MetPrep Application Notes – Application Note – 003

The Preparation of Carbon Fibre Composites using a Semi-automatic Preparation System

Kevin Smith - March 2020

Introduction

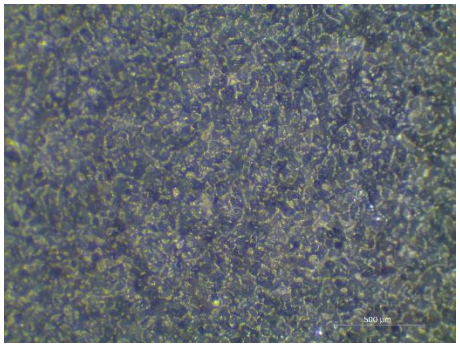
To prepare a sample for metallographic examination requires an understanding of both your sample properties and the properties of your consumables. This allows you to create a preparation procedure from first principles.

Carbon fibre composites do come in a variety of forms but they tend to be based on a ductile 'gummy' matrix with an assorted range of bundled fibres that are long, soft and brittle.

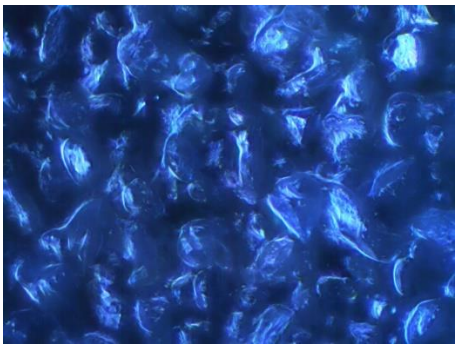
Primary grinding.

Primary grinding is employed for several reasons. Removal of damage created by sectioning, removal of excess mounting material which may have encroached over the sample surface, or if central pressure is being employed to get your samples planar. Let's consider the choices for the primary grinding stage.

Carbon Fibre Composites can be easily ground by Silicon Carbide, Zirconia and Fixed Diamond. It is known that Silicon Carbide is the sharpest abrasive and is also both very efficient and 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. If you have 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. 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 P320g (46 um) size would be an ideal compromise. The choice of grade will be dependent on the existing surface finish and the amount of material needed to be removed.



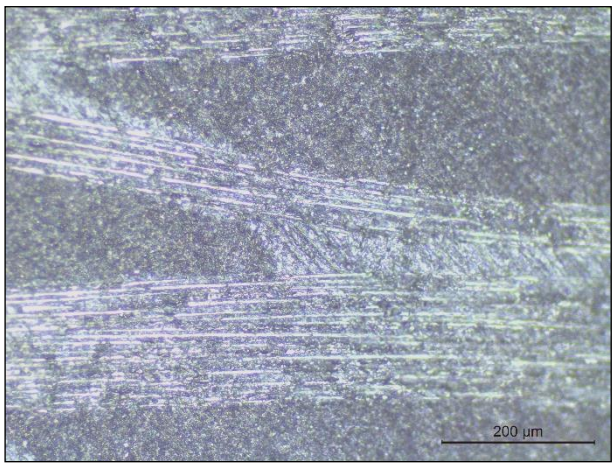
P320g Silicon Carbide – Stereo 40x



P320g Silicon Carbide – DMLM 20x Obj

Being a sharp abrasive silicon carbide will have more chance of cutting both the Carbon Fibres & the gummy matrix, in this case PEEK, 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. A clean sharp scratch pattern is clearly obtained. Due to the nature of the sample & the surface finish there is little to see apart from that the fibres can be seen in the matrix.

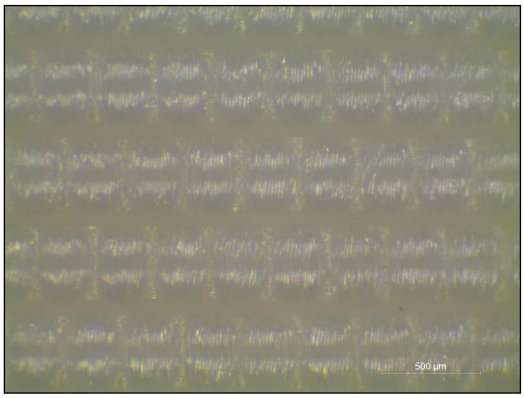


P320g SiC finishP240g - 10x Objective – Brightfield illumination

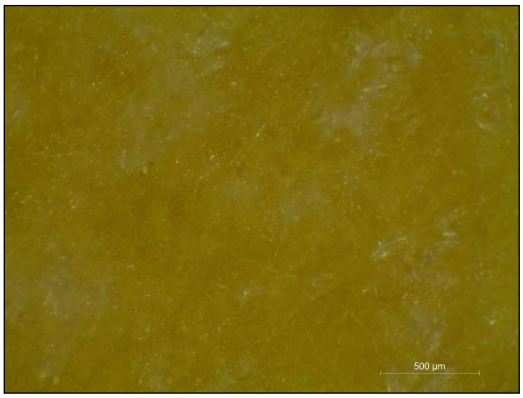
Secondary Grinding

As we are preparing the sample with a semi-automatic preparation system whilst we could continue through the grades of Silicon Carbide to maybe P1200g (16um) but this will require several paper changes and multiple washings.

Therefore, what are the options for a secondary grinding stage? MetPrep provide two cloths typically employed for secondary grinding stages, the coarse cross woven polyester - Abracloth and the more forgiving and less aggressive chemotextile - Planocloth H. Both cloths are hard wearing and ideal for the secondary grinding stages for metallic materials. As the Carbon Fibre Composite contains both soft fibres whose adhesion to the matrix is unknown, and has a matrix that is relatively soft it is good practice to err on the side of caution and use the less aggressive cloth, the Planocloth H. Care must also be taken regarding rubbing.

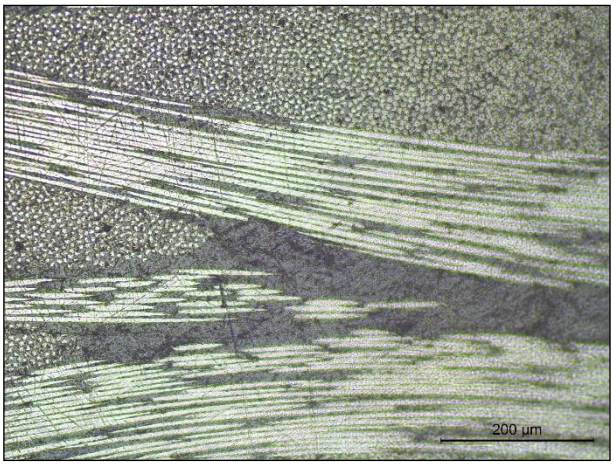


Abracloth 40x Stereo microscope



Planocloth H 40x Stereo microscope

Stepping down from a P320g (46 um) silicon carbide stage to a 9um diamond abrasive is a typical step but it will require some time to remove the damage from the previous stage. Whilst it may take a few minutes the stock removal rate is very consistent and no intermediate washing is required thus saving on time. 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 photomicrographs at this point will allow comparison to any further steps viewed. Having assessed the surface microscopically after 3 minutes at this stage the samples should be should further prepared for a couple more minutes and again examined accordingly. When microscopical examination confirms that further work is not improving the surface only then should a tertiary grinding stage can be considered.



Planocloth H - 9um finish - 20x Objective – Brightfield illumination

Tertiary grinding

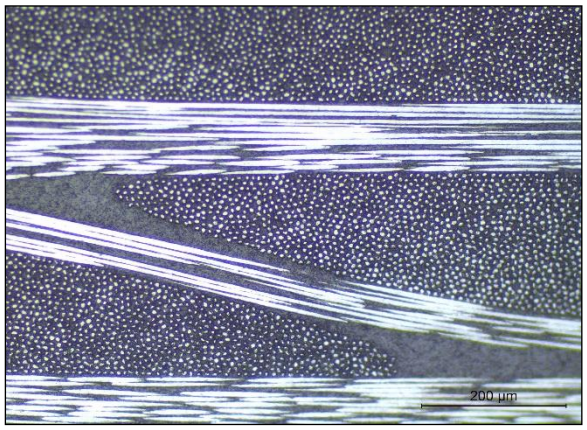
Having determined the sample is correctly prepared at the 9um stage we can now consider the tertiary stage. MetPrep provide a choice of three cloths useful the tertiary grinding stages of ductile materials, Planocloth, Nylap, and Durasilk. All these cloths are suitable for the tertiary stages but again there are differences. The more aggressive Planocloth that is chemotextile in nature could possibly cause rubbing which could be an issue with the brittle graphite nodules at this stage, the fine woven Durasilk is designed more for the softer ductile materials as it is less aggressive and less hardwearing, therefore the compromise cloth would be the Nylap. Nylap is a fine cross woven cloth and should remove material without leaving excessive damage in the more brittle fibres but still remove material from the whole sample efficiently. It will certainly have less chance of rubbing like the smoother chemotextile Planocloth option.

Planocloth 40x

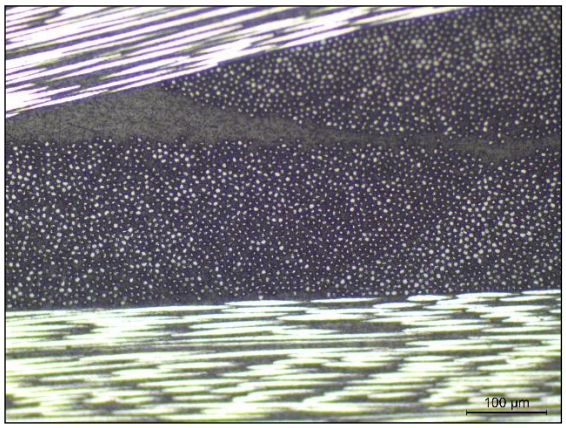
Nylap 40x

Durasilk 40x

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 is recommended. 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 now that 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.

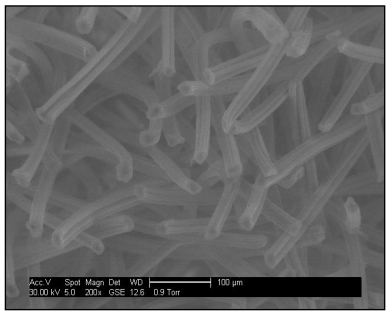


Surface finish at 3um Nylap 10x Objective - BF

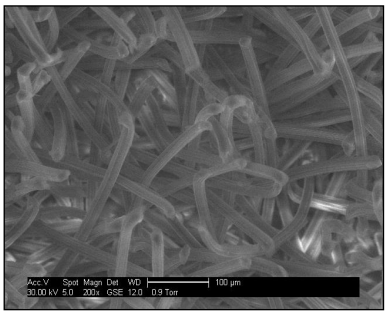


Surface finish at 3um Nylap 20x Objective - BF

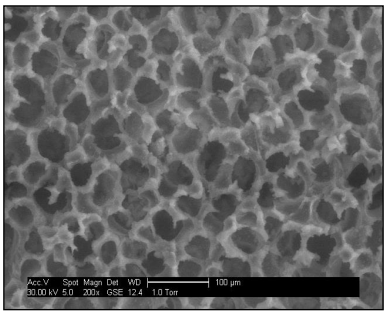
When it comes to a final polish with dissimilar materials in the sample it is best to consider a cloth with a short nap or no nap and a fine abrasive. From our microscopical evaluation we can see that Multicloth and Memphis are low napped in nature and will leave less relief when compared to the longer napped Alphacloth . In addition, the Chemicloth with Colloidal Silica is an option. The nature of the Chemicloth is completely different being more sponge like and having next to no nap but giving a good surface finish with increased flatness and reducing problems associated with polishing samples containing dissimilar materials.



Alphacloth SEM 200x



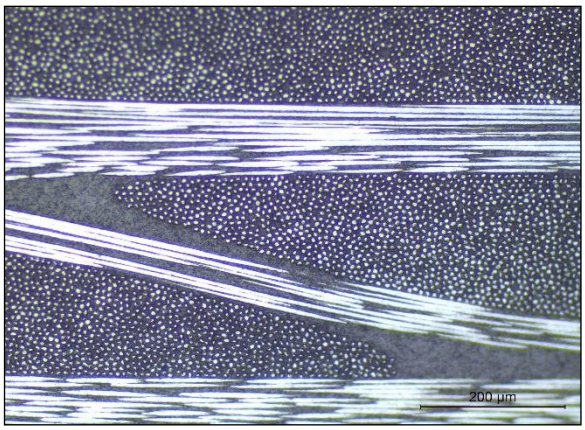
Multicloth SEM 200x



Chemicloth SEM 200x

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 isn't usually a problem. The result should be a scratch free surface, free of damage and showing the true microstructure. All will give a good final finish.

Whilst the 3um structure produced would be acceptable in most laboratories above there is still some considerable damage left. The matrix, which would clean up with a polishing cloth is still a bit damaged and whilst the fibres are reasonable in the longitudinal direction, the transverse ones are ill defined and not clear at all. Measurements taken in such a condition with additional possible rounding by the polishing stage would not be correct. Therefore, we need to consider an alternative tertiary grinding stage. A stage that will take the 9um finish from Planocloth H to a better quality finish for the tertiary condition.

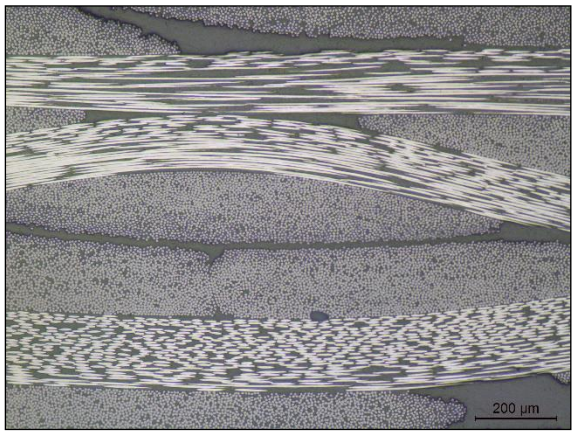


Surface finish at 3um Nylap 10x Objective - Brightfield .

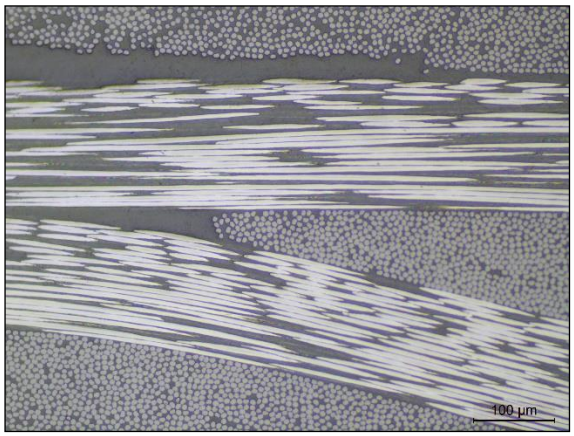
It is known that whilst diamond is hard it is not very sharp and it appears that the sample is not being ground efficiently with the Nylap / 3um diamond abrasive / surface combination. What is required is a sharper abrasive to cut both the gummy matrix and the carbon fibres

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 for such materials. Typically, at this stage a 0.3um sized abrasive size is used and with a hard tertiary grinding cloth such as the chemotextile Planocloth it is possible to remove the damage from the 9um Planocloth H stage and leave the sample flat and damage free. The nature of the suspension will reduce the possibilities of rubbing.



Planocloth 0.3um Alumina 10x Objective



Planocloth 0.3um Alumina 20x Objective

The resulting surface finish after the 0.3um Alumina grinding stage is now much cleaner. It is damage and scratch free and a final polish is actually not really required. Technically a polishing stage would tend to degrade the sample surface by introducing relief between the fibres and the matrix but a Multicloth with Colloidal Silica could be used if really necessary.

It is possible to argue here that with the 0.3um Alumina - Planocloth stage in the CFC procedure 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.

Completed Preparation Procedure for Carbon Fibre Composites

P.E.E.K. Carbon Fibre Composite

	Surface	Abrasive	Pressure		Speed – Dir	Time
Primary Grinding Stage	Paper	P320g SiC	Psi	N	150 – Comp	Until Planar
			5	25		

	Surface	Abrasive	Pressure		Speed – Dir	Time
Additional Grinding Stages	Planocloth H	9 μm (WB) Diamond	Psi	N	150 – Comp	4 mins
			5	25		
	Planocloth	0.3 μm Alumina	5	25	100 – Comp	4 mins

	Surface	Abrasive	Pressure		Speed – Dir	Time
Polishing Stage	Multicloth	0.06 μm Silco	Psi	N	80 – Comp	2 mins
			3	15		

Summary

By using the understanding of a materials properties and the properties of the metallographic consumables it is possible to prepare a sample of Carbon Fibre Composites correctly from first principles.

Understanding the difference of using cloths in both grinding modes as well has the polishing mode allows the use of multiple grinding stages effectively carried out with a semi-automatic preparation system such as the Saphir 250 A1 eco. Minimising time and improving quality is the main benefit of using such a system. Add to that removing operator variability, it is the ideal way to generate correct preparation results. It is also worth reminding oneself that an abrasive is just an abrasive and it depends on the surface it is used on as to how it will perform.

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