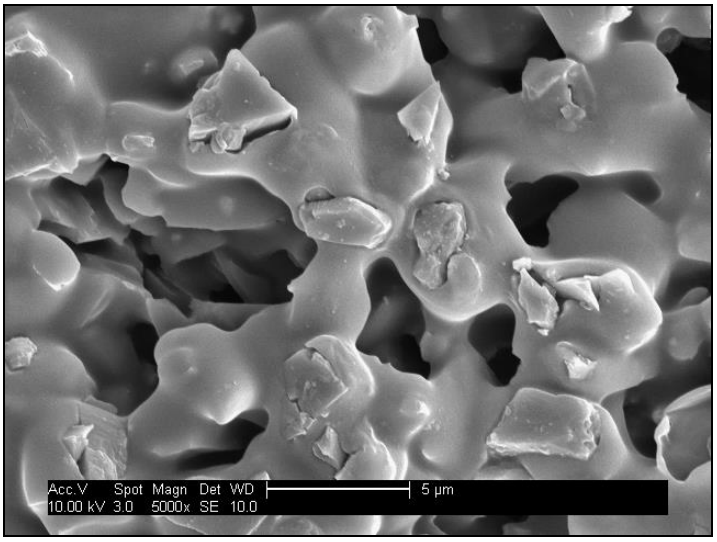
Fig 14. Silicon Carbide Paper – P4000g SEM Examination



P4000g 1000x

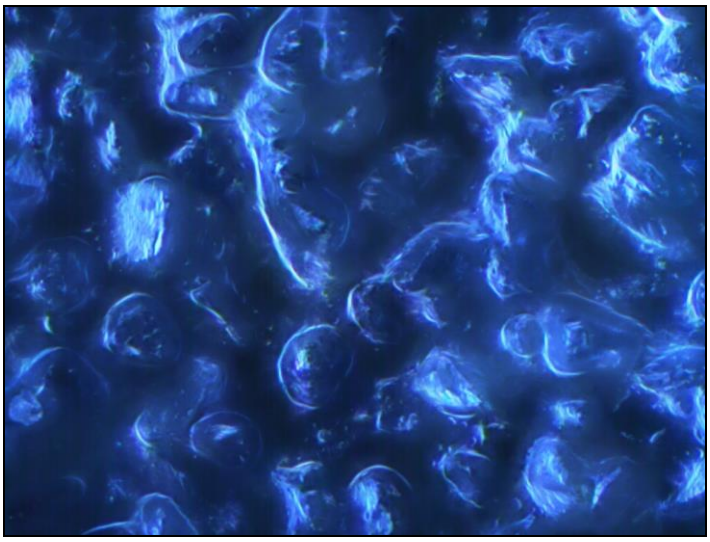


P4000g 2500x

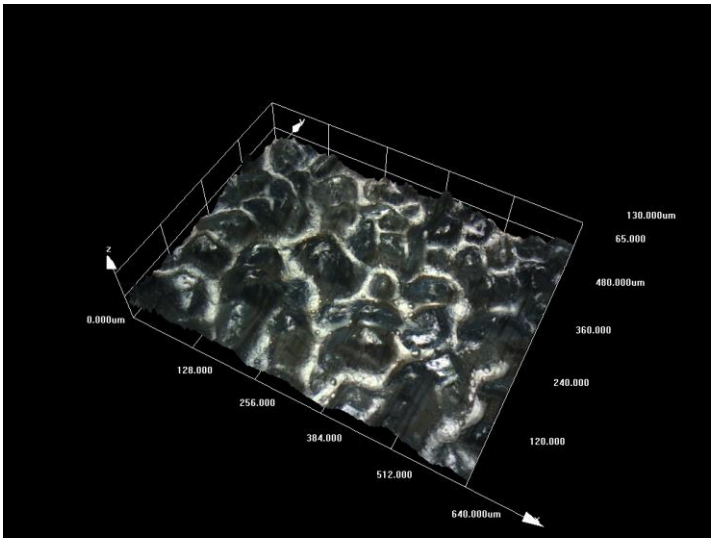


P4000g 5000x

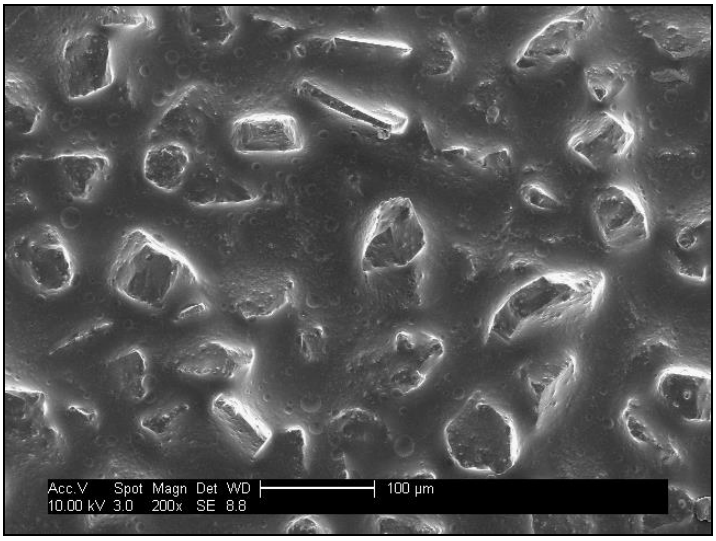
Fig 15. Silicon Carbide Paper - Technique comparison



P240g 20x Objective Brightfield

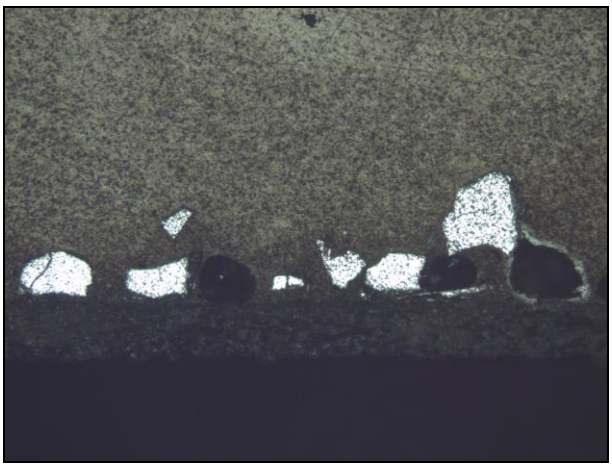


P240g 20x Obj – LSCM 3D image

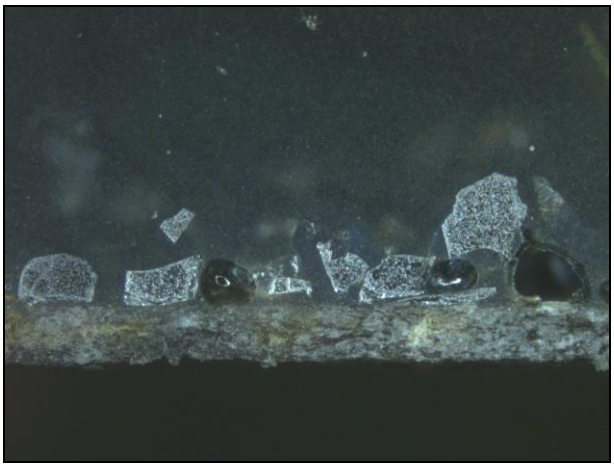


P240g 200x – SEM image

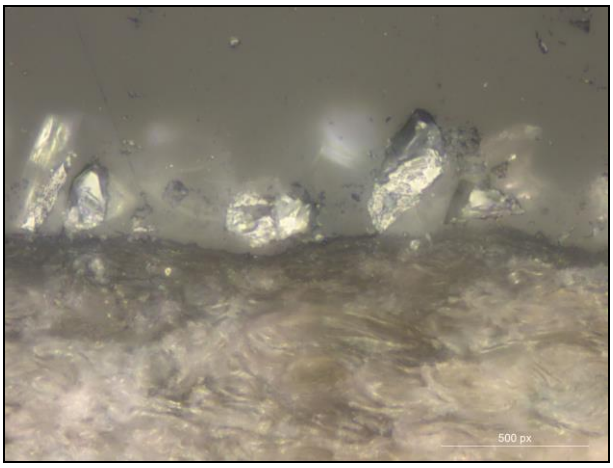
Fig 16. Silicon Carbide Paper - Metallurgical Microscope Metallographic Examination



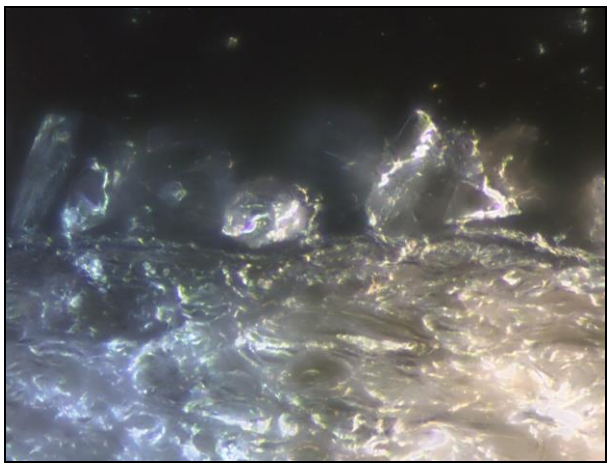
P60g 5x objective - Brightfield



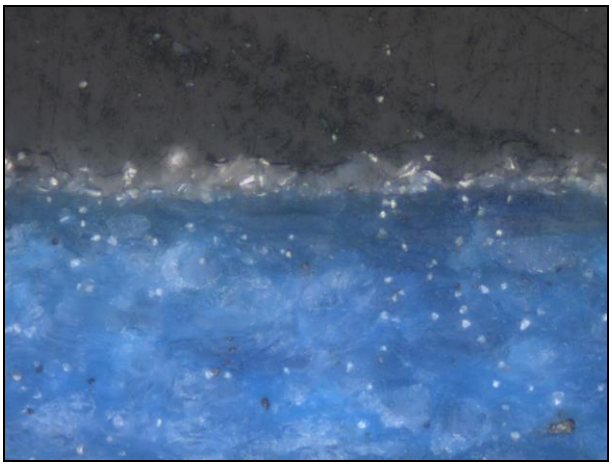
P60g 5x objective - Darkfield



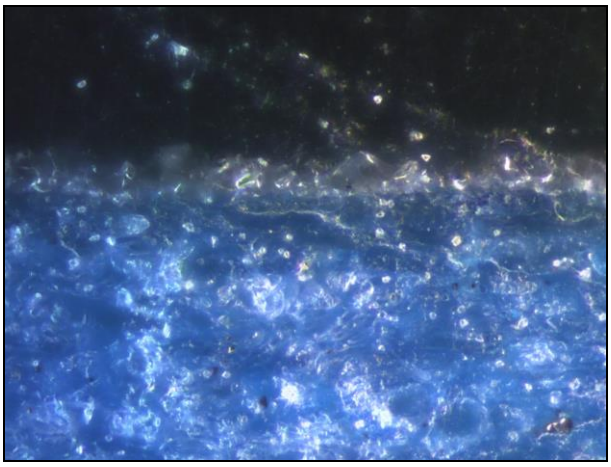
P400g 50x Objective - Brightfield



P400g 50x Objective – Darkfield

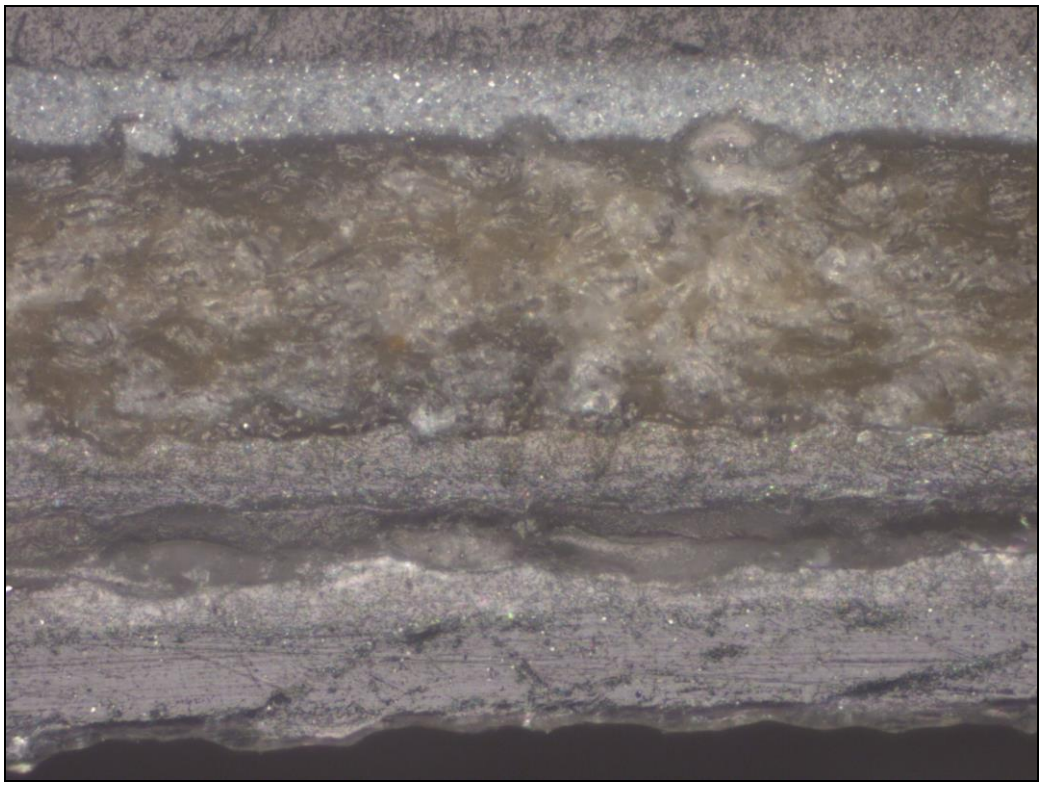


P2500g 50x Objective - Brightfield

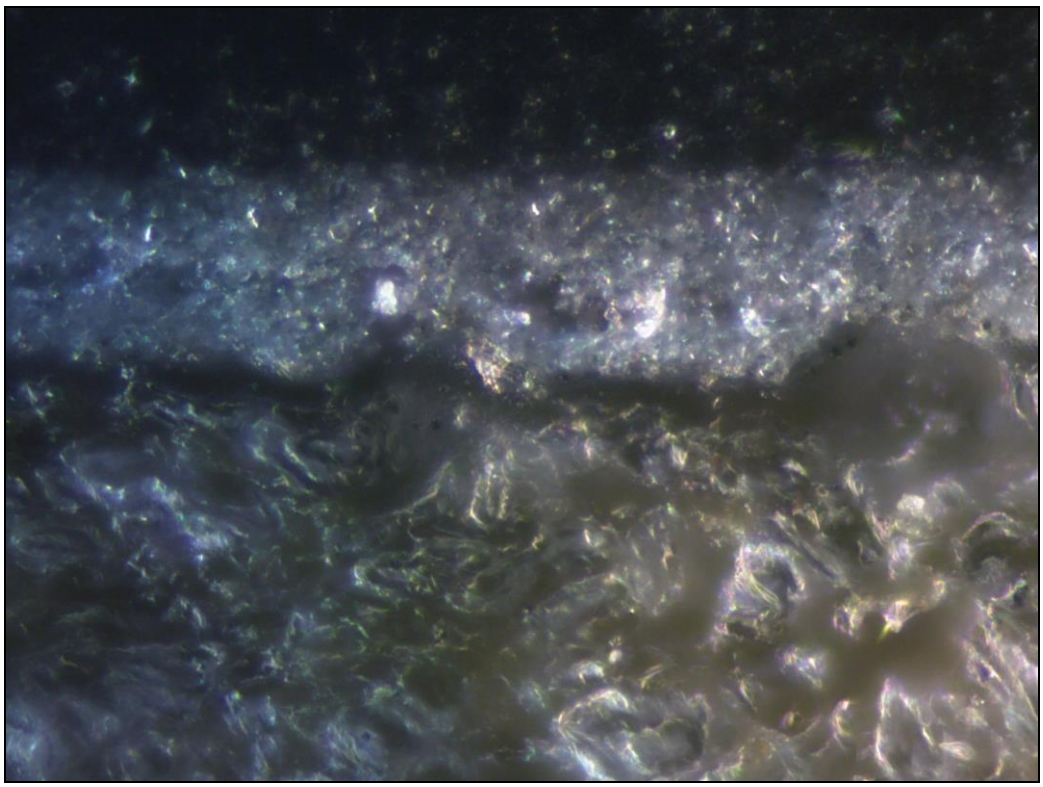


P2500g 50x Objective - Darkfield

Fig 17. P4000g Silicon Carbide Paper - Metallurgical Microscope Metallographic Examination



P4000g 50x objective - Brightfield



P4000g 50x objective - Darkfield

(b) Zirconia Paper

Zirconia paper is similar in nature to that of Silicon Carbide and consists of grains of Zirconia (or on occasion combined with Alumina) to create a coarse hard wearing abrasive. Zirconia is used primarily when a large amount of material has to be removed from a sample and when greater damage can be accepted. It is hard wearing abrasive surface and whilst not used a lot in Metallography it is a very useful abrasive for the early stages of preparation when considerable material needs removing at the start of a preparation.

Examinations using a typical laboratory Stereo microscope, a Leica Sd6 (*figs18*) again shows the presence of particles in a regular arrangement almost sunk within a reflective layer. As these are larger grades than used in the Silicon Carbide evaluation it is easier to resolve detail and in some cases the cutting edges.

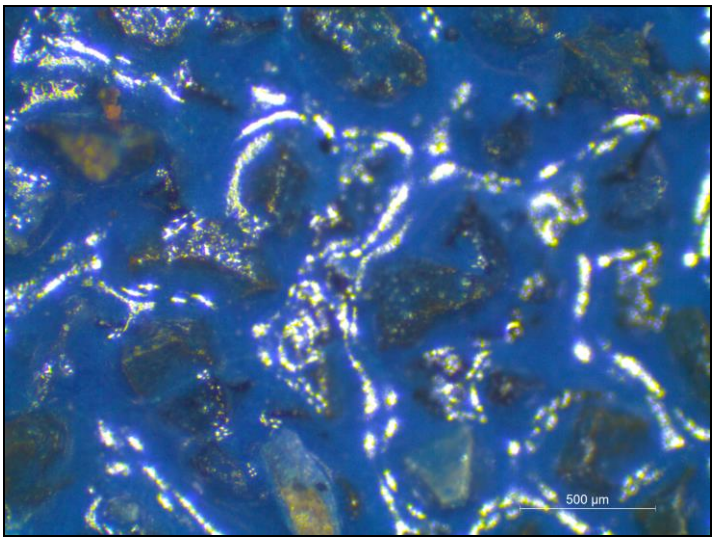
Examinations with a typical Metallurgical microscope DM2500M in Brightfield illumination (*fig19*) was quite difficult with poor views that were diffuse in nature and is probably due to the amount of reflective coating. Examination carried out using Darkfield illumination however picked up further information (*fig20*) and showed the particles more clearly. Examination was kept to a low magnification objective - 10x due to the reduced depth of field & the large nature of the abrasive.

Examination using the Laser Scanning Confocal Microscope was carried using a 20x objective (*fig21*). Again, the LSCM gave good 3D & 2D images showing the particle morphology and surface detail. Measuring data would again be possible if required.

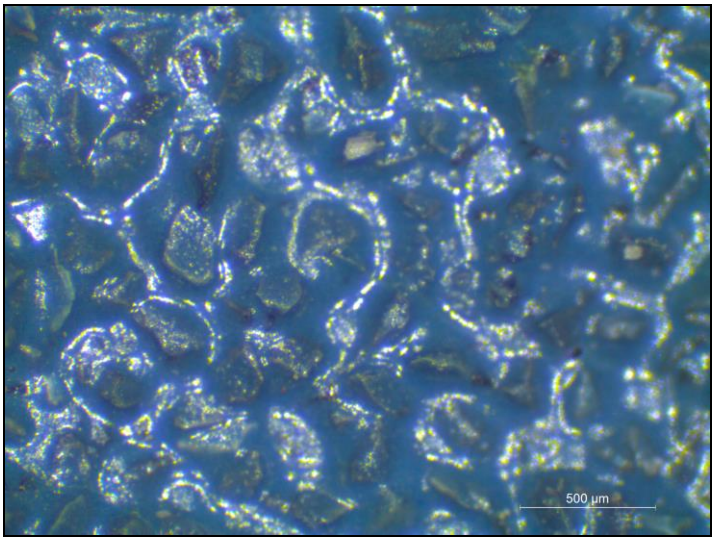
Examination with the SEM showed the greater depth of field that can be captured by using the secondary electrons to generate an image. Observed at the same 200x & 1000x magnification (*fig 22*) as used in the Silicon Carbide examination allows comparison of the abrasives rather than the technique. It is now possible to see the individual particle detail that shows how much cleaner and sharper the Silicon Carbide product is. (*fig23*).

Cross sectional analysis using a Metallurgical microscope (*Fig24*) again reveals the angular nature of the particles and how they are attached to the paper. It also reveals the particles are again covered in a bond coat. Whilst Brightfield illumination showed some detail, the use of Darkfield illumination highlighted details within the paper base and a clearer view of the bond coat. It also reveals the bond coat is greater in thickness than in the silicon carbide. This indicates a greater longevity of the paper.

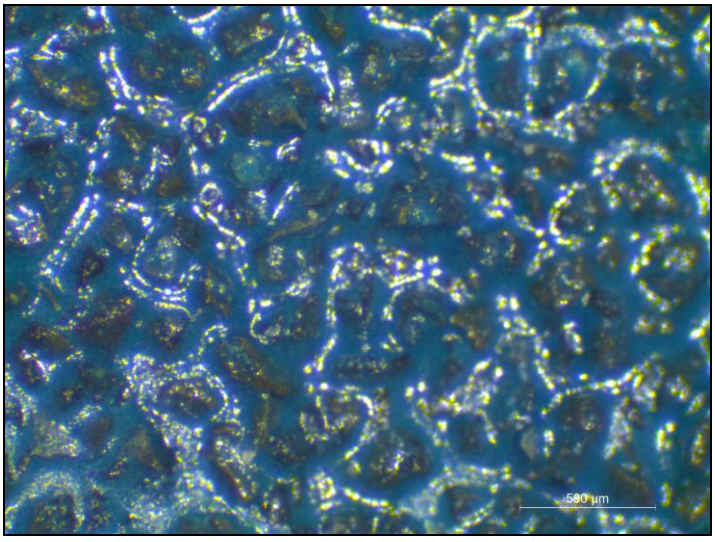
Fig 18. Zirconia Paper - Stereo Microscope Examination



P60g Stereo at 40x

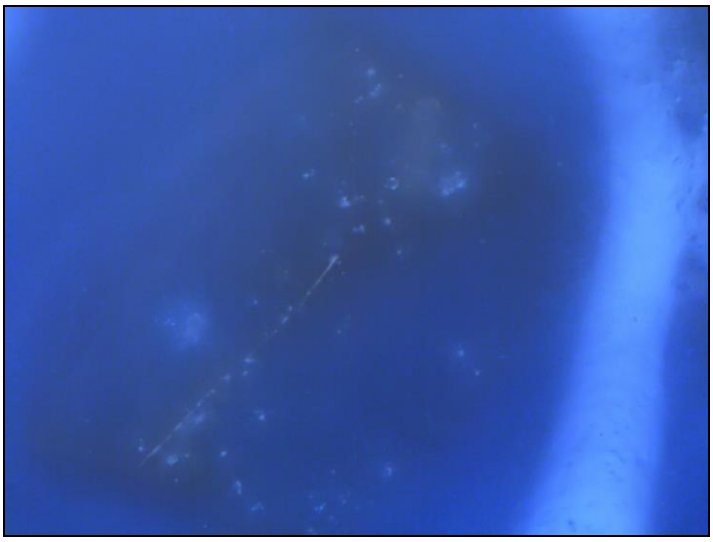


P80g Stereo at 40x

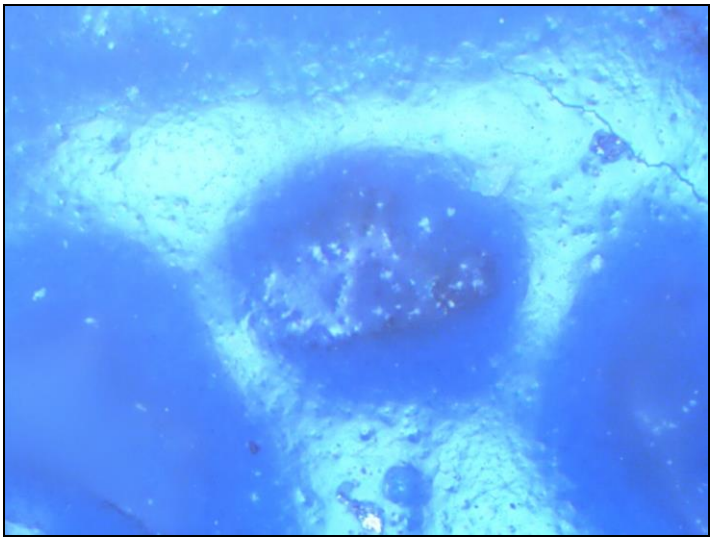


P120g Stereo at 40x

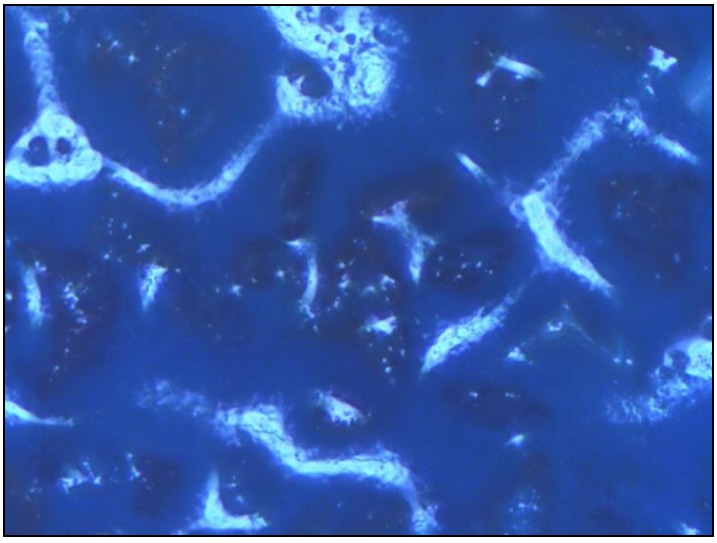
Fig 19. Zirconia Paper - Metallurgical Microscope Examination – Brightfield



P60g 10x objective - Brightfield

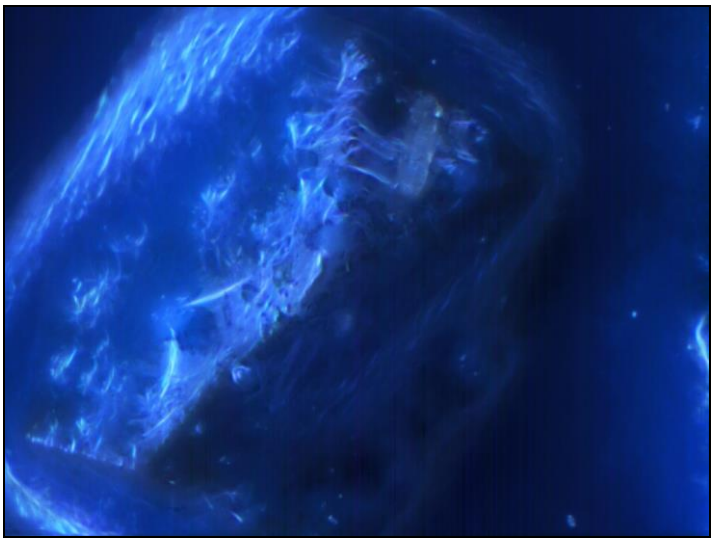


P80g 10x Objective - Brightfield

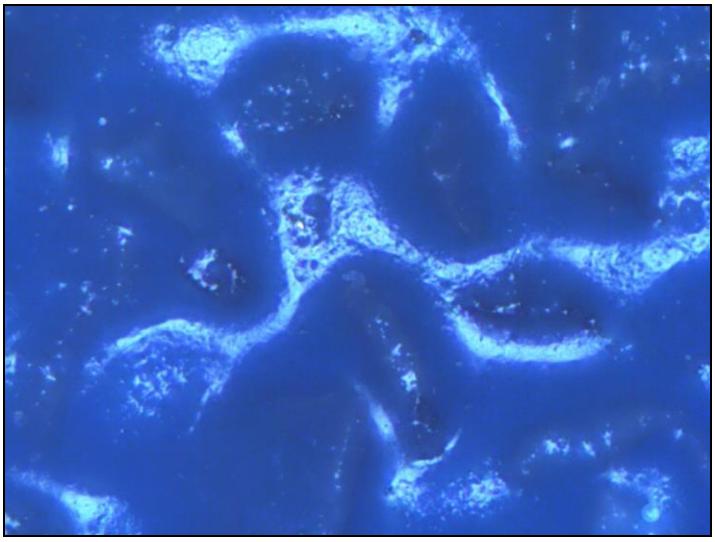


P120g 10x Objective – Brightfield

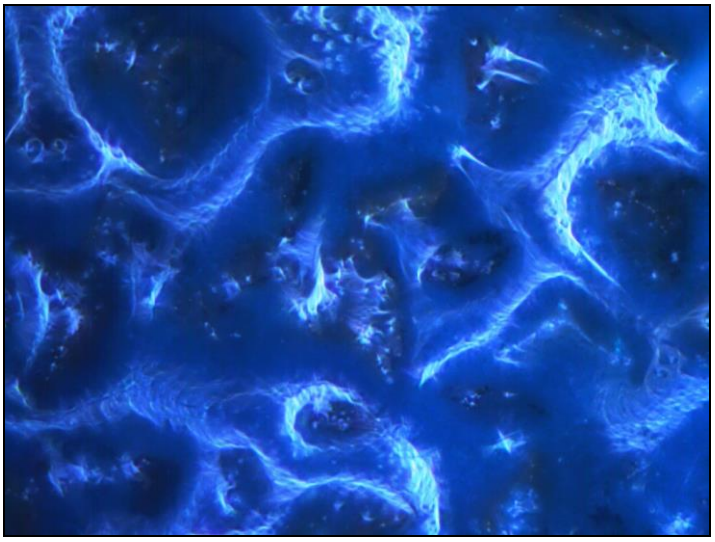
Fig 20. Zirconia Paper - Metallurgical Microscope Examination - Darkfield



P60g 10x objective - Darkfield

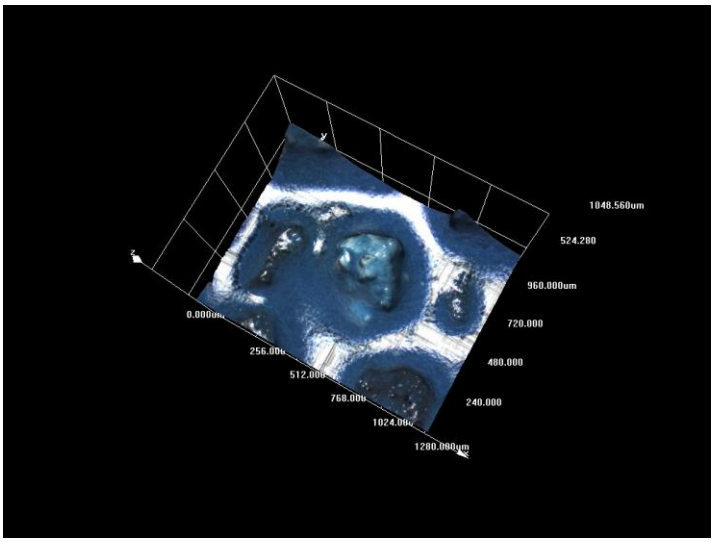


P80g 10x objective - Darkfield

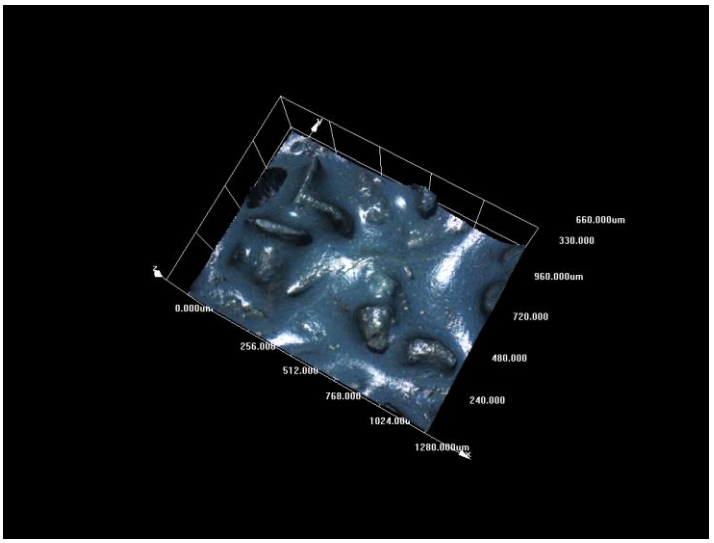


P120g 10x objective - Darkfield

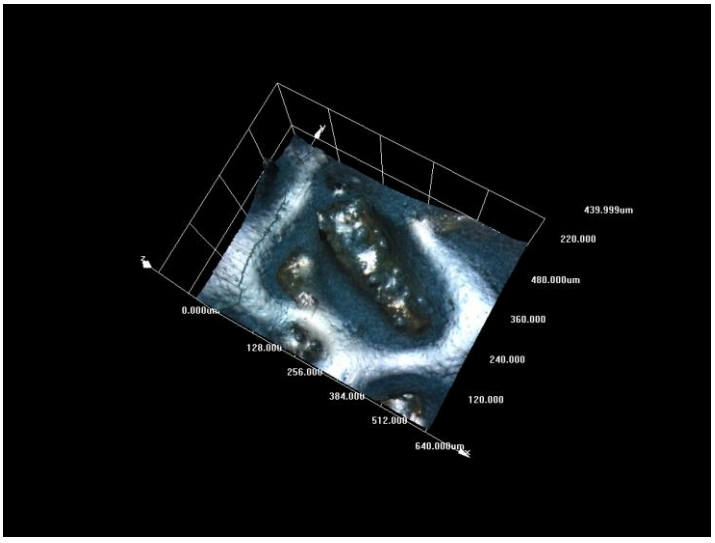
Fig 21. Zirconia Paper - Laser Scanning Confocal Microscope Examination



P60g 20x objective

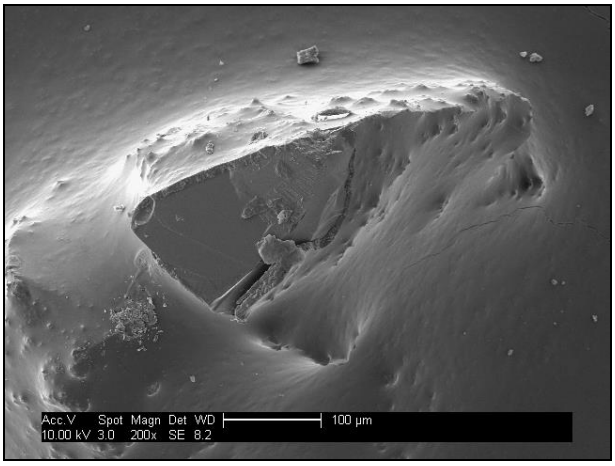


P80g 20x Objective

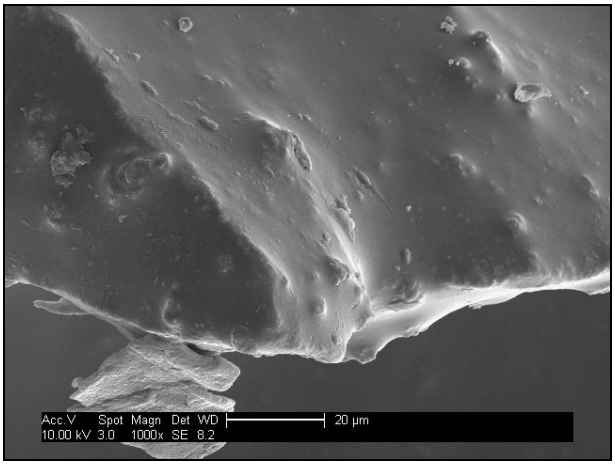


P120g 20x Objective

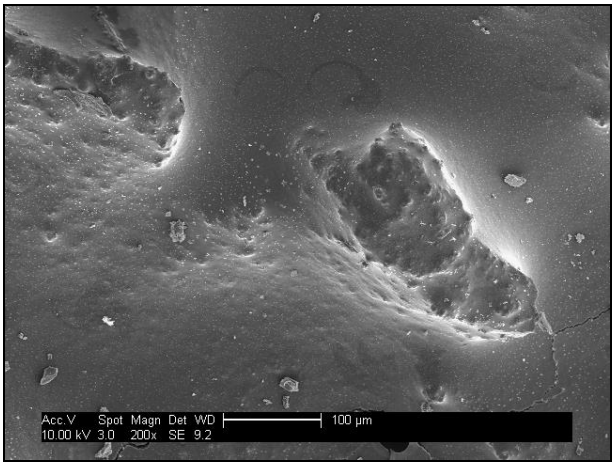
Fig 22. Zirconia Paper - SEM Examination 200x & 1000x



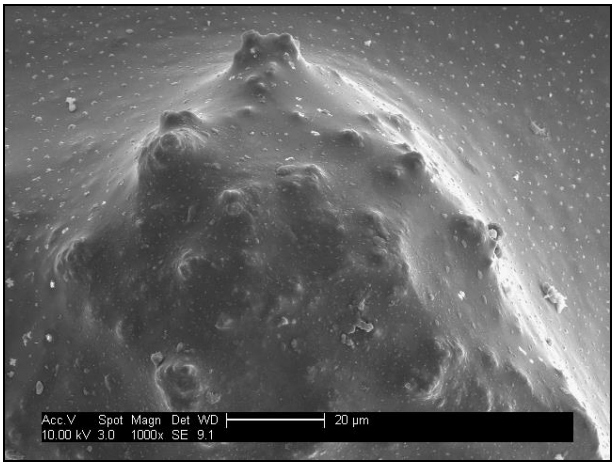
P60g 200x



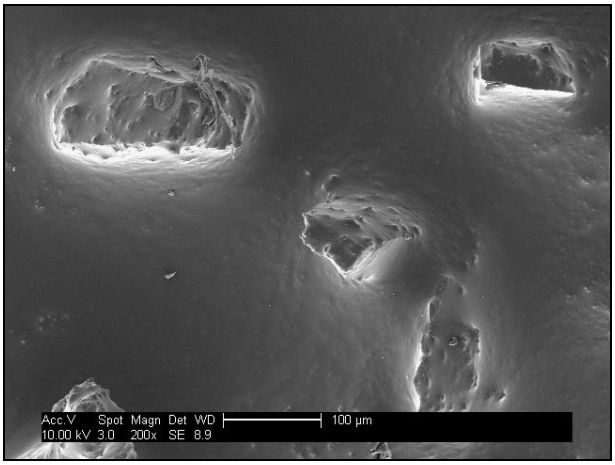
P60g 1000x



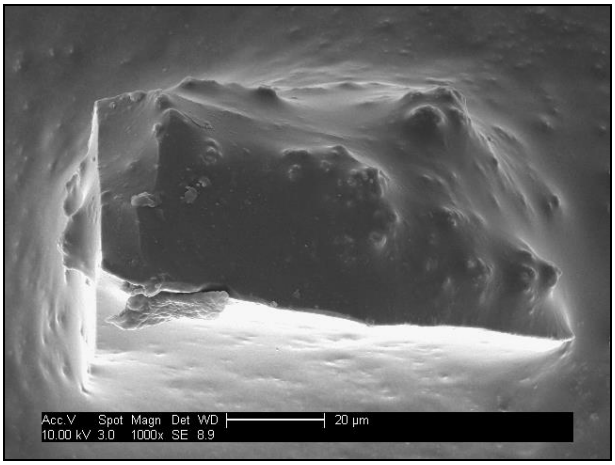
P80g 200x



P80g 1000x

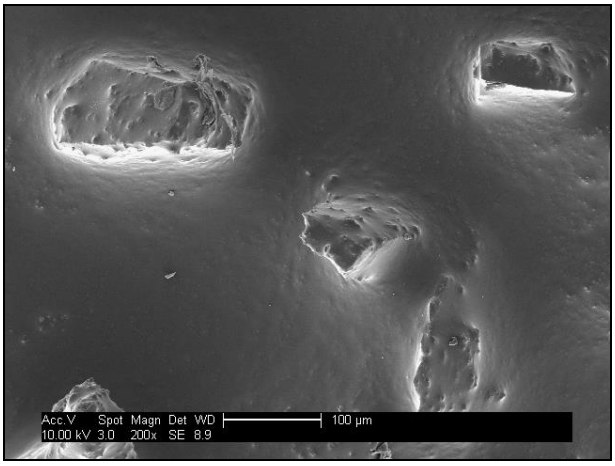


P120g 200x

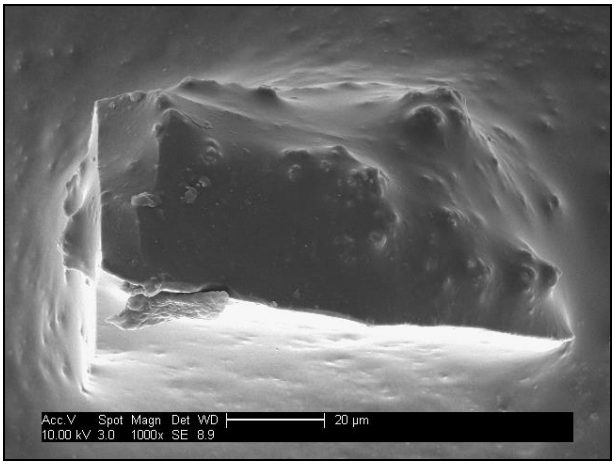


P120g 1000x

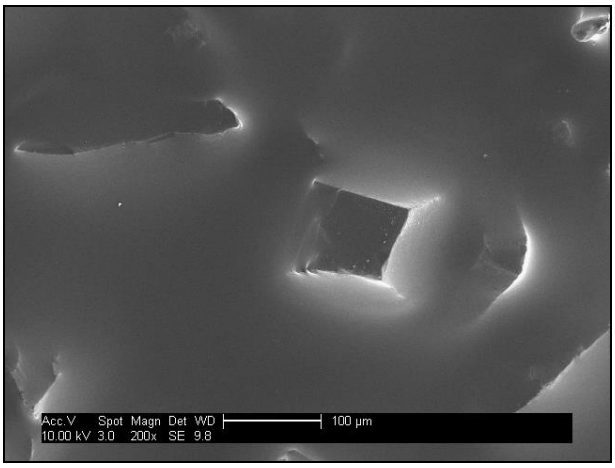
Fig 23. Zirconia Paper compared to Silicon carbide Paper P120g - SEM & LSCM Examination



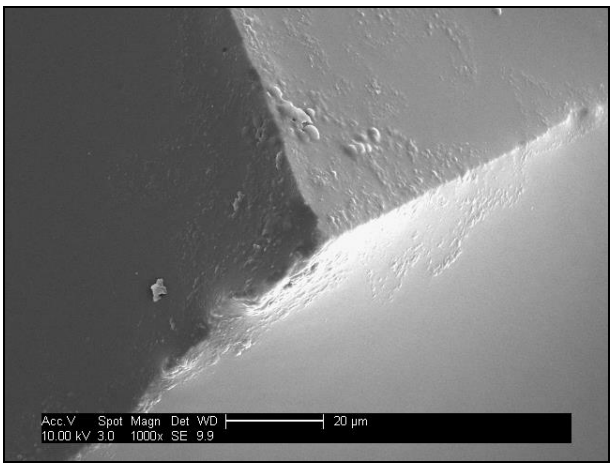
P120g Zirconia 200x



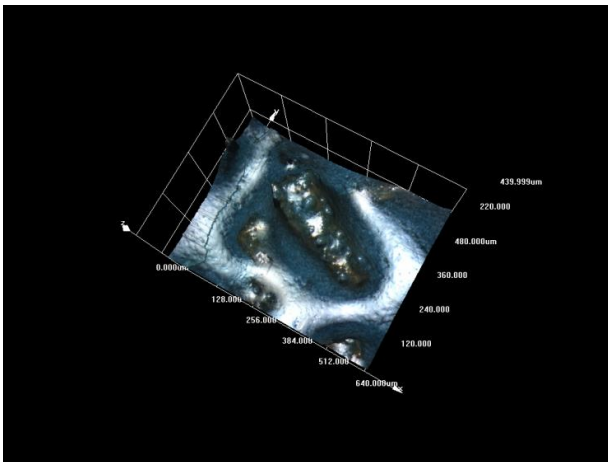
P120g Zirconia 1000x



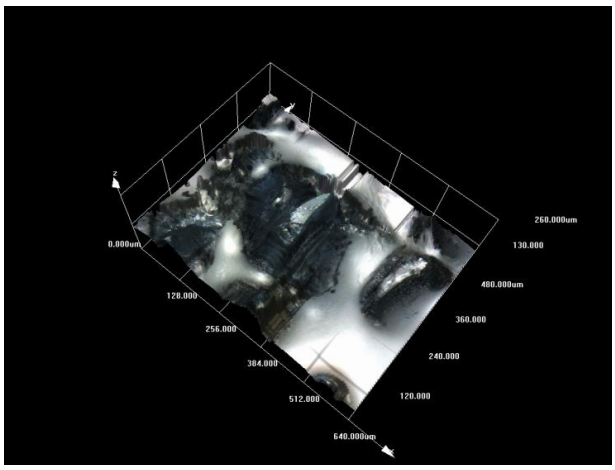
P120g Silicon Carbide 200x



P120g Silicon Carbide 1000x



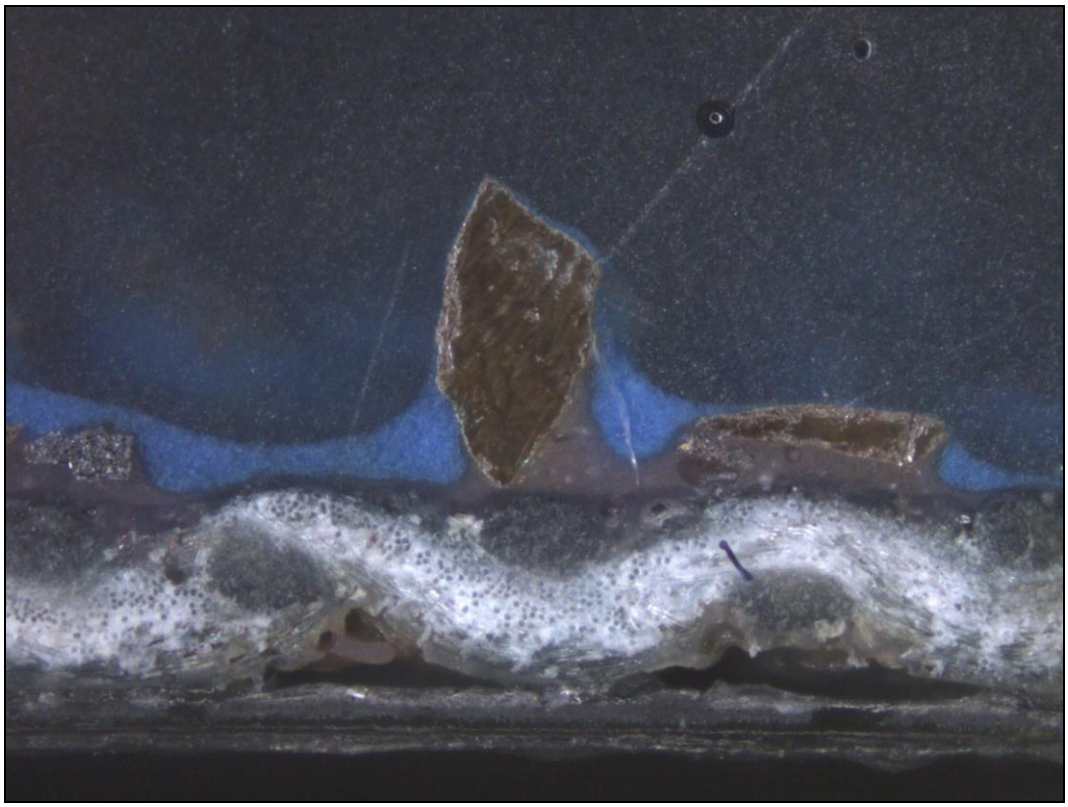
P120g Zirconia – LSCM 20x Objective



P120g SiC – LSCM 20x Objective

Fig 24. P60g Zirconia Paper - Metallurgical Microscope Metallographic Examination

P60g Zirconia 5x objective - Brightfield



P60g Zirconia 5x objective - Darkfield

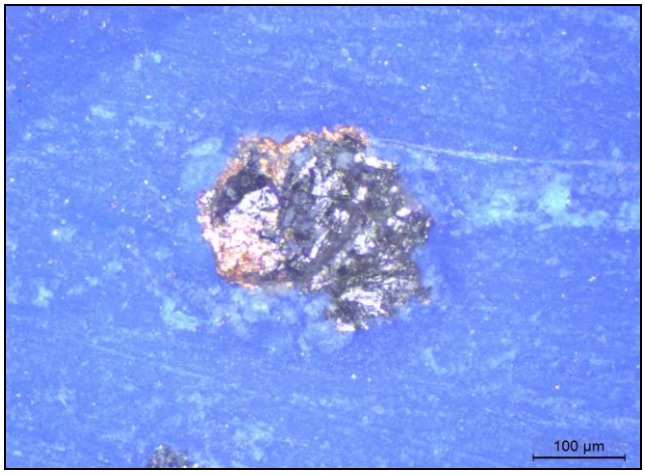
Fixed Grinding Diamond Surfaces

Many suppliers now offer fixed diamond abrasive surfaces. These are relatively new products in the market and contain diamond abrasives embedded into a matrix and usually fixed to a metal backing. These surfaces are used in various grinding stages raging from the primary stage through to the tertiary or even quaternary stage depending on the material being prepared. Diamond is harder than the Silicon carbide but it is not as sharp. It also has greater longevity as the Silicon carbide degrades and wears out. As these items are supplied as discs between 200 & 300 mm in diameter putting them into an SEM is not always possible. In addition, it would take some justification to cut up a product of such value just to examine in an SEM. With that in mind observations are be based around the other techniques of the Metallurgical microscope & the Laser Scanning Confocal microscope

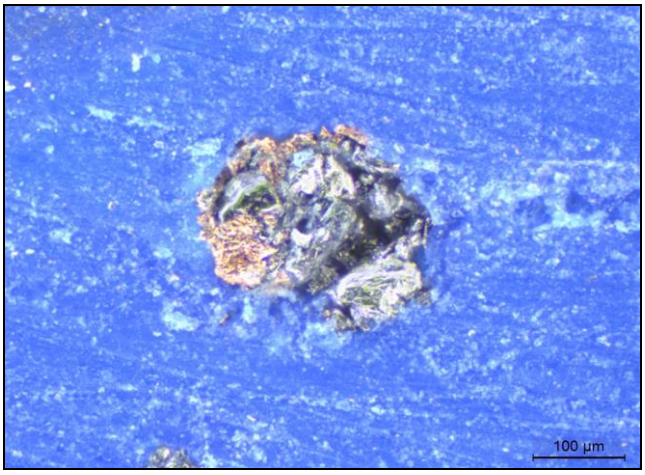
Metallurgical microscope examination. Typically these types of surface are generally used in the size range of P120g – P320g (95-46um) though coarser and finer grades are available. Examination of typical surfaces using both Brightfield and Darkfield illumination using a 20x objective clearly shows the blocky nature of the abrasive and also how well the diamond is dispersed across the platen (*fig25*). With such clarity it also means it is possible to also monitor the wear and appearance of these surfaces as they are regularly used. Comparison between the appearances of the surface in Brightfield & Darkfield illumination techniques gives the Darkfield image the edge due to greater contrast. If examination were only possible using Brightfield illumination it would not really be a problem in revealing the necessary information.

Considering a typical view using the LSCM technique it is clear to see how the general morphology of the abrasive is much more clearly visible (*fig26*). The ability to use a laser as well as the colour camera data to create a 3D image shows the much greater detail than can be obtained of the abrasive shape using this technique. If measurement of the individual abrasives is required or greater detail of the abrasives morphology is required then this is surely the best technique to evaluate this product. If however only a general examination was required then the simple metallurgical microscope would be good enough to assess and compare or even monitor wear during operation.

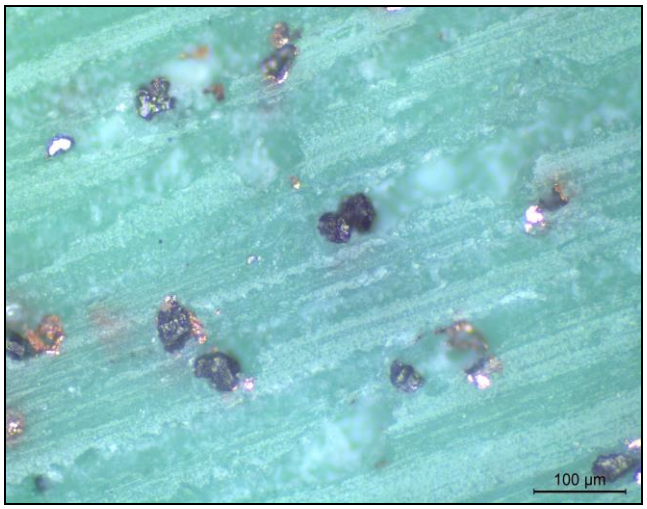
Fig 25. Fixed Diamond grinding surfaces - Metallurgical Microscope Examination



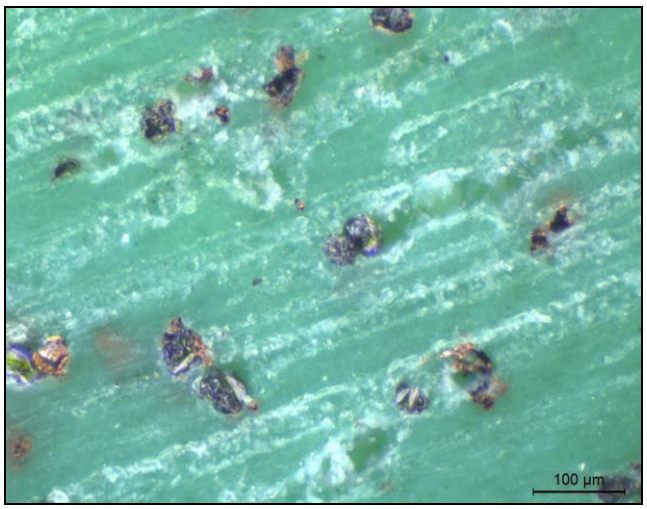
Cameo Blue P120g 20x Objective - Brightfield



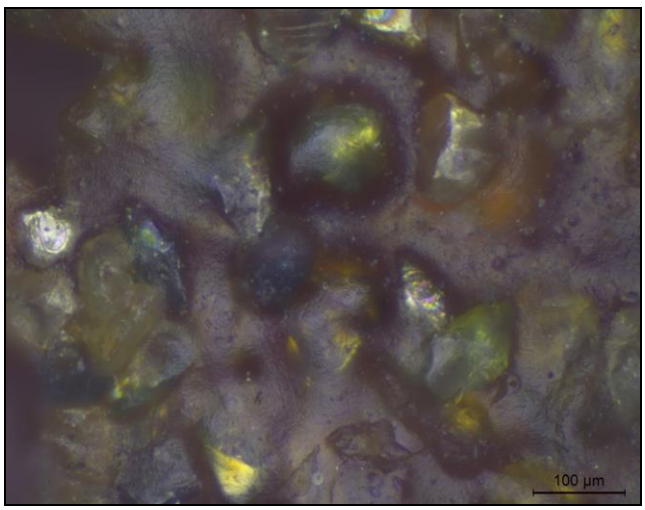
Cameo Blue P120g 20x Objective - Darkfield



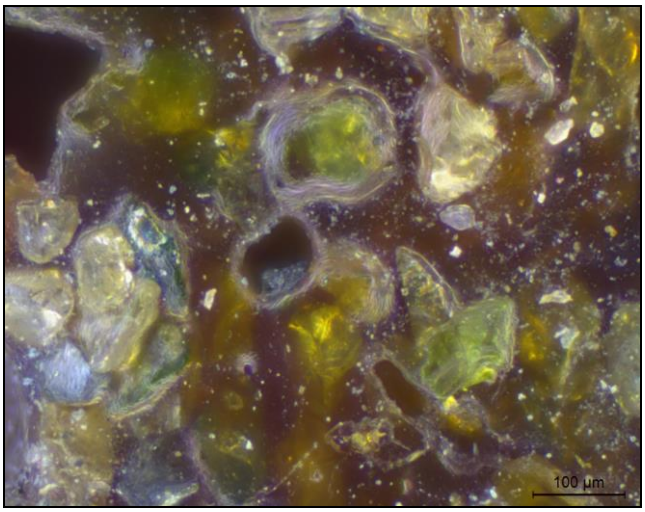
Cameo Green P320g 20x Objective - Brightfield



Cameo Green P320g 20x Objective - Darkfield

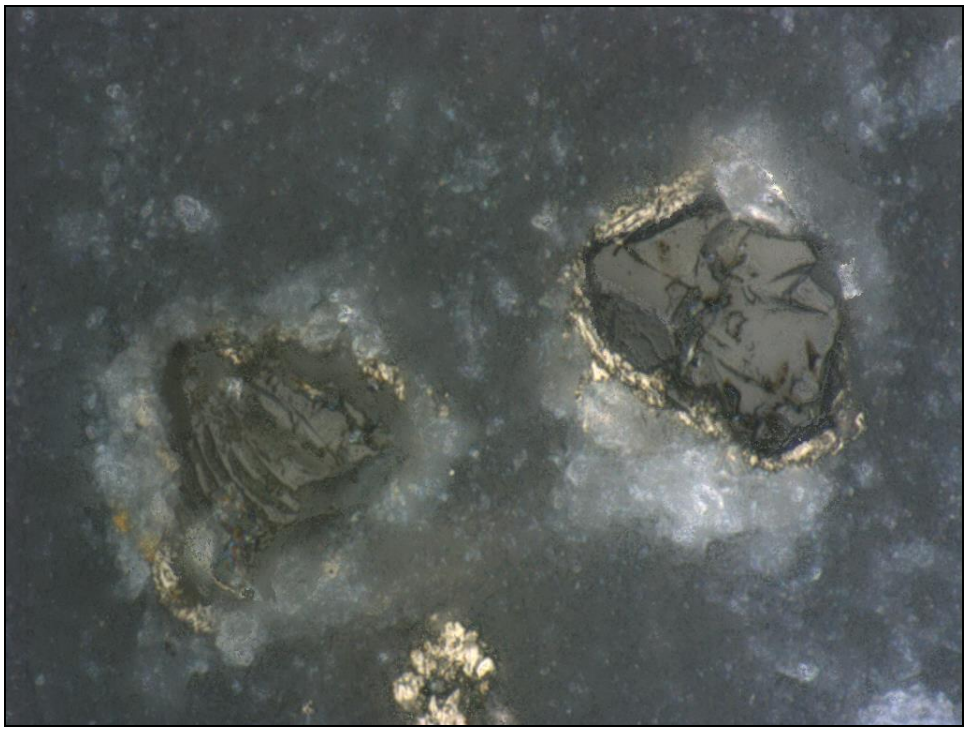


ReflexP80g Brown 20x Objective-New Brightfield

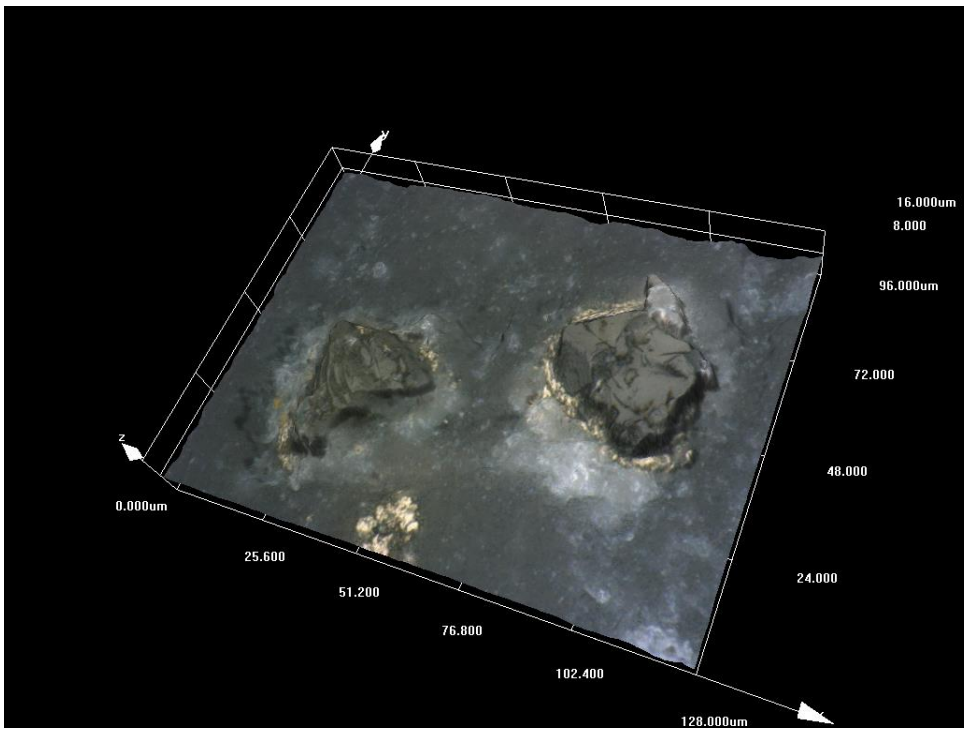


ReflexP80g Brown 20x Objective-New Darkfield

Fig 26. Fixed Diamond surface - 2D & 3D Laser Scanning Confocal Microscope Examination



100x Objective



100x Objective

Polishing cloths

As mentioned in the introduction the term polishing cloth is really a misnomer. In many cases when we are actually using these cloths they are being employed as a grinding tool. The only time polishing occurs is when a final napped cloth is used to remove scratches from an otherwise damage free prepared sample.

Most metallographic consumable suppliers will supply these grinding cloths in several forms. These variants correspond to the amount of stock they will remove, how hardwearing they are and how much structural damage they will leave on a material for a given abrasive size. Usually grouped to fit into Secondary, Tertiary & Quaternary grinding stages these cloths will support progressively finer abrasives as you progress towards producing a damage free surface. The position that these cloths hold within a preparation procedure will be material dependent and, on many occasions, not all stages will be needed to gain a damage free sample. The most common abrasive used on such cloths is diamond though Alumina & Colloidal silica products can be used when necessary.

Additional to the Grinding cloths there are the Final polishing cloths. These are more correctly named as these are designed to remove final scratches from the otherwise damage free samples. These cloths are usually identified by a nap that covers their surface though other surfaces do exist.

Secondary Grinding Cloth Surfaces

Secondary grinding cloths are designed to follow a Primary grinding stage. For ductile materials this primary stage is often a coarse Silicon Carbide paper such as P180g – P240g for an example. These cloths are usually a hard chemotextile material or coarse woven polyester and are employed with a coarse diamond abrasive of 15um or 9um in size. The actual chosen abrasive size being dependent on sample size or hardness / toughness of the material to be prepared.

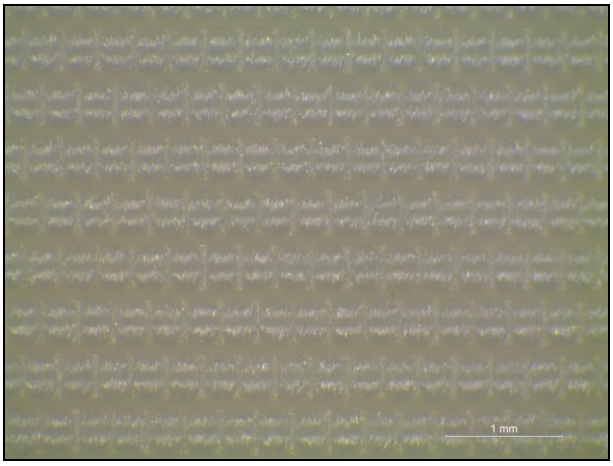
Examinations using a typical laboratory Stereo microscope reveals a considerable amount of information even at low powers such as 20x or 40x (*fig27*). It is clearly possible to determine whether a cloth is of a cross woven type or chemotextile type. Any operator could easily compare various manufactures cloths an allocate them a typical grinding stage by using a stereo microscope and simply running their finger nails across the surface to gauge the relative severity and hardness.

Examinations with a typical metallurgical microscope using both Brightfield & Darkfield techniques also gave a good indication of the nature of the cloths (*fig28*). Even with a 20x objective there is reasonable depth of field but using a Z axis image stacking program a better view can be obtained. Whether you can see more in Brightfield or Darkfield is debateable but to get a better understanding it is always worth trying both techniques if available. On this occasion one will give slightly more information than the other depending on the cloth examined.

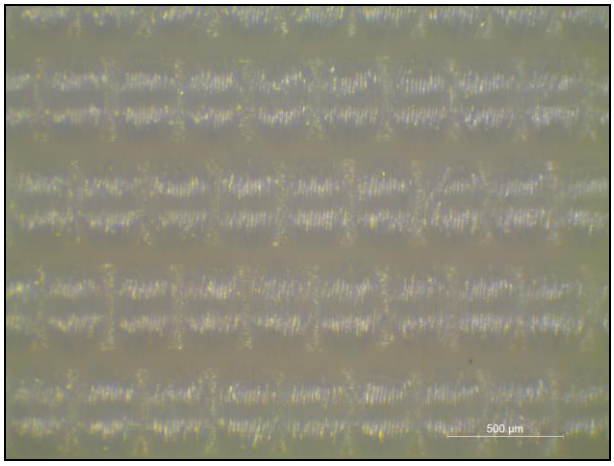
Examination with the Laser Scanning Confocal Microscope was again carried out using a 20x objective (*fig29*). Again the LSCM allowed the construction of 3D or 2D images showing the cloth morphology. Unfortunately, as the illumination on the cloth is only from directly above the 3D effect does not show the intricate nature of the cloth. This is always a restriction of the LSCM. Again the ability to take measurements for comparing fibre sizes for instance is useful and it is possible to determine the nature of the individual cloths.

Examination with an SEM gives a really detailed view of the cloths (*Fig30*). Despite the extra requirement to vacuum coat the samples with a thin layer of gold to prevent the cloths charging the detail resolved is excellent. Anyone wishing to make a critical comparison of the nature and construction of their cloth will find the SEM a potent tool in doing so.

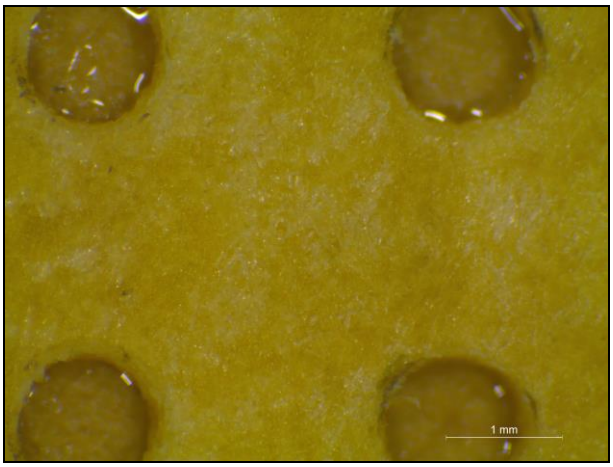
Fig 27. Secondary Grinding Cloths - Stereo Microscope Examination



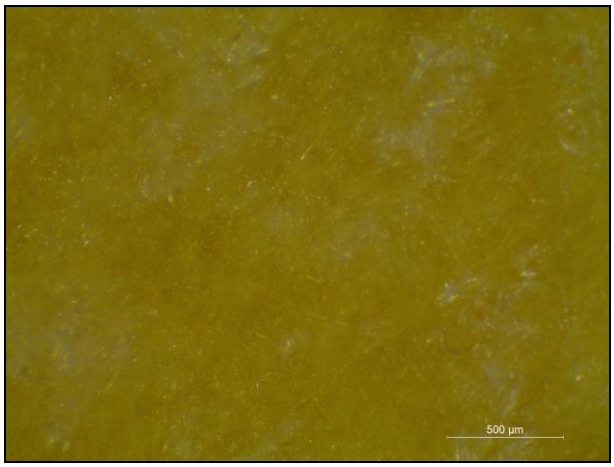
Abracloth Stereo Microscope at 20x



Abracloth Stereo Microscope at 40x

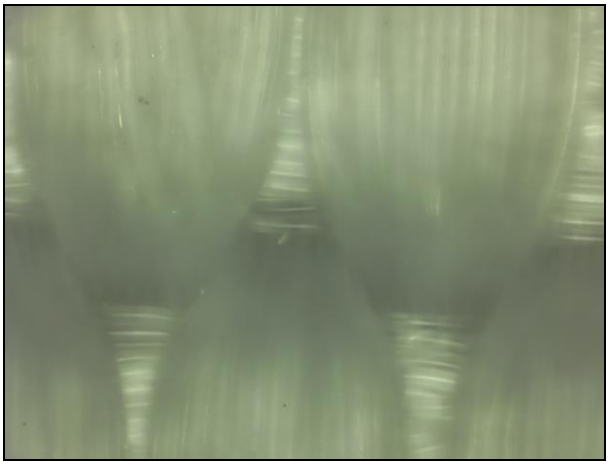


Planocloth H Stereo Microscope at 20x

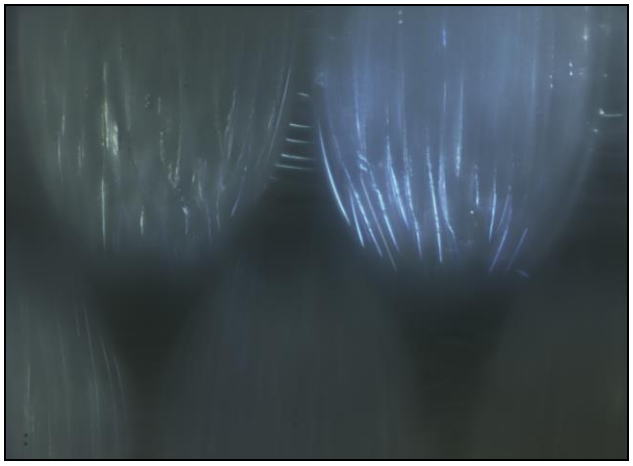


Planocloth H Stereo Microscope at 40x

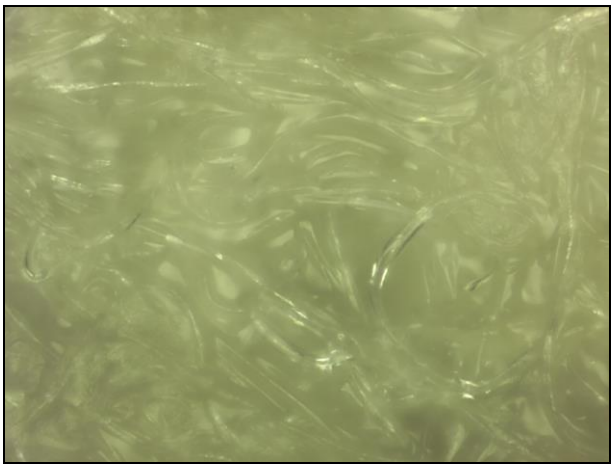
Fig 28. Secondary Grinding Cloths - Metallurgical Microscope Examination



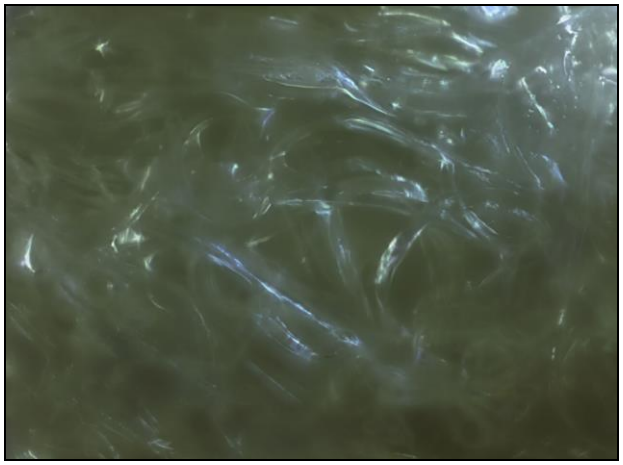
Abracloth 20x Objective - Brightfield



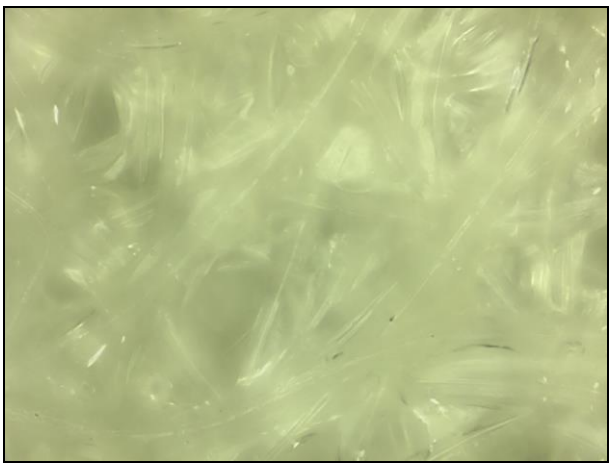
Abracloth 20x Objective - Darkfield



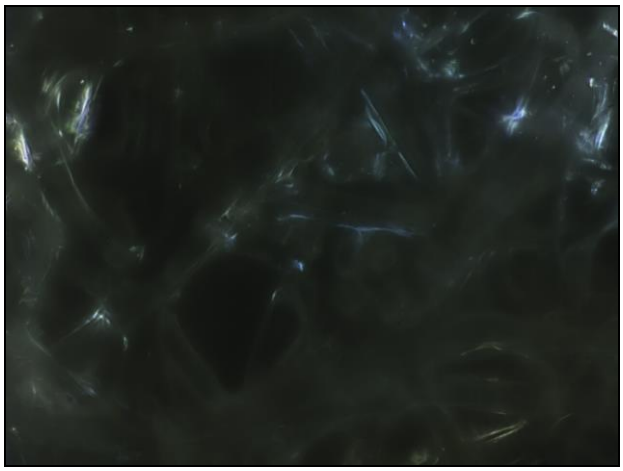
Planocloth H 20x Objective - Brightfield



Planocloth H 20x Objective – Darkfield

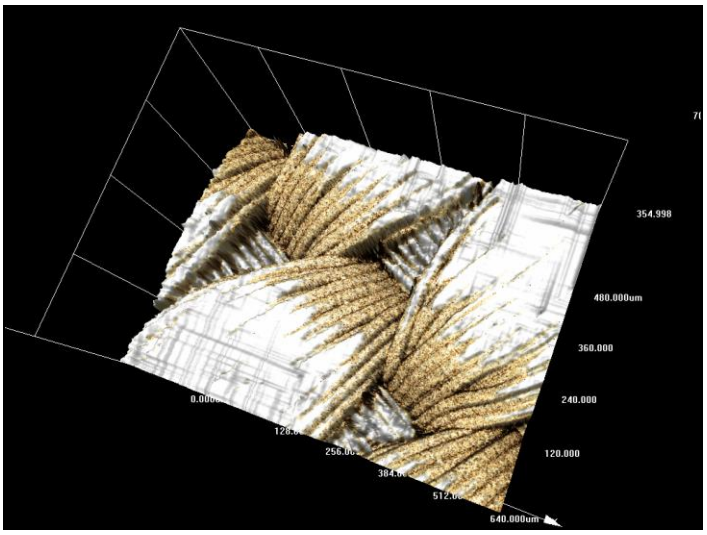


PAW 20x Objective - Brightfield

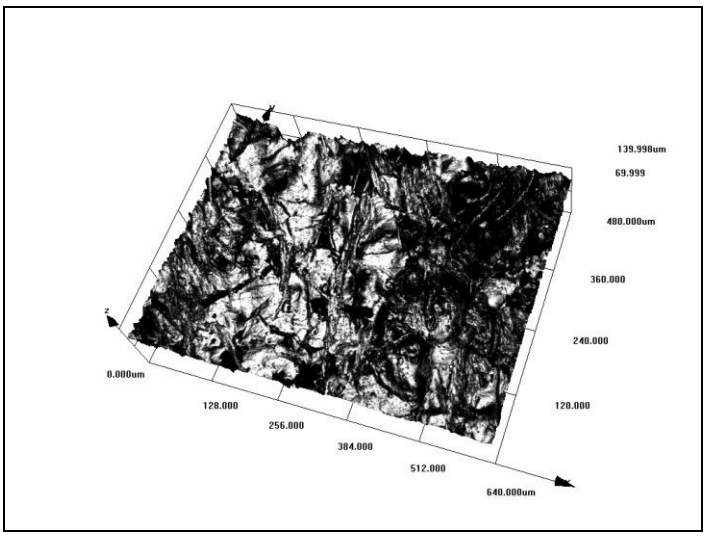


PAW 20x Objective – Darkfield

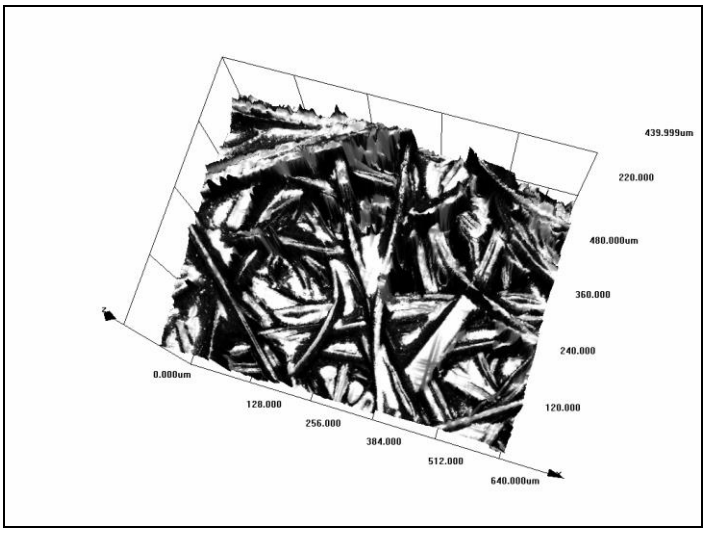
Fig 29. Secondary Grinding Cloths - LSCM Examination



Abracloth 20x 3D Colour

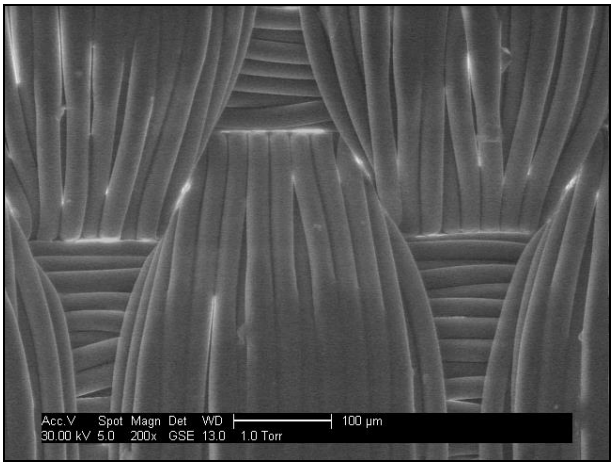


Planocloth H 20x 3D Black & White

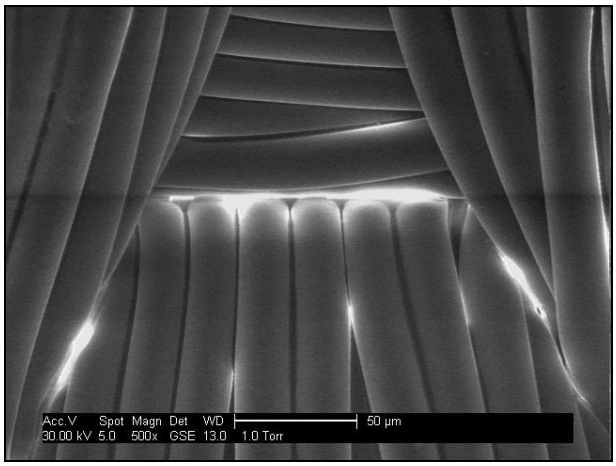


PAW 20x 3D Black & White

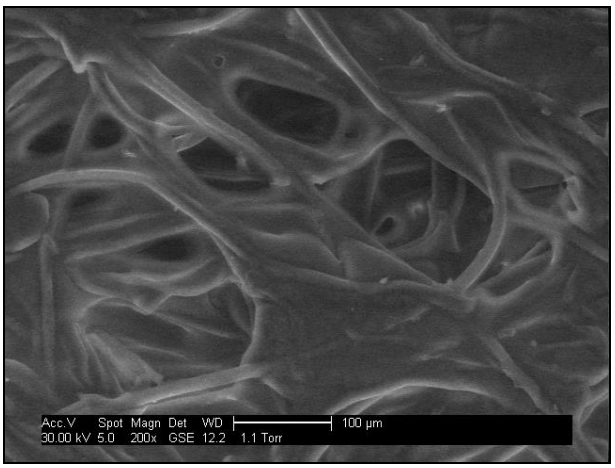
Fig 30. Secondary Grinding Cloths - SEM Examination



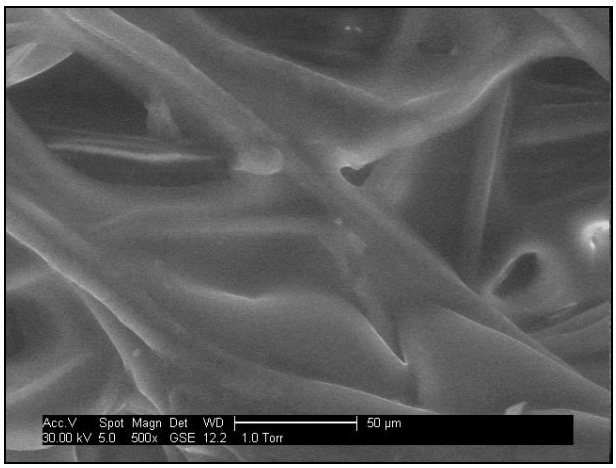
Abracloth 200x



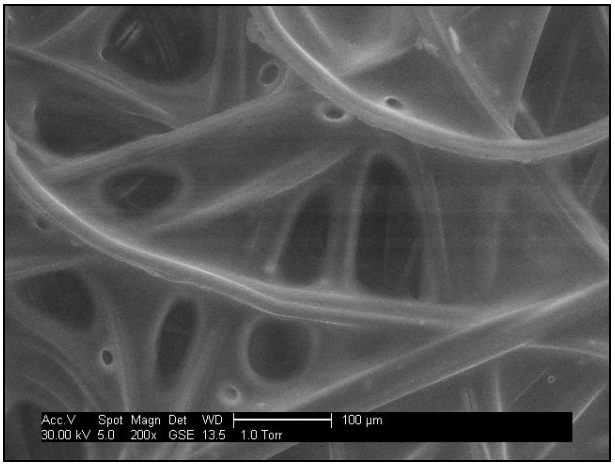
Abracloth 500x



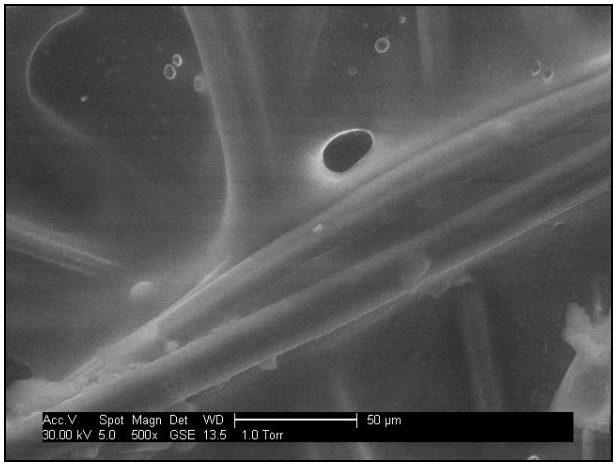
Planocloth H 200x



Planocloth H 200x



PAW 200x



PAW 500x

Tertiary Grinding Surfaces

The tertiary grinding cloths are designed to follow a secondary grinding stage. A secondary stage surface usually consists of a fixed diamond or a more aggressive cloth. In some softer materials a tertiary cloth may be used as a secondary stage as all these stages are guidelines based on the material being prepared. Again, cross woven cloth such as polyesters & chemotextiles are employed. They will however tend to be finer in nature and less aggressive thus imparting less damage to the sample than their secondary counterparts. In addition, another fibre type that fits into this category is silk. Whether the silk is artificial or natural, these fine cloths can be used on softer materials or even friable materials that are highly susceptible to damage.

In general diamond in the size range of 6um or 3um is used on such cloths but again this is dependent on sample size or the number of samples being prepared. In certain other cases even a 1um abrasive size may be employed. This can be particularly helpful in removing the final traces of damage in ceramic type materials. In such conditions it will often be employed as a quaternary grinding stage. In many cases the finish of such a quaternary grinding stage may mean there is no need to polish with a napped cloth as the stage often leaves no visible scratches when viewed with a metallurgical microscope.

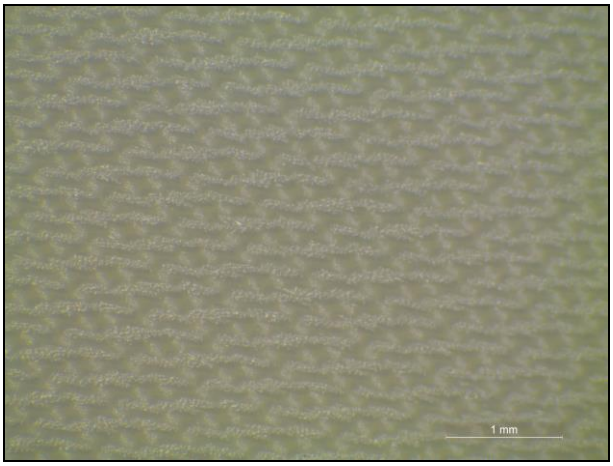
As with the secondary cloths examination with a typical laboratory Stereo microscope shows a considerable amount of information even using low powers such as 20x & 40x (*fig31*). It is again possible to determine whether a cloth is of a cross woven type or a chemotextile type though the finer nature of the cloth means it is more difficult to resolve the complexities. As with the secondary cloths an operator could still be able compare various manufactures cloths and allocate them a typical grinding stage by using a Stereo microscope and again by using touch to gauge severity & hardness.

Again, as with the secondary cloths examination with a typical Metallurgical microscope using both Brightfield & Darkfield techniques gives a good indication of the nature of the cloths (*fig32*). Examination using a 20x objective and image stacking software recorded fine details of the weave or the chemotextile nature of the cloth. As with the secondary cloths whether you can see more in Brightfield or Darkfield illumination is debateable but it is worth trying both if available. Subtle differences between the cloths can be seen in both contrast techniques.

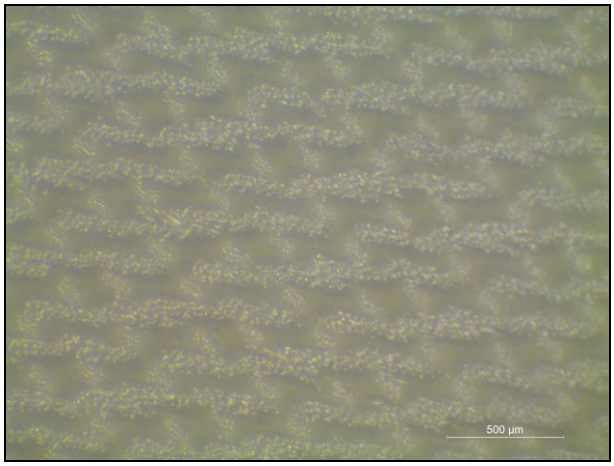
Examination with the Laser Scanning Confocal Microscope was again carried out using a 20x objective (*fig33*). Again, the LSCM allowed the construction of 3D or 2D images showing the cloth morphology but again as the illumination on the cloth is only from above the 3D effect does not really show the detailed nature of the cloth. Again, the ability to take measurements for comparing fibre sizes for instance is useful and it is possible to determine the nature of the individual cloths examined.

Examination with an SEM again gives stunning views of the cloths (*fig34*). As before the samples were coated with a thin layer of gold that of course is yet another stage to carry out, but the detail resolved is worth it. As with the secondary grinding cloths, anyone wishing to make a critical comparison of the nature and construction of their cloth will find the SEM an ideal instrument for doing so.

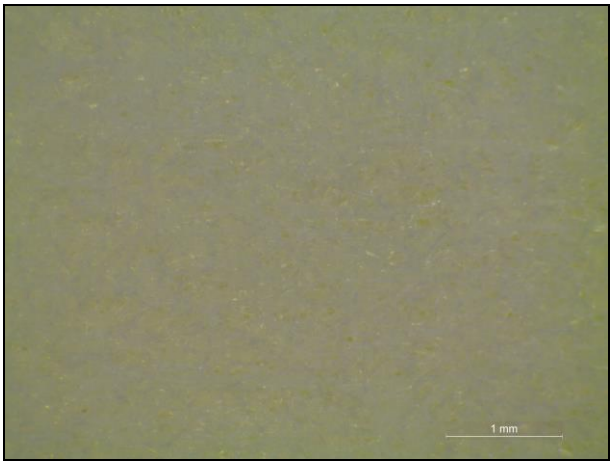
Fig 31. Tertiary Grinding Cloths - Stereo Microscope Examination



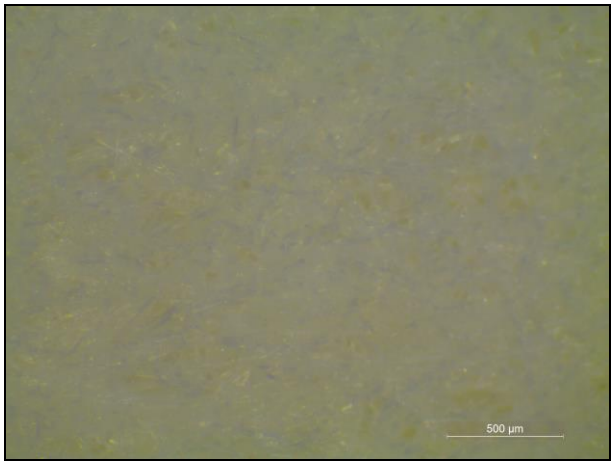
Nylap Stereo Microscope at 20x



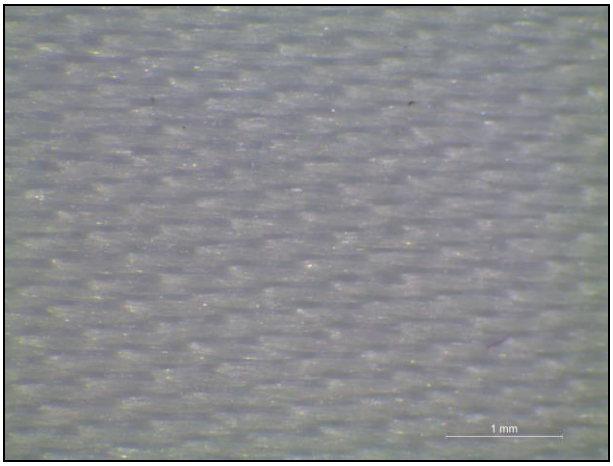
Nylap Stereo Microscope at 40x



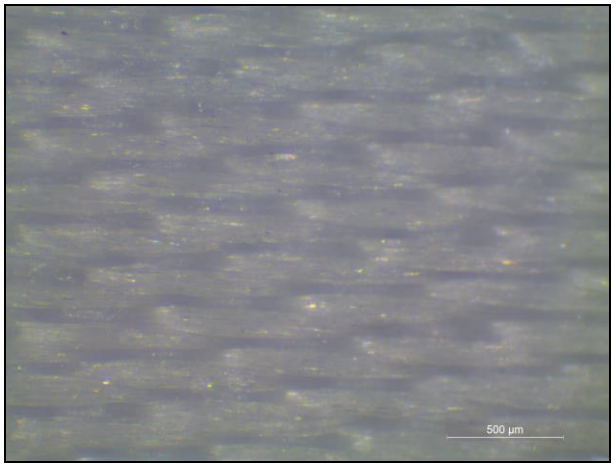
Planocloth H Stereo Microscope at 20x



Planocloth H Stereo Microscope at 40x

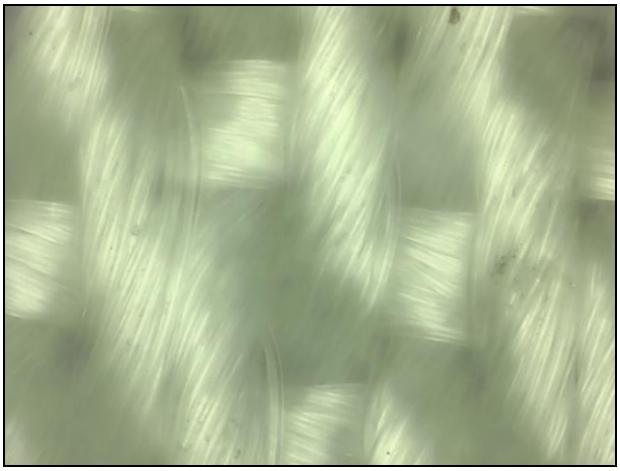


Durasilk Stereo Microscope at 20x



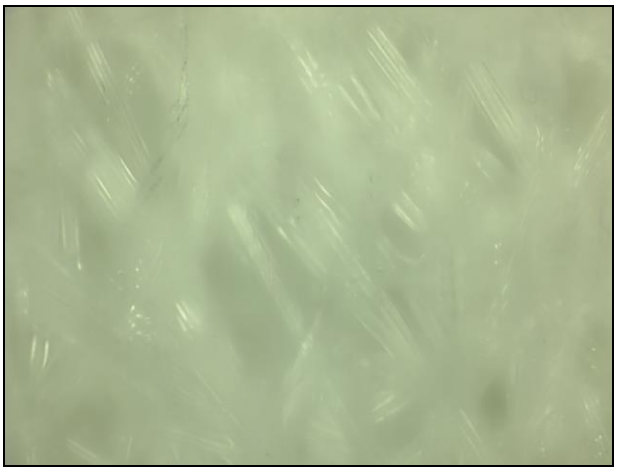
Durasilk Stereo Microscope at 40x

Fig 32. Tertiary Grinding Cloths - Metallurgical Microscope Examination

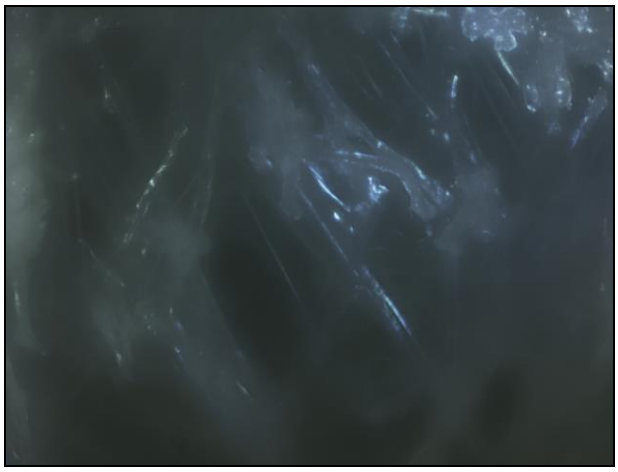


Nylap 20x Objective - Brightfield

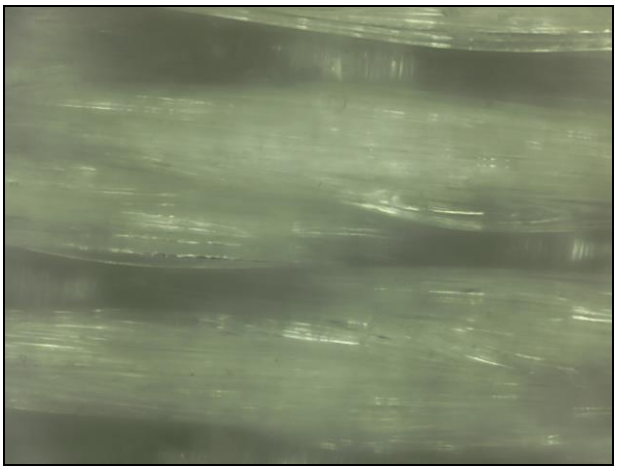
Nylap 20x Objective - Darkfield



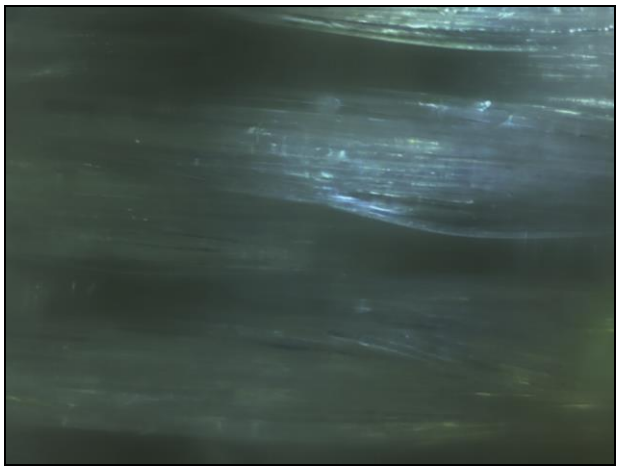
Planocloth 20x Objective - Brightfield



Planocloth 20x Objective – Darkfield

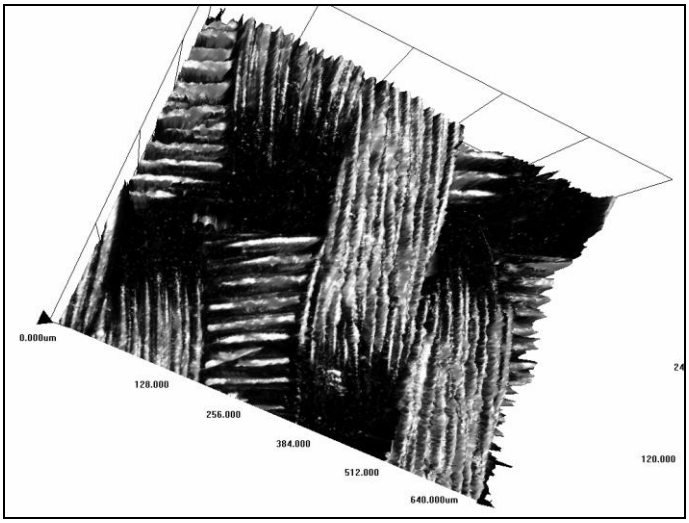


Durasilk 20x Objective - Brightfield

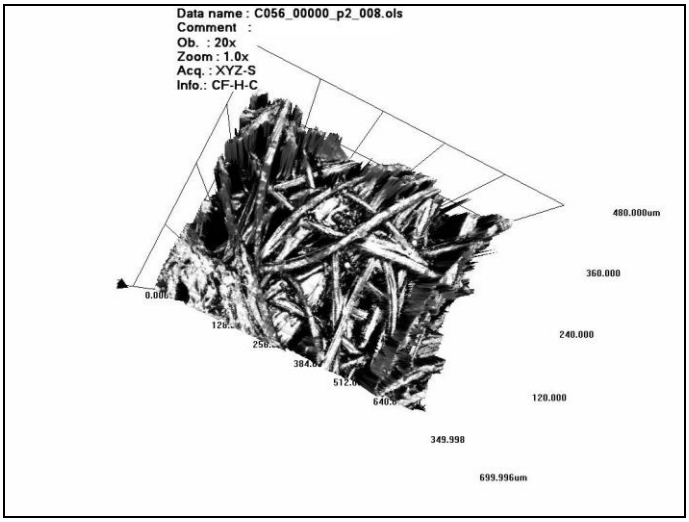


Durasilk 20x Objective - Darkfield

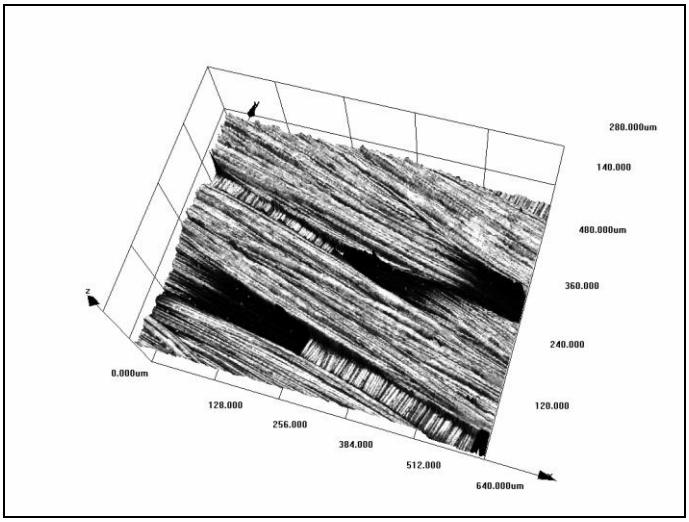
Fig 33. Tertiary Grinding Cloths - LSCM Examination



Nylap 20x 3D Black & White

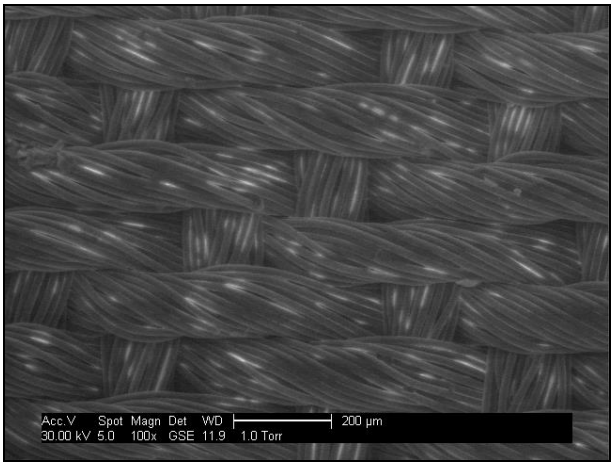


Planocloth 20x 3D Black & White

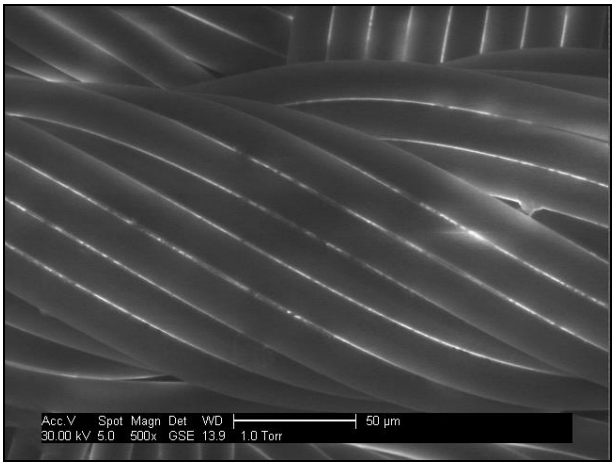


Durasilk 20x 3D Black & White

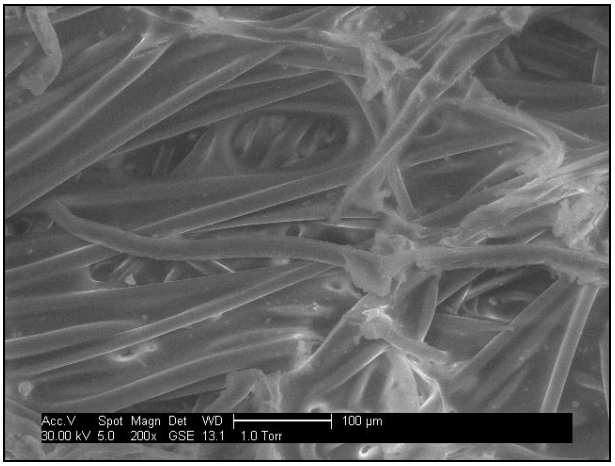
Fig 34. Tertiary Grinding Cloths - SEM Examination



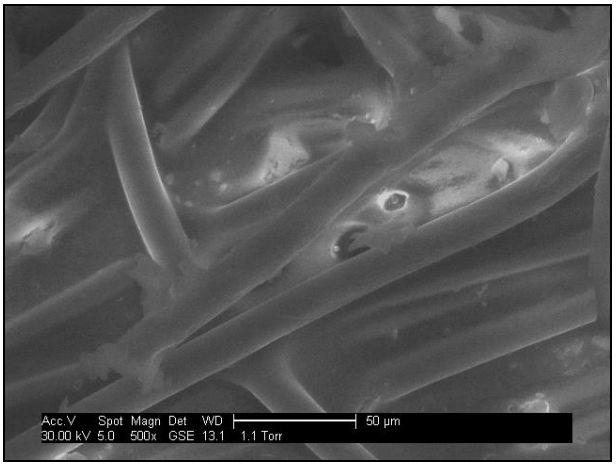
Nylap 200x



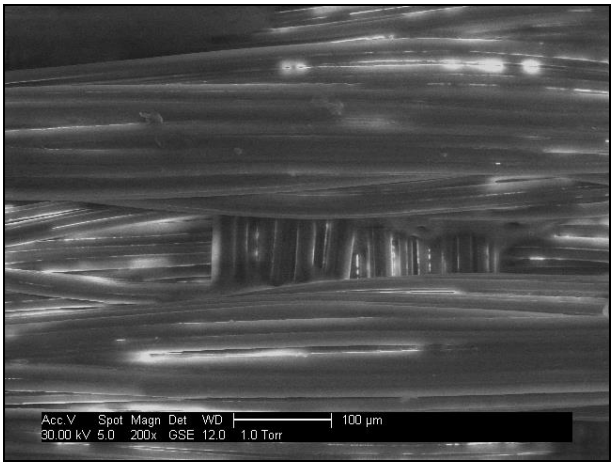
Nylap 500x



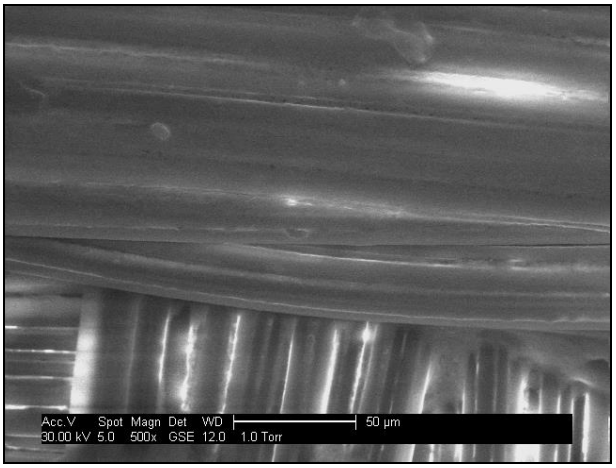
Planocloth 200x



Planocloth 200x



PAW 200x



PAW 500x

Final Polishing Cloths

Ensuring that a sample has been prepared free from damage, it is possible that it is still necessary to remove any scratches to create a micrograph suitable for publication. In many other instances this is not necessary as operations such as the measurement of a layer, determination of a cast iron type and even some image analysis etc can be assessed when scratches still exist. Final polishing is only there to remove any final scratches therefore if you haven't any scratches or you don't need to remove them then there is no need to polish.

There are now numerous final polishing cloths available to the Metallographer. Most of these clothes have a nap or raised soft surface and it is this nap that holds the abrasive and creates the polishing action. The excessive use of force or time at this stage can generate relief between different constituents within materials. Relief is the differential surface height of various sample constituents and is caused by the differential abrasion of these constituents. Consequently, it is necessary to use low pressures and short times to get the best results. In general, the longer the nap of the cloth the greater the relief generated in a given time, for a given abrasive size and surface combination. Preparing samples with dissimilar materials for example coated samples or metal matrix composites need to be particularly careful at the final polishing stage. Care taken on your cloth choice at this stage will reduce relief and additional possible edge rounding.

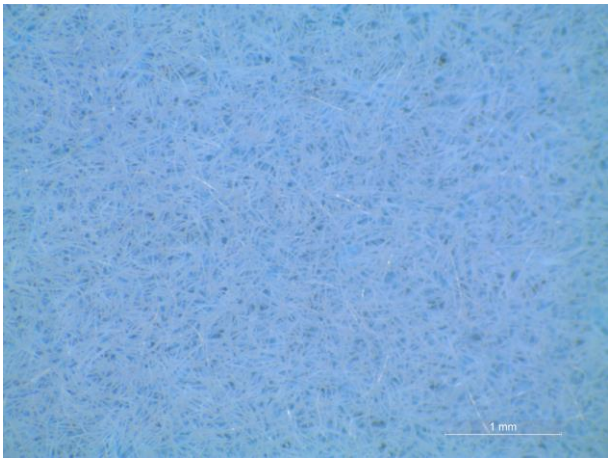
As with the other cloths, examination with a typical laboratory Stereo microscope shows a considerable amount of details even using low powers such as 20x & 40x (*figs 35&36*). It is again possible to differentiate cloths from one another by the type and amount of nap. This will be at least a useful guide when combined with touch to see how the cloths compare and even perform.

The use of the metallurgical microscope again using Brightfield and Darkfield contrast techniques revealed details of the type of fibres used. Again, images were stacked to get as much detail as possible but the illumination can only illuminate part of the fibre structure. That said in Darkfield, the technique did I believe give more information than Brightfield (*figs37&38*). Whilst it was possible to compare these cloths, the value of the information is rather questionable.

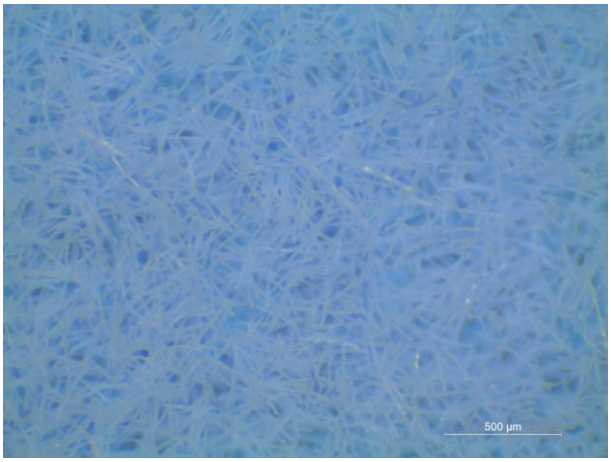
With the LSCM again 3D images were generated from all cloths examined (*figs39&40*). As ever illuminating a 3D object from above reduced the information that could be obtained but the technique did give a good idea on fibre shape and density. It is certainly be possible to compare cloths using this technique and in addition accurate fibre measurement could be carried out.

Examination with an SEM again generated excellent images of the final polishing cloths (*figs41&42*). As before the samples were coated with a thin layer of gold to allow correct examination and reduce charging. Clear detail of the fibre shape, the fibre density & the general morphology is achieved. This microscopical technique allows detailed evaluation of the cloths so that even the smallest differences can be assessed.

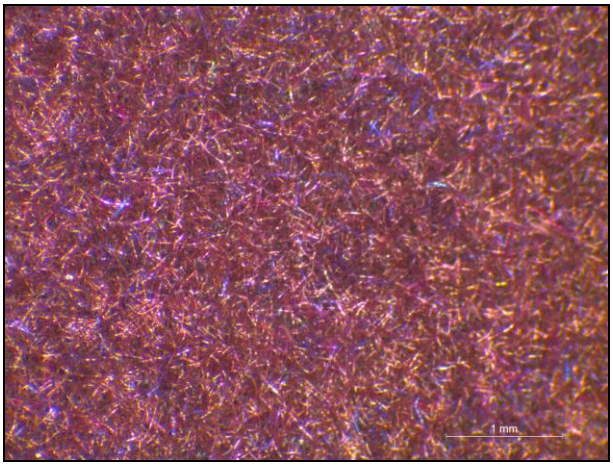
Fig 35. Final Polishing Cloths - Stereo Microscope Examination



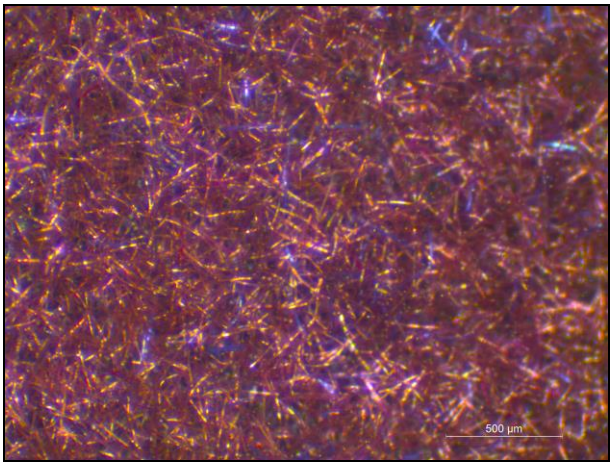
Memphis Stereo Microscope at 20x



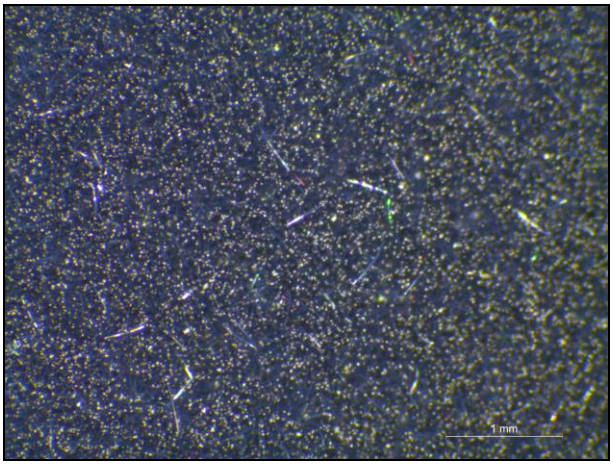
Memphis Stereo Microscope at 40x



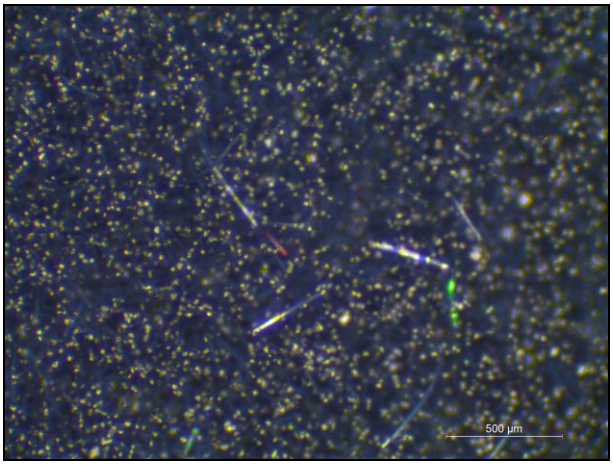
DP Nap Stereo at 20x



DP Nap Stereo at 40x

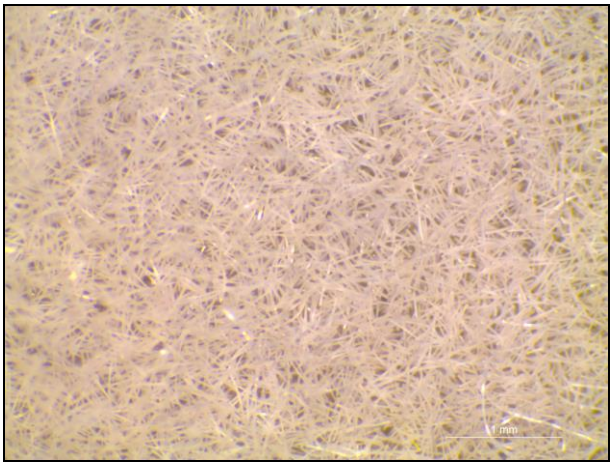


Trounoir Stereo Microscope at 20x

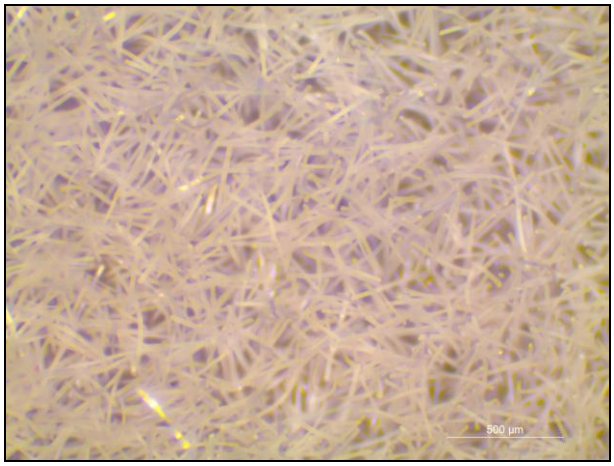


Trounoir Stereo Microscope at 40x

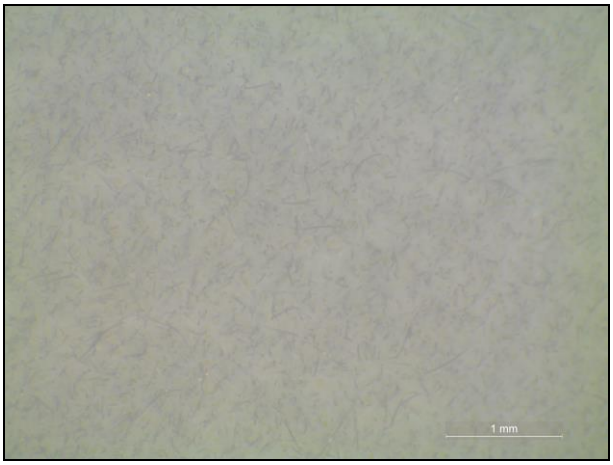
Fig 36. Final Polishing Cloths - Stereo Microscope Examination



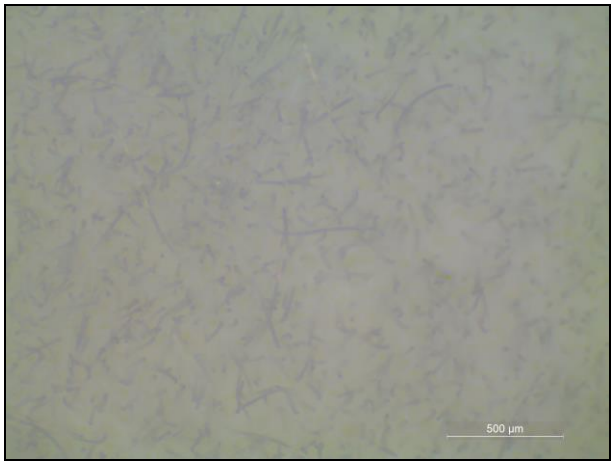
Alphacloth Stereo Microscope at 20x



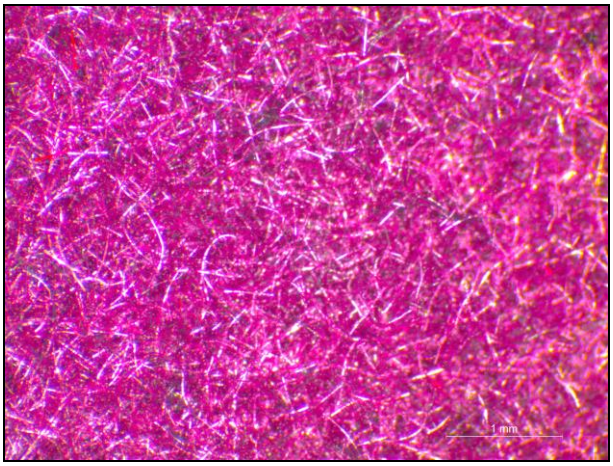
Alphacloth Stereo Microscope at 40x



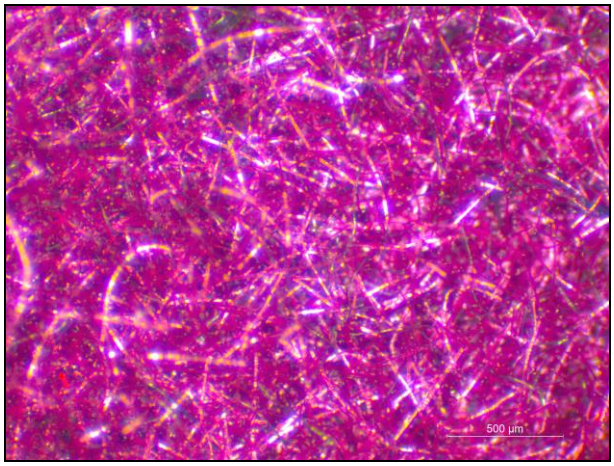
Multicloth Stereo Microscope at 20x



Multicloth Stereo Microscope at 40x

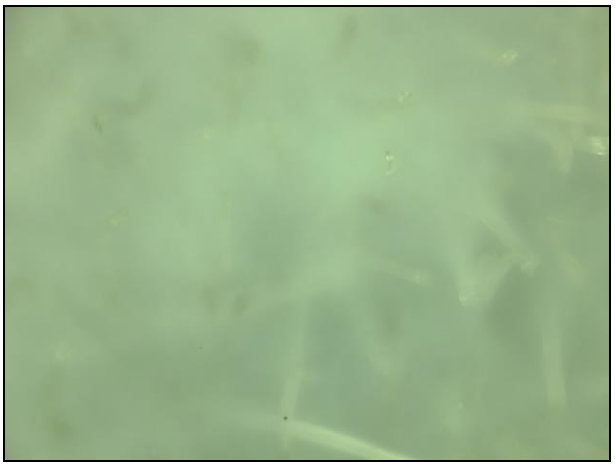


Royal Stereo Microscope at 20x

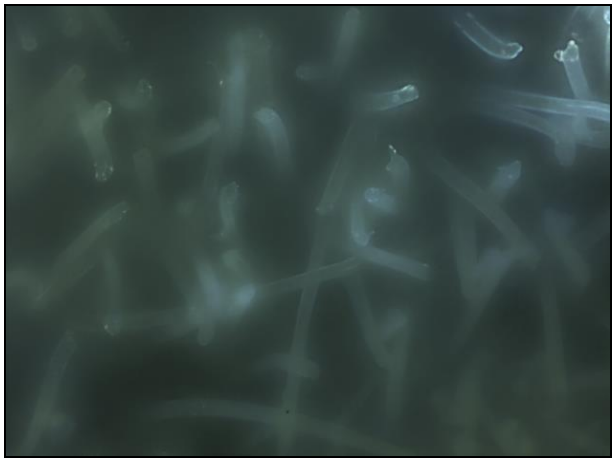


Royal Stereo Microscope at 40x

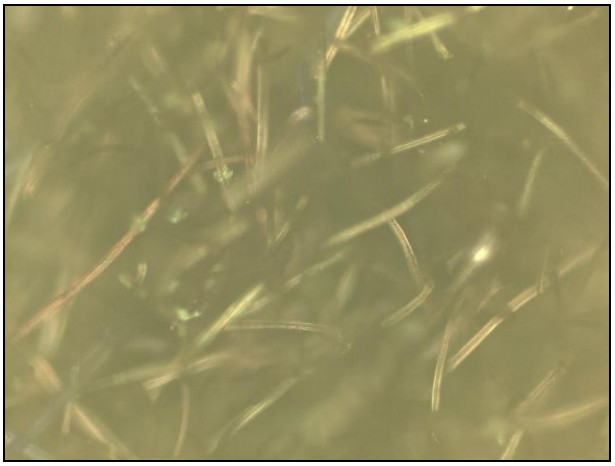
Fig 37. Final Polishing Cloths - Metallurgical Microscope Examination



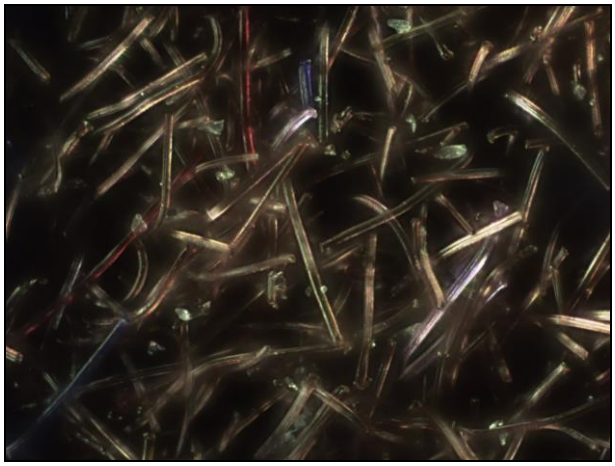
Alphacloth 20x Objective - Brightfield



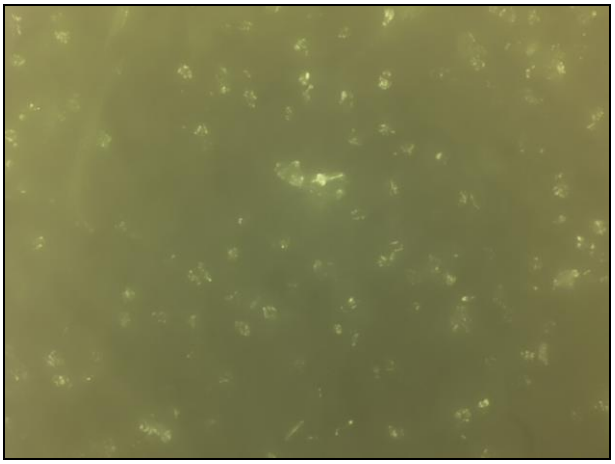
Alphacloth 20x Objective – Darkfield



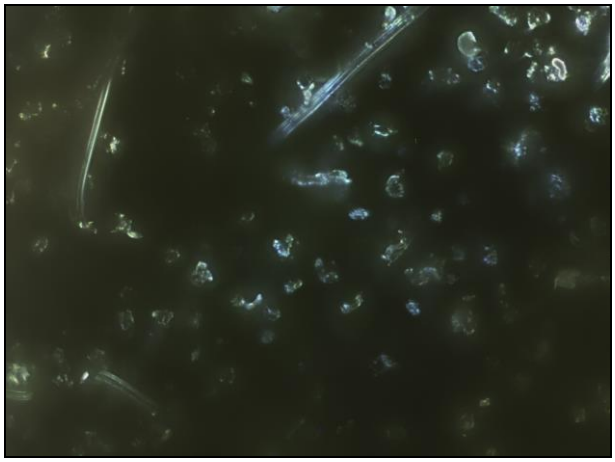
DP Nap 20x Objective - Brightfield



DP Nap 20x Objective – Darkfield

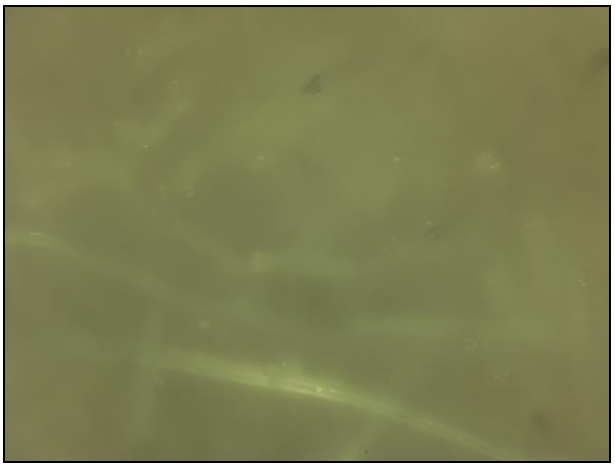


Trounoir 20x Objective - Brightfield

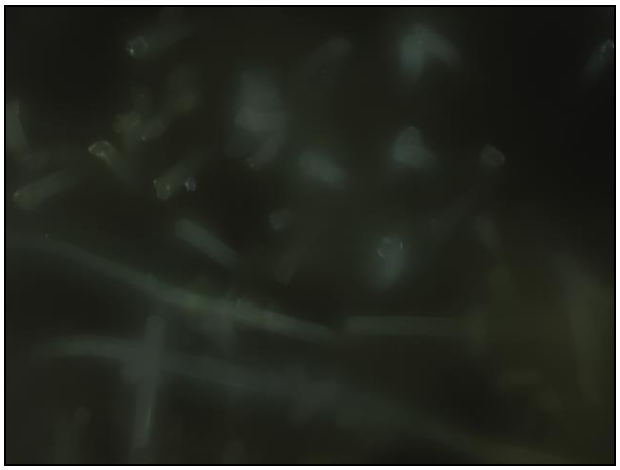


Trounoir 20x Objective – Darkfield

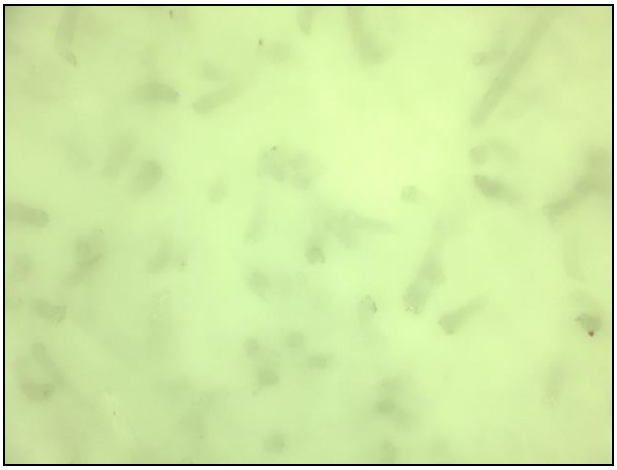
Fig 38. Final Polishing Cloths - Metallurgical Microscope Surface Examination



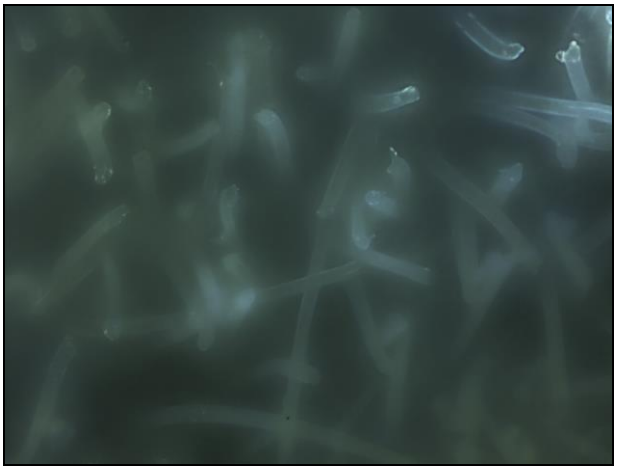
Alphacloth 20x Objective - Brightfield



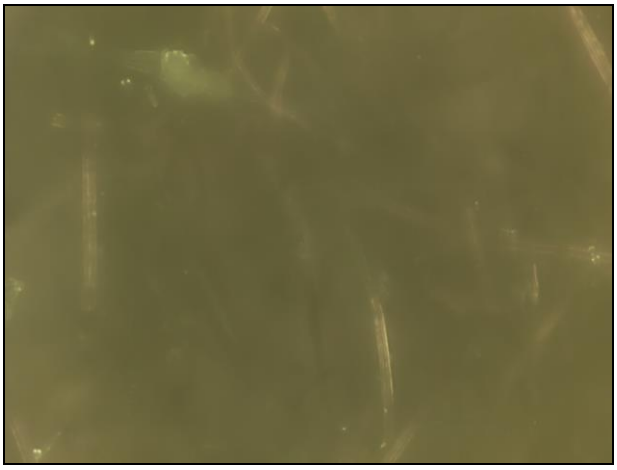
Alphacloth 20x Objective - Darkfield



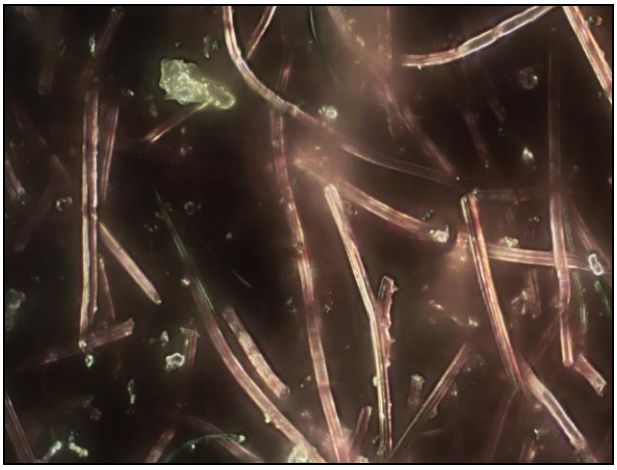
Multicloth 20x Objective - Brightfield



Multicloth 20x Objective - Darkfield

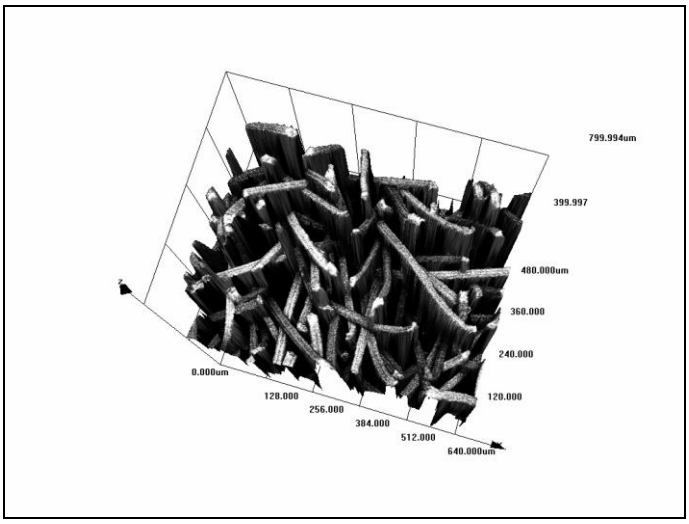


Royal 20x Objective - Brightfield

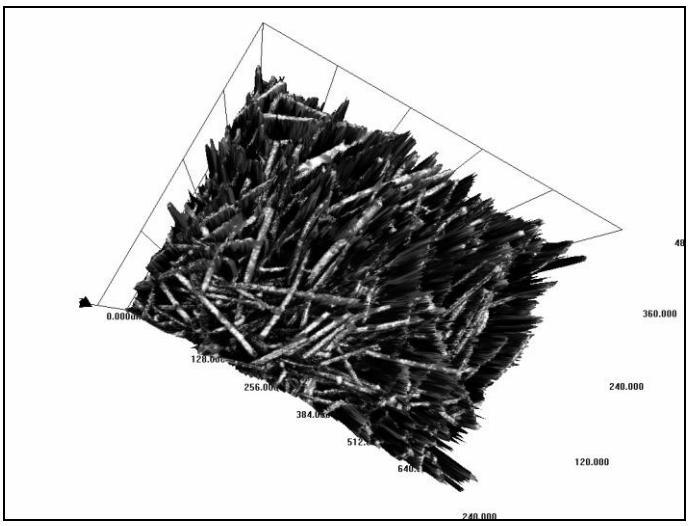


Royal 20x Objective – Darkfield

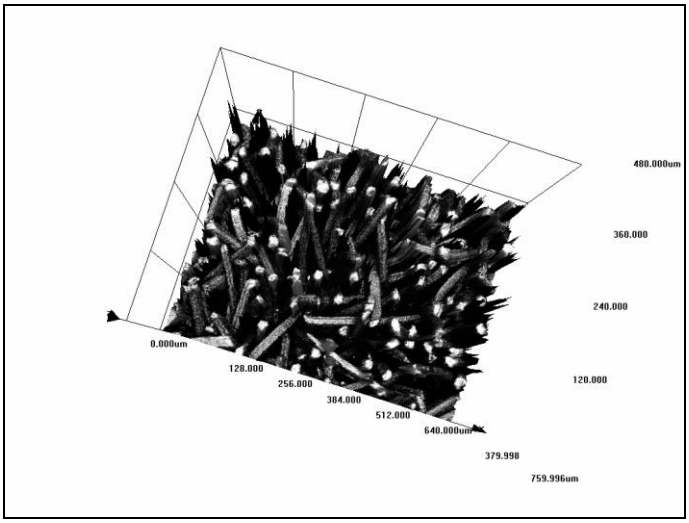
Fig 39. Final Polishing Cloths - LSCM Surface Examination



Memphis 20x 3D Black & White

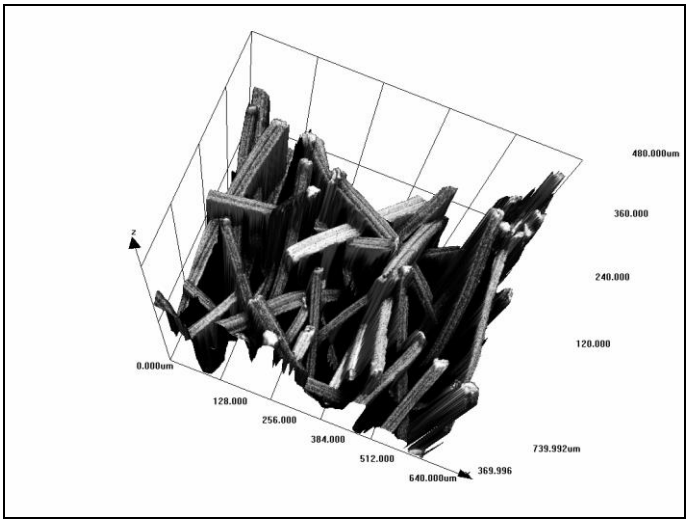


DP Nap 20x 3D Black & White

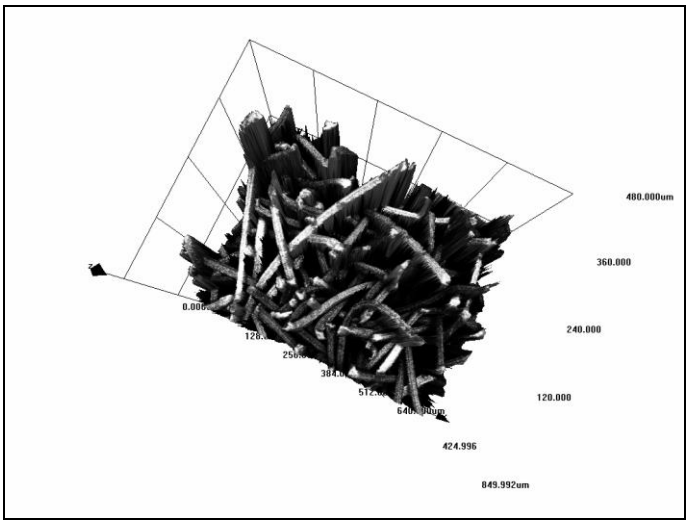


Trounoir 20x 3D Black & White

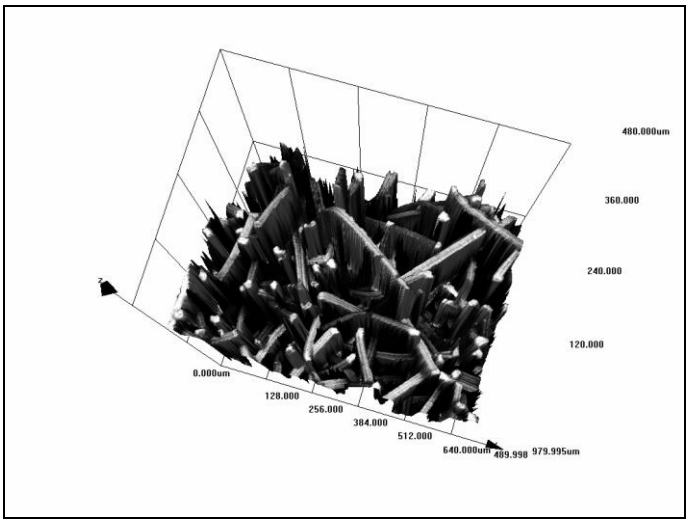
Fig 40. Final Polishing Cloths - LSCM Surface Examination



Alphacloth 20x 3D Colour

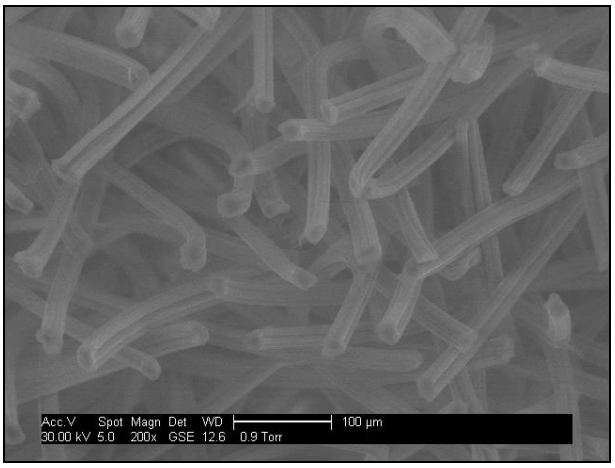


Multicloth 20x 3D Black & White

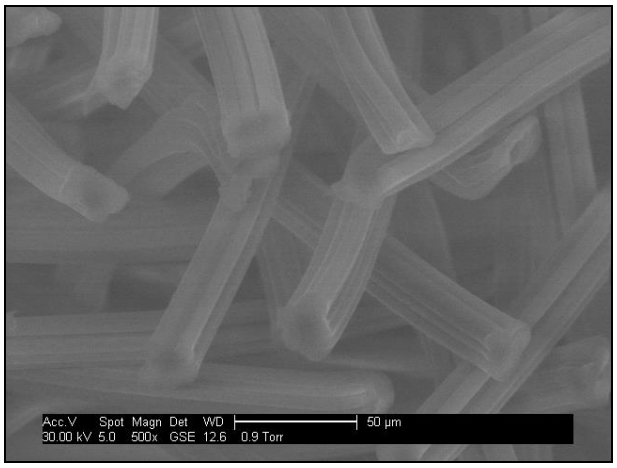


Hacotex 20x 3D Black & White

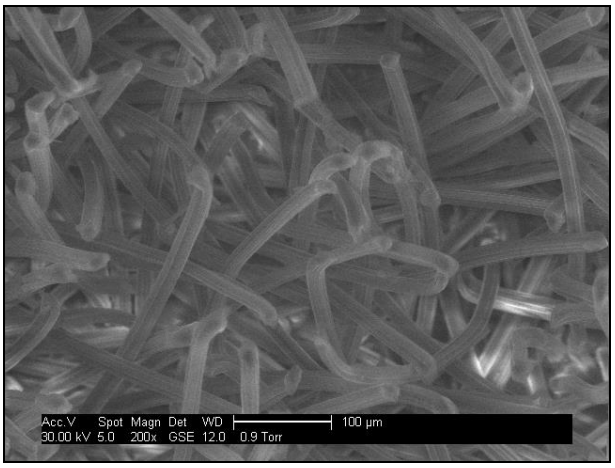
Fig 41. Final Polishing Cloths - SEM Surface Examination



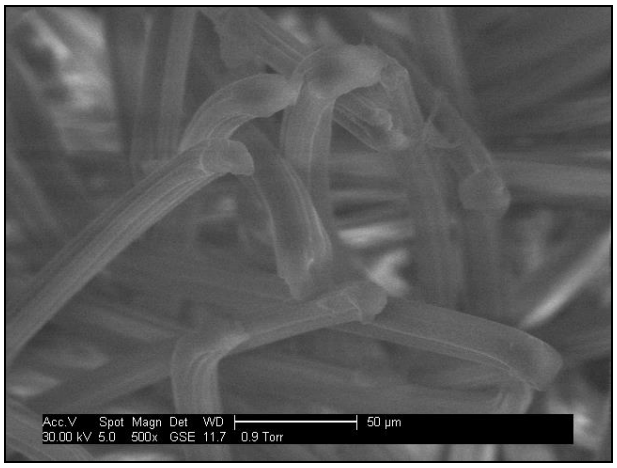
Alphacloth 200x



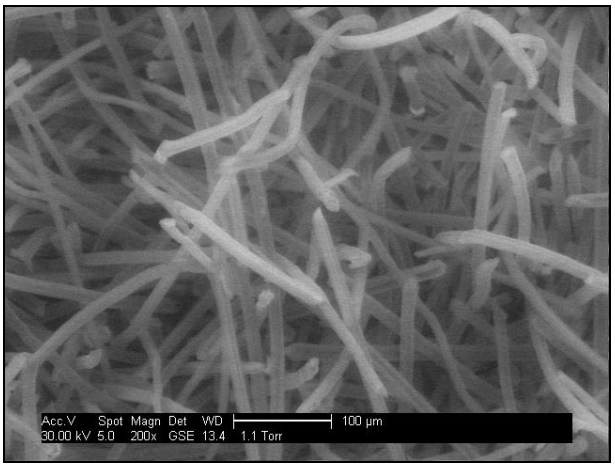
Alphacloth 500x



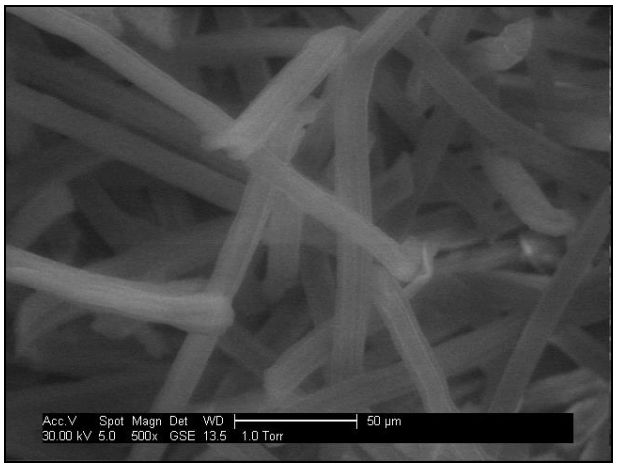
Multicloth 200x



Multicloth H 200x

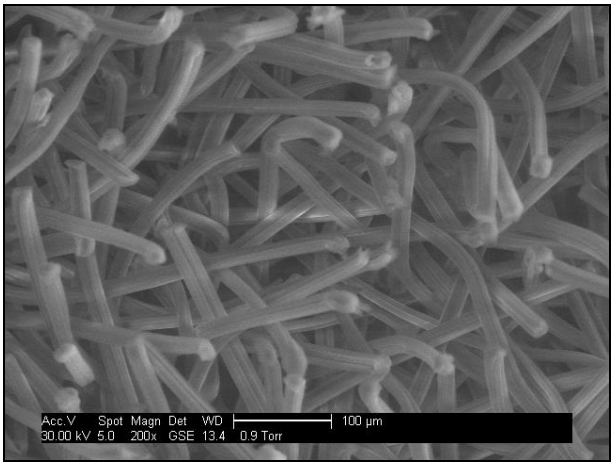


Royal 200x

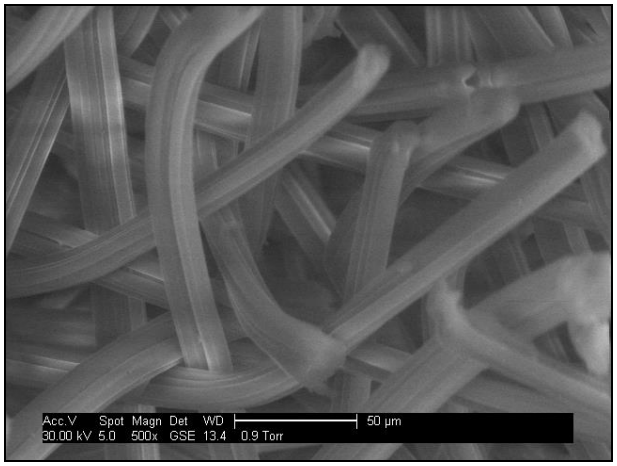


Royal 500x

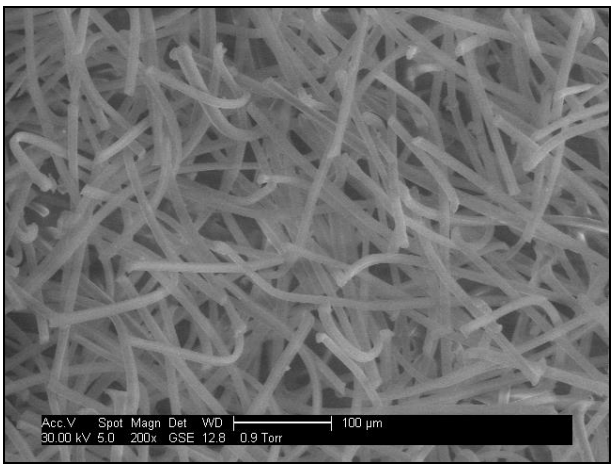
Fig 42. Final Polishing Cloths - SEM Surface Examination



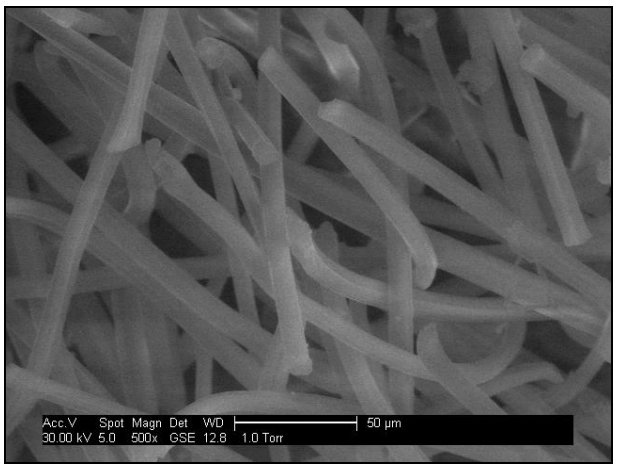
Memphis 200x



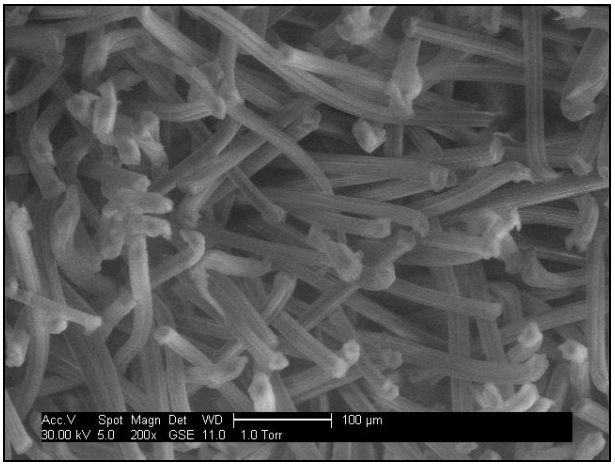
Memphis 500x



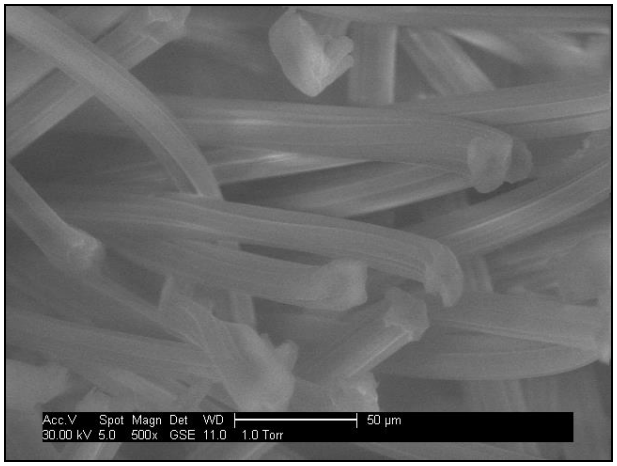
DP Nap 200x



DP Nap 200x



Trounoire 200x



Trounoire 500x

Cloths in operation

It has been stated earlier in the discussion regarding whether cloths were acting as a grinding operation. To operate in a grinding mode the abrasives need to be fixed at the point of contact with the material. To confirm this is the case, cloths that have been used in preparation have been examined using the LSCM. The size of the used platen omits the use of the SEM unless the surface is destroyed to get it in. Therefore to look at this occurrence the LSCM was chosen to get the best resolution and depth of field. The additional option of being able to measure any abrasives present was a bonus. As illustrated in (*fig43*) it is possible to observe how the abrasive is actually fixed into the cloth allowing it act as a fixed point cutting tool and thus leave a long straight scratch.

Another aspect worth looking at is the choice between using a diamond paste or a diamond suspension. The latter is more common now as with the option of semi-automatic preparation machines. The ability to dispense the abrasive regularly & evenly across the platen when needed gives a regular and controlled amount of abrasive which is both independent and in the absence of the operator. It is possible with the LSCM to reveal how the diamonds are concentrated in the paste medium close together and that with a suspension they are more evenly dispersed (*fig44*).

With a diamond suspension the abrasive can be dispersed evenly across the surface whereas a diamond paste will remain in the patches applied as applied by the operator. Obviously if the diamond is in a patch the abrasive can only cut the sample when the patch is under the sample. With a suspension the abrasive is dispensed evenly across the surface and this is cutting on a continuous basis. Using the LSCM it is clearly possible to illustrate the difference on how this occurs on the cloth.

Further examination of diamond in use on Planocloth H with the LSCM illustrates how the diamond is located on a smoother Secondary grinding cloth (*fig45*). We can of course now make measurements of the abrasive in the cloth if required (*fig46*).

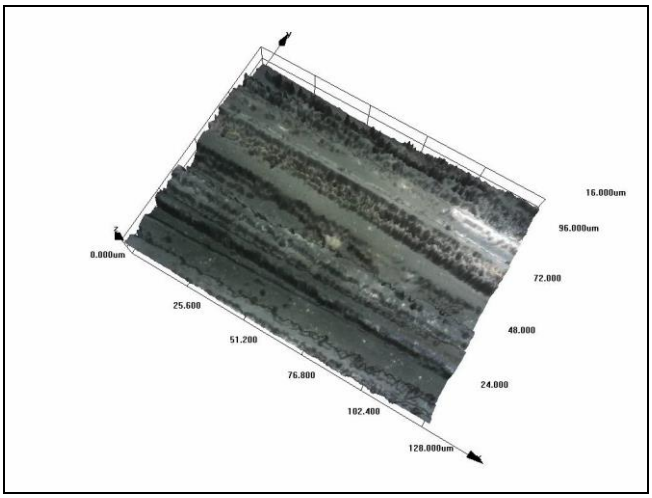
In addition to the napped cloths already discussed here there is another type of cloth used for final polishing. This is a black porous synthetic cloth used with abrasive solutions such as Colloidal silica. Colloidal silica has a typical micrometer size of 0.04 – 0.06 um and also has a pH of 9.8 – 10.2 and is ideal for polishing soft metals such as Aluminium & Copper. As this abrasive solution has an alkaline pH this process is often referred to as chemo-mechanical polishing. The cloth itself is soft but has no nap & appears smooth to the touch. The lack of a nap gives both a fine finish and a flat sample surface with minimal relief.

Examination with both the stereo microscope and a metallurgical microscope gave a good indication of this porous nature of this cloth type (*fig47*). LSCM examination also highlights the nature of this cloths structure (*fig48*). Two different cloths from two different suppliers indicate that the cloths are almost identical. Examination using the SEM again shows excellent detail highlighting the porous nature of the cloth (*fig49*).

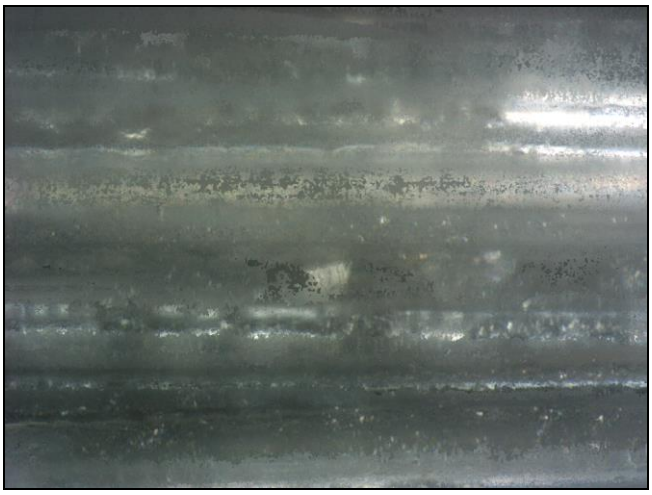
All the microscopical techniques employed give information as to the porous nature of this cloth type and it illustrates how it will soak up & hold the Colloidal silica for the task of preparation.
As employed earlier with the Silicon Carbide & Zirconia surfaces, to get a better understanding of the nature of these cloths it is again possible to carry out a cross sectional analysis using standard metallographic techniques. Using cross sectional techniques reveals details of any weave, supporting materials and its even possible to measure their thickness (*fig50*). Examination of the porous smooth black cloth cross section again gives a good understanding of

the form and in particular the pore structure that characterises the nature of this cloth (*fig51*). Metallographic examination of cross sections is an ideal way of comparing different suppliers' cloths.

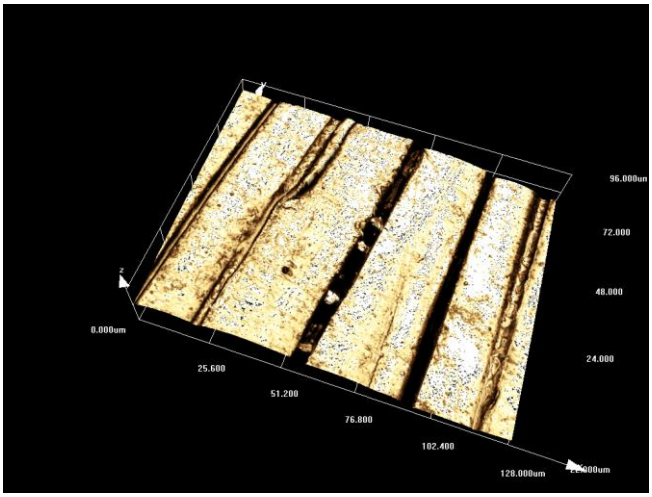
Fig 43. Polishing Cloths in action - LSCM Examination



Fine diamond embedded in Durasilk cloth - LSCM 100x Objective 3D monochrome image

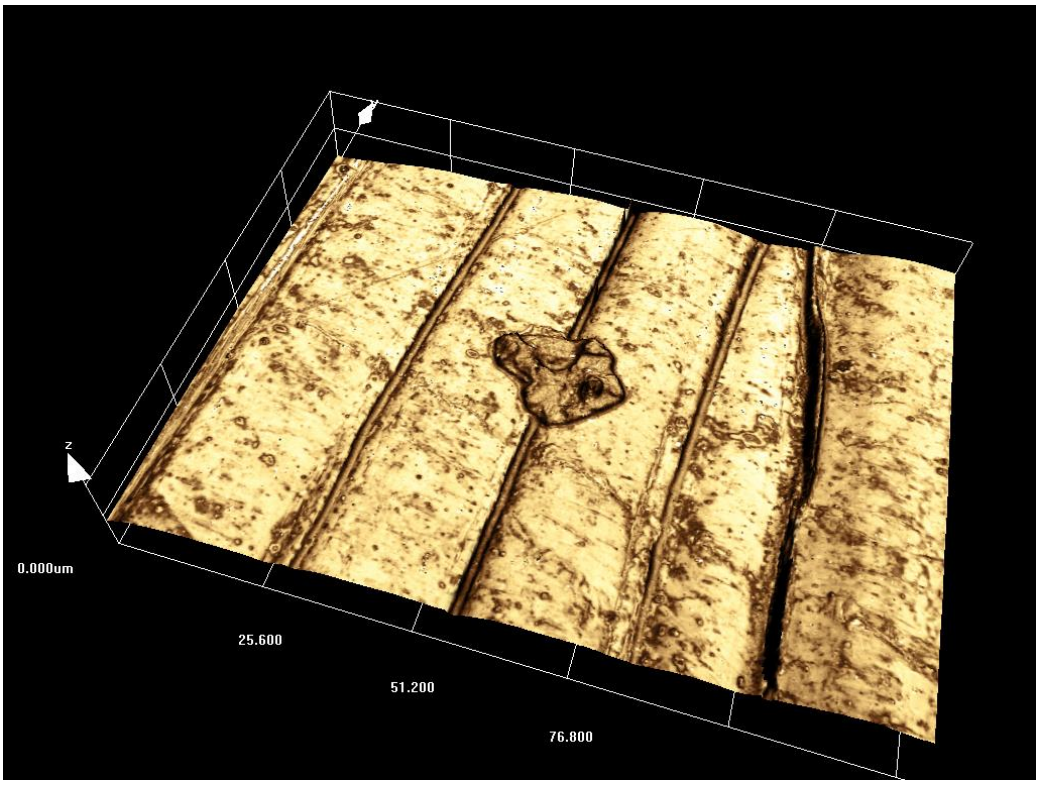


Fine diamond embedded in Durasilk cloth - LSCM 100x Objective 2D Colour image

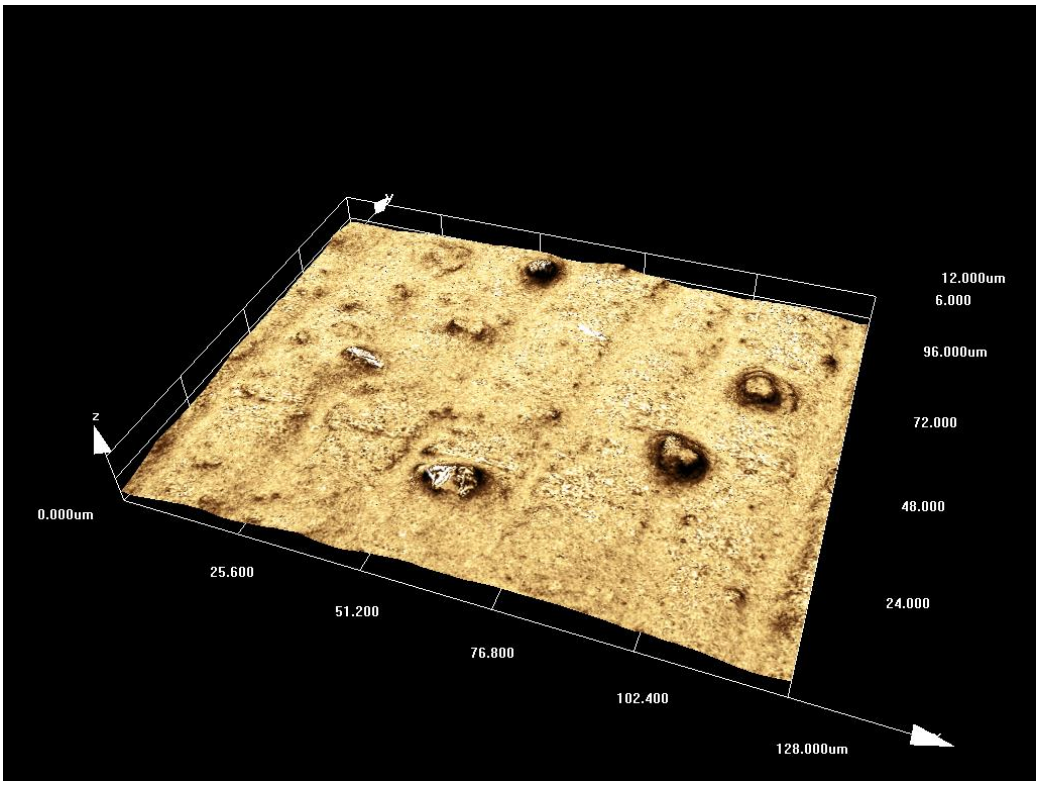


6um Diamond trapped in the weave of an Abracloth - LSCM 100x Objective

Fig 44. Polishing Cloths in action - Diamond Suspension versus Diamond Paste LSCM Examination

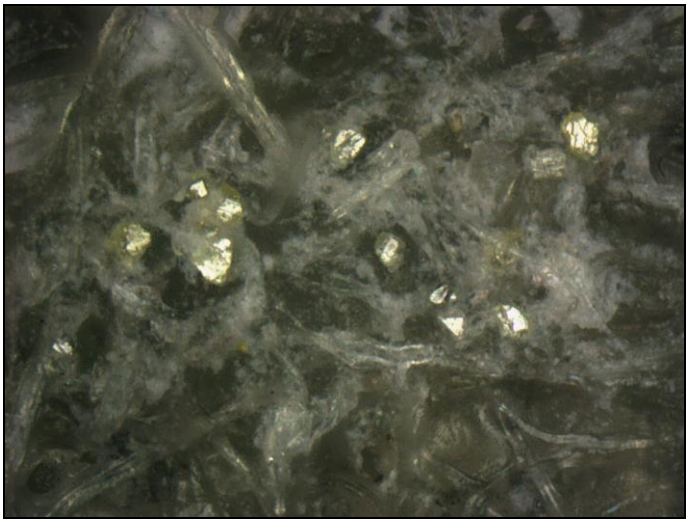


6um Diamond suspension abrasive lodged in Abracloth weave – LSCM 100 x Objective

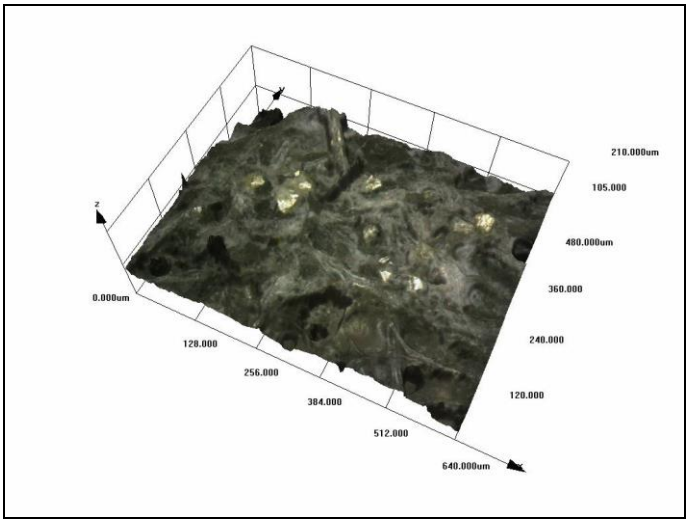


6um Diamond paste abrasives lodged in & over Abracloth weave – LSCM 100 x Objective

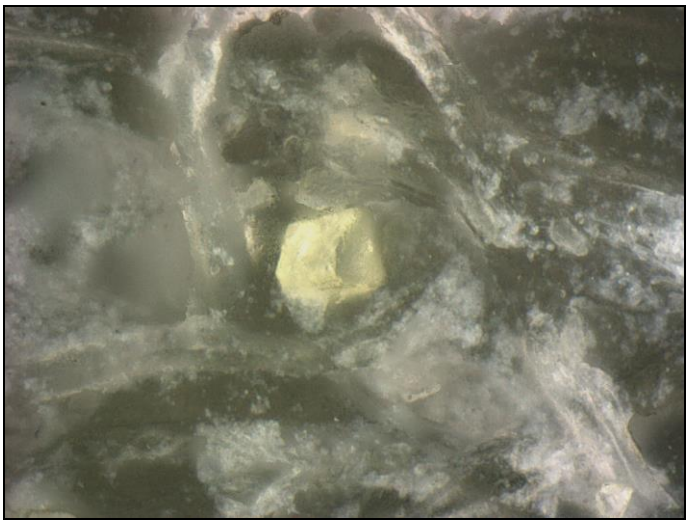
Fig 45. Polishing Cloths in action - 9um Diamond on Planocloth H - LSCM Examination



9um Diamond embedded in Planocloth H LSCM 20x Objective 2D Colour image

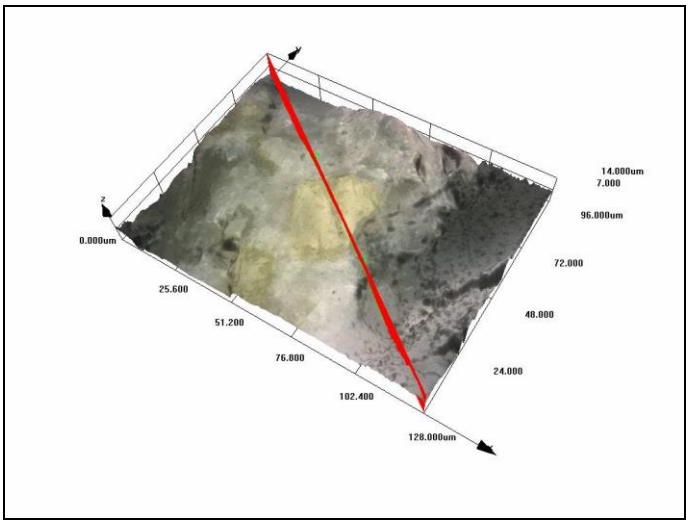


9um Diamond embedded in Planocloth H LSCM 2ox Objective 3D Colour image

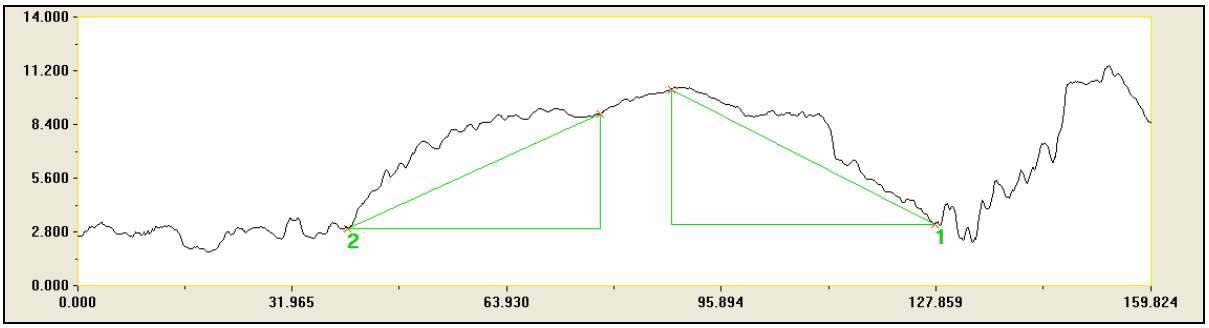


9um Diamond embedded in Planocloth H LSCM 50x Objective 2D Colour image

Fig 46. Polishing Cloths in action - LSCM Examination



9um Diamond embedded in Planocloth H LSCM 50x Objective with measuring line present



Profile across measuring line

#	Judge	Width[um]	Height[um]	Ler
1		39.176	7.034	
2		37.615	5.927	

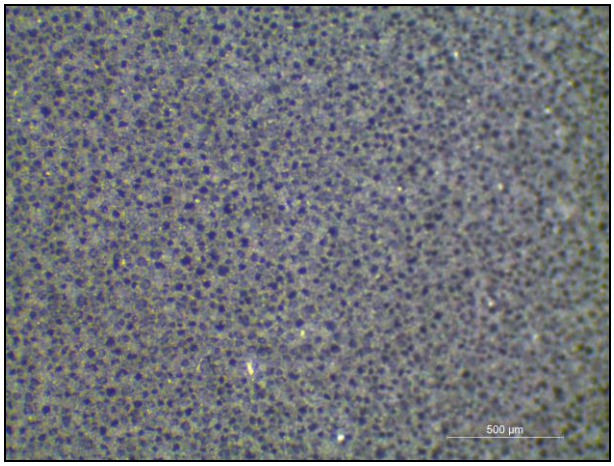
#	2	2
Average	38.395	6.481
Max.	39.176	7.034
Min.	37.615	5.927
Range	1.561	1.107
Sigma	1.104	0.783

Result	Off	Off	Off
Upper limi			
Standard			

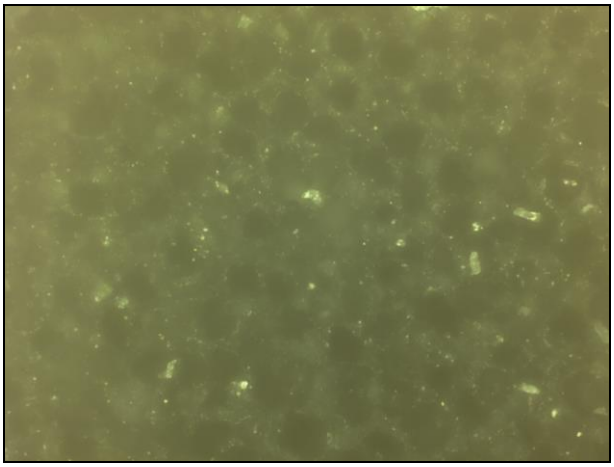
Resulting data indicating an abrasive size in the correct regime for a 9um diamond particle embedded.

Fig 47. Chemicloth - Black Napless Porous Synthetic Polishing Cloth – Multiple techniques

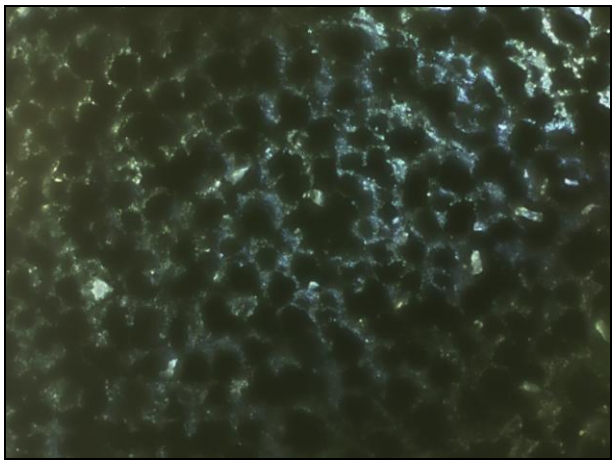
Chemicloth Stereo Microscope at 20x



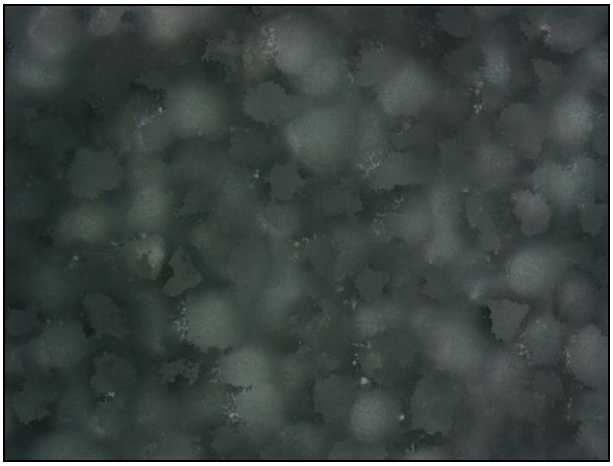
Chemicloth Stereo Microscope at 40x



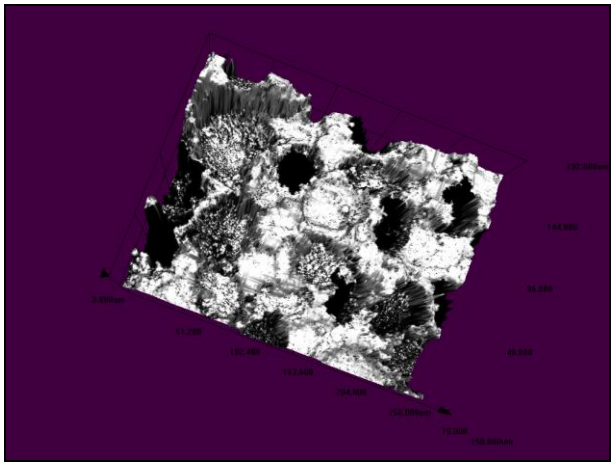
Chemicloth 20x objective Met Mic Brightfield



Chemicloth 20x objective Met Mic Darkfield

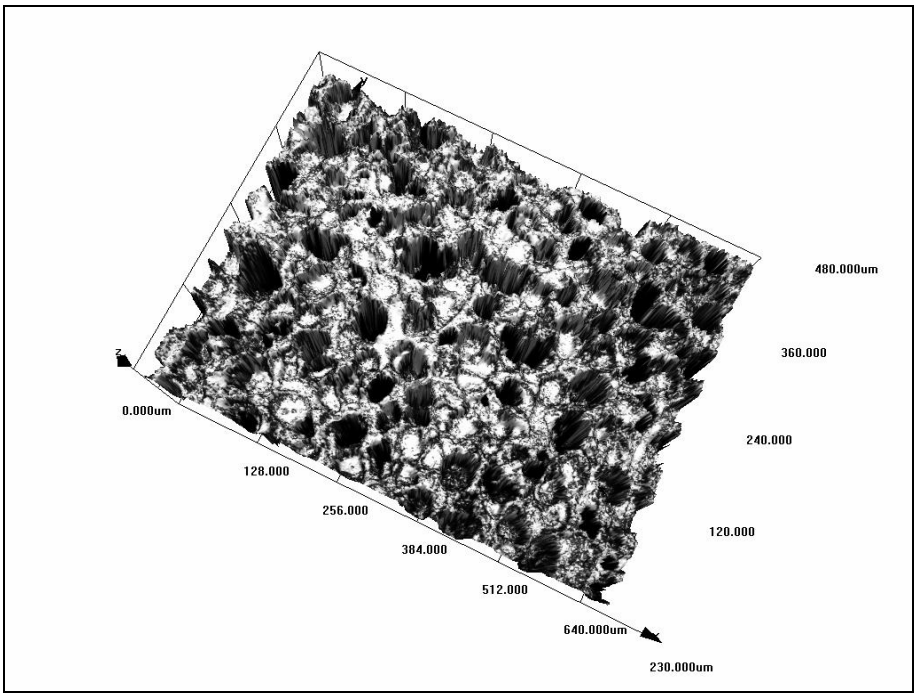


Chemicloth 20x Objective LSCM 2D

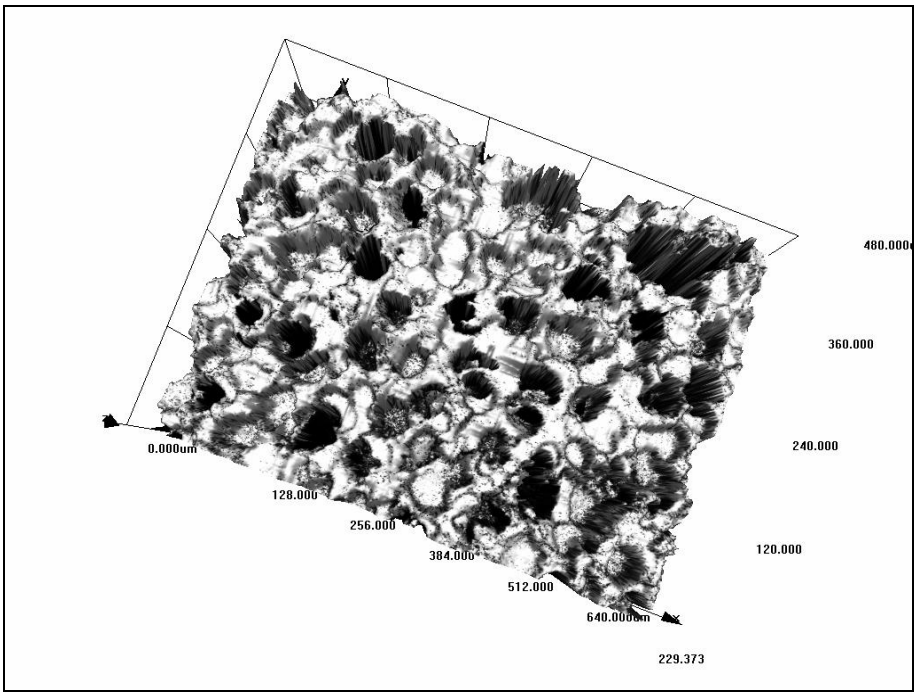


Chemicloth 50x Objective LSCM 3D

Fig 48. Black Napless Porous Synthetic Polishing Cloth - Multiple supplier comparison

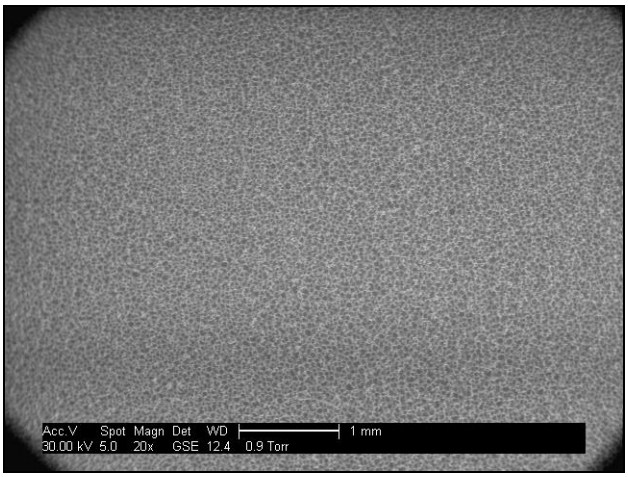


MD Chem 20x Obj - LSCM

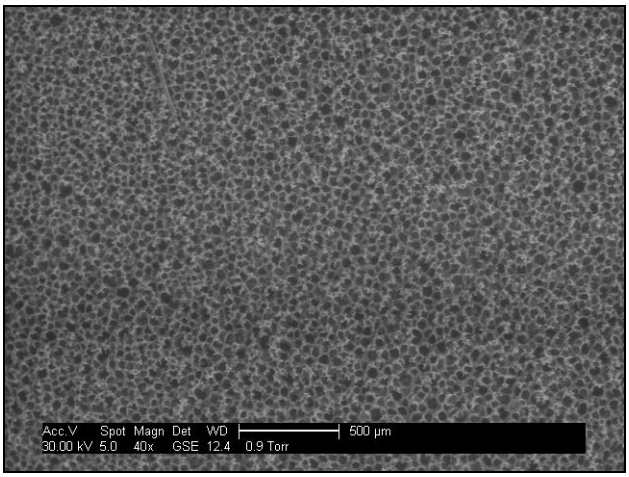


Chemomet 20x obj - LSCM

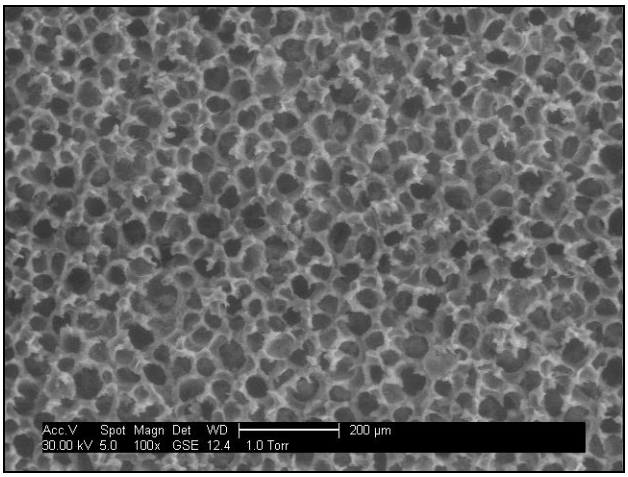
Fig 49. Black Napless Porous Synthetic Polishing Cloths - SEM



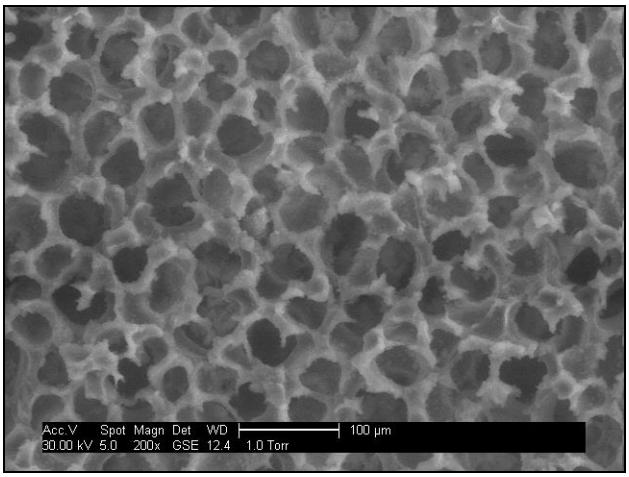
Chemicloth 20x SEM



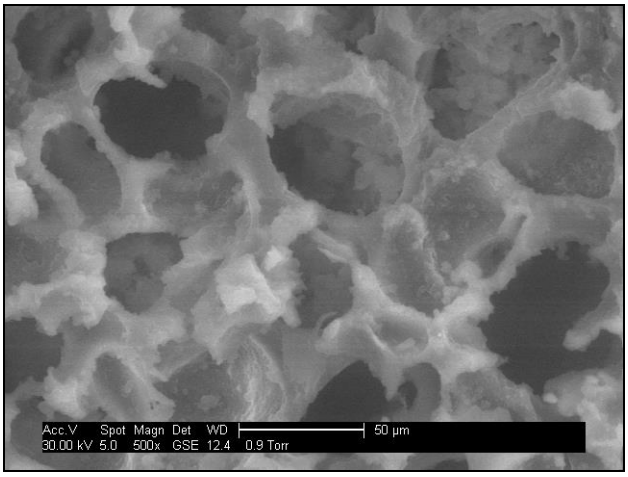
Chemicloth 40x SEM



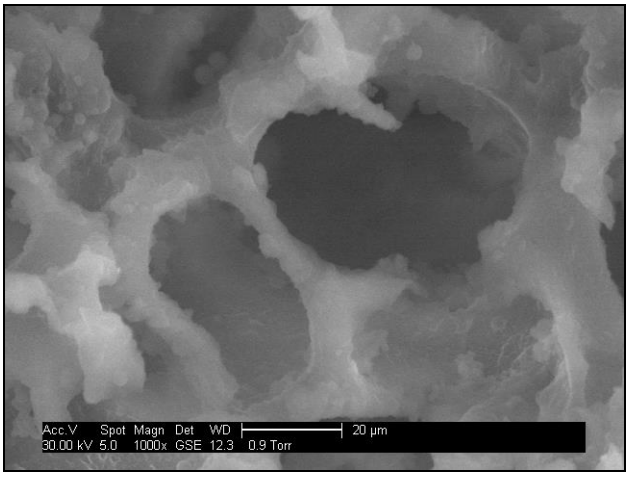
Chemicloth 100x SEM



Chemicloth 200x SEM

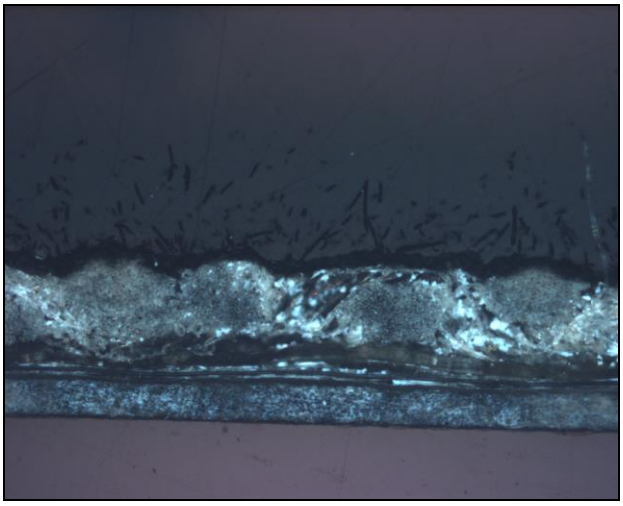


Chemicloth 500x SEM

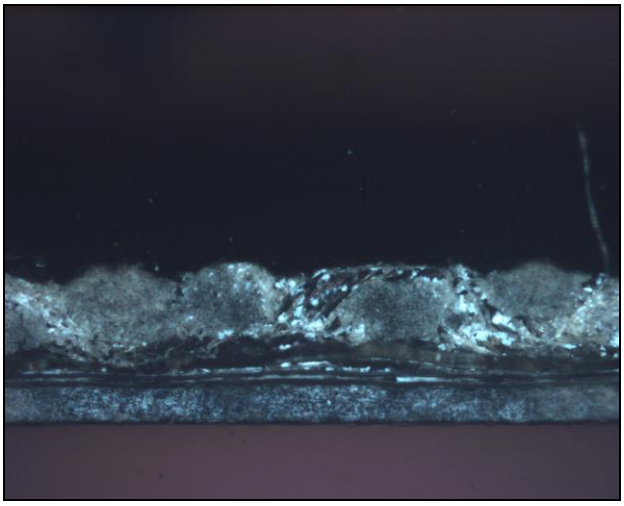


Chemicloth 1000x SEM

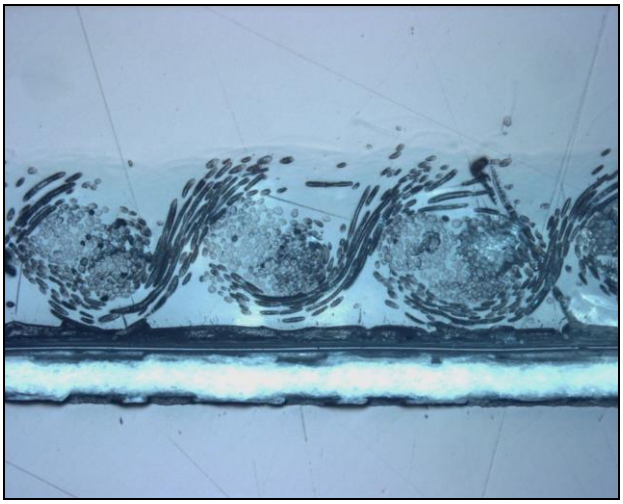
Fig 50. Polishing Cloths - Metallographic Microscope Cross Sectional Examination - Brightfield/Darkfield



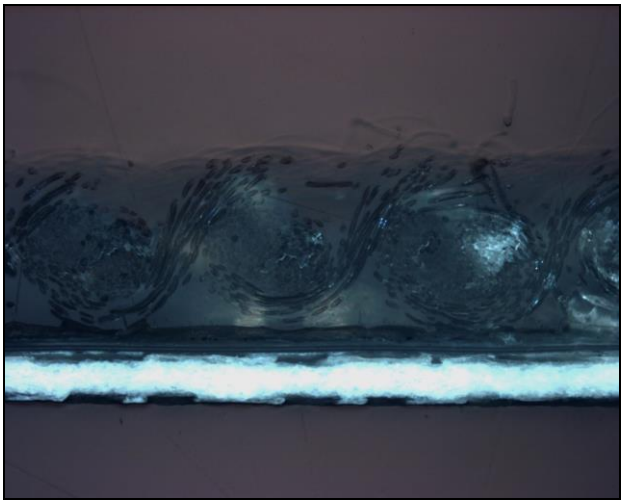
Alphacloth 5x Objective - Brightfield



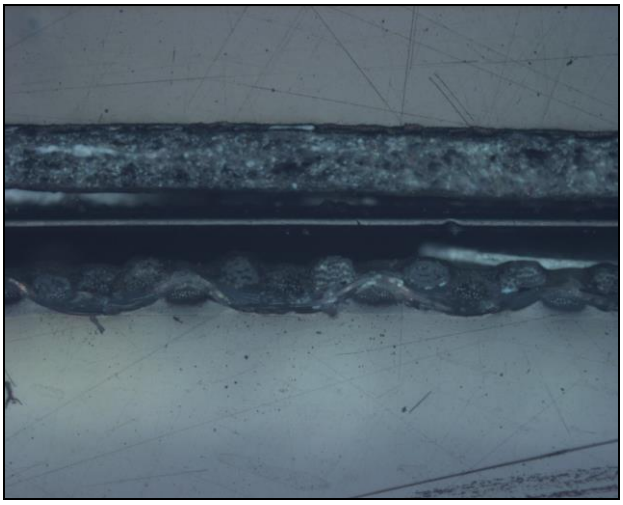
Alphacloth 5x Objective - Darkfield



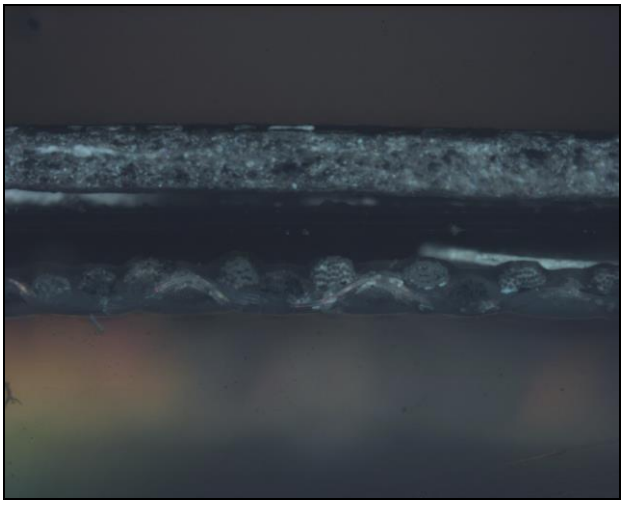
BAA cloth 5x Objective - Brightfield



BAA cloth 5x Objective – Darkfield

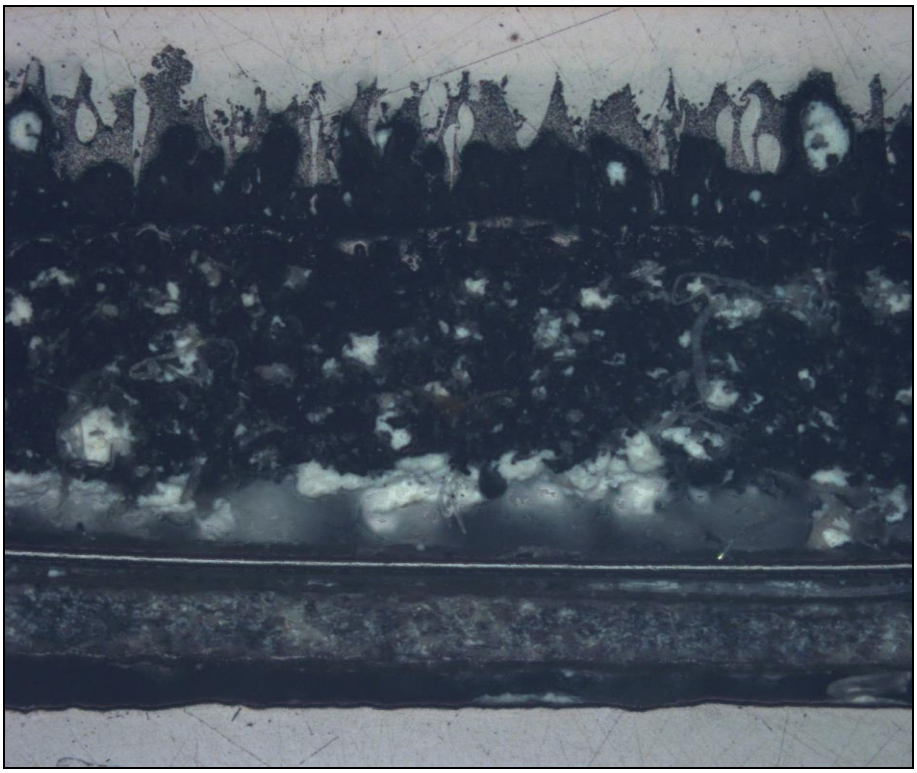


Nylap 5x Objective - Brightfield

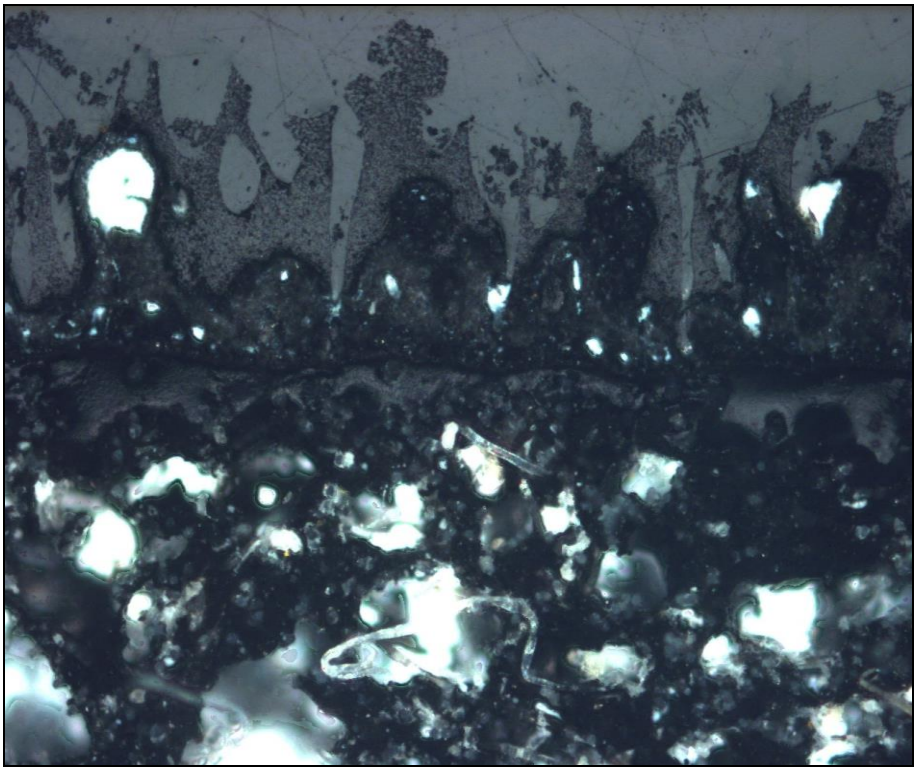


Nylap 5x Objective – Darkfield

Fig 51. Chemicloths - Metallographic Microscope Cross Sectional Examination - Brightfield / Darkfield



Chemicloth 5x Objective – Brightfield



Chemicloth 10x Objective – Darkfield

Abrasives

Having looked at Silicon Carbide & Zirconia where the abrasive is supplied fixed to a surface and also the use of cloths where a diamond abrasive is dispensed on to the surface and lodged in to the cloth, to get a better understanding of the process we also need to look at the diamond abrasive too.

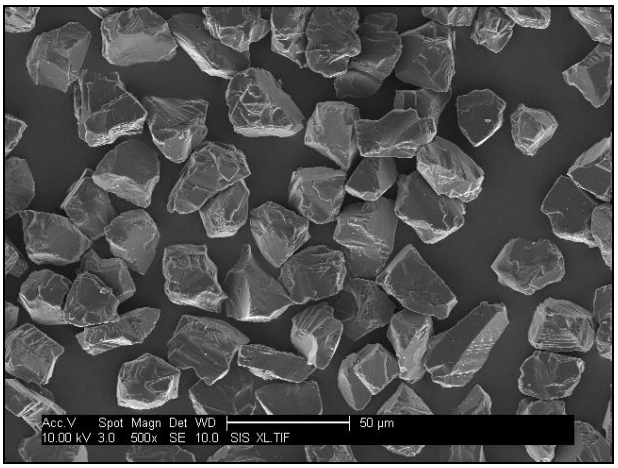
Diamond in the metallographic world is usually supplied in two forms, Polycrystalline & Monocrystalline. Both types are supplied regularly with the monocrystalline being more popular as it is the least expensive. Whilst it would be difficult to assess these differences using simple optical techniques, the SEM will give us the option of great depth of field and the ability to use higher magnifications and achieve greater resolution revealing any differences between the two types.

Diamond abrasives tend to be supplied in either a suspension or a paste therefore a sample of the raw product prior to processing was obtained. Examination of both 30um & 3um samples was carried out using the SEM to see any differences in morphology.

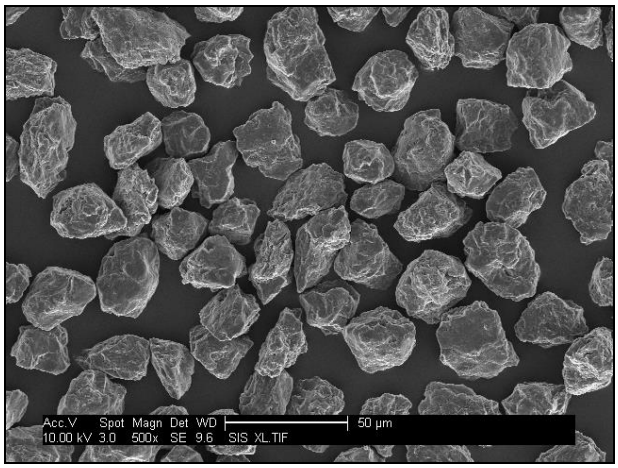
Two completely different processes are employed to manufacture Monocrystalline and Polycrystalline diamond abrasives. To manufacture the Polycrystalline variant a process called Shock Synthesis is used (Rohr. N). The Shock Synthesis process takes graphite and by the use of explosions creates diamond microcrystallites as small as 0.01um. These microcrystallites are then combined to create particles of the required abrasive size. Monocrystalline diamond on the other hand is grown under high temperature & high pressure conditions and is then sorted into the corresponding abrasive size grades.

Examination using the SEM reveals the typical morphology of the two types of diamond (*figs52&53*). The polycrystalline diamond shows multiple facets when compared to its monocrystalline equivalent and illustrates the reason for the greater stock removal and better surface finish produced (Lamplan 2006).

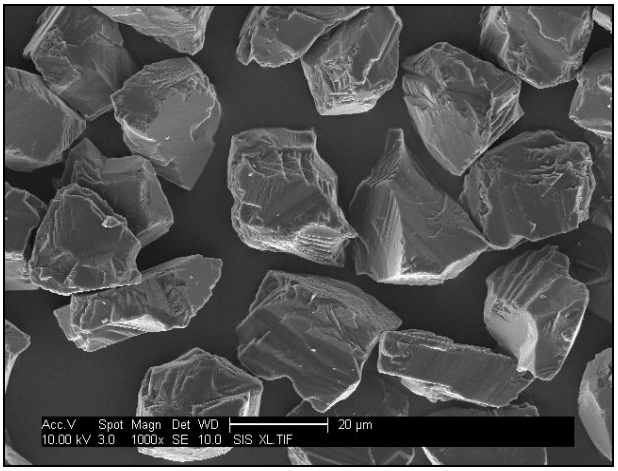
Fig 52. Polycrystalline - Monocrystalline Diamond Comparison 30um - SEM examination



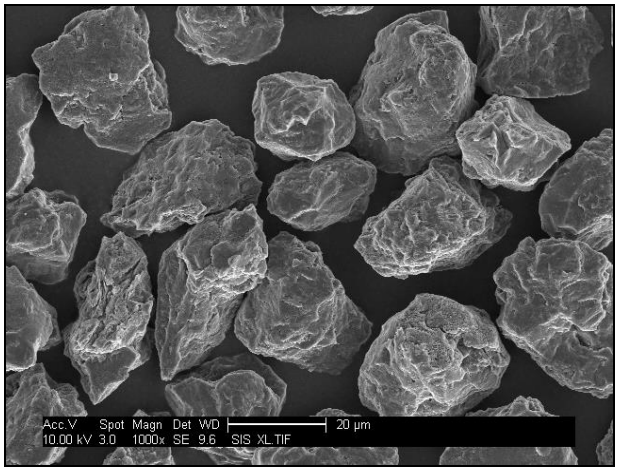
30um Monocrystalline diamond – 500x



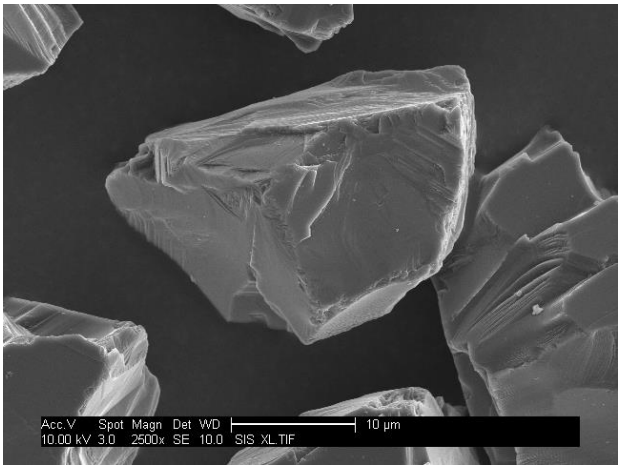
30um Polycrystalline diamond – 500x



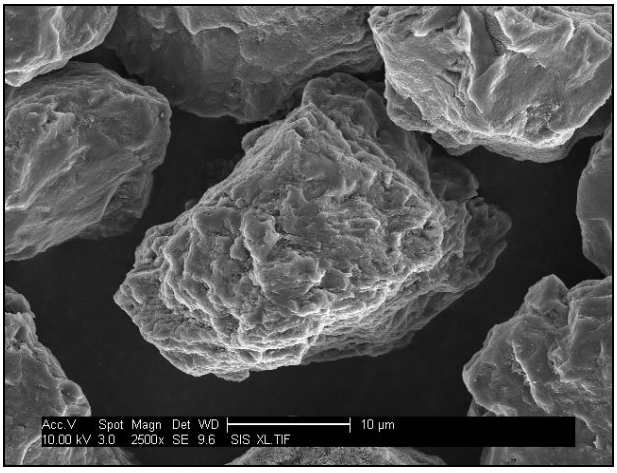
30um Monocrystalline diamond – 1000x



30um Polycrystalline diamond – 1000x

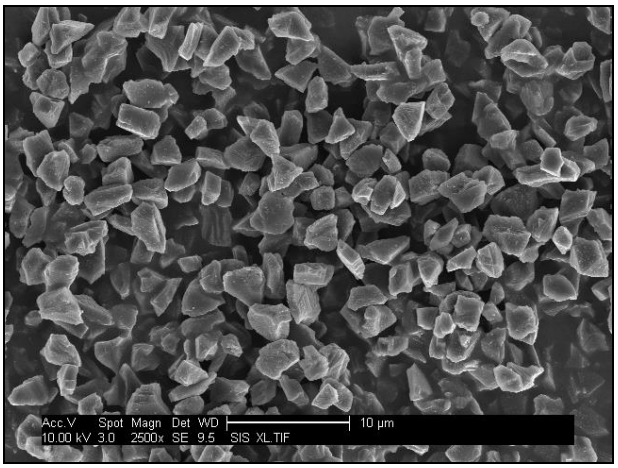


30um Monocrystalline diamond – 2500x

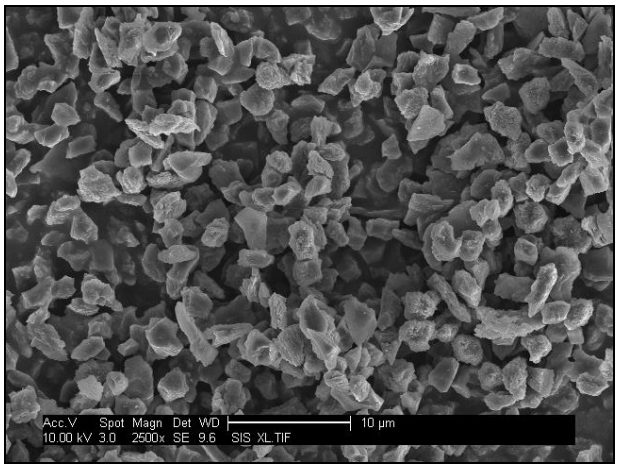


30um Polycrystalline diamond – 2500x

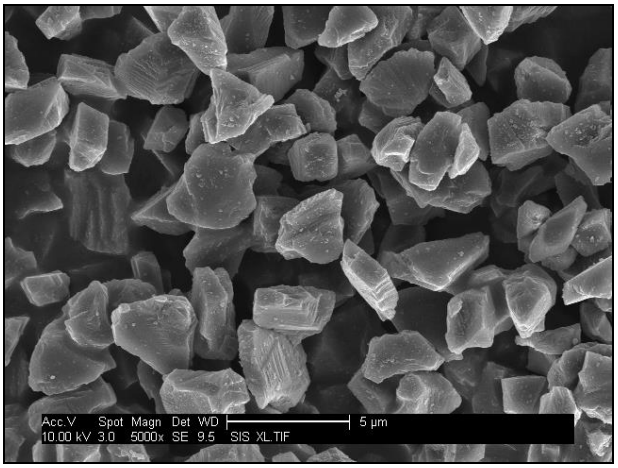
Fig 53. Polycrystalline - Monocrystalline Diamond Comparison 3um - SEM examination



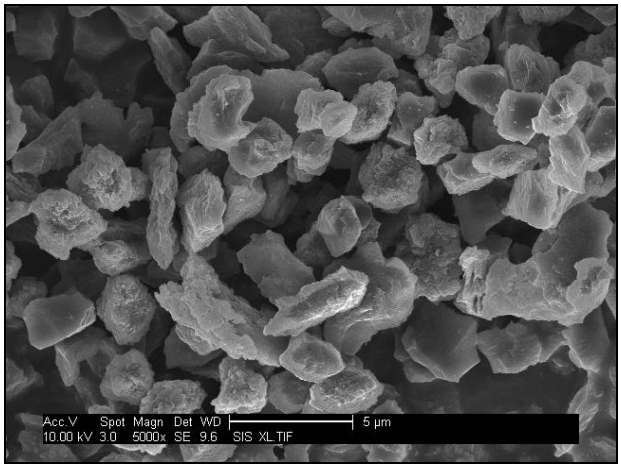
3um Monocrystalline diamond – 2500x



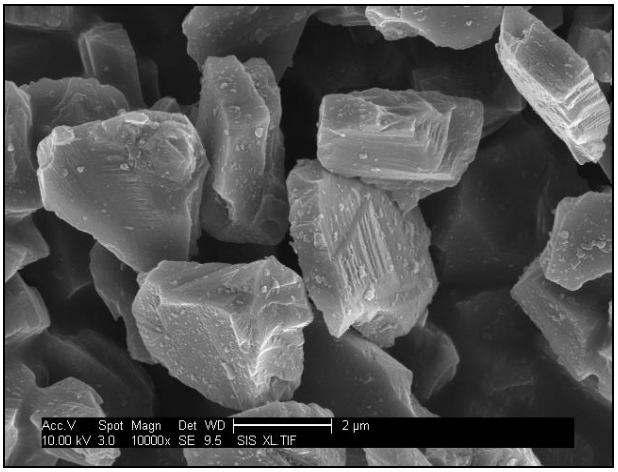
3um Polycrystalline diamond – 2500x



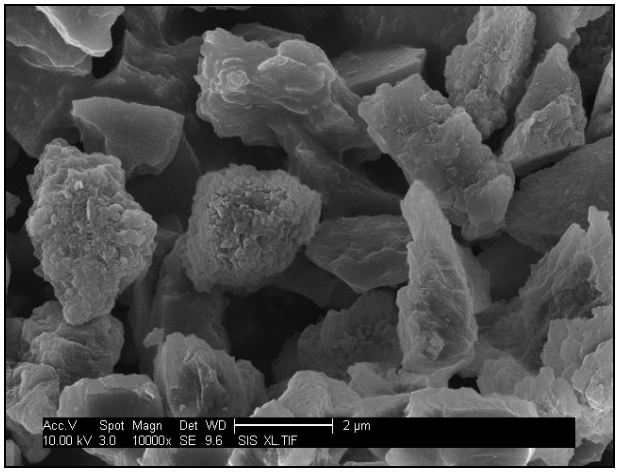
3um Monocrystalline diamond – 5000x



3um Polycrystalline diamond – 5000x



3um Monocrystalline diamond – 10000x



3um Polycrystalline diamond – 10000x

Generating & Evaluating Preparation Procedures

Knowing the properties of the material to be prepared and now having an understanding of the surfaces & abrasives based on our use of microscopy, we can now start to think on how we can prepare our materials. Most metallographic suppliers will provide typical preparation procedures for a wide range of materials. These indicate not just the machine parameters but also the surfaces and the abrasive combination that are recommended for particular materials. This means the materials scientist doesn't have to start from a zero position. The suppliers have already done most of the work for the Metallographer. With knowledge of metallographic consumables achieved via the various microscopical techniques, it is now possible to understand the thinking behind a supplier's generated procedure and apply them to other materials.

Using microscopy, it is possible to examine the recommended surfaces and abrasives and start to understand why the various surface / abrasive combination have been chosen. It also allows comparison by various suppliers' products and also allows the Metallographer to modify these standard routes to suit their particular material.

If a preparation is started with one of these suppliers recommended procedures and on completion it reveals a surface that is not correctly prepared, knowing how the various consumables behave on the surfaces becomes a key to modifying the route to suit the actual material. In addition, and particularly on the first occasion of preparing a sample looking at the final surface at the end of the preparation is of little use when things go wrong. To prevent such situations arising and to get a better understanding of what is happening during a preparation it is better to examine the specimen after the various individual preparation stages. By doing this it is possible to examine the way in which the specimens' surface is being affected by the abrasive / surface combination. It also builds a deeper understanding of the process that is taking place. Even observing the scratch pattern can indicate how efficient the cutting action is and highlight potential problems. Other features that can be checked for is the level of damage left at each stage maybe manifesting itself as pull out, relief or even chemical attack.

For example, consider a secondary grinding stage after a primary grind. It makes sense for the operator to initially run the stage for 3 minutes and check the specimen surface for progress using a metallurgical microscope. This could be to examine for example the type of scratch pattern that the abrasive surface / surface combination leaves. Examination to see whether there is any pullout, whether there is any edge rounding or differential abrasion due to different materials with different properties, or even just to check that the damage from the previous stage has been removed. If the preparation appears to be going well and there is no evidence of trouble it is advisable to go back to the same stage for slightly longer to see if prolonged preparation is beneficial. If there is an improvement then repeat until either there is no advantage seen or the specimen surface actually degrades. This is the only way to determine what the best preparation period is for your specimen at this preparation stage. It is only by the use of a metallurgical microscope that it is possible for the Metallographer make a valid judgement of progress.

In addition to using the metallurgical microscope to assess damage and progress towards a damage free sample, close examination can illustrate how the material behaves during the grinding process. Microscopical examination of scratches gives a useful indication of how a material is being cut. Ideally the scratches should be straight and continuous with clean edges indicating an efficient cut. In some instances, the perfect cut might not be possible but it is always preferable and if it isn't ideal it is possible to see how poor it is.

Again, a wide range of microscopical techniques can be used to examine scratch patterns and whilst it might only be practical in the laboratory during routine preparation to use a

metallurgical microscope, other techniques can be employed when necessary. A small matrix of experiments was carried out to see how three different grinding surfaces, Silicon Carbide, Zirconia & the Cameo fixed diamond disc effect three different materials, a steel, a copper alloy and a white metal bearing alloy with regard to scratch pattern produced. Whilst these samples are all ductile materials, their hardness is different and the way they will grind will also vary.

To record these scratch patters, equipment including the SEM, the LSCM and the metallurgical microscope were employed. The matrix of results illustrates how the softer white metal bearing material is considerably more damaged in all three grinding operations and this is shown clearly in all techniques with the SEM producing the greater detail. (*figs54-55-56*) The copper alloy is then the next in hardness and this displays less damage than seen on the white metal bearing (*figs 57-58-59*). The Steel sample is harder still and demonstrates the cleanest scratch pattern (*figs 60-61-62*). Also revealed is that the Silicon Carbide gives the cleanest cut on all three materials but the cameo fixed diamond disc gives the more consistent surface finish.

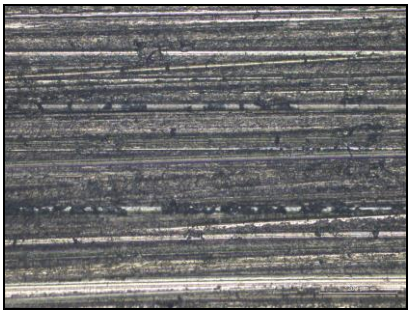
As in the majority of occasions only a Metallurgical microscope will be available to check whilst preparation is being conducted a series of images obtained at various magnifications has been included. The other techniques whilst comprehensive are included to give an understanding of the information obtainable. In addition, if one is looking for surface conditions the LSCM can provide extra data as well. This can give quantitative data for these surfaces similar to a roughness measurement.

Understanding how the SEM, LSCM & Metallurgical microscope can be used to look at the surfaces, it shows how effective the metallurgical microscope is in providing the information you need with the minimum of work or expense. The detail obtained clearly indicates you can assess different preparation surfaces on a range of materials to assess the cutting action and damage levels. It is normal to have access to this instrument in a metallographic laboratory, as this is how the completed preparation will be examined. It is therefore clear that in most circumstances more advanced techniques are probably not required.

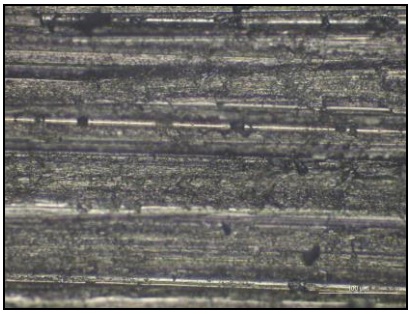
With that in mind examination of the surfaces of these three materials using a metallurgical microscope after a typical secondary grinding stage – (Planocloth H with 9um diamond) - it is clear to see that there is a variation in the amount of damage that is left following this stage and it can easily be assessed optically. This is best illustrated when comparing the copper alloy with the more difficult to resolve structure of the white metal bearing material (*fig63*). With little to view in an as ground Steel we have to rely on the clarity and definition of the scratches themselves rather than on the background microstructure damage.

Using such evidence on how a material interacts during these grinding processes it is possible to create and monitor a preparation procedure.

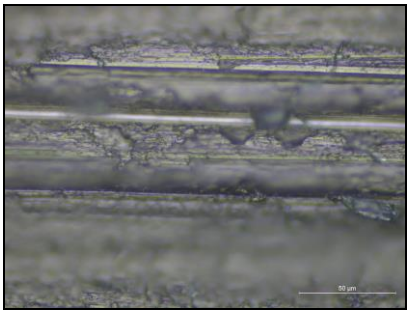
Fig 54. Scratch Patterns on WMB using various P120g surfaces revealed by the Metallurgical microscope



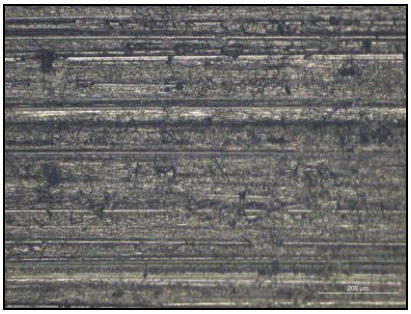
Silicon Carbide 10x Objective



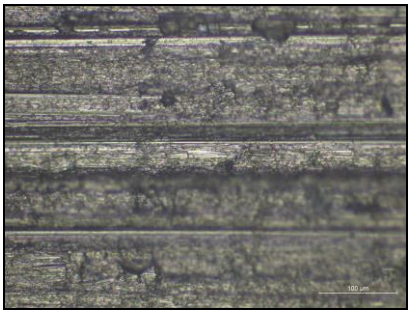
Silicon Carbide 20x Objective



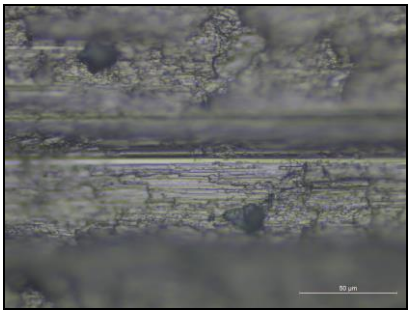
Silicon Carbide 50x Objective



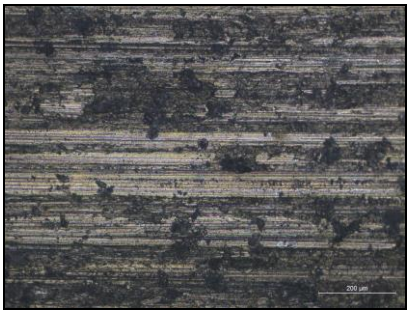
Zirconia 10x Objective



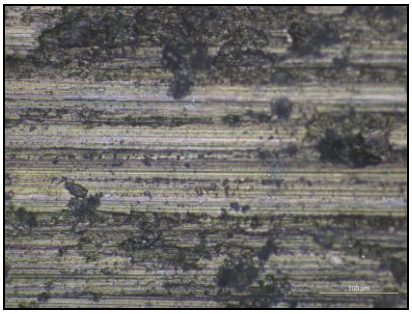
Zirconia 20x Objective



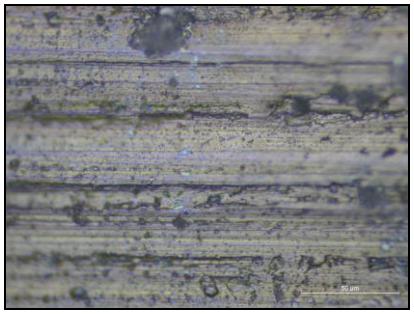
Zirconia 50x Objective



Cameo 10x Objective

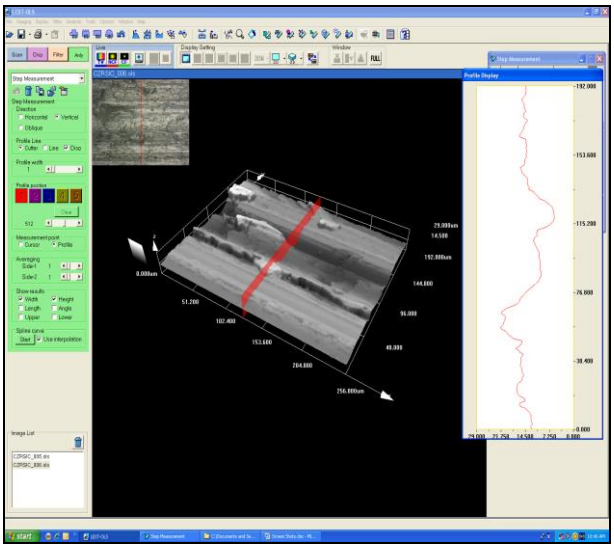


Cameo 20x Objective

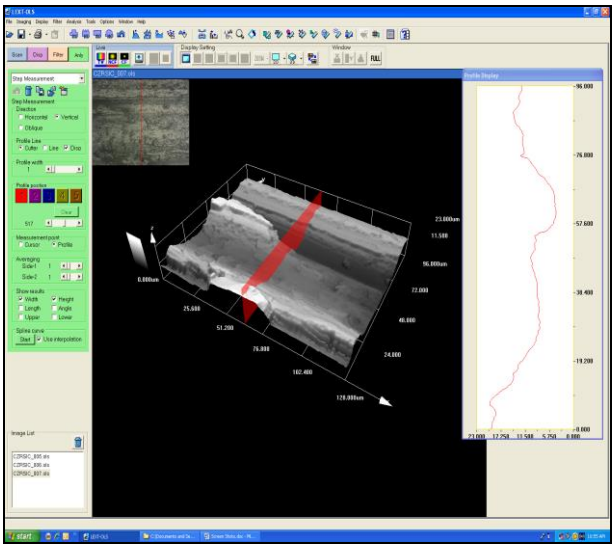


Cameo 50x Objective

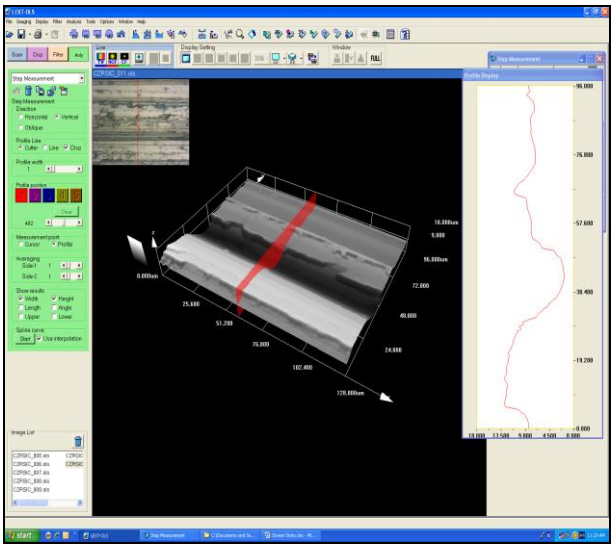
Fig 55. White Metal Bearing alloy - Surface profile 50x & 100x objectives - LSCM



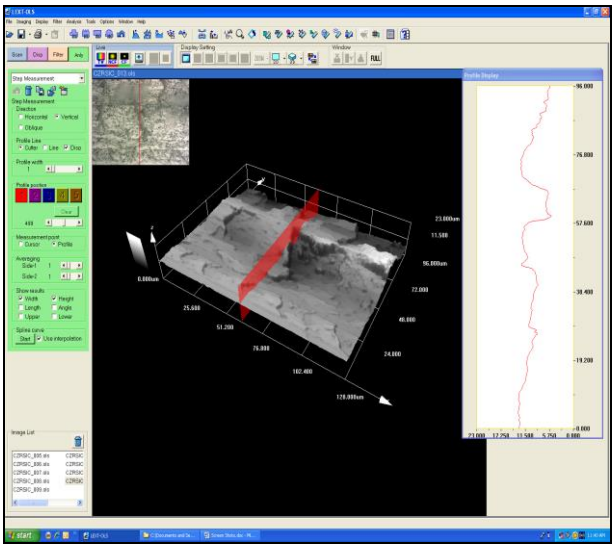
WMB SiC P180 grit 50x objective



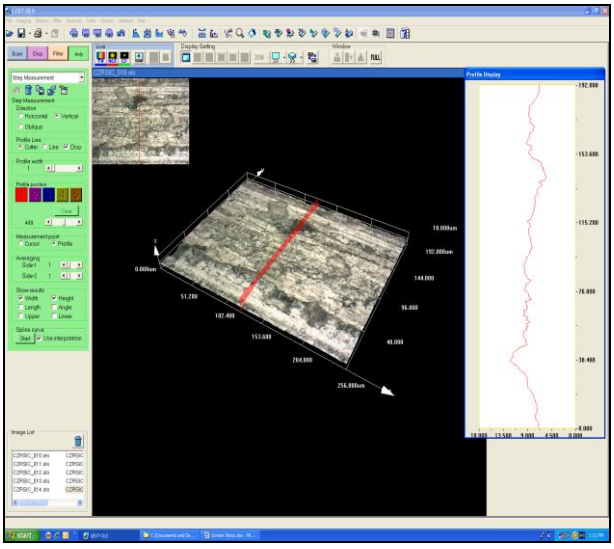
WMB SiC P180 grit 100x objective



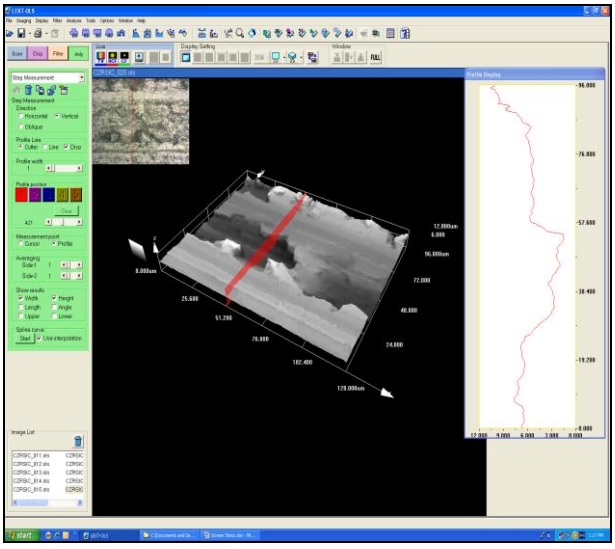
WMB Zr 80g 50x objective



WMB Zr 80g 100x objective

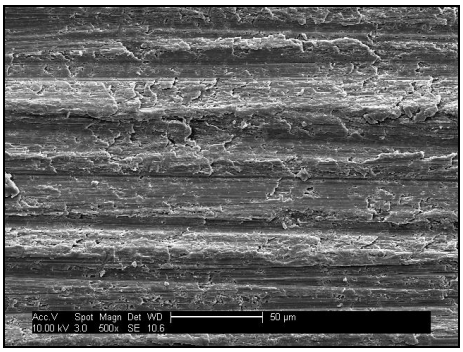


WMB Cameo Blue 50x objective

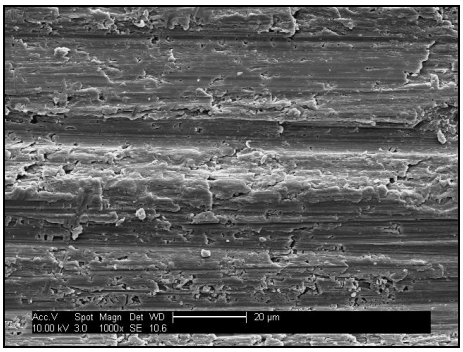


WMB Cameo Blue 100x objective

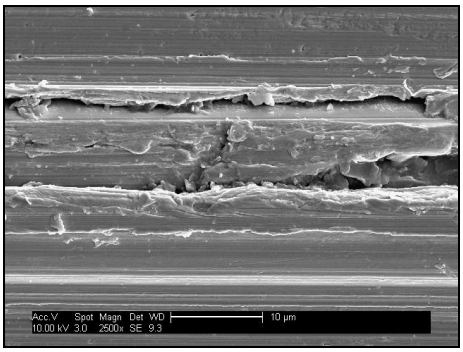
Fig 56. Scratch Patterns on White Metal Bearing sample using various grinding surfaces revealed by the SEM



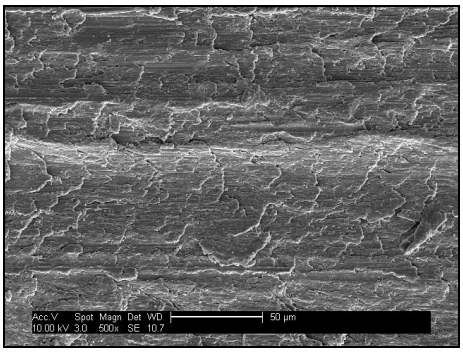
Silicon Carbide P120g - 500x



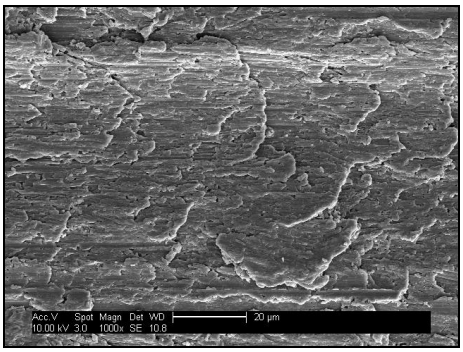
Silicon Carbide P120g - 1000x



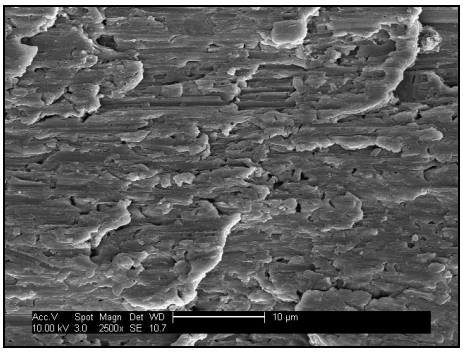
Silicon Carbide P120g - 2500x



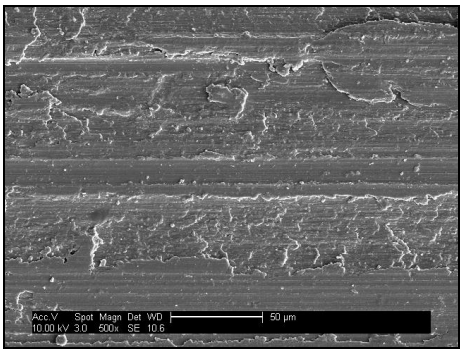
Zirconia P120g - 500x



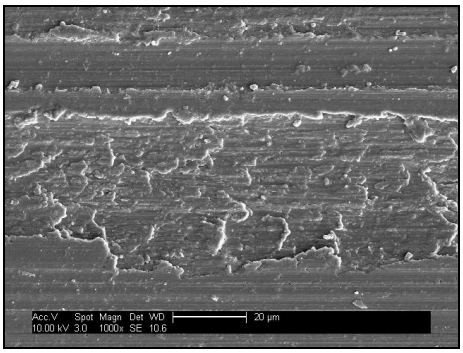
Zirconia P120g - 1000x



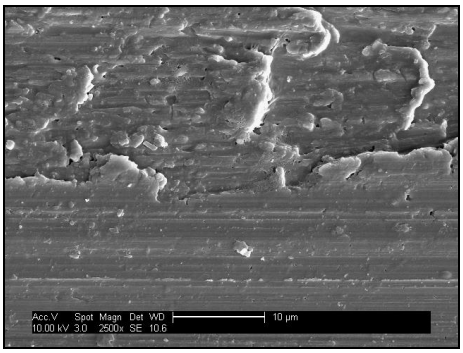
Zirconia P120g - 2500x



Cameo P120g - 150g - 500x

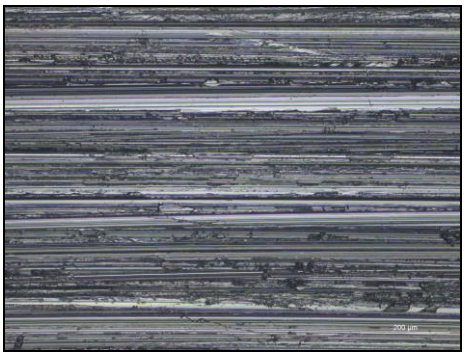


Cameo P120g - 150g - 1000x

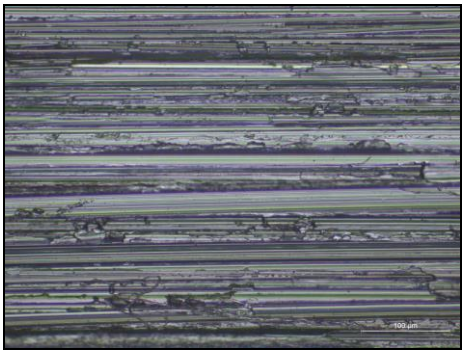


Cameo P120g - 150g - 2500x

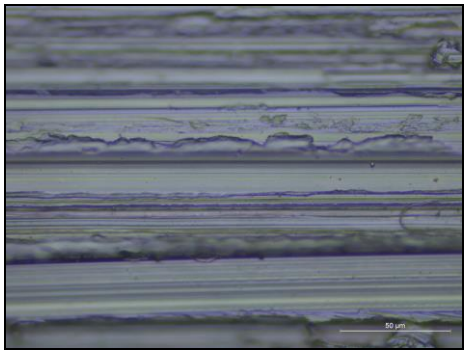
Fig 57. Scratch Patterns on Copper using various P120g surfaces revealed by the Metallurgical microscope



Silicon Carbide 10x Objective

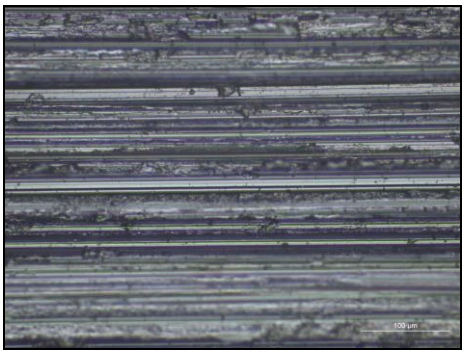


Silicon Carbide 20x Objective

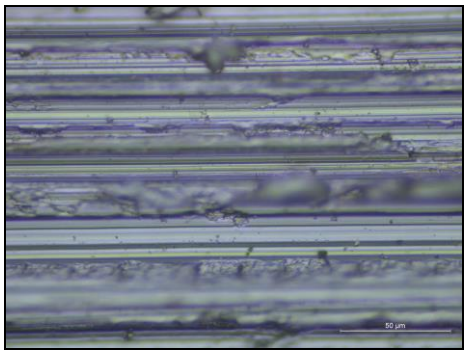


Silicon Carbide 50x Objective

Zirconia 10x Objective

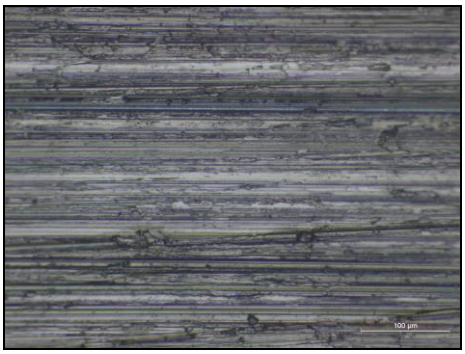


Zirconia 20x Objective

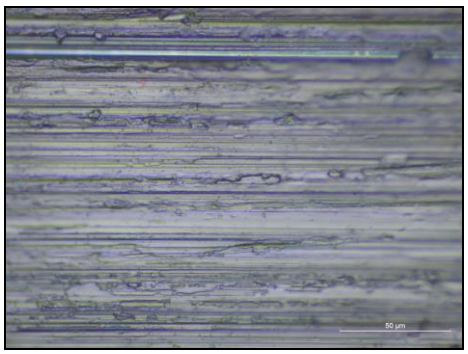


Zirconia 50x Objective

Cameo 10x Objective

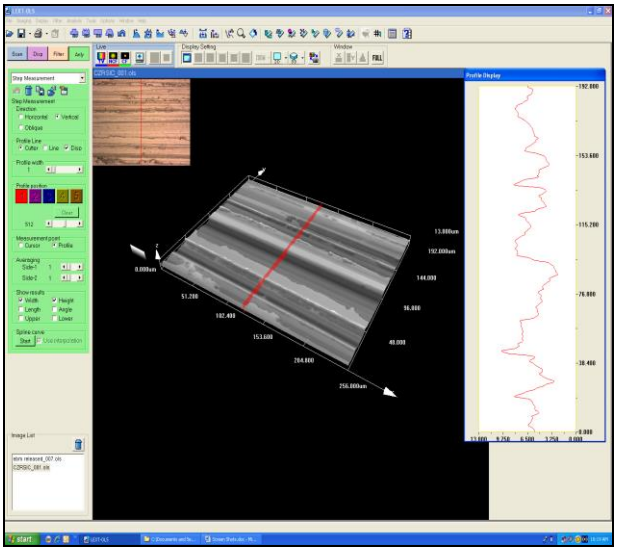


Cameo 20x Objective

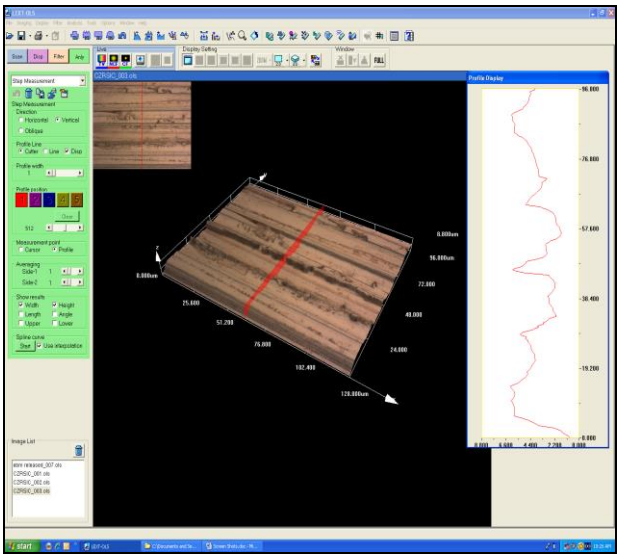


Cameo 50x Objective

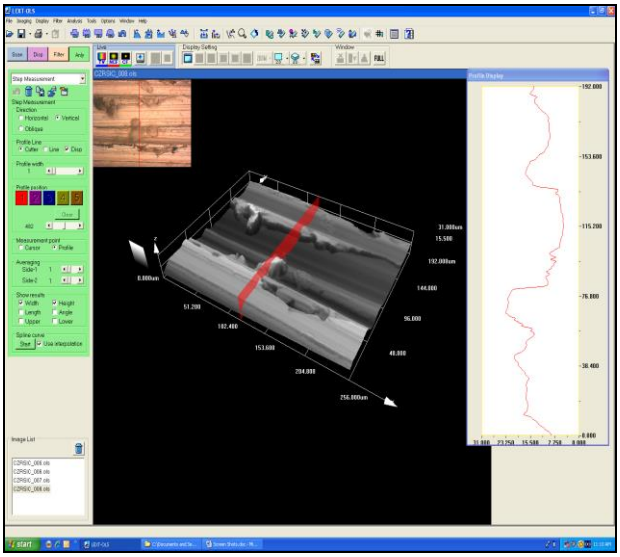
Fig 58. Copper alloy - Surface profile 50x & 100x objectives LSCM



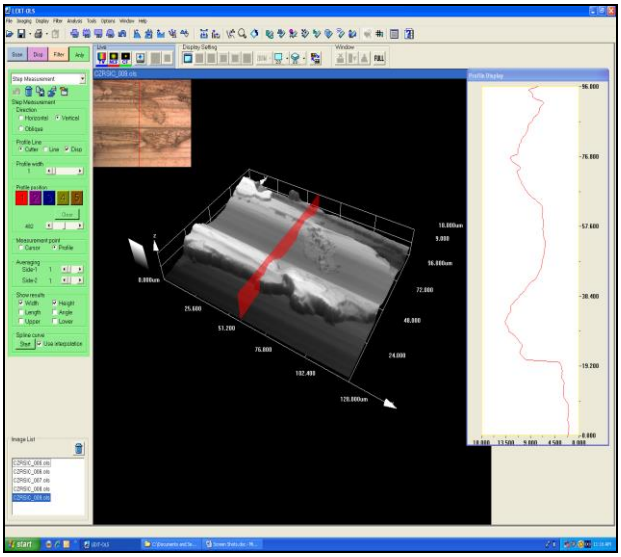
Copper SiC P180 grit 50x objective



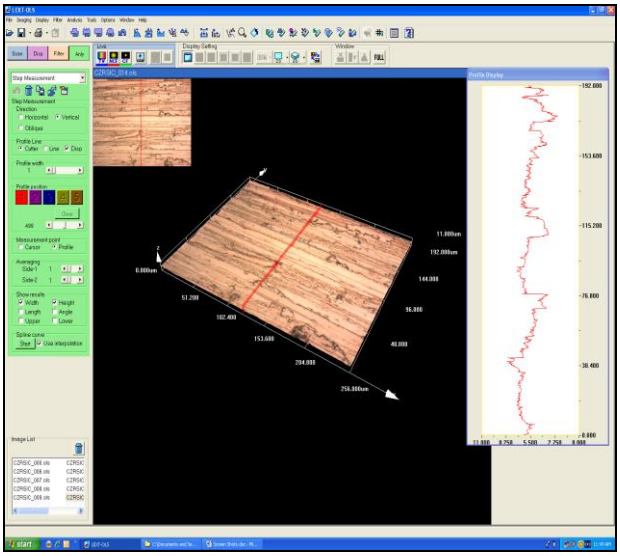
Copper SiC P180 grit 100x objective



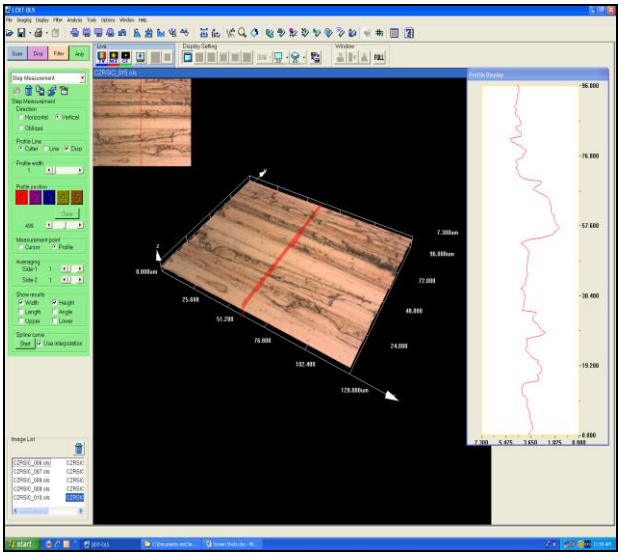
Copper Zr 80g 50x objective



Copper Zr 80g 100x objective



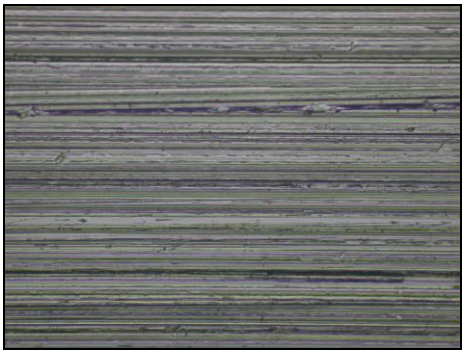
Copper Cameo Blue 50x objective



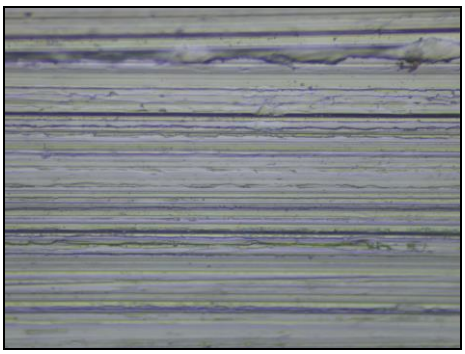
Copper Cameo Blue 100x objective

Fig 60. Scratch Patterns on Steel using various P120g surfaces revealed by the Metallurgical microscope

Silicon Carbide 10x Objective

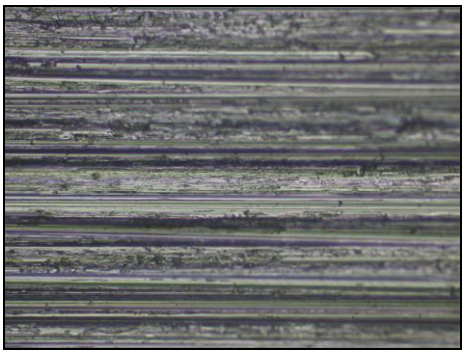


Silicon Carbide 20x Objective

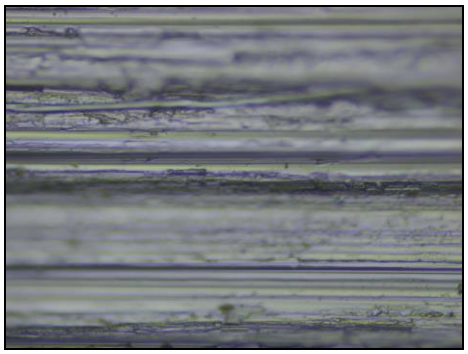


Silicon Carbide 50x Objective

Zirconia 10x Objective

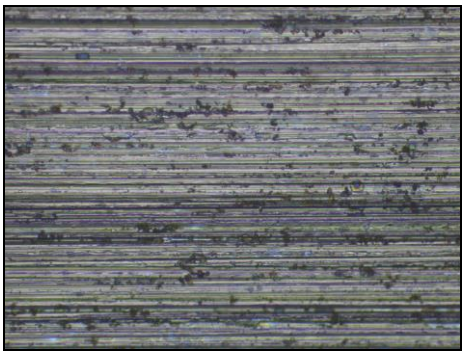


Zirconia 20x Objective

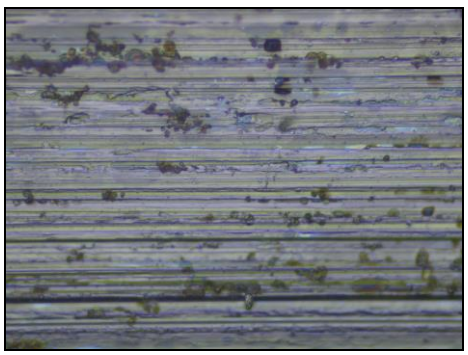


Zirconia 50x Objective

Cameo 10x Objective

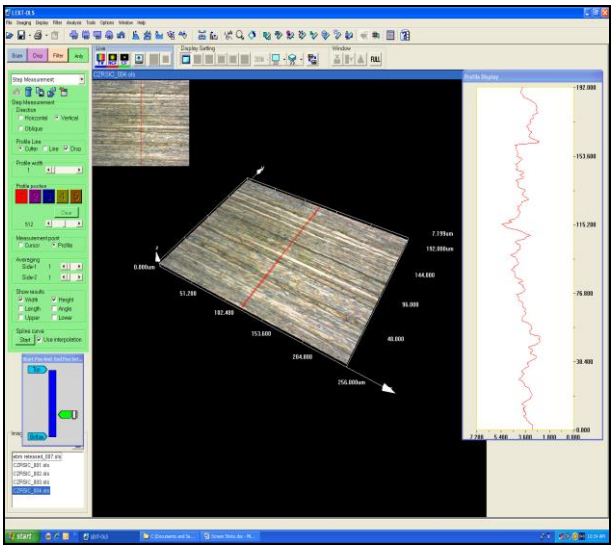


Cameo 20x Objective

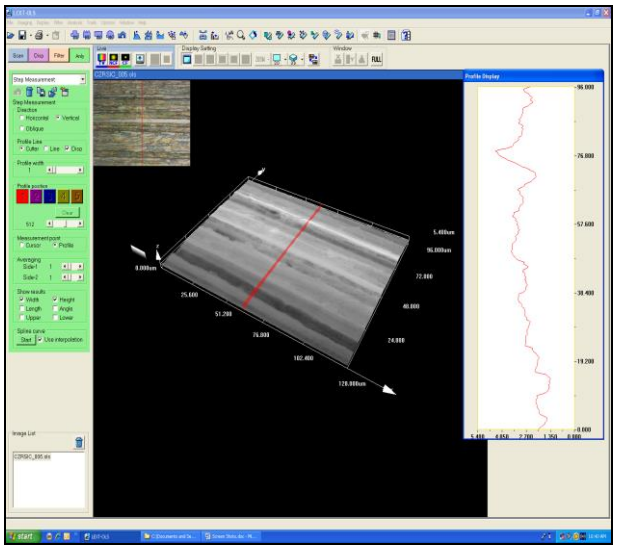


Cameo 50x Objective

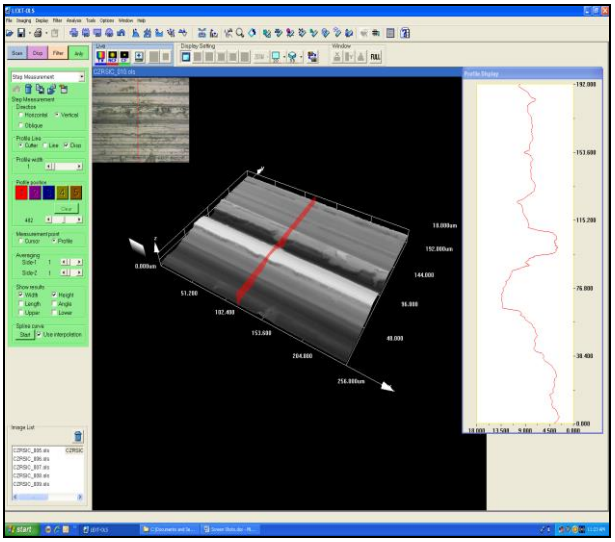
Fig 61. Steel alloy - Surface profile 50x & 100x objectives LSCM



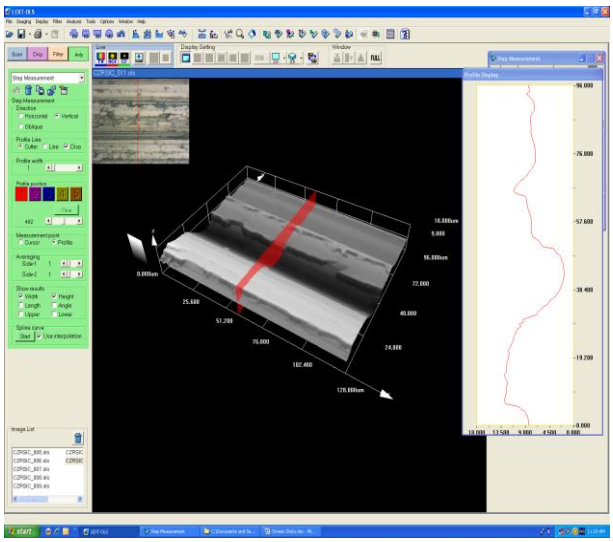
Steel SiC P180 grit 50x objective



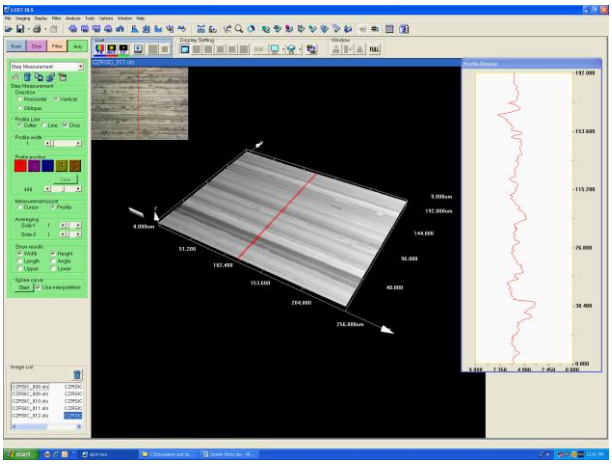
Steel SiC P180 grit 100x objective



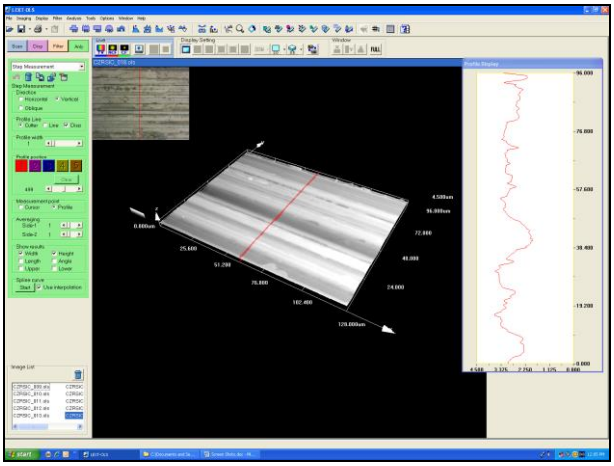
Steel Zr 80g 50x objective



Steel Zr 80g 100x objective

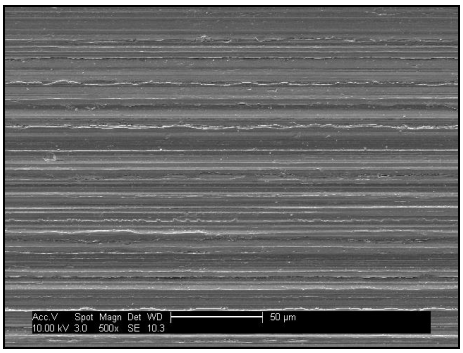


Steel Cameo Blue 50x objective

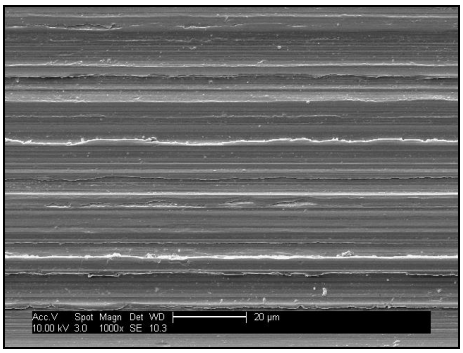


Steel Cameo Blue 100x objective

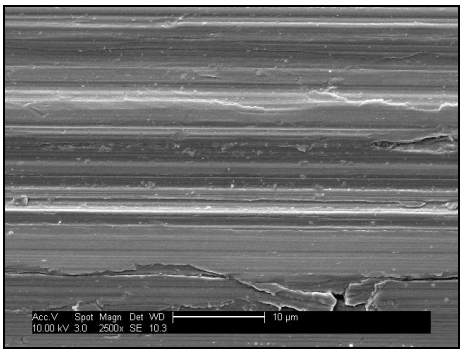
Fig 62. Scratch Patterns on Steel sample using various grinding surfaces revealed by the SEM



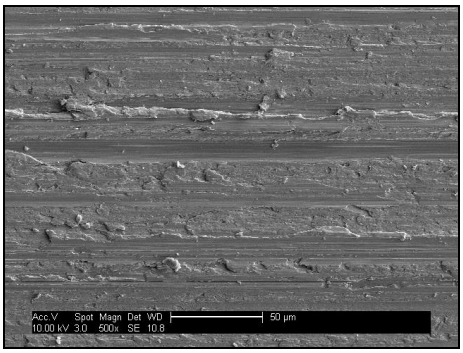
Silicon Carbide P120g - 500x



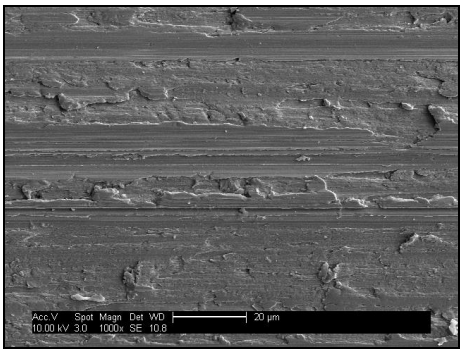
Silicon Carbide P120g - 1000x



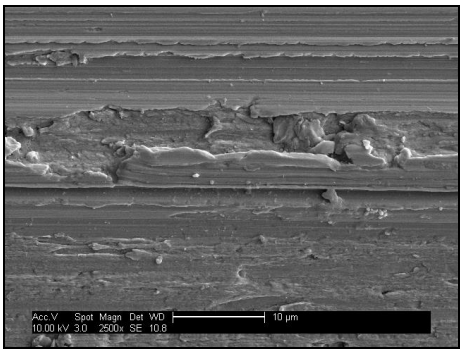
Silicon Carbide P120g - 2500x



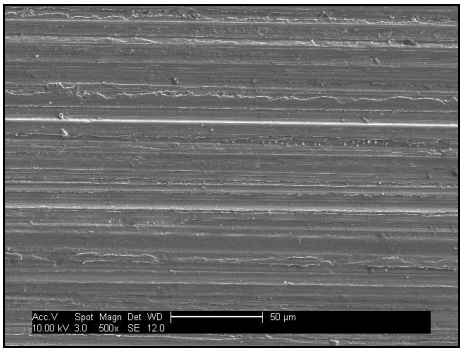
Zirconia P120g - 500x



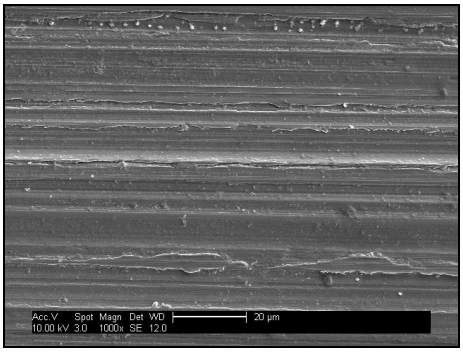
Zirconia P120g - 1000x



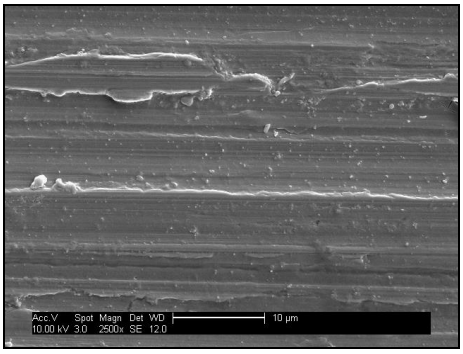
Zirconia P120g - 2500x



Cameo P120g - 150g - 500x

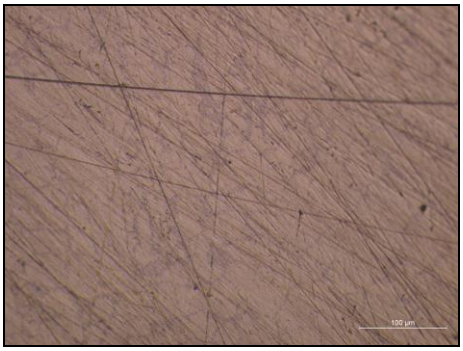


Cameo P120g - 150g - 1000x

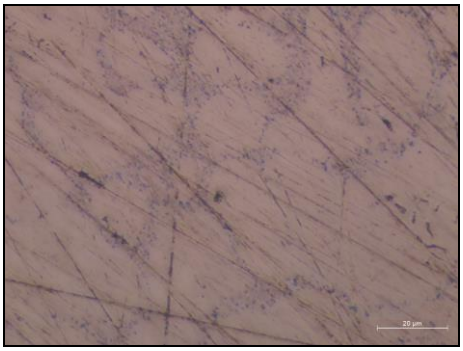


Cameo P120g - 150g - 2500x

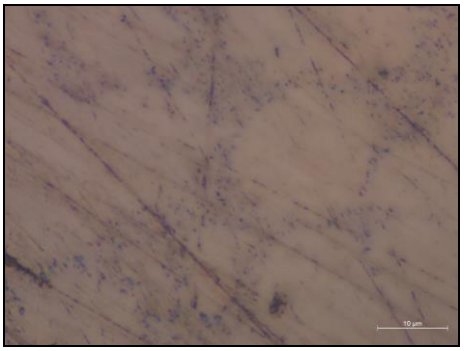
Fig 63. Scratch Patterns on various Materials using Planocloth H & 9um Diamond - Metallurgical microscope



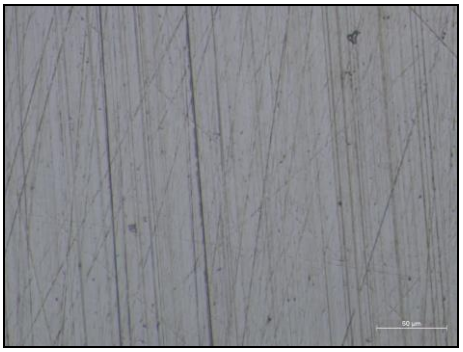
Copper 20x Objective



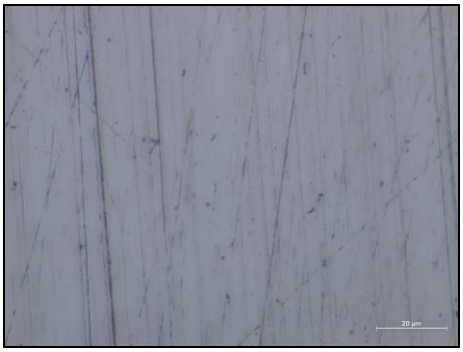
Copper 50x Objective



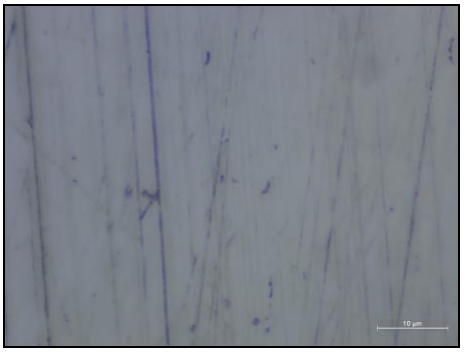
Copper 100x Objective



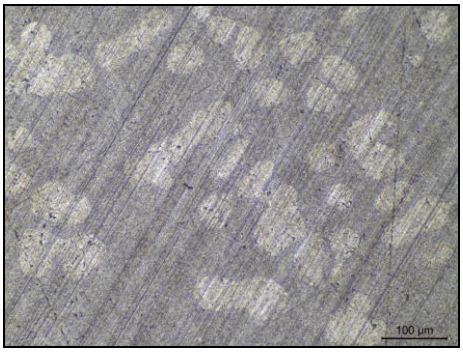
Steel 20x Objective



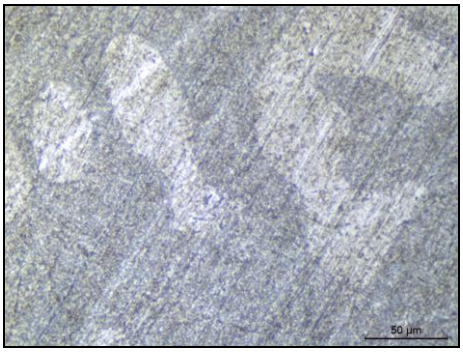
Steel 50x Objective



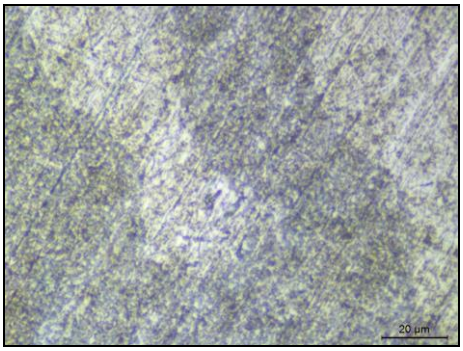
Steel 100x Objective



WMB 20x Objective



WMB 50x Objective



WMB 100x Objective

Overview and examples of creating a preparation procedure

Understanding your sample properties and the properties of your consumables enables creating a preparation procedure from scratch more straightforward. For example, let us consider the preparation procedure for Spheroidal graphite cast iron or SG iron.

SG iron is a medium hardness metallic material consisting of an iron - usually ferritic / pearlitic matrix - with individual nodules of graphite present. On cutting cut it behaves in a ductile manner though it is necessary to consider the soft brittle nature of the graphite.

Considering first a primary grinding stage. SG iron can be easily ground by silicon carbide, zirconia and fixed diamond. From the earlier microscopical examination it is known that silicon carbide is the sharpest abrasive and is also quite economical. Diamond is hard but relatively blunt as is zirconia. In the event of a lot of material is required to be removed from the surface, then the latter grinding options have a place but with a correctly sectioned sample silicon carbide will provide an excellent cutting action through both the matrix and the graphite nodules causing as little damage as possible and it is also economical. An effective sized abrasive size will be needed to remove any cutting damage and excess moulding material but care is required to minimise damage. There is no point in cutting carefully to reduce damage to then put in more damage in the primary grinding stage. With this in mind a P240g (58 um) size would be an ideal compromise.

Being a sharp abrasive Silicon Carbide will have more chance of cutting both the metallic & graphite material equally and at a similar rate. Following this stage, the sample can be examined under a metallurgical microscope and when confirmed the sample surface is as good as it can be at this stage it can be considered for the next stage

Regarding the Secondary grinding stage. There is here an opportunity to use one of two grinding cloths identified both in the earlier microscopical examination and also from available supplier information. One is the coarse cross woven polyester - Abracloth and the more forgiving and less aggressive - Planocloth H. Both cloths are hard wearing and ideal for this secondary grinding stage. With the SG iron containing graphite nodules and graphite being quite brittle it is good practice to err on the side of caution and use the less aggressive cloth. Stepping down from a P240g (58 um) silicon carbide stage to a 9um diamond abrasive will require some time to remove the damage from the previous stage. If the preparation is for just one or two samples then an initial time of 3 minutes preparation is a good start. Follow this with a microscopical examination to determine what damage remains. Taking a series of photomicrographs at this point will allow comparison to any further steps. Having assessed the surface microscopically after 3 minutes at this stage the samples should be should further prepared at this stage for a couple more minutes and again examined accordingly. When microscopical examination confirms that further work is not improving the surface only then a tertiary grinding stage can be considered.

For the tertiary stage a choice of three cloths previously examined are available. Planocloth, Nylap, Durasilk. All these cloths are suitable for the tertiary stage but the chemotextile nature of Planocloth as viewed microscopically could cause rubbing and the fine woven Durasilk is designed more for softer materials according to the supplier notes. The compromise cloth would be the Nylap. From the microscopical observation already conducted it is known that the Nylap is a fine cross woven cloth and should remove material without leaving excessive damage in the more brittle nodules. It will also have less chance of rubbing like the smoother chemotextile Planocloth option. Again, the process of preparing for a short time followed by microscopical

examination, recording of micrographs and returning to the preparation surface for a short time. Completion is determined by microscopical examination confirming the surface is damage free. If the surface isn't free of damage a Quaternary stage would be needed where the aggressive nature of the cloth and the abrasive is reduced even further.

Assuming a surface free of structural damage has been produced it will no doubt still have some scratches. It is now acceptable to consider using a polishing cloth if a scratch finish is required.

When it comes to a final polish with dissimilar materials in the sample it is best to consider a cloth with a short nap or no nap and a fine abrasive. From our microscopical evaluation we can see that Multicloth and Memphis are low napped in nature and will leave less relief when compared to the longer napped Alphacloth (*fig 38*). In addition, the Chemicloth with Colloidal Silica is an option. Keeping the relief to a minimum and wanting to get the finest scratch free finish the latter combination would be best. There will be some chemical attack by the Colloidal Silica on the surface due to the pH but this usually isn't a problem. The result should be a scratch free surface, free of damage and showing the true microstructure. Evidence showing the optical microstructure through a metallurgical microscope for this preparation including the various stages discussed is illustrated in (*fig64*)

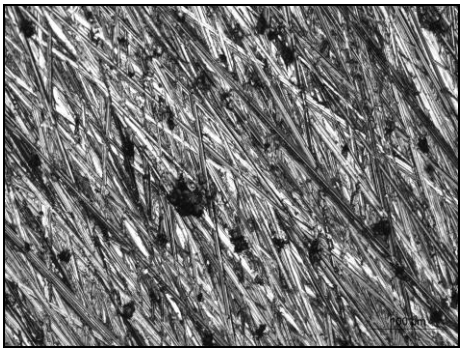
Whilst the structure produced would be acceptable in most laboratories above there is still some very minimal damage left in some of the graphite nodules.

In the final polished condition, the soft napped cloth with colloidal Silica has removed the scratches but not this last minimal damage in the graphite. As has already been highlighted - final polishing cloths are designed for removing scratches not damage. If the same abrasive – Colloidal silica is used but instead used on a 'hard' cloth such as the Planocloth - a chemotextile cloth with no nap, then the colloidal Silica will operate in a grinding mode. This fine grinding is capable of removing the final damage in the graphite. Whilst the chemotextile Planocloth can cause problems by rubbing with a diamond suspension, the more viscous nature of the colloidal silica solution means this does not occur.

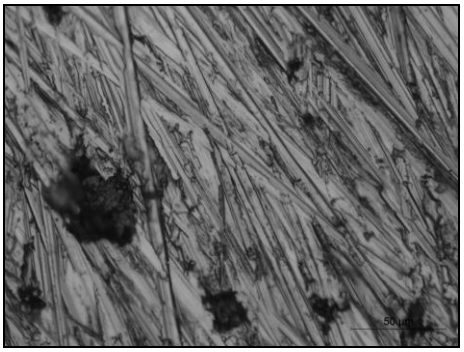
Comparison between the 'final as polished' stage using a napped cloth – Multicloth and the 'quaternary grinding' stage with a 'hard' cloth – Planocloth shows that the fine grinding quaternary stage results in less damage present in the graphite nodules and additionally reflectivity.

Consequently, having less damage in the graphite nodules allows the use of alternative contrast techniques to Brightfield illumination. As graphite is a birefringent material it is classed as optically active and will respond well to polarised light illumination (*Fig65*). This technique is only possible when the surface damage is completely removed. This is yet another way of using the metallurgical microscope to investigate the quality of the preparation.

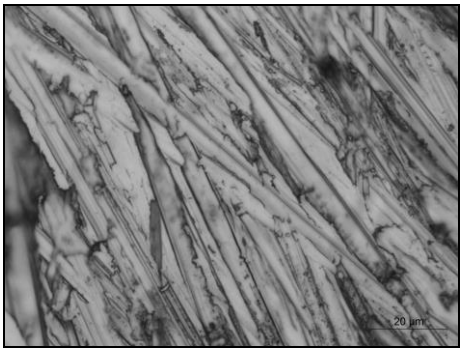
Fig 64. Preparation of Spheroidal Cast Iron - Examination during preparation - Metallurgical microscope



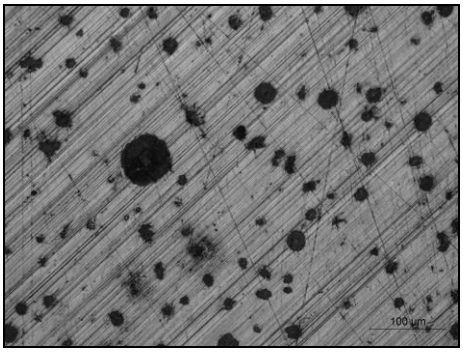
P240g - 20x Objective



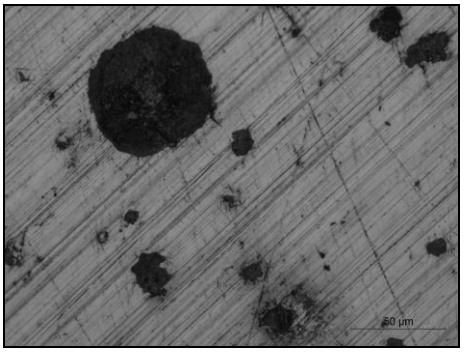
P240g - 50x Objective



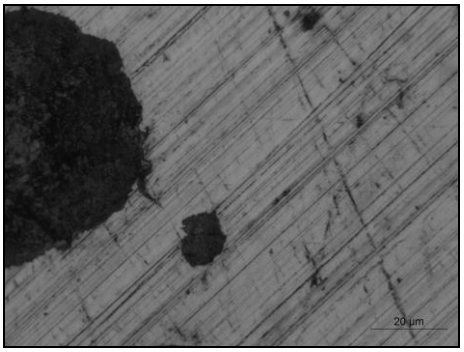
P240g - 100x Objective



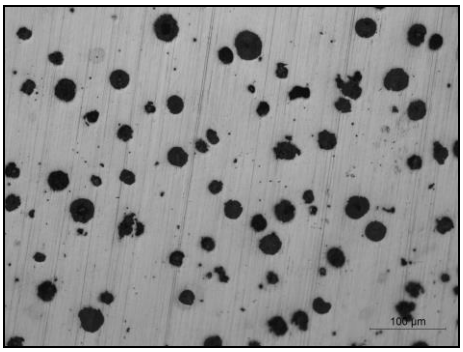
Planocloth H - 20x Objective



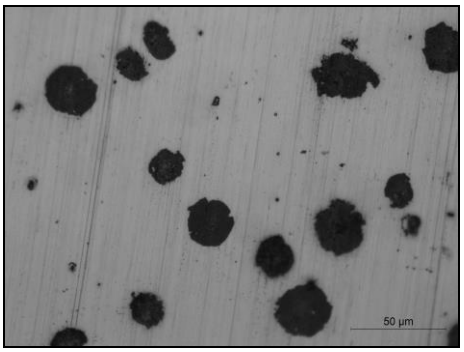
Planocloth H - 50x Objective



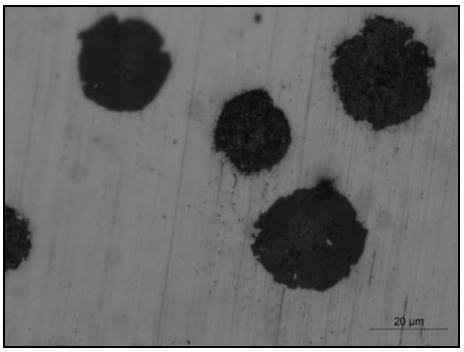
Planocloth H - 100x Objective



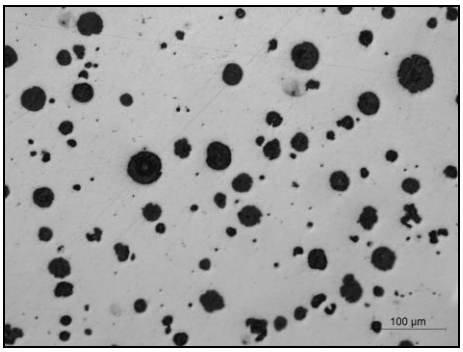
Nylap 20x Objective



Nylap 50x Objective

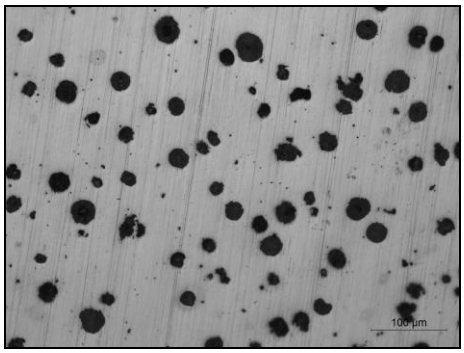


Nylap 100x Objective

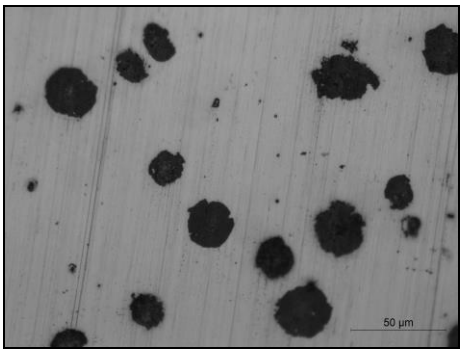


Multicloth 20x Objective

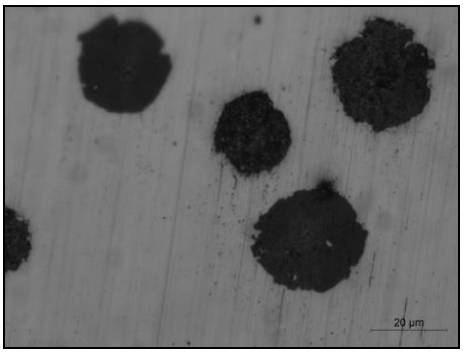
Fig 65. Preparation of Spheroidal Cast Iron - Metallurgical microscope & Polarised Light



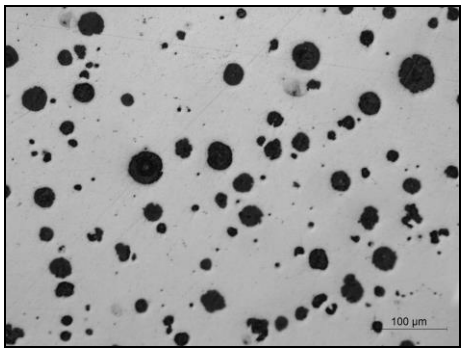
Nylap 20x Objective



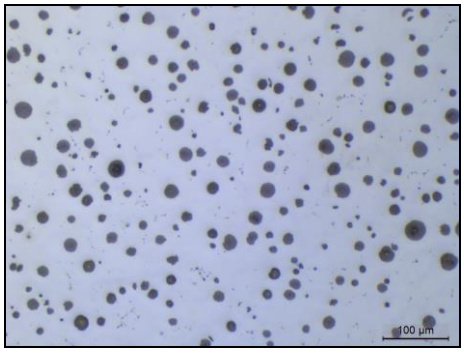
Nylap 50x Objective



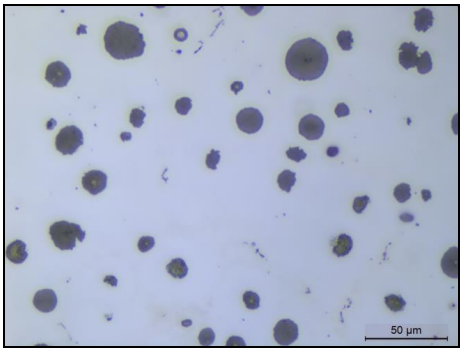
Nylap 100x Objective



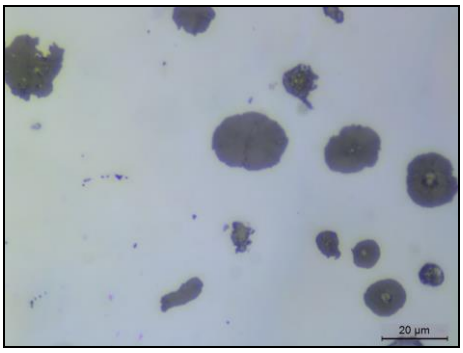
Multicloth 20x Objective



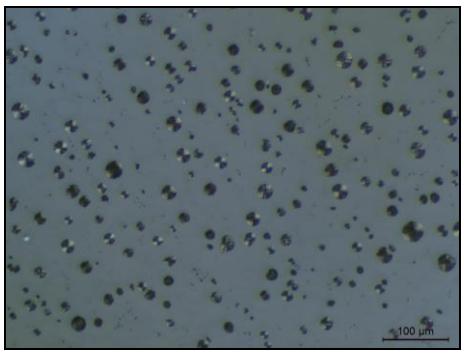
Planocloth 20x Objective
Brightfield only



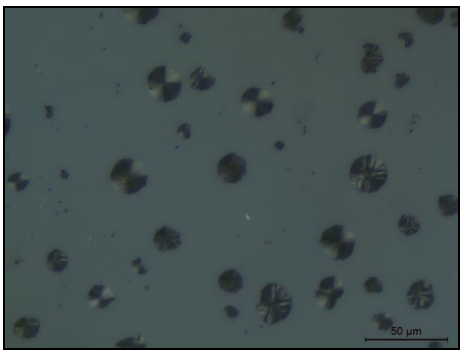
Planocloth 50x Objective
Brightfield only



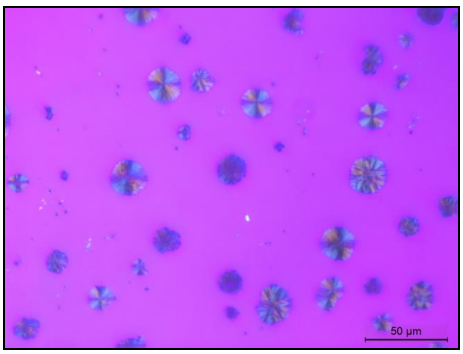
Planocloth 100x Objective
Brightfield only



Planocloth 20x Objective
Brightfield - Polarised



Planocloth 50x Objective
Brightfield - Polarised



Planocloth 50x Objective
Brightfield - Polarised + Wavepla

Considering another material, a carbon fibre composite CFC. The approach to the preparation is the same way as the cast iron preparation procedure.

The PEEK Carbon fibre composite chosen is a combination of soft brittle carbon fibres in a soft gummy polymer matrix. It is again necessary to consider both characteristics in the preparation procedure.

Firstly choosing a primary grinding stage. The PEEK carbon fibre composites can be comfortably cut by Silicon Carbide, Zirconia and a fixed diamond grinding disc. It is known from the earlier microscopical examination that the silicon carbide is the sharpest abrasive and is also quite economical. Diamond is hard but relatively blunt as is Zirconia and the latter will tend to leave greater damage. The last thing required is to create excessive damage at the start of the preparation. Silicon Carbide being sharp will cut through both the soft & brittle carbon fibres and the gummy matrix. The clean cut will minimise damage to both constituents. If a significant amount of material had been required to be removed then a fine grained fixed diamond Cameo disc could be employed.

As with the cast iron a reasonable sized abrasive size will be required to remove any cutting damage and excess moulding material whilst also limiting additional damage to the sample. There is no point in cutting carefully to reduce damage to then put in more damage in the primary grinding stage. With this in mind a P320g (46um) size abrasive would be a sensible compromise between stock removal and residual damage.

Choosing a secondary grinding stage is conducted in the same manner as before. Again, there is an opportunity to use one of two grinding cloths we are familiar with following our microscopical examination and supplier information. The coarse woven polyester - Abracloth and the more forgiving and less aggressive - Planocloth H. Both cloths are hard wearing and ideal for this stage. With the carbon fibre composite having the brittle carbon fibres again it would make sense to err on the side of caution and use the less aggressive cloth. Stepping down from a P320g (46 um) stage to a 9um abrasive stage it will again take some time to remove the material. As before if the preparation is for just one or two samples then an initial time of 3 minutes preparation is a good start. Again, it should be followed by a microscopical examination and determine what damage remains and what can be seen microscopically. Taking a series of micrographs at these stages allows later comparison to any additional steps. Having assessed the surface microscopically after 3 minutes at this stage the samples should be should further prepared and examined accordingly. One or two minutes would be typical. When microscopical examination confirms that further work is not improving the surface at this stage a tertiary grinding stage can be considered.

For a Tertiary stage a choice of three cloths that have been examined and are available. Planocloth, Nylap, Durasilk. All these cloths are suitable for the tertiary stage but the chemotextile nature of Planocloth as seen microscopically might cause rubbing and the Durasilk is designed more for softer materials. The compromise would be the Nylap typically with an abrasive in the region of 3um. We know from the microscopical observation & the earlier cast iron preparation that the Nylap is a fine cross woven cloth and should remove material without leaving excessive damage in the more brittle parts of the material. Again, the process of preparing for a short time followed by microscopical examination, recording of micrographs and returning to the surface for a short time whilst checking progress microscopically is necessary.

Unfortunately, examination after the 3um - Nylap stage (*fig 66*) reveals that the preparation is not going well. There is considerable relief caused by the differential abrasion rates between the

matrix and the fibres. The fibres are ill defined particularly at their ends and the matrix is riddled with damage.

It has been previously determined microscopically that whilst diamond is hard it is not very sharp and it appears that the sample is not being ground efficiently with this abrasive / surface combination. What is required is a sharper abrasive to cut both the gummy matrix and the carbon fibres equally and more efficiently. At the higher grinding stages, it would have been possible to use silicon carbide paper but down at this stage of preparation that is not an option.

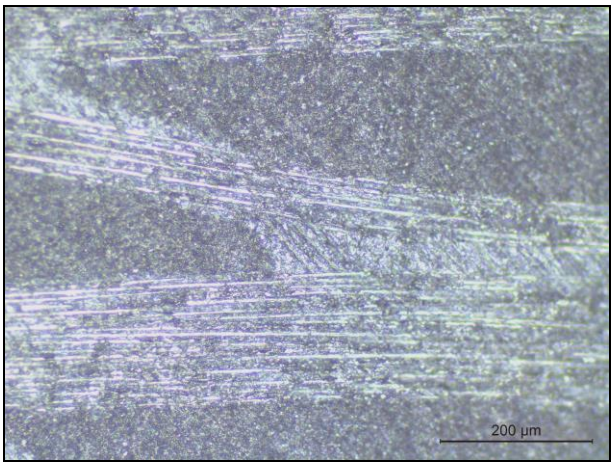
A useful alternative abrasive in such conditions is Aluminium Oxide or Alumina, as it is often known. Whilst not as hard as diamond it is very sharp and will slowly reduce in size as well. Two alternatives are available, the gamma Alumina usually at a size of 0.05um and the alpha Alumina at sizes of 0.3um, 1um & 5um. The hardest form is the alpha at 9 on the Mohs scale - (Diamond is 10) and the gamma version of alumina is 8.

This abrasive is often referred to as polishing alumina and whilst this would be correct if it was being used on a soft napped cloth, when it is used on a hard cloth it can be used as a grinding abrasive. Typically, at this stage a 0.3um sized abrasive size is used and with a hard tertiary grinding cloth such as the Planocloth it is possible to remove the damage from the 9um Planocloth stage and leave the sample flat and damage free (*fig 66*).

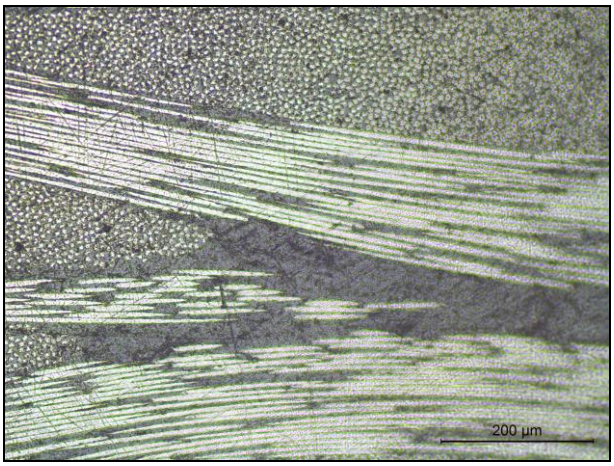
The resulting surface finish after the 0.3um Alumina grinding stage is now damage and scratch free and a final polish is not required. Technically a polishing stage would tend to degrade the sample surface by introducing relief between the fibres and the matrix.

It is possible to argue here that with the 0.3um Alumina - Planocloth stage in the CFC procedure and also with re the colloidal silica – Planocloth stage in the SG iron procedure whether these stages are grinding or polishing with regard to the abrasive being as fixed when in contact with the sample. The important detail however is what is trying to be accomplished, damage removal or scratch removal. In many instances an abrasive is referred to as a polishing or grinding abrasives but they are really just abrasives. When used with a soft napped cloth they can act in a polishing manner but on a hard cloth they can behave in a grinding mode to remove damage. Sticking to this fundamental principle when developing a preparation procedure will ensure success with numerous materials.

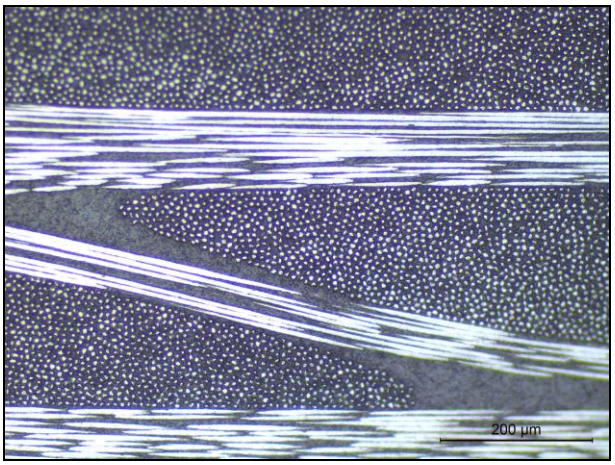
Fig 66. Preparation of a Carbon Fibre Composite - Examination during preparation - Metallurgical microscope



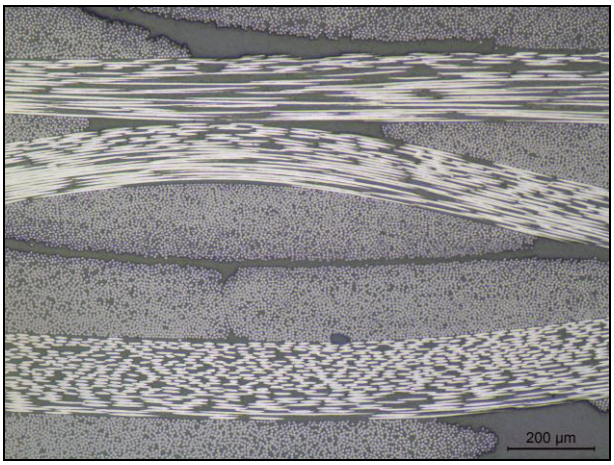
P320g - Silicon Carbide 10x Objective



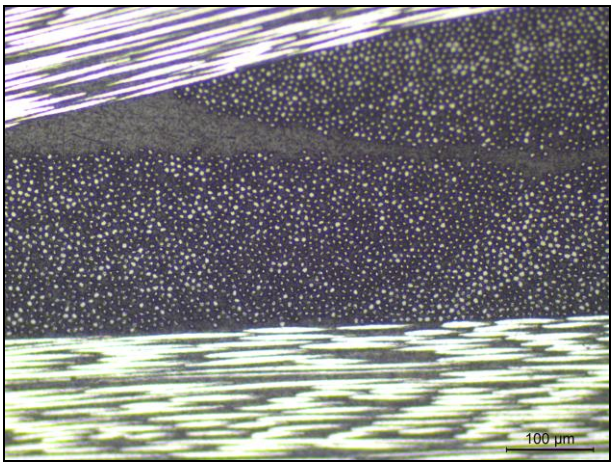
Planocloth - H 9um Diamond 10x Objective



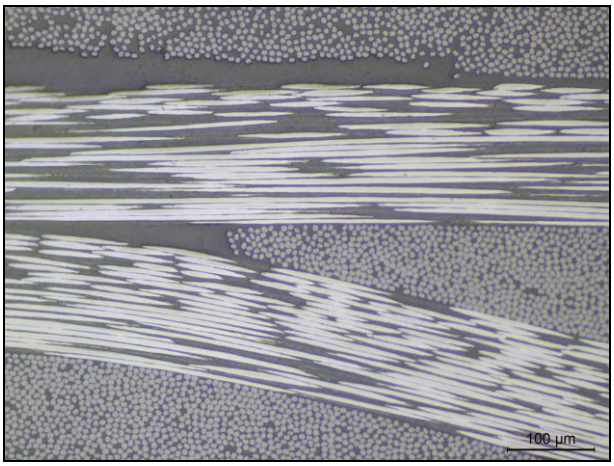
Nylap – 3um Diamond 10x Objective



Planocloth 0.3um Alumina 10x Objective



Nylap – 3um Diamond 20x Objective



Planocloth 0.3um Alumina 20x Objective

When preparing materials such as ceramics a fine abrasive is often used on a ‘hard’ cloth for a quaternary grinding stage to remove the final fine levels of damage. This damage usually appears as fine porosity. As the material being prepared is so hard a scratch free finish is often obtained at this stage. In cases such as this there is no need for a final polishing stage.

It isn’t possible to go through numerous different material procedures within the scope of this work but a series of typical preparation procedures generated by the technique described here have been added as an addendum. These can be used reference procedures complete with photomicrographs of the associated structures generated. The creation of these procedures was totally dependent on examining the surfaces microscopically at each preparation stage. Without this microscopical examination it would have been impossible to generate these procedures.

There can even be many preparation procedures for preparing a single material and as long as they follow the correct principles, then they are all valid procedures. It would have been possible for both the Spheroidal graphite cast iron & the PEEK carbon fibre composite materials to have manually progressed through a range of ever decreasing sized Silicon carbide paper surfaces and a couple of cloth stages as has been used traditionally for many years. When a Metallographer is just moving down a row of manual grinder polishers with a single sample then this might be quite sensible but when preparing several samples this requires lots of short stages and operator interactions as well also regular cleaning of the samples between stages. Not an ideal option when using a semi automatic machine.

Surface comparisons

There are numerous surfaces and abrasives supplied by many different companies. All these companies offer products with different degrees of both technical support and marketing material. By the use of the various microscopical techniques employed in this project it is possible to see behind the marketing information and assess the metallographic consumables in a new and clear technical light. This gives the materials scientist the opportunity to evaluate various suppliers’ goods, assess similar products and compare both appearance and performance like for like. It can also highlight whether they are paying more for the same goods just because of the name on the product.

As an example, comparing the popular ‘Chemi’ type cloths (*fig48*) indicated an almost identical structure from different suppliers. The reason they are so similar is that they need to fulfil the same task.

Microscopical examination cannot fully guarantee that the performance of an item will be the same. It does however give the materials scientist a great indicator as to the behaviour of the surface and also an indication of how much like an alternative surface it is.

Artefacts

Earlier it was demonstrated that it is possible to follow a structured preparation procedure using microscopy to observe the progressive removal of damage. Equally important is known that all our actions will impart damage to the material being prepared. This damaged can create artefacts that could lead to erroneous assessment of a prepared sample. Using the metallurgical microscope to examine between stages minimises this but it is important to be vigilant in looking

out for such artefacts. Artefacts can include, impressed abrasives, damage from earlier preparation stages (*fig67*), phase pull out and even chemical attack (*fig68*). Even initial sectioning operations can create serious damage at the start and this damage can be retained in the structure throughout the preparation procedure (*fig69*).

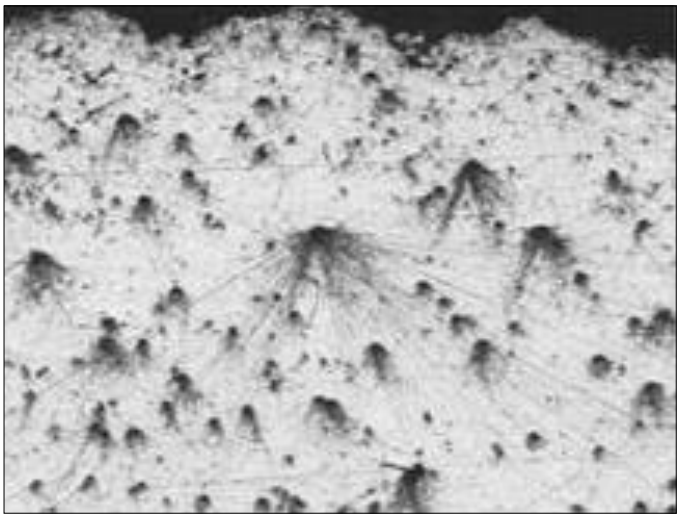
In the event that the produced a sample has unknown impressed abrasives lodged in the material, it is possible to use microscopical techniques to examine the sample and determine how to proceed.

Considering a wrought Titanium alloy, an alloy renowned for its susceptibility to impressed abrasives. Examination of the sample using a Metallurgical microscope in both Brightfield & Darkfield illumination shows the morphology to be more in keeping with Diamond abrasive than Silicon Carbide (*fig70*). Examination with the LEXT LSCM also indicates the morphology to be more Diamond like (*fig71*). It also provides the opportunity to measure the protrusion from the surface. In this instance it appears the abrasives are protruding out of the surface by a couple of micrometers (*fig72*). The use of the SEM increases the resolution (*fig73*) and as well as showing the morphology of the impressed abrasives using the option of the Back Scattered Electron technique. This highlights the dissimilar nature of the abrasive and the matrix due to their different atomic numbers (*fig74*)

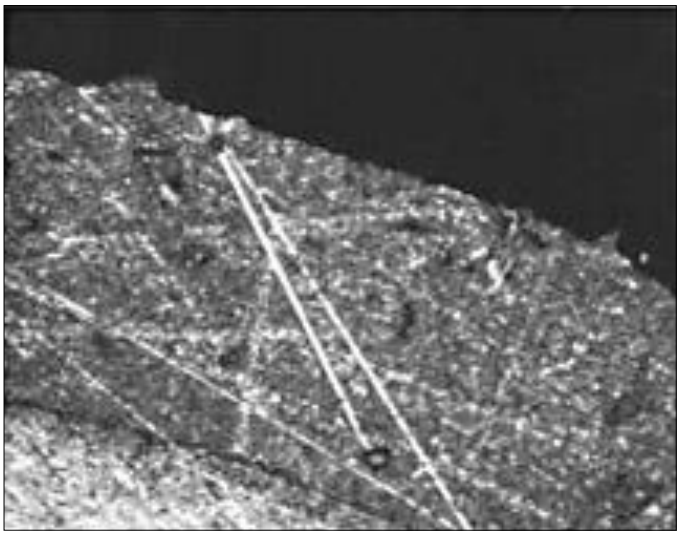
Additional to being able to separate elements by atomic number many SEMs have other analysis capabilities. This can be a real benefit when one is trying to identify impressed abrasives. Visual examination using the techniques above allows the abrasives to be identified by their morphology as Diamond but the option to use X-Ray microanalysis means we can confirm this. Producing X-Ray maps for the elements Titanium, Carbon and Silicon reveals that there is carbon present in the particles but no trace of Silicon (*fig75*). This eliminates Silicon Carbide and proves it is Diamond particles that have become impressed in the Titanium matrix.

If diamond is found to be the offending impressed abrasive then it is possible to cut these out with a sharp alumina abrasive. If silicon carbide is impressed into the sample it is usually best to cut the sample further back and start again. The sharp pointed nature of silicon carbide means it tends to just push further in.

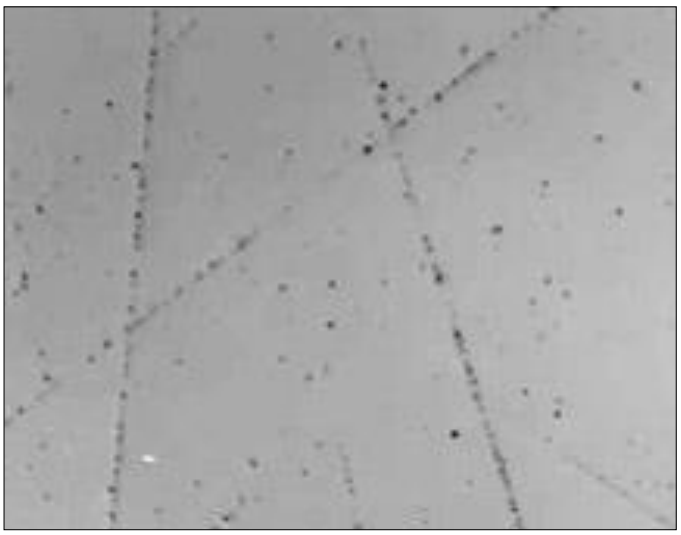
Fig 67. Preparation Artefacts - Impressed abrasives - Primary stage scratches



Impressed abrasives Diamond in Aluminium
Metallurgical microscope 20x objective – Brightfield

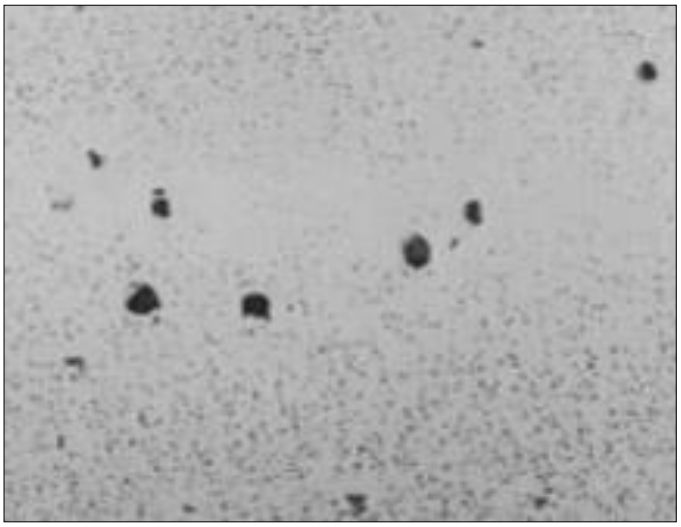


Impressed abrasives Diamond in Aluminium coating
Metallurgical microscope 50x objective – Brightfield

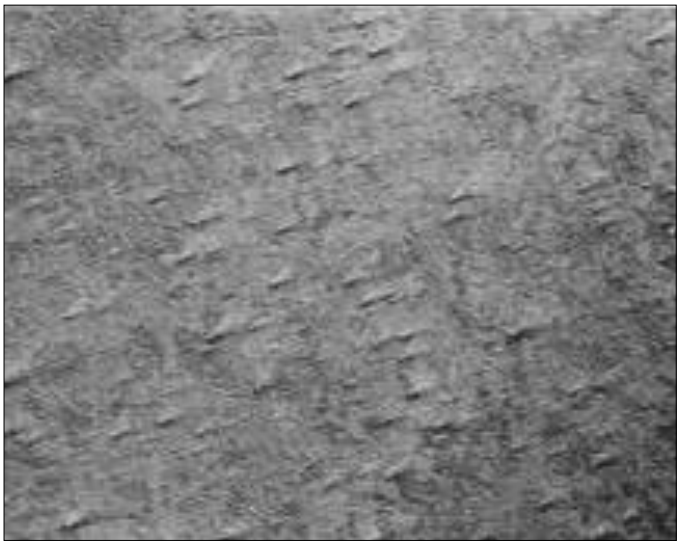


Scratches in steel left from Primary grinding stage.
Metallurgical microscope 50x objective – Brightfield

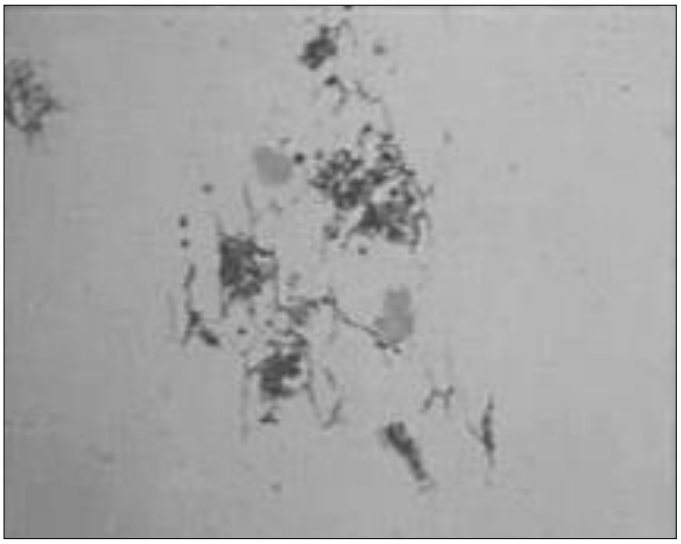
Fig 68. Preparation Artefacts – Phase pull out – Smearing – Chemical attack



Phase pull out – Silver 10% Cadmium wire
Metallurgical microscope 50x objective – Brightfield

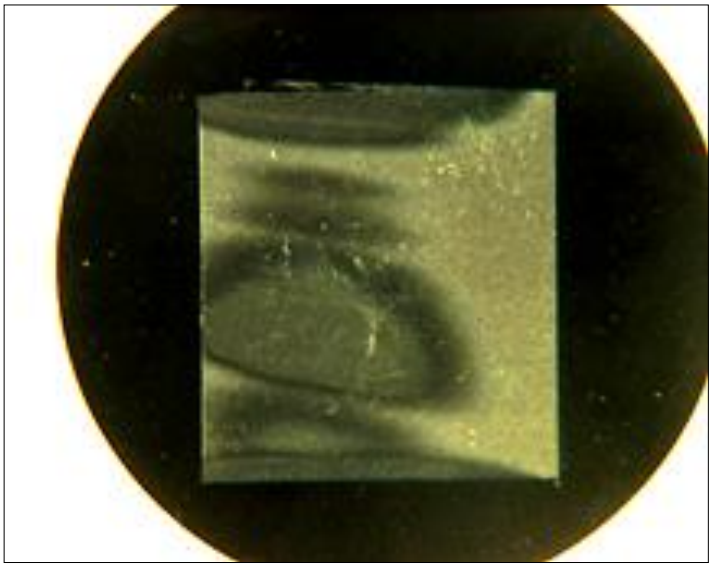


Comet tails- Excessive directional force - etched steel
Metallurgical microscope 20x objective – Brightfield

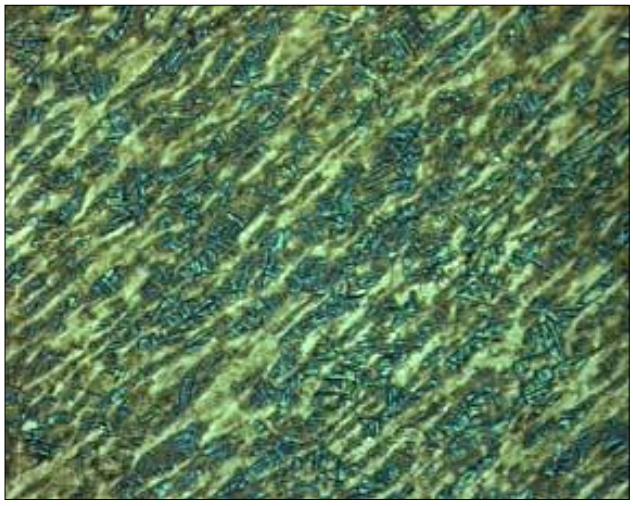


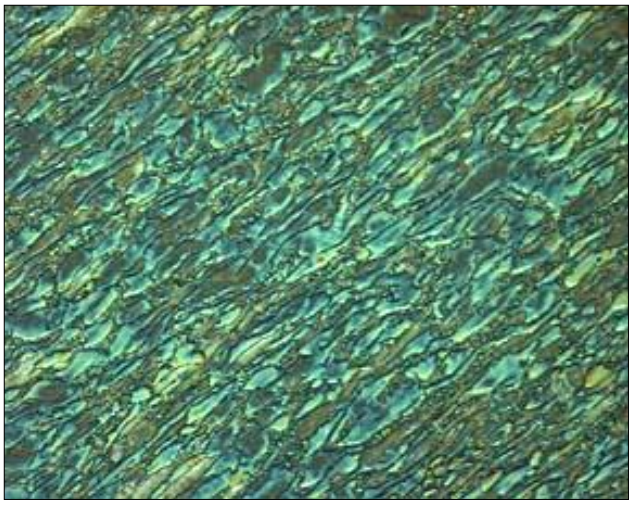
Chemical attack of phases in Aluminium alloy.
Metallurgical microscope 100x objective – Brightfield

Fig 69. Preparation Artefacts - Sectioning damage in Titanium alloy - Etched in Krolls reagent



Preparation beings as soon as the material arrives – Damage through sectioning can profoundly affect a microstructure leading to erroneous assessment of the completed preparation procedure. (32mm dia).



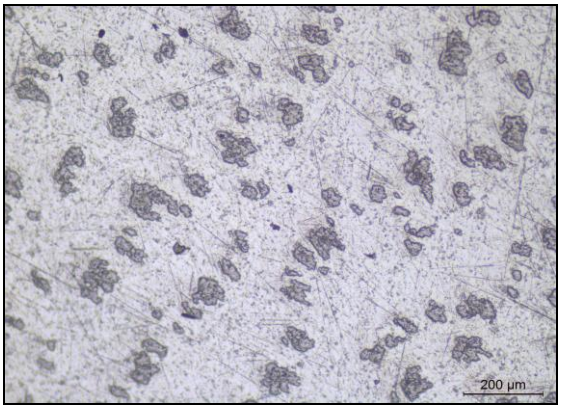


Microstructure of Titanium alloy etched in Krolls reagent to create contrast in Brightfield illumination. Metallurgical microscope 20x Objective

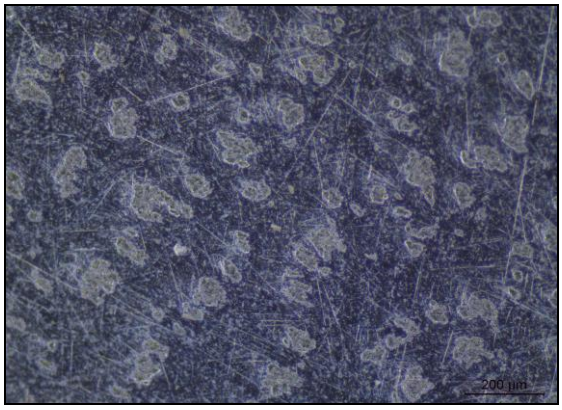
Which is the correct structure?

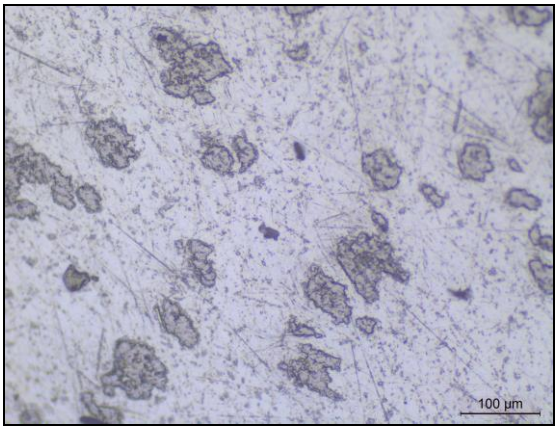
The early stages of preparation including Sectioning & Encapsulation an have a dramatic effect on the final result. Damage imparted here may not be removed during any further grinding & polishing stages.

Fig 70. Metallurgical Microscope Brightfield & Darkfield Examination of Impressed Abrasives in Titanium

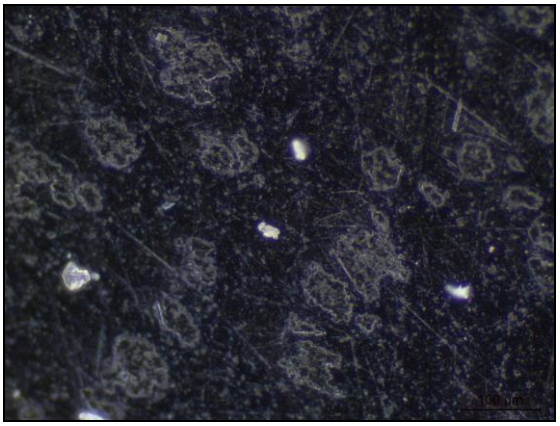


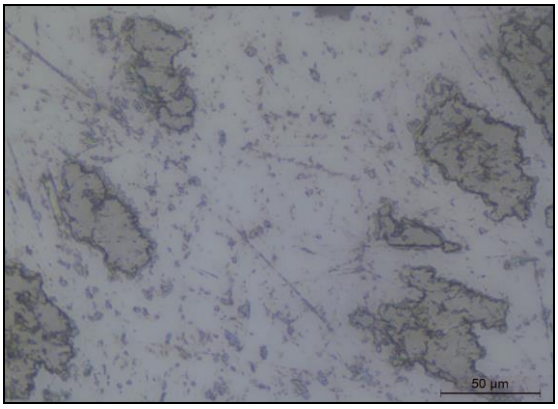
BF 10x DF



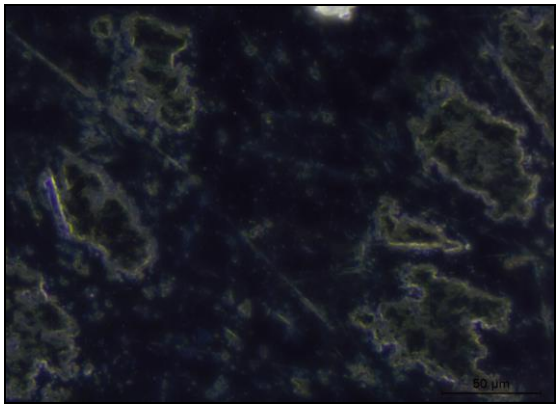


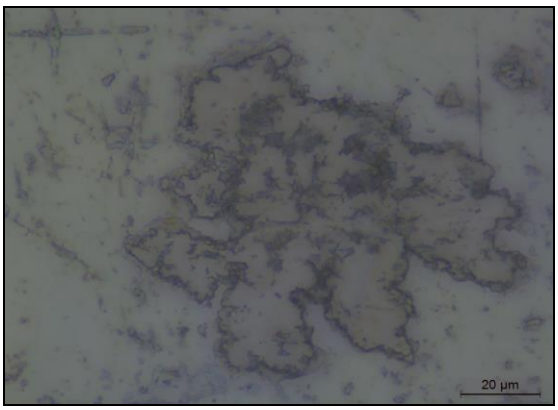
BF 20x DF





BF 50x D





BF 100x DF

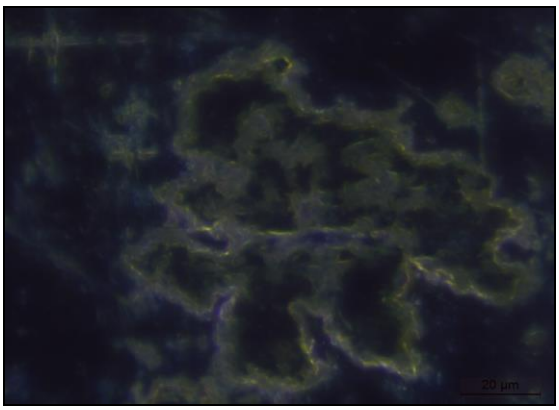
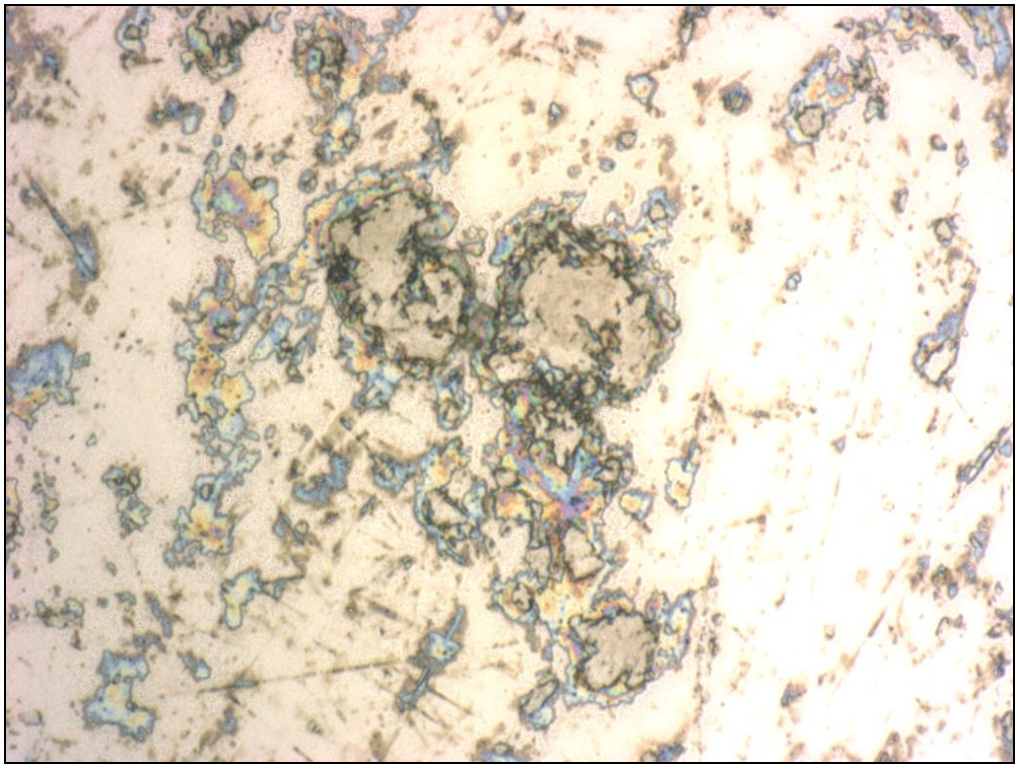
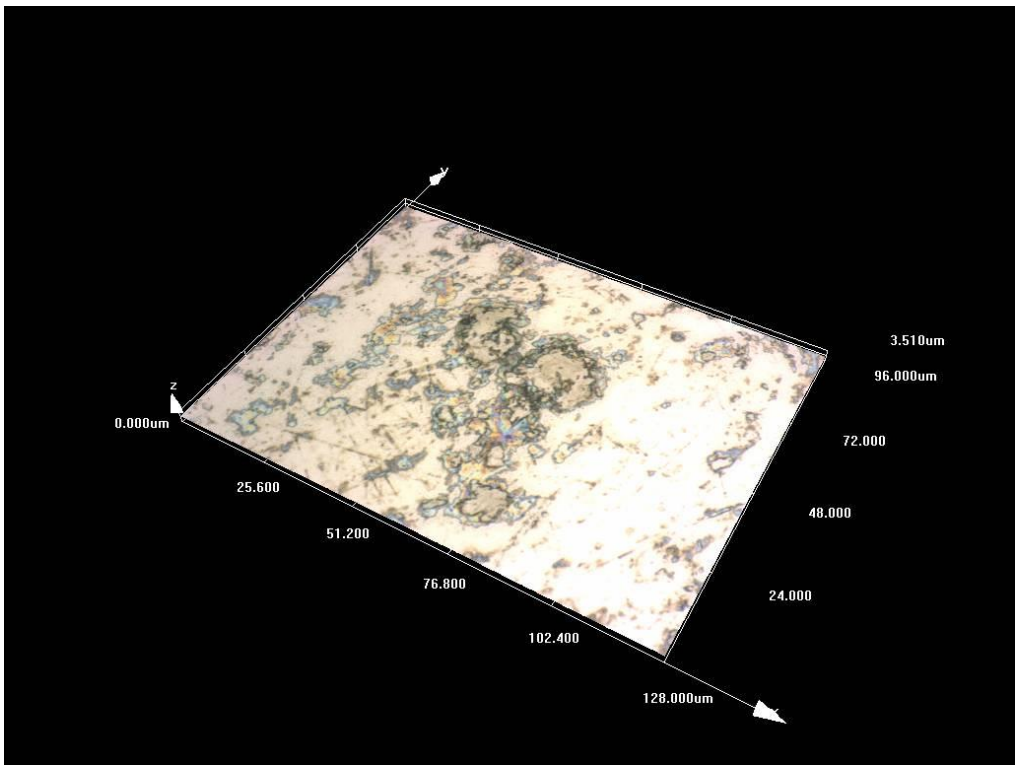


Fig 71. LSCM Examination of Impressed Abrasives in Titanium

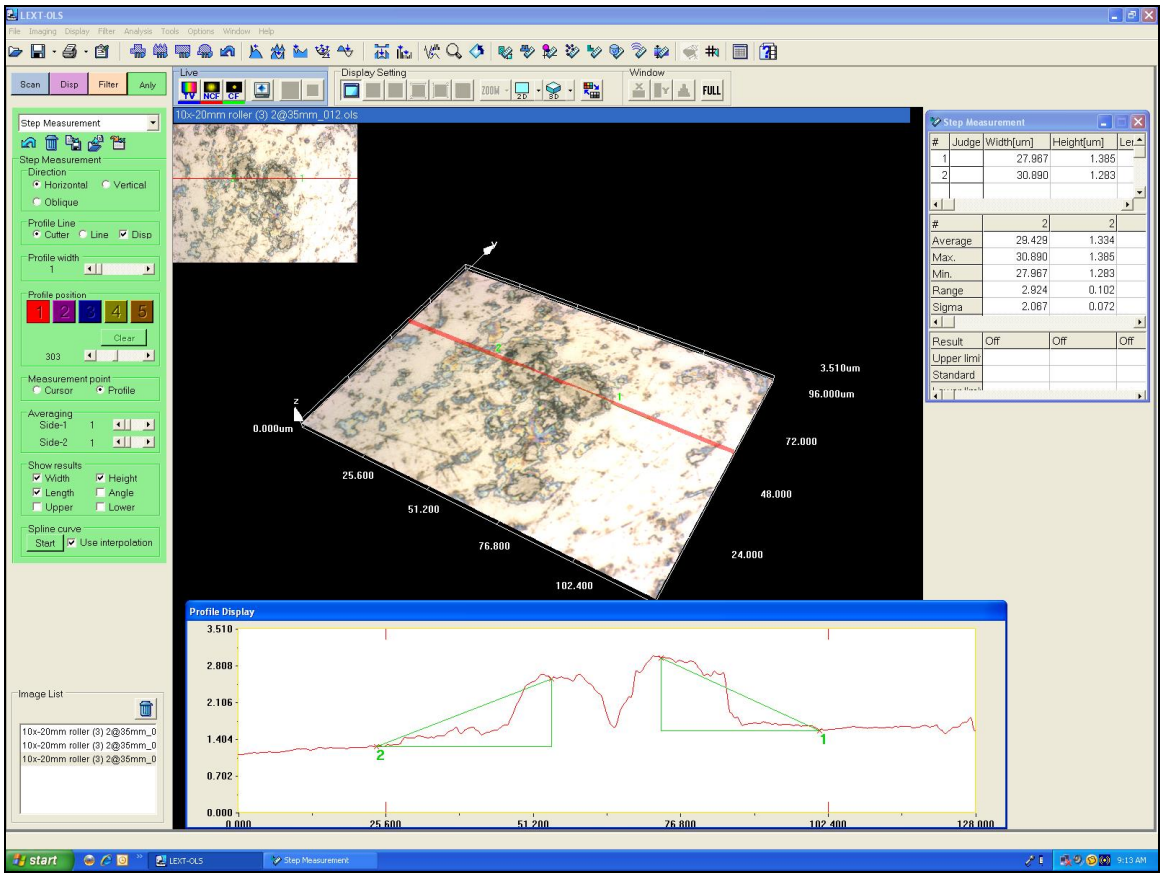


100x Objective 2D view



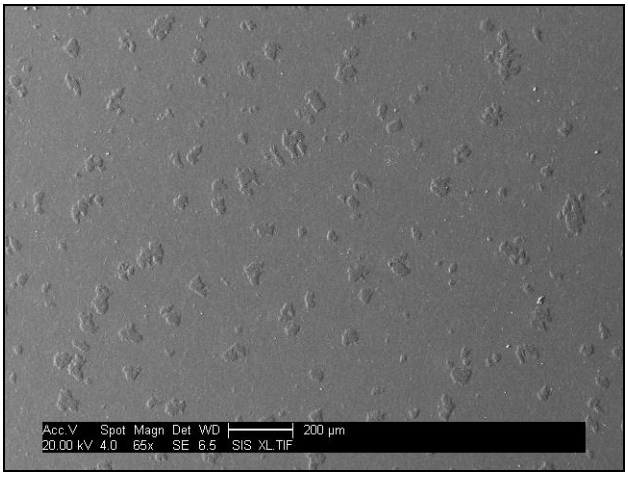
100x Objective 3D view

Fig 72. LSCM Examination of Impressed Abrasives in Titanium

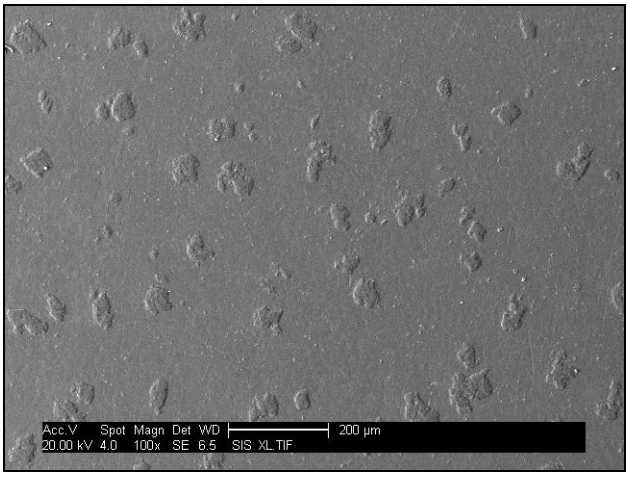


Measurement indicating that the Impressed abrasive is protruding above the matrix by approximately 1 micrometer.

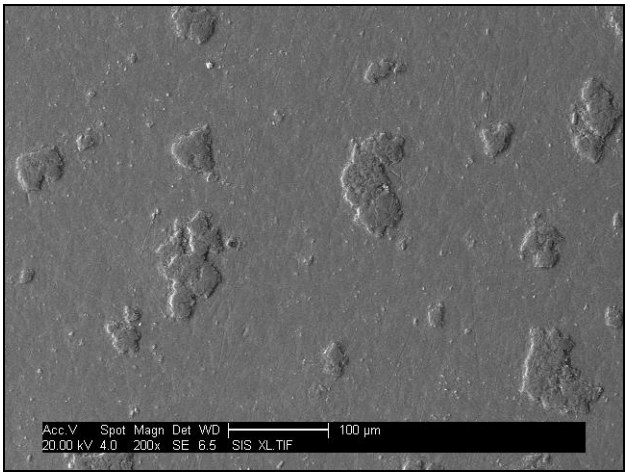
Fig 73. SEM Examination of Impressed Abrasives in Titanium



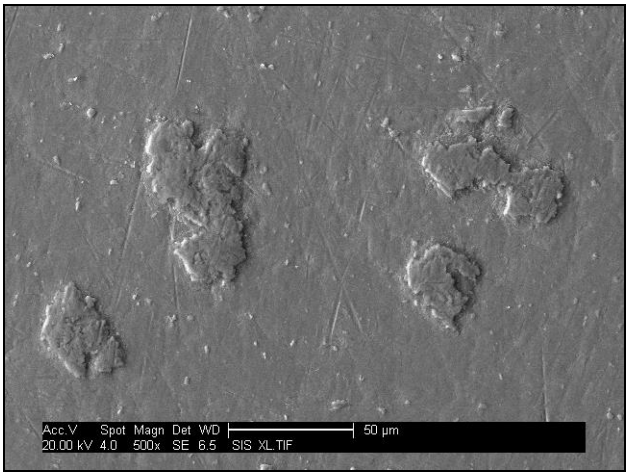
65x



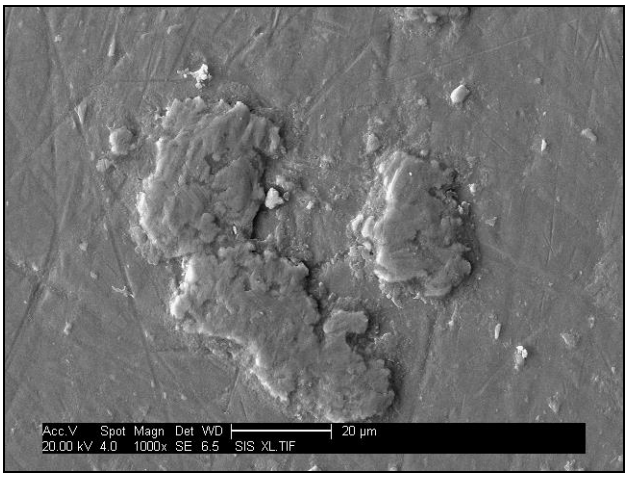
100x



200x



500x

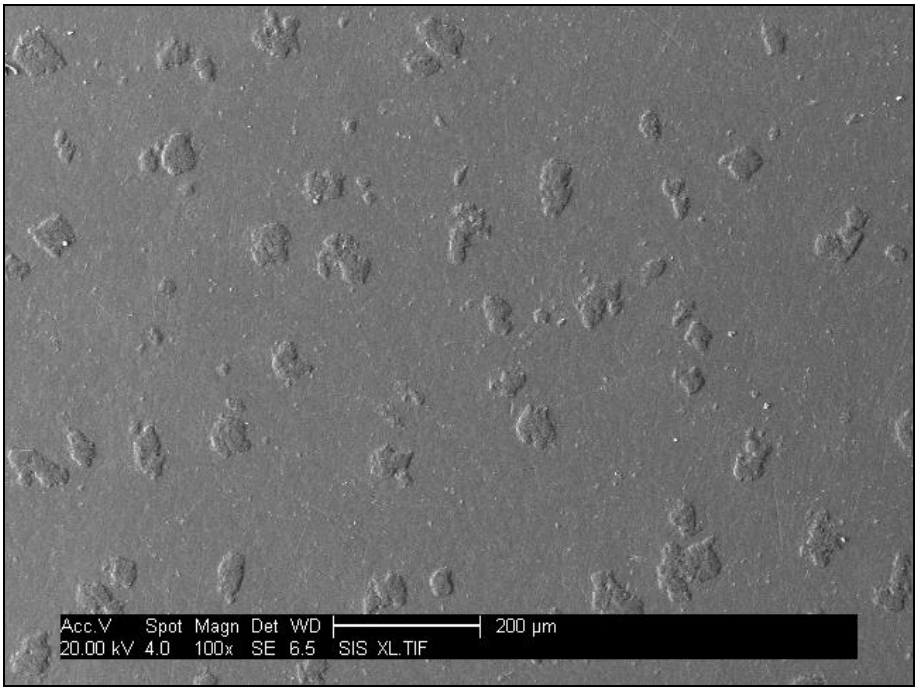


1000x

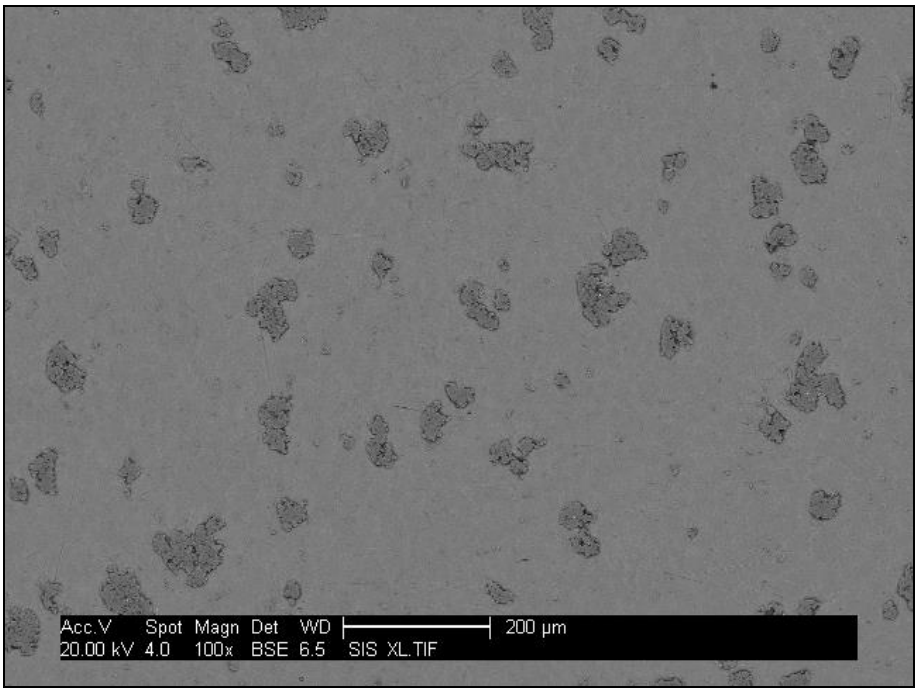
SEM examination shows as in the LSCM examination both the morphology and an indication material is impressed into the surface.

Fig 74. SEM Examination of Impressed Abrasives in Titanium

Comparison of Secondary & Back Scattered SEM images at 100x



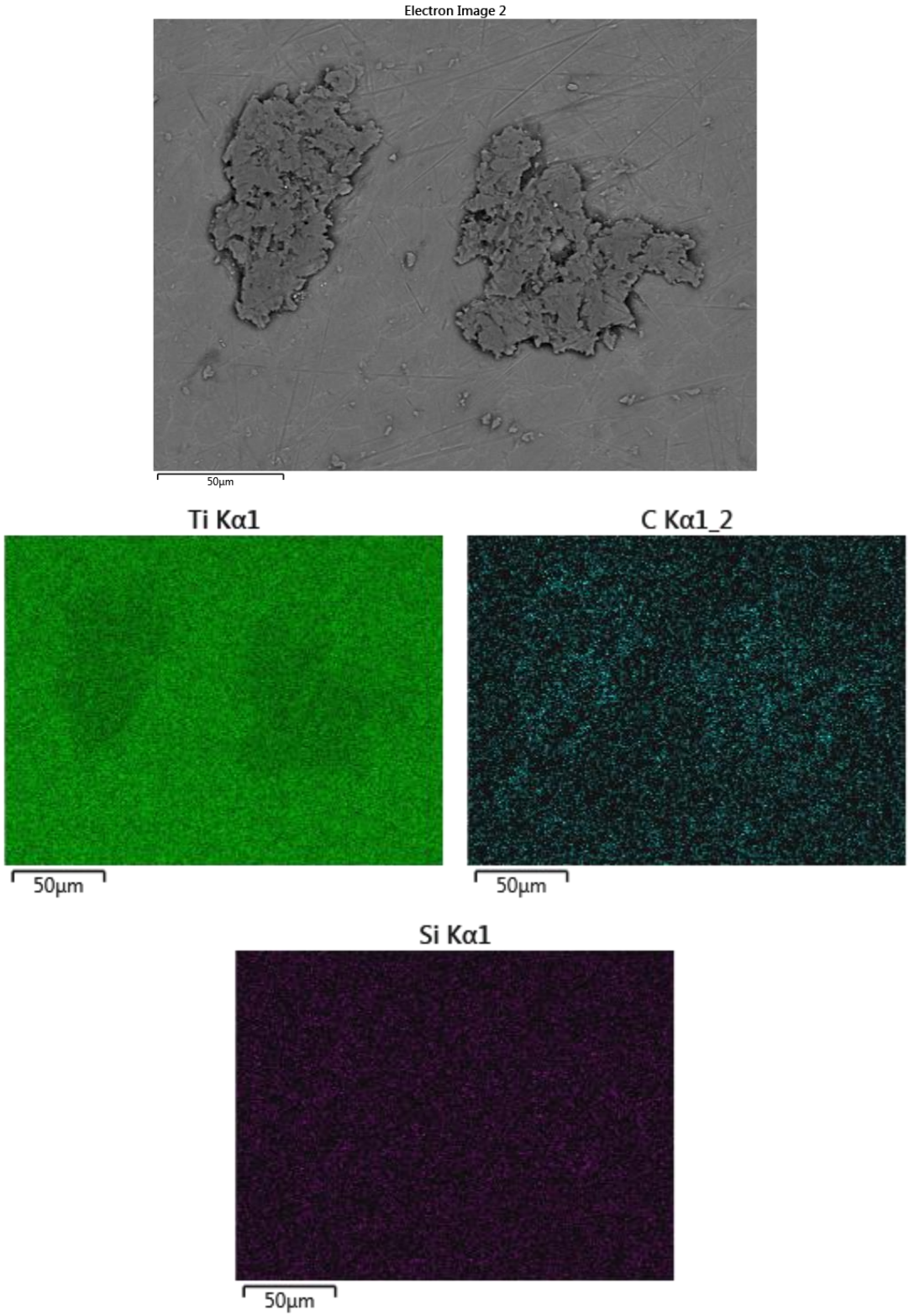
100x Secondary Electron Image



100x Back Scattered Electron Image

The Back Scattered Electron image gives greater contrast as it separates material by atomic number.

Fig 75. SEM Examination & X-Ray Microanalysis of Impressed Abrasives in Titanium



X ray analysis carried out for Titanium Carbon and Silicon shows that the material present is Carbon rich but not containing Silicon. Thus indicatiing the impressed abrasive is Diamond & not Silicon Carbide.

Microstructual analysis of the prepared sample. Contrast techniques in optical microscopy

It has been demonstrated in this work how the use of the optical microscope is fundamental in revealing the true microstructure of a prepared sample and how we can use the metallurgical microscope to assess the structure & morphology of metallographic consumables. During this process a couple of light microscope contrast techniques, Brightfield and Darkfield have been employed and polarised light has also been mentioned.

Having the knowledge to prepare damage free samples allows various microscopical contrast techniques to be employed. This allows the materials scientist or Metallographer extract the maximum data from the prepared samples. Several contrast techniques exist which can greatly aid the materials scientist in his evaluation. Brightfield illumination, Darkfield illumination, Polarised light and Differential Interference Contrast are the main techniques used in the materials environment. Each technique offers something different and not all techniques will be ideal for all samples. In addition, only by using a correctly configured instrument is it possible to get the most from these microscopical techniques. For those needing advice on such techniques the RMS Light Microscopy Course and the MetPrep Introduction to Microscopy & Digital Imaging are highly recommended. Details pertaining to the MetPrep IMDI course are included in the additional course notes included with this project and will not be discussed here.

Before going into further detail, the examination of an SG iron (*fig76*) illustrates how the various contrast techniques have be employed on the recently prepared material. It is not intended within the scope of this work to go into great detail regarding the physics of these techniques but instead to give an overview and illustrate how these techniques can be employed to aid the materials scientist in retrieving information from correctly prepared samples.

Brightfield illumination

Brightfield illumination is typically the standard and most used common type of illumination used in materials microscopy. In this configuration the correctly collimated light is sent through a partially reflecting mirror set at 45 degrees and directed through the objective to the specimen surface. This partially reflecting mirror is usually housed in a simple cube that can be inserted in to the optical path when required. The light strikes the sample and is reflected back up through the objective passing through the partially reflecting mirror and arriving at the eyepiece or camera to generate the final image (*fig77a*).

When a simple highly polished surface is viewed in Brightfield illumination a completely white image is seen all light is completely reflected back as though looking at a mirror. To view any information from the sample, some form of contrast is required. Materials such as aluminium alloys have a considerable amount of precipitates or intermetallic phases present. These individual components do not reflect the light back through the optical axis in the same manner and so appear darker. This is how the contrast is generated and some of the structure revealed.

In materials such as steel there are no intermetallic particles or phases visible and therefore no information on the microstructure can be obtained using Brightfield illumination in the as prepared condition. Other constituent particles in the steel such as inclusions will be revealed if present in the same way as the aluminium's intermetallics. These inclusions often appear orientated in a particular axis giving an indication of any working direction. It is the difference in colour and reflectivity again that create the contrast. In such instances viewing the steels microstructure is impossible and an therefore an etchant is required. An etchant is a chemical that

is used to attack part of the material and leave other parts unaffected. This creates the contrast in Brightfield illumination allowing the microstructure to be examined. If the correct etchant is used then the features of the microstructure such as grain structure, hardened layers and segregation can be viewed. Spheroidal graphite (*fig76*).

Many etchants exist and are available for a wide range of materials (Petzow. G 1999) and whilst some require chemicals that are not pleasant to handle, they do provide an excellent way to reveal the microstructure. The one drawback with using an etchant is that you have now actually damaged the surface by chemical attack. Therefore, if it is required to view again in the as polished condition the sample will have to be prepared once again.

Darkfield illumination

Darkfield illumination again requires a collimated light beam to be fed through an optical cube but in this case the half reflecting mirrored surface is replaced with an elliptical front surfaced mirror at 45 degrees. This prevents the light reaching the surface directly as in Brightfield illumination but it does allow the light to travel down the periphery of the objective striking the surface of the specimen at an angle. If this angled light strikes a highly polished surface, all the light striking the surface is reflected out of the optical axis and thus the background appears completely black (*fig77b*). If however, there are scratches, particles and phases etc on that surface, the light path will be disturbed and the light deflected into the optical axis. Particles will now appear as bright dots in a dark background. A simple analogy is that of Darkfield being like bright stars in the night sky and Brightfield being dark stars in a bright sky.

In addition to scratches and particles reflecting light into the optical axis, if a sample surface when prepared is partially transparent then when illuminated by a metallurgical microscope, in Darkfield conditions some of the light that strikes the sample actually penetrates the surface of the material and is then deflected into the optical axis. As light is reflected from within the material and into the optical axis it can be seen. This can be employed when examining materials such as glassy slags and various geological materials. A typical example to compare with the Brightfield technique is the Spheroidal graphite (*fig 76*)

Polarised light

It has been shown when the graphite of Spheroidal cast iron is examined in cross polarised light conditions that the resultant image shows a classic Maltese cross structure. To achieve this effect and to assess a material for its optical activity we need to configure the metallurgical microscope with a polariser and analyser. The polariser and analyser only allow light vibrating in one direction through the optical path.

Usually this will be in a North - South and East - West direction respectively. In a plane polarised condition, the collimated light striking a highly polished sample for instance will only allow one direction of vibration to strike the sample and return to the eyepiece in the usually manner. It will of course be fainter than when observed without the polariser. If the analyser is now inserted into the optical path and is rotated 90 degrees to the polariser, the light will be completely extinguished and nothing will be seen (*Fig78a*). If one now observes a sample that by its nature changes the nature of the illuminating lights vibration; i.e. a material that is birefringent then depending on the anisotropic nature of that material the light will be bent slightly and thus be visible in what would have otherwise been a black background. Typical

causes for such birefringence are crystal orientation and thin films. Even internal stresses in plastics can cause such anisotropy to occur.

As well as the obvious examination of graphite in cast irons, other such graphite can be viewed and assessed using polarised light techniques including carbon – carbon composite materials as used in the automotive & aerospace industry as brake materials and also in carbon based ceramic refractory applications.

One of the most important uses of Polarised light in materials microscopy is that of the examination of aluminium alloys. It has been shown how when polished, aluminium alloys show a series of particles or phases but unless these are distributed at the grain boundaries no grain structure can be seen. Using etchants such as in the case of steels discussed earlier Brightfield and Darkfield still fail to reveal any indication of the grain structure.

The only real technique that can be employed to reveal the grain structure of an aluminium alloy is that of anodising of the sample and then examining in Polarised light. This involves an electrolytic process where the sample is suspended in a solution of 2% Tertaflouroboric acid - commonly known as (Barkers Reagent). The sample is made the anode and a Stainless Steel cathode is added. An electrical current – typically 20v DC is passed through the solution for two minutes and the solution stirred with a magnetic stirrer to disperse any bubbles generated.

If attempting this procedure for the first time, do not use the solution when freshly made up as I have found the results poor. Instead mix the solution up the day before use and add a small sample of aluminium foil into the solution and leave overnight. I understand introducing some additional aluminium ions into the solution must help the process work. This is a tip I picked up at the Alcan International Research Laboratories – Banbury and I have no reference for it.

Examining the anodised sample in Brightfield illumination reveals the typical particles within the material and somewhat attacked similar as when they are viewed in the etched or polished condition. In addition, there is a slight indication of the presence of a grain structure but they are indistinct and one certainly wouldn't like to assess the grains structure from such a surface condition. The addition of the Polariser and rotating the Analyser towards the extinction position suddenly reveals the grain structure in full detail. The individual grains show up as a series of grey levels ranging from black to white. In the past this effect has been attributed to the anodising operation creating an interference film but this appears not to be the case and occurs due to a roughening of the surface by pitting and the orientation of the grains (Smithels. 2003 - Vander Voort. 2005). If a waveplate is also introduced into the optical path then the background moves from the first order grey position in the optical spectrum to the first order red position and a range of colours instead of shades of grey are seen. *(fig 80)*

Differential Interference Contrast - DIC

Often called Nomarski Interference Contrast after Georges Nomarski (1919-1997). This is one of the most powerful contrast techniques available to the materials scientist and relies heavily on a high quality sample preparation that is defect free and very flat. Whilst users unfamiliar with the technique may think the samples are exhibiting relief caused during the preparation stages this is far from true. Samples with excess relief will not respond favourably. It is most important that the samples are totally flat.

To employ DIC it is necessary to configure the microscope as used for the Polarised light extinction position. When viewing a sample that isn't birefringent nothing will be viewed and the

surface will appear black. For instance, the metal matrix in the Spheroidal graphite as examined earlier. The background is clearly free from any microstructural information (*Fig 78a*).

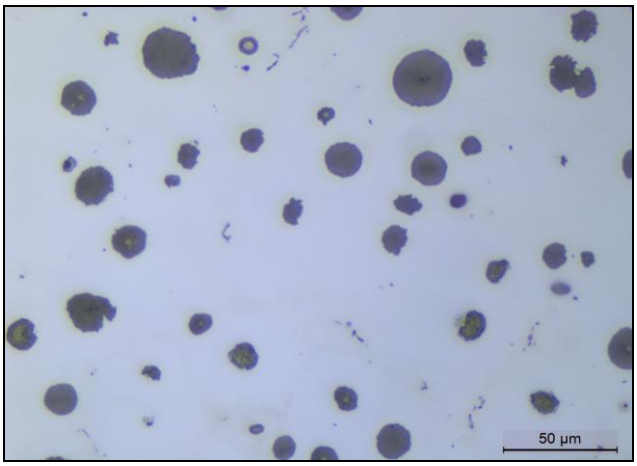
It is now necessary to insert the Wollaston or Nomarski prism into the light path just above the microscope objective at the correct conjugate plane. Here the light is parallel and in infinity space i.e. before it reaches a Telan lens. The result is that the prism creates two orthogonal beams of polarised light at 45 degrees relative to polariser as they strike the sample surface. If the surface is perfectly flat then nothing is seen. If however there is slight sub micron topography invisible in Brightfield illumination such as a small step between a particle and its matrix then the returning beams will be slightly out of sync and one beam will travel a longer distance than the other. These rays are often referred to as ordinary & extraordinary rays. On their return the beams then pass through the objective and prism and reach the analyser (Rottenfusser et al). At this point an interference image is generated and these path differences are displayed in shades of grey (*fig78b*). Again, as in the case of polarised light a waveplate can be added to move the first order grey position to the first order red position of the coloured spectrum. The prism or wedge is adjustable and it can set to the position that gives the microscopist the most information and occasionally the most pleasing colours. This technique is again illustrated on the Spheroidal graphite sample (*fig76*).

To illustrate how the various contrast techniques can be applied to a range of different materials a selection of photomicrographs has been included to illustrate the various techniques outlined.

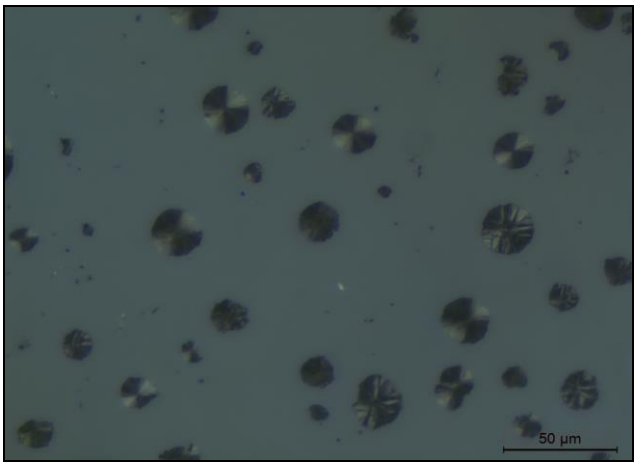
These include a glass fibre composite (*fig79*), a wrought aluminium alloy (*fig80*), a carbon – carbon brake pad (*fig81*) and a selection of medieval glass slags (*figs 82,83&84*).

It can be seen that the materials scientist has a wide range of contrast techniques available to examine materials but all these materials require high quality sample preparation. Not all techniques will be suitable to all materials but it takes only a few minutes to try these techniques to see which is most beneficial to the sample being examined. One only has to think of the glass fibre composite when viewed in Darkfield illumination to realise how critical the choice of contrast technique can be. Even the task of trying to assess grain size in aluminium alloys would be impossible in most instances if polarised light was not available.

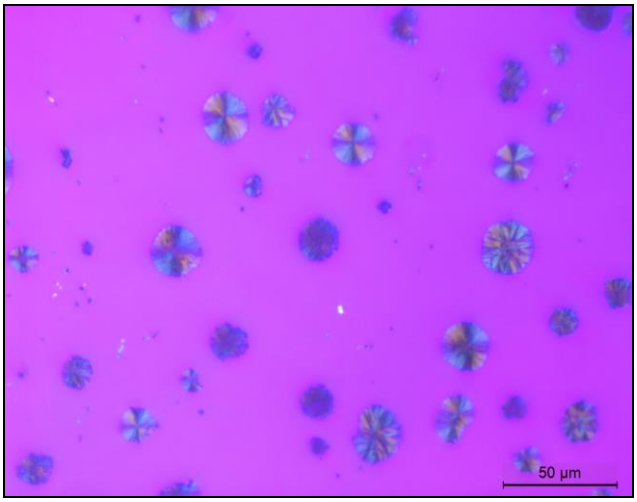
Fig 76. Contrast Techniques in Materials Preparation - Spheroidal Graphite Cast Iron



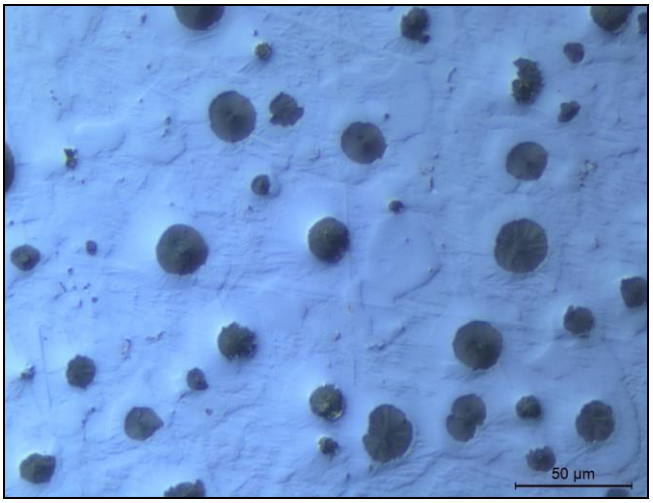
Brightfield illumination 50x objective



Brightfield – Crossed Polarised light 50x objective

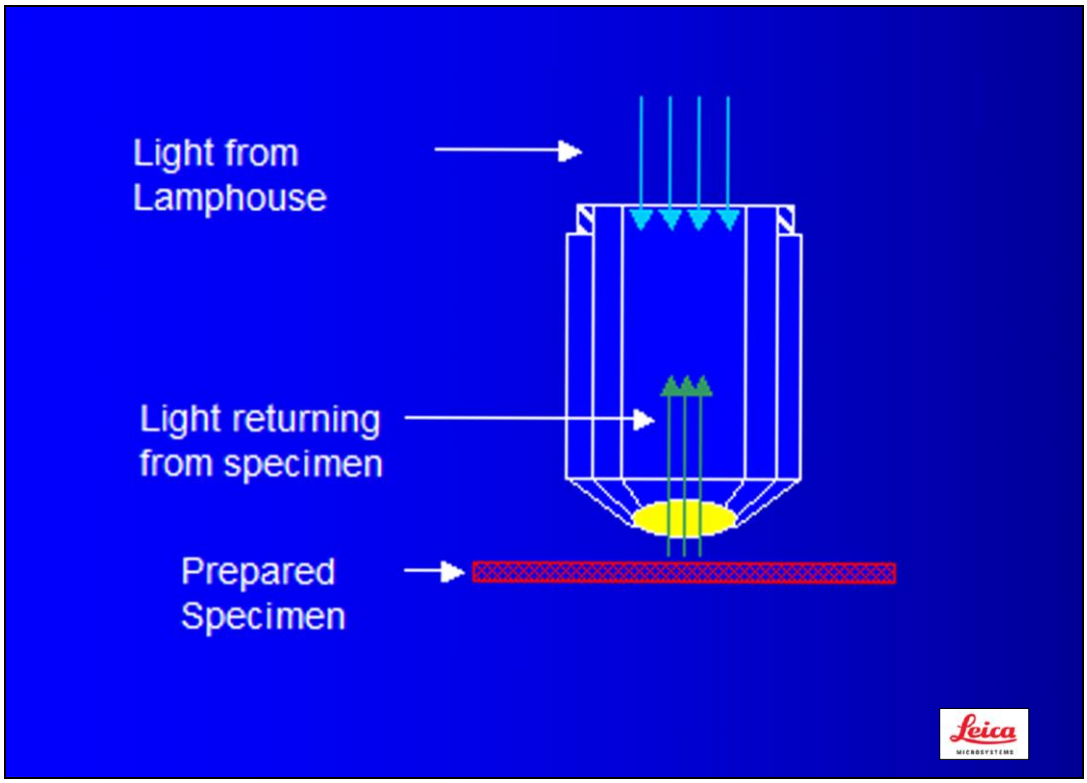


Brightfield – Polarised & Waveplate 50x obj

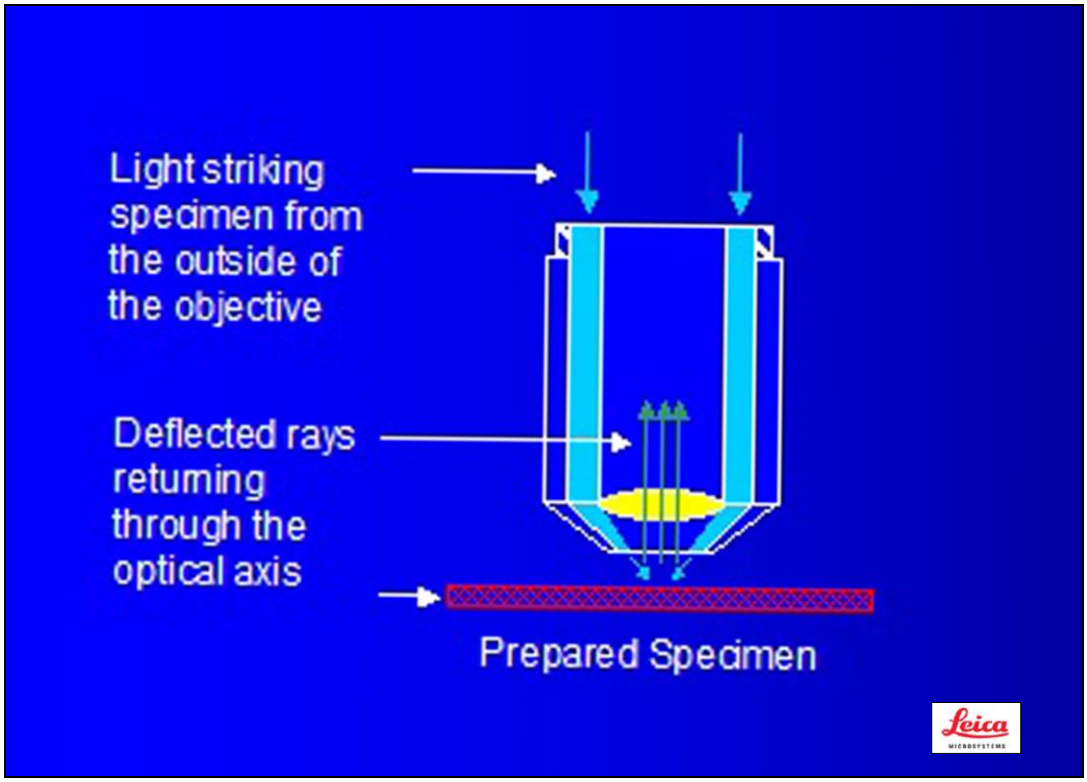


Differential Interference Contrast 50x objective

Fig 77 a-b Contrast Techniques in Materials Microscopy - Brightfield & Darkfield illumination

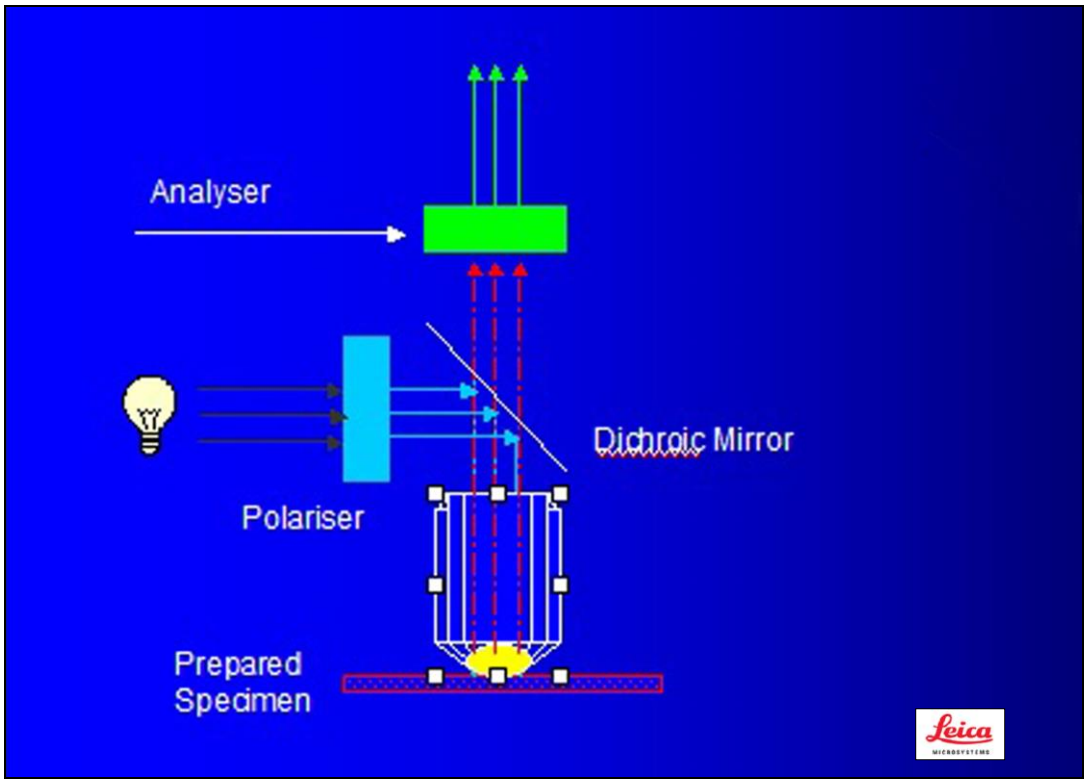


Brightfield illumination – Ray diagram

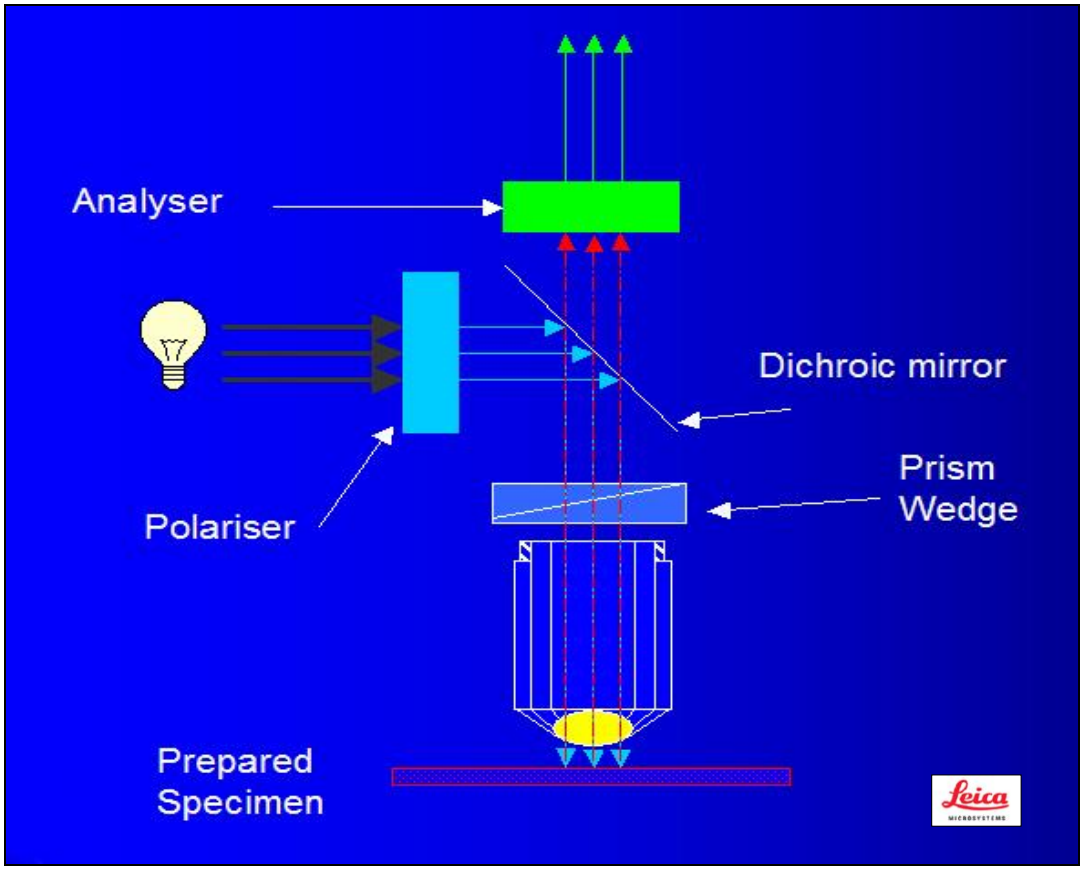


Darkfield illumination – Ray diagram

Fig 78 a&b. Contrast Techniques in Materials Microscopy - Polarised & Differential Interference Contrast

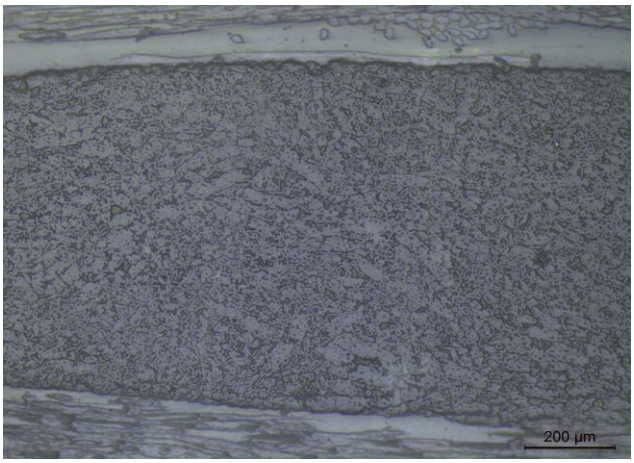


Polarised light – Ray diagram

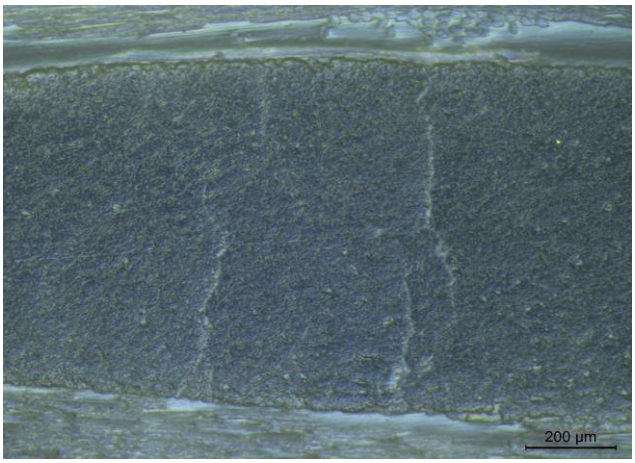


Differential Interference Contrast – Ray diagram

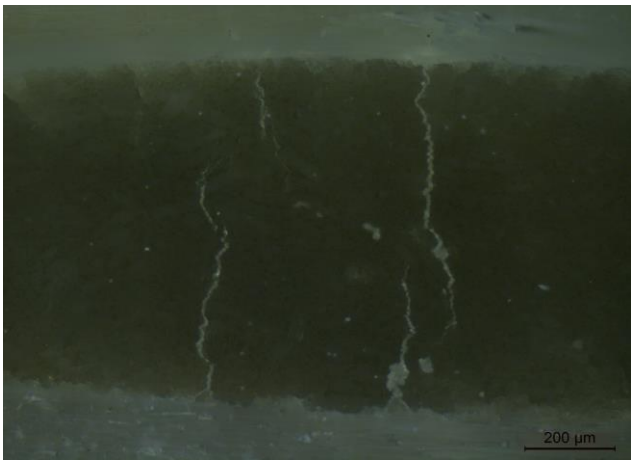
Fig 79. Contrast Techniques in Materials Preparation - Glass Fibre Composite



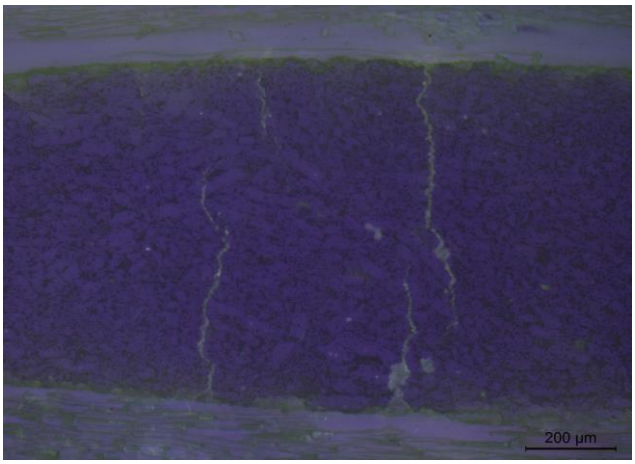
Brightfield illumination - 10x objective



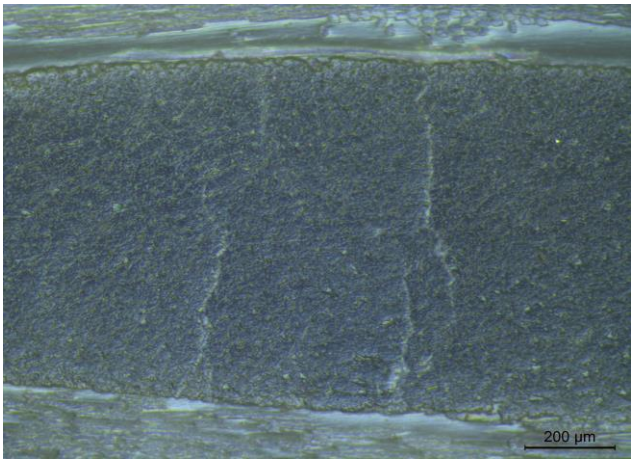
Darkfield illumination - 10x objective



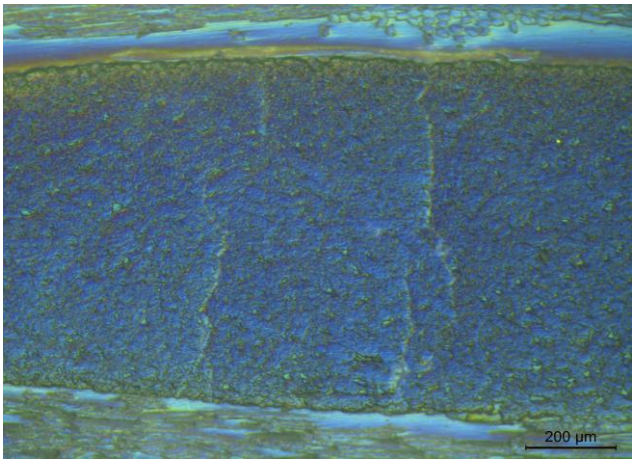
Crossed Polars - 10x objective



Crossed Polars + Waveplate - 10x objective

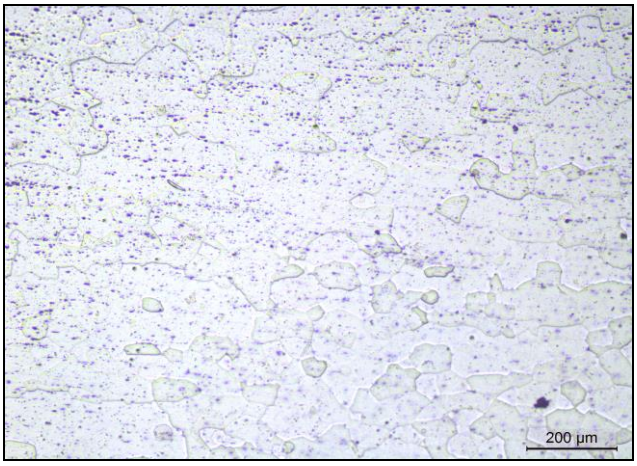


DIC - 10x Objective

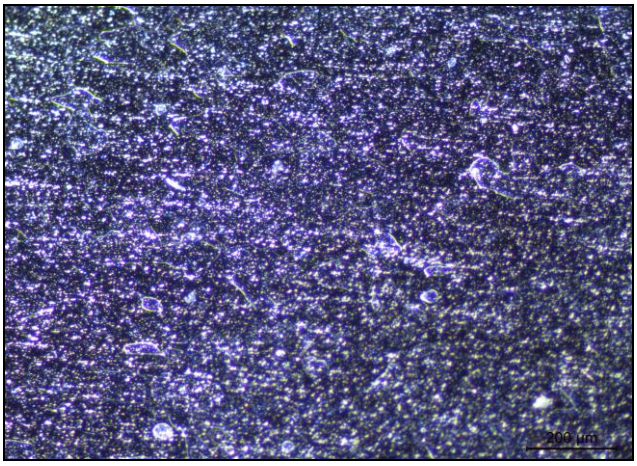


DIC + Waveplate - 10x Objective

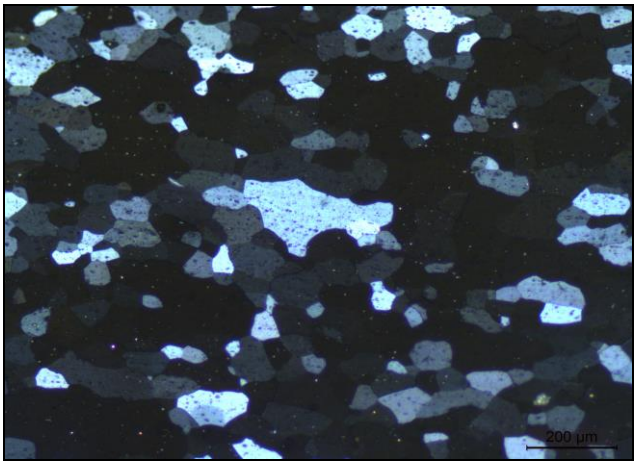
Fig 80. Contrast Techniques in Materials Preparation - Wrought Aluminium alloy - Anodised



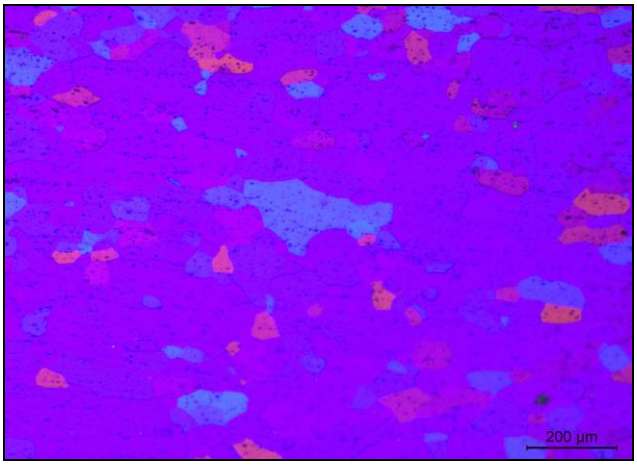
Brightfield illumination - 10x objective



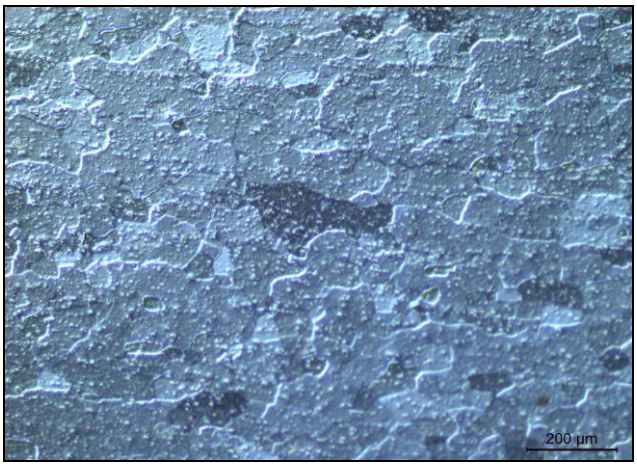
Darkfield illumination - 10x objective



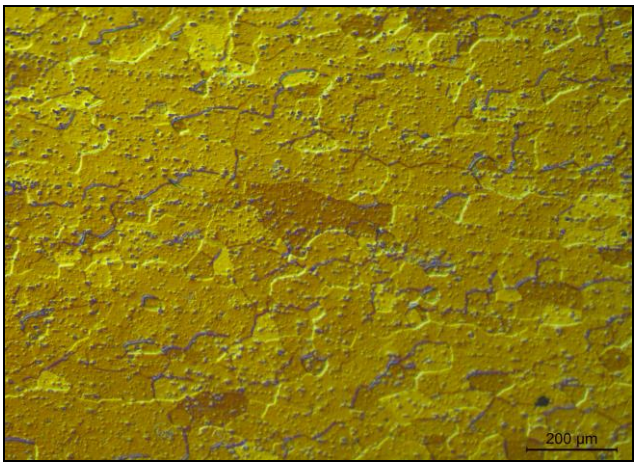
Crossed Polars - 10x objective



Crossed Polars + Waveplate -10x objective

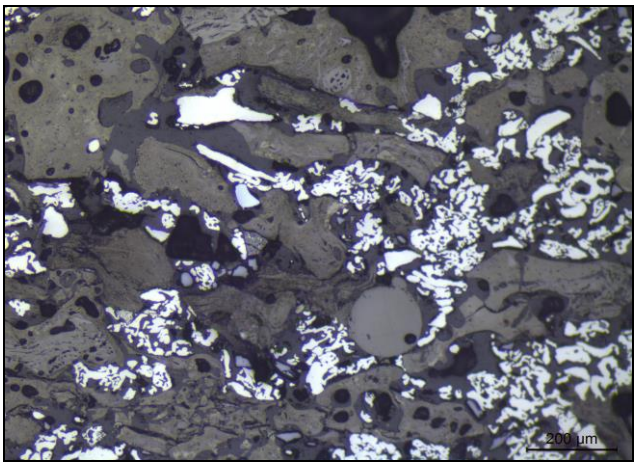


DIC - 10x Objective

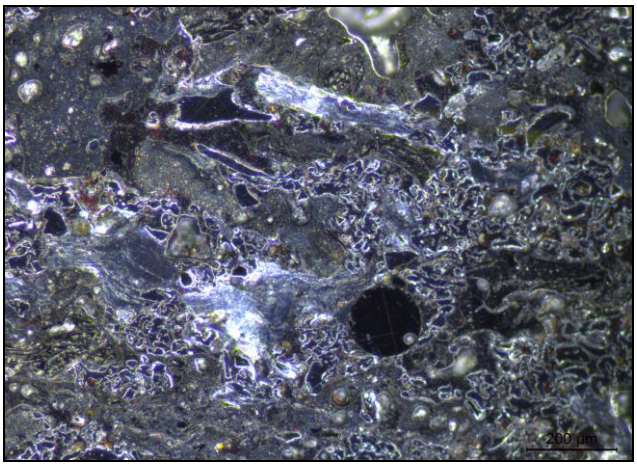


DIC + Waveplate - 10x Objective

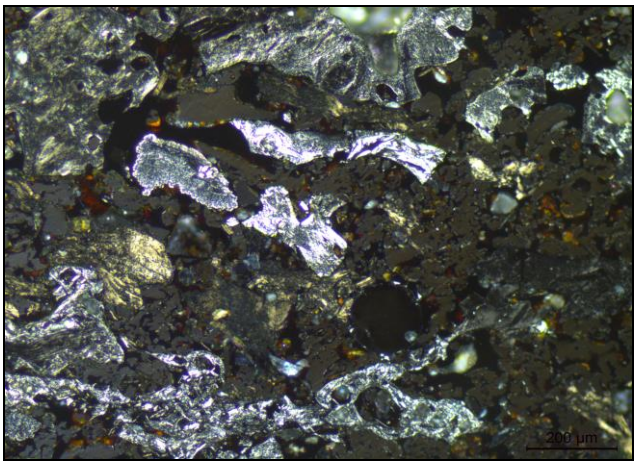
Fig 81. Contrast Techniques in Materials Preparation - Carbon / Carbon Brake Pad



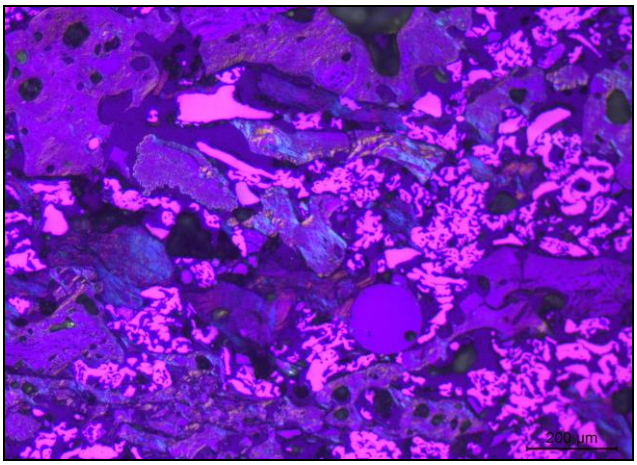
Brightfield illumination - 10x objective



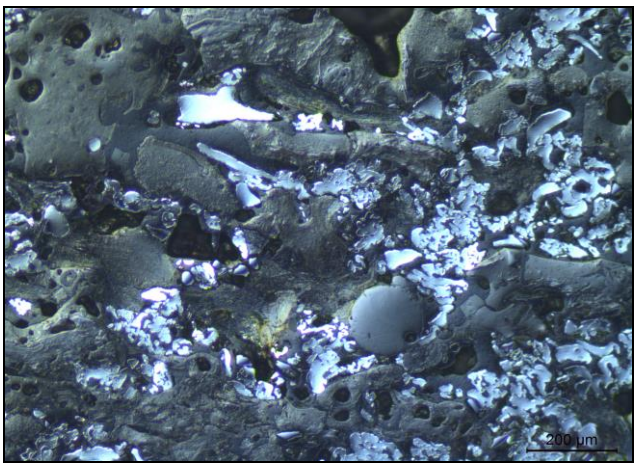
Darkfield illumination - 10x objective



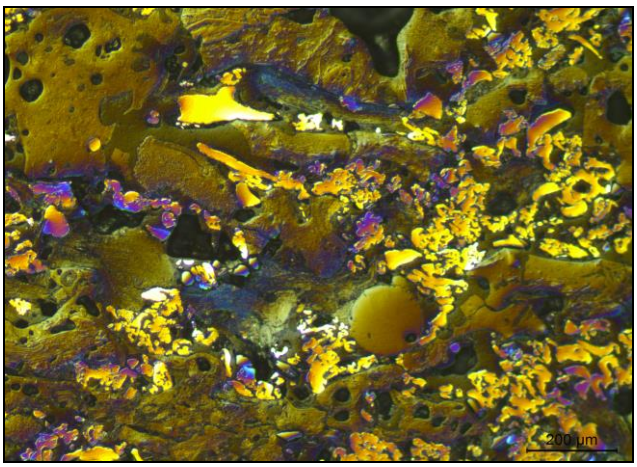
Crossed Polars - 10x objective



Crossed Polars + Waveplate - 10x objective

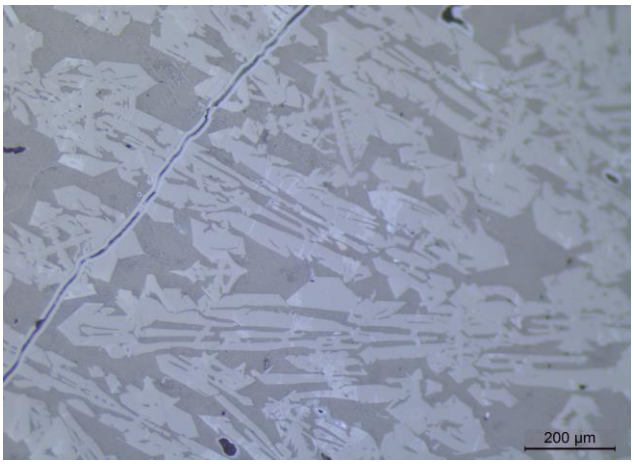


DIC - 10x Objective

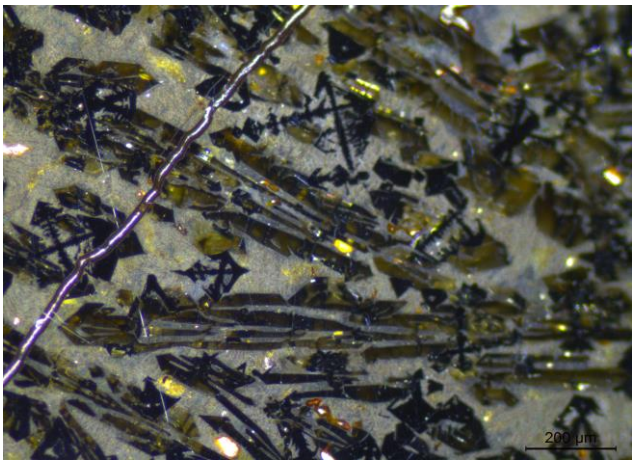


DIC + Waveplate - 10x Objective

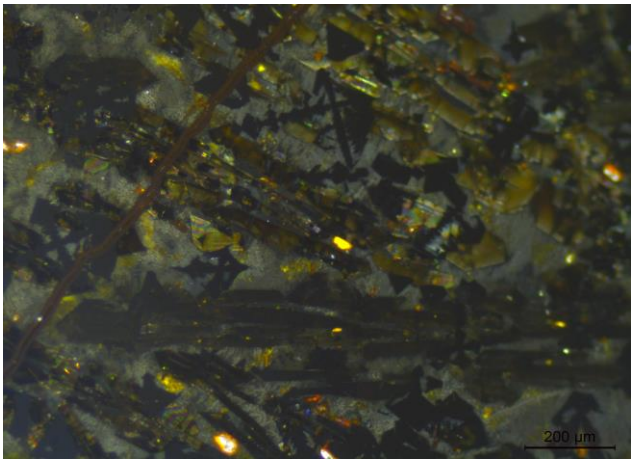
Fig 82 Contrast Techniques in Materials Preparation – Iron Rich Medieval Glass Slag



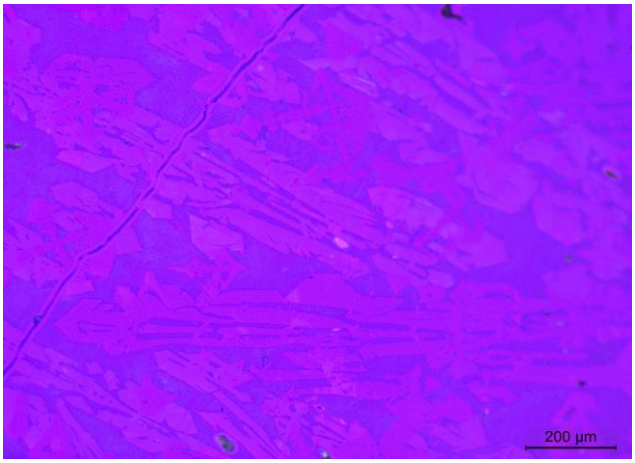
Brightfield illumination - 10x objective



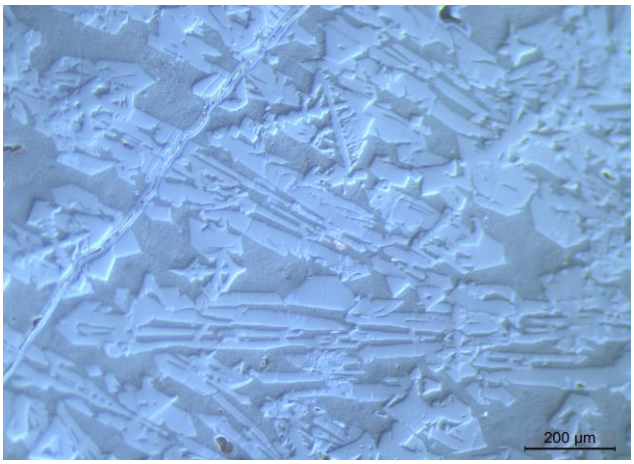
Darkfield illumination - 10x objective



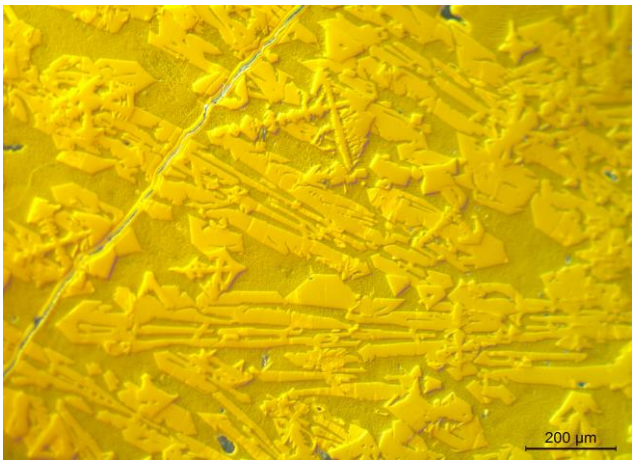
Crossed Polars - 10x objective



Crossed Polars + Waveplate -10x objective

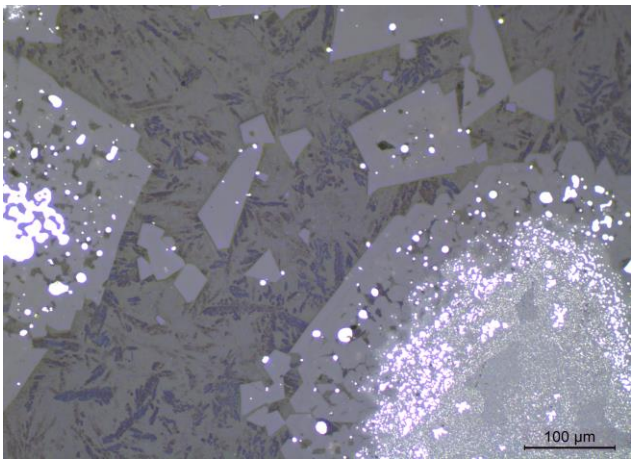


DIC - 10x Objective

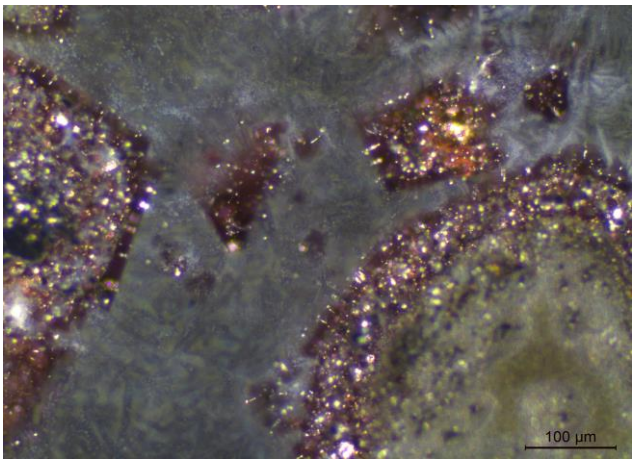


DIC + Waveplate - 10x Objective

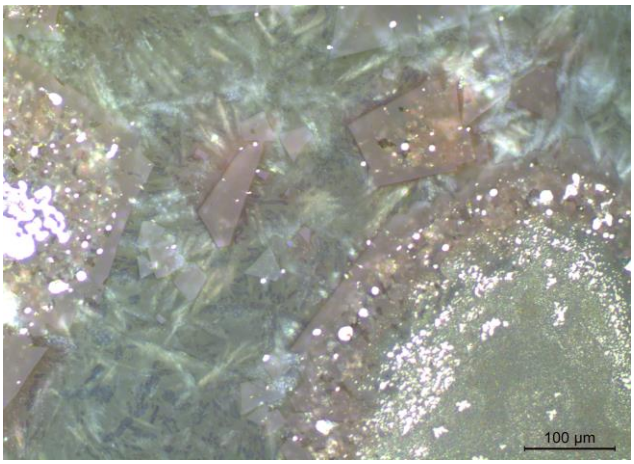
Fig 83 Contrast Techniques in Materials Preparation – Iron Rich Medieval Glass Slag



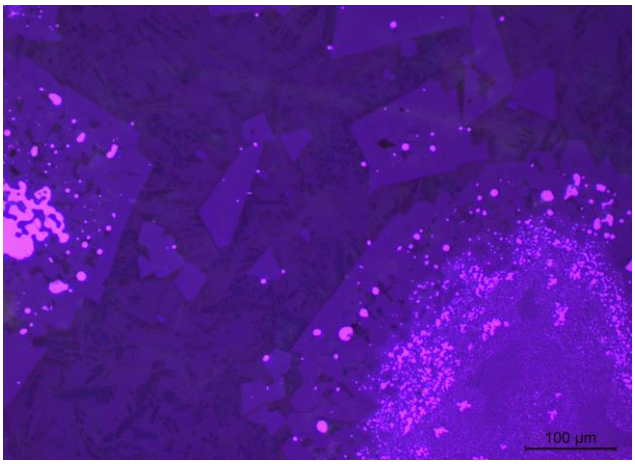
Brightfield illumination - 10x objective



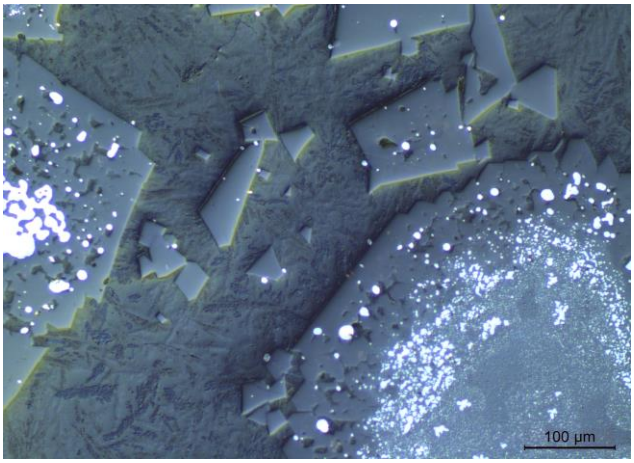
Darkfield illumination - 10x objective



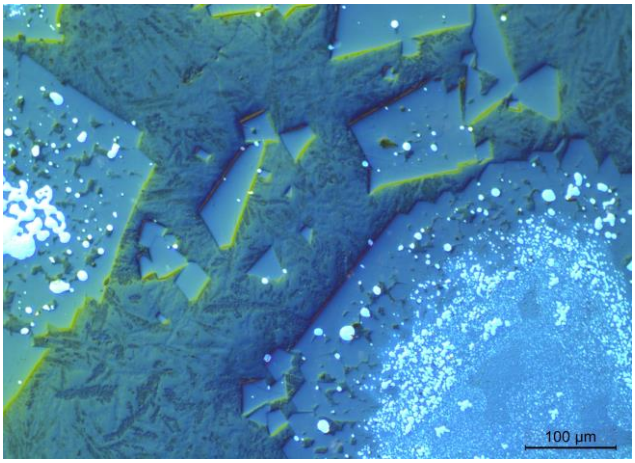
Crossed Polars - 10x objective



Crossed Polars + Waveplate -10x objective

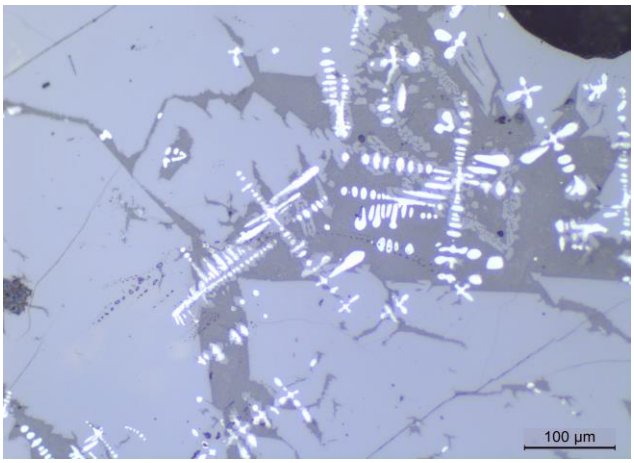


DIC - 10x Objective

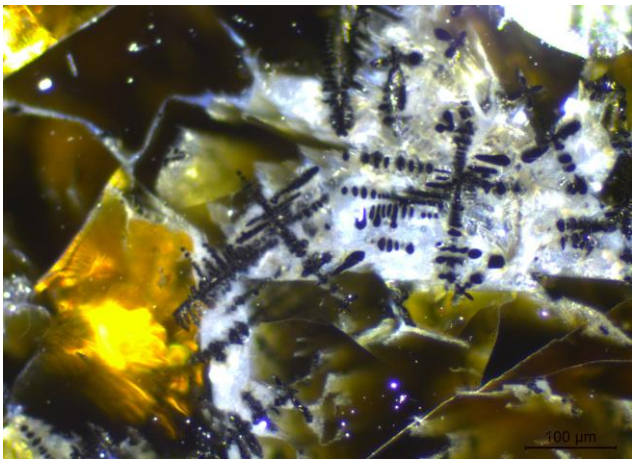


DIC + Waveplate - 10x Objective

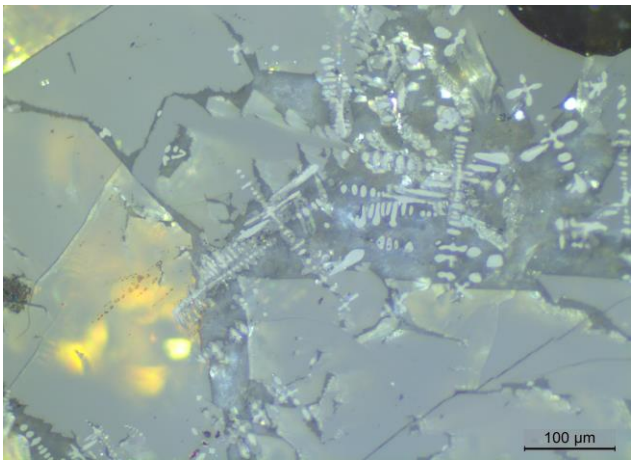
Fig 84 Contrast Techniques in Materials Preparation – Iron Rich Medieval Slag



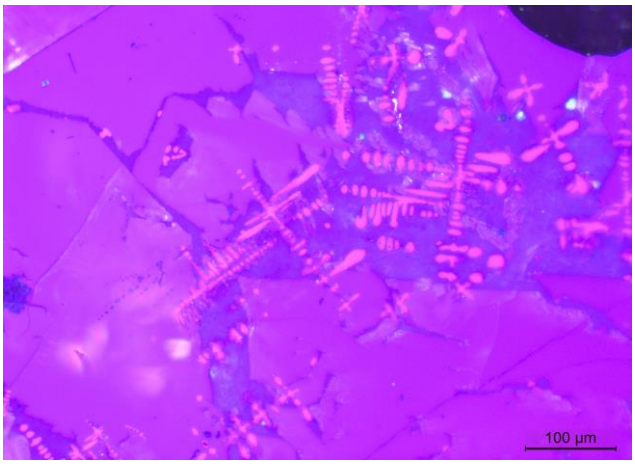
Brightfield illumination - 20x objective



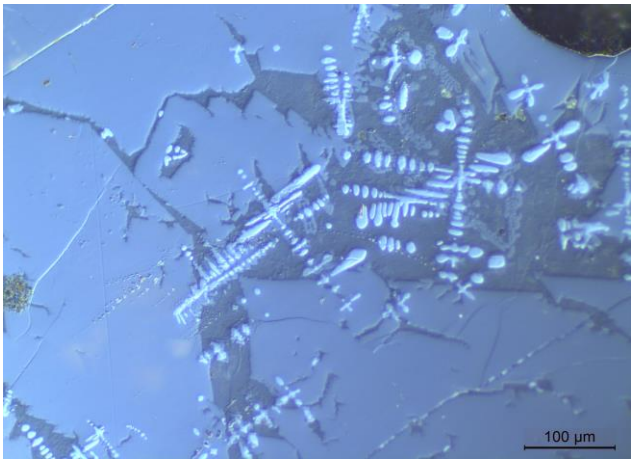
Darkfield illumination - 20x objective



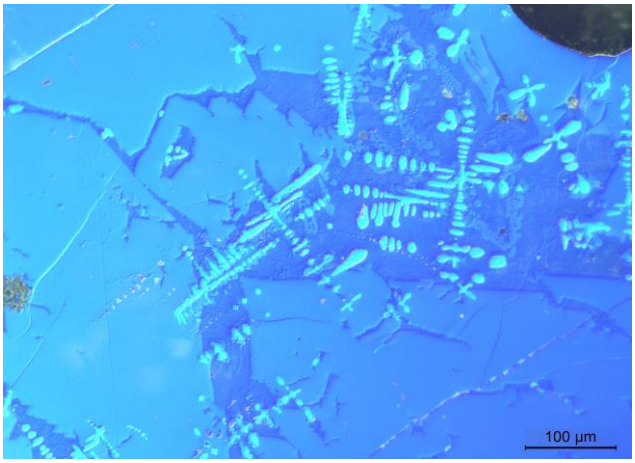
Crossed Polars - 20x objective



Crossed Polars + Waveplate - 20x objective



DIC - 20x Objective



DIC + Waveplate - 20x Objective

In addition to gaining information from a correctly prepared sample some of the microstructures revealed are breath taking in their appearance. These can be equally as beautiful as that generated in any other area of microscopy. If time permits it is certainly worth trying your hand at taking some photomicrographs purely for aesthetic reasons. A selection of materials generated by various contrast techniques are illustrated in the following figures (Phot 1 - Phot 20) to show some of the wonderful images that can be obtained by a metallurgical microscope. The preparation procedures used to prepare these materials have also been included.

Due to restrictions on file size the items Phot 1 – Phot 2 have been added as an addendum and is listed as an independent file under the Diploma on the web site

Summary

Within this piece of work it has been possible to show how important microscopy is not only in examining a prepared sample but also in determining how to prepare the samples.

Using techniques such as simply viewing with a low power stereo microscope can provide detailed information on cloth types and applications. The standard metallurgical microscope can also assist in the examination of many surfaces including silicon carbide, fixed diamond & cloths of various types. Whilst only low magnifications were really practical it was possible to assess such surfaces. Contrast techniques of Brightfield & Darkfield illumination gave variable information on these surfaces and both were considered to be of use.

Additional examination of the consumables using the Laser Scanning Confocal microscope allowed detailed measurements & excellent 3D images of a wide range of consumables. With the illumination only coming from above the cloths really didn't lend themselves to this technique. An area where this technique really scores is on the large diameter fixed diamond surfaces. It not only allows a 3D image to be generated but the colour aids greatly when compared to an SEM. Being able to examine surfaces often up to 300mm in diameter either at the start or when monitoring the morphology during a preparations a considerable bonus.

The use of an SEM to examine all consumables possible was also of value. The high resolution of this technique giving remarkable depth if field allowed comparison of surfaces & even revealed the differences in the manufacturing procedure of the silicon carbide papers. The SEM was the only technique capable of comparing and distinguishing between the monocrystalline and polycrystalline diamond types. For comparing the nature of the cloths the SEM was ideal for both comparison & assessment. The fibre type, size & detail were clearly resolved more than in any other technique. The only drawbacks were the necessity to coat non-conducting materials, the time taken to actually get an image and the size restriction of the samples that can be examined.

When looking at monitoring the actual metallographic preparation during the various grinding and polishing stages, whilst the SEM and LSCM gave very detailed information it was possible to assess progress & make decisions by simply using a metallurgical microscope in Brightfield conditions. Anything above this was overkill & the stereo microscope was obviously unable to resolve anywhere near the detail required.

It has been demonstrated in this work how fundamental microscopy is in preparing materials for microstructural examination both in the consumables used and their interaction with the material to be prepared. In addition it has been demonstrated how the assorted contrast techniques in optical microscopy can be used to obtain far more detail than is often expected. The simple comparison of Brightfield & Darkfield in the fractured Glass fibre composite exemplifies how these techniques should be more widely used in our materials laboratory.

I hope this work will both aid and inspire others to look in more detail at the process of Metallography and even encourage others to develop further consumables and advance this wonderful area of materials investigation.

The progress made over the last few years follows on directly from those early days of materials investigation and hopefully more will follow. What is important is that people need to be educated in the areas of materials preparation & how to use the available microscope techniques correctly to observe the detail provided by correct sample preparation

References

Bousfield, B. 1992. *Surface Preparation and Microscopy of Materials*. Wiley.

Clinging, V. 2009. Henry Clifton Sorby - *Sheffield's Greatest Scientist*, The Sorby Natural History Society.

Diez, D. 2013. *Metallography an Introduction* – Leica Microsystems publcation

Gifkins, R. C. 2001. *Materials Australia*. 33(1), 11-13.4).

Hammond, C., 1989. The contribution of Henry Clifton Sorby to the study of reflected light microscopy of iron and steel. *Historical Metallurgy*. 23(1), 1-8.

Hardwick, D. and Williams, W. M., 1980. The birth of metallography – The work of Henry Clifton Sorby (1826 - 1908). *Bulletin of the Canadian Institute of Mining and Metallurgy*. 73(813), 143-144.

Lamplan Publication – *Diamond Information sheet* - January 2006

Petzow. G – *Metallographic etching* - 1999 ISBN 0871708334

Raith M.M, Raase,P, Reinhardt.J – *Guide to Thin Section Microscopy* 2012 ISBN 978-3-00-037671-9 (PDF)

Rohr. N– Powder Particulars article - *Warren Diamond Powder Co. Inc*

Rosenhain, W. 1920. The metallurgical microscope. *Journal of the Royal Microscopical Society*. 40(4), 128-134.Rottenfusser- Wilson – Davidson – Contrast modes in Reflected Light Microscopy – *Zeiss Education Article*

Samuels, L. E. 1967. *Metallographic Polishing by Mechanical Methods*. Pitman & Sons, London.

Sheppard, T., 1906. *Prominent Yorkshire workers*. 1 - Henry Clifton Sorby. *The Naturalist*, May 1906, p.137-230.

Smithells *Metals reference book 8th Edition* - Page 988 – December 2003

Townsend. N - *Private email communication* – 15th October 2012

Vander Voort, G. 1999. *Metallography Principles and Practice*.

Vander Voort. G.F – *Color Metallography* – Microscopy Today - Nov 2005

Vilella, J. R. 1938. *Metallographic technique for steel*. American Society for Metals.

Acknowledgements.

I would first like to thank my employer MetPrep Ltd and in particular my Operations Director Keith Ashby for his steadfast and continued support. Be it time or resources he has made it possible for me to complete this diploma.

I would also like to thank my mentor Eric Bennet, my RMS diploma coordinator Kate Wooding and in particular Susan Anderson for their input, support and encouragement. The assistance has been particularly helpful in getting everything together in the format required.

Additional thanks must also go to Christine Kimpton & Andrew Dyer at the School of Applied Sciences Cranfield University. To Christine for her tireless assistance in obtaining the SEM material and to Andrew for allowing me regular access to the Olympus LEXT Laser Scanning Confocal Microscope. Without their a kindness, support and assistance much of the this investigation would not have been possible.

Thanks also to Leica for the use of the images of the various contrast techniques,

Finally I would like to thank my partner Debbie for putting up with the endless days of being ignored as well as having to listen to the endless murmurings of microscopical related material as I tried to put this project together.

I would also just like to add a small thank you note to Brian Bousfield who in my earlier years, now quite some time ago, helped me to develop my skills and encourage my enthusiasm in the microscopy of materials.