SUMNotes

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METALLOGRAPHIC PREPARATION OF FIBER REINFORCED COMPOSITES WITH/WITHOUT METALLIC INSERTS

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Introduction

Composites materials are finding wider applications in aerospace, building construction and automotive sectors attributed to their high strength, light weight, ability to mould complex shapes, and corrosion resistance among others. The most widely used type of composites are fiber reinforced polymers (FRPs). The reinforcing fibers generally used in FRPs are carbon or glass fibers but boron, Kevlar, silicon nitride, alumina fibers are also used. The reinforcing components can also be particulate-reinforced or structural as found in sandwich structures. Typically, these fibers make up about 60% of a PMC by volume. Composite materials also extend to metal matrix (MMC) or ceramic matrix (CMC) materials, which may have very different properties and therefore different preparation challenges but are not within the scope of this article. The following article will address preparation of FRP's and structural FRP's with metallic inserts. Structural composites are manufactured into hybrid laminate structures with the addition of metallic inserts to improve their strength compared to structures without metallic inserts. This has been shown by Fink et.al [1] to be up to 91% when compared to that of a full FRP laminate, Koleniskov et.al [2] also reported similar improvements.

Metallographic Analysis

Metallographic examination of fiber reinforced structures involves the analysis of separation between constituent components, porosity and transitions between components. Common problems with preparation are related to relief and lack of flatness between the different components. Moreover, certain composite structures with metallic inserts/component can smear the surface masking interfacial defects such as micro-voids/pores or cracks. Preparation procedures involve sectioning on an abrasive or precision saw, followed by mounting in appropriate mounting media where necessary and then grinding and polishing either manually or semi-automatically before macroscopic and microscopic analysis.

Sectioning

Sectioning of larger composite samples should be carried out on a specialized abrasive cutter using a diamond or abrasive wheels to attain a manageable size. The use of aggressive techniques such as band saws should be avoided, as they can cause severe damage to a great depth in the specimen. Even abrasive cutters may cause some damage, and so further re-sectioning on a precision saw is recommended [3]. Precision cutters, such as the IsoMet High Speed saw (left), cut with minimal force. Not only does this ensure that minimal damage is introduced, it also allows sectioning closer to the area of interest, reducing the amount of work needed in later stages. The effect of different cutting techniques on the resultant surface finish is shown in Figure 1, with the precision cut surface exhibiting excellent surface finish suitable for routine macro-analysis of the laminate structure.

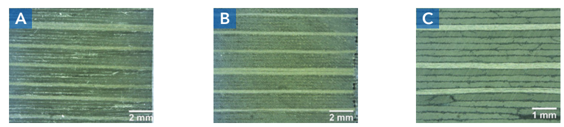

Figure 1 showing sectioning using different blades types with (a) diamond blade on abrasive cutter (b) thin abrasive blade on an abrasive cutter and (c) sectioning on a IsoMet high speed precision saw with a 15LC diamond wafering blade.

For structures containing metallic components, the thickness and material type of the inserts used will have a bearing on the blade type used for sectioning. For titanium or aluminium containing laminates will require a suitable non-ferrous blade for sectioning. Thus, a silicon carbide blade with a suitable bond should be selected for use on an abrasive cutter, whereas for a precision saw, a high concentration (HC) diamond blade would be recommended. The diamond micron size used should be around 10µm to 15µm to ensure a consistent cut through the metallic insert whilst presenting a surface with minimal damage on both metallic and polymeric structure.

Mounting

Where samples have to be mounted before grinding and polishing stages, these must be cleaned and dried to improve mounting media wettability, as well as minimising the shrinkage gaps that may otherwise form after curing. For mounting fiber reinforced composite structures, low curing and low viscosity epoxies, such as EpoThin 2 or EpoxiCure 2 are recommended. Use of vacuum equipment such as the Cast 'n Vac (right) for mounting, and pressure during the cure cycle, is also recommended for better impregnation of the resin into the composite structure. Addition of dyes/pigments to the resin improves contrast during optical analysis and helps facilitate identification of cracks or pores. Avoid hot compression mounting due to the high pressure and cure temperatures that would affect the integrity of composite structures.

Grinding and Polishing

SiC paper is commonly used for the initial grinding steps but it must be noted that for composite structures with varying differences in hardness (low to high) of constituent components, SiC paper might not be ideal and diamond grinding discs should be used. For fiber reinforced composites, a typical 4-step procedure as recommended by Buehler SumMet [6] is given in table 1. This procedure starts with a 320grit (P400) SiC paper followed by a rigid hard non-woven perforated disk TexMet P using 9µm diamond suspension. This removes the damage caused after step 1, but also ensures the resultant surface finish has excellent flatness. This is followed by a medium hard woven polishing surface VerduTex cloth with 3µm diamond suspension and finishing with 0.05µm alumina step on a napped cloth such as MicroCloth.

Semi-Automatic preparation method for composites (PMC's) - *Table 1*

Step No.	Surface	Abrasive	Lubricant/ Extender	Force (Per Specimen)	Time (min:sec)	Platen Speed (rpm)	Head Speed (rpm)	Rotation
1	SiC Paper	320grit (P400)	Water	25N	until plane	300	60	>>
2	TexMet P	9µm MetaDi Supreme	MetaDi Fluid	25N	05:00	150	60	><
3	VerduTex	3µm MetaDi Supreme	MetaDi Fluid	25N	04:00	150	60	>>
4	MicroCloth	0.05 µm MasterPrep (Alumina)	•	25N	01:30	150	60	><

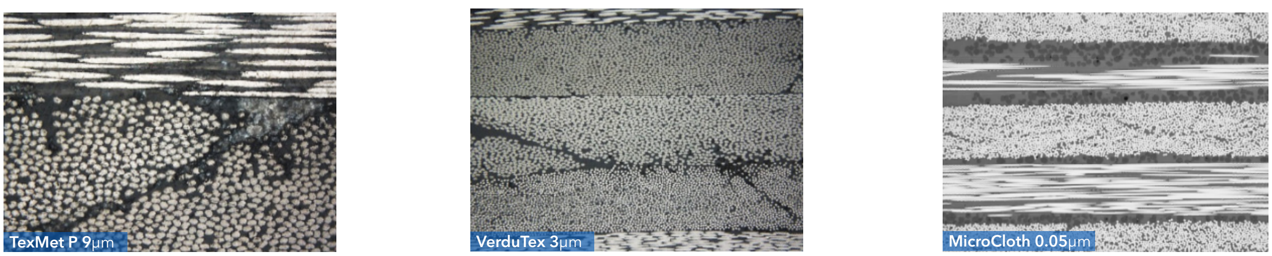

Figure 2 showing surface finish after semi-automatic preparation after 9µm, 3µm and 0.05µm step.

Figure 2 illustrates the change in surface finish after step 2, 3 and 4, with the latter step resulting in a scratch free surface and retaining excellent fiber geometry from cross-sectional view and the desirable optical interferometry bands on the ends of longitudinally lying fibers for validating flatness after polishing. For manual preparation, Table 2 illustrates a 4 step procedure involving two grinding steps with SiC paper followed by a polish using 0.3µm alumina on TexMet C before a final polish using 0.05µm alumina on Microcloth. The force applied is light to medium during grinding and medium to light for polishing stages. Surface finish after the 0.3µm alumina step is generally good for standard analysis. Figure 3 below shows the surface finish after hand preparation.

Manual preparation method for composites (PMC's) - *Table 2*

Step No.	Surface	Abrasive	Lubricant/ Extender	Force (Per Specimen)	Time (min:sec)	Platen Speed (rpm)	Rotation
1	SiC Paper	320grit (P400)	Water	light - medium	until plane	200	><
2	SiC Paper	600grit (P1200)	Water	light - medium	01:30	200	><
3	TexMet C	0.3µm Alumina	MetaDi Fluid	medium	04:00	150	>>
4	MicroCloth	0.05 µm MasterPrep (Alumina)	•	light	01:30	100	><

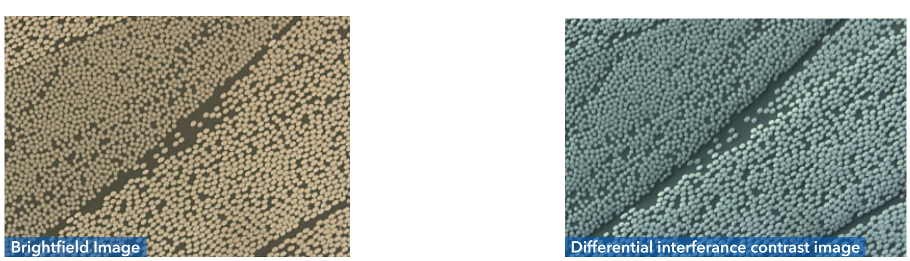

Figure 3 showing surface finish after 0.05µm alumina on Microcloth prepared by manual preparation.

Glass fiber reinforced composites present different challenges during metallographic preparation. The glass fibers are prone to cracking due to their brittle nature. Table 3 presents a procedure that ensures good flatness whilst ensuring minimal or no damage to the fibers. Use of diamond suspensions could also result in fiber damage and should be minimised and where employed, ensure subsequent steps recover the damage caused by preceding coarser step.

Semi-Automatic preparation method for composites (PMC's) - *Table 3*

Step No.	*Surface*	*Abrasive*	*Lubricant/ Extender*	*Force (Per Specimen)*	*Time (min:sec)*	*Platen Speed (rpm)*	*Head Speed (rpm)*	*Rotation*
1	SiC Paper	320grit (P400)	Water	20N	until plane	250	60	>>
2	SiC Paper	400grit (P800)	Water	20N	01:00	250	60	>>
3	SiC Paper	600grit (P1200)	Water	20N	01:00	250	60	>>
4	TexMet C	1 µm MasterPrep (Alumina)	•	20N	02:00	150	60	><
5	MasterTex	0.05 µm MasterPrep (Alumina)	•	20N	02:00	100	60	><

Figure 4 illustrates glass fiber reinforced structure prepared semi-automatically on an EcoMet/AutoMet grinder polisher using the procedure in table 3.

Figure 4 (a) low magnification and (b) high magnification showing glass fiber reinforced composite (GFRP) after 0.05µm alumina polishing on Microcloth prepared semi-automatically on a grinder polisher.

Typical applications of FRC's with metallic components

For FRC structures with metallic inserts, metallographic preparation would follow procedures shown in Table 1 but modified slightly to ensure that both the FRC and metallic components are polished flat and without deformation or damage. For titanium inserts, step 2 can be replaced with UltraPad, a hard woven no nap cloth instead of TexMet P followed by 3µm on VerduTex and finishing off with 0.05µm MasterMet colloidal silica for 5 – 8min on ChemoMet. For aluminium inserts follow procedure in Table 1 with an additional step for 1µm on VerduTex and replacing the last step with 0.05µm MasterMet colloidal silica. For an excellent finish on titanium inserts, attack polish using hydrogen peroxide can be added with MasterMet colloidal silica that aids in removing remnant scratches and deformation.

Honeycomb structure

A honey comb structure, Figure 5, is characterised by a composite panel sandwiching a honeycomb core, either titanium or aluminium, resulting in a structure with low density and high out-of-plane compression and shear properties. These are widely used in aerospace and metallographic inspection is generally carried out to check the integrity of the bond between the honeycomb core structure and the fiber reinforced composite outer skin, defects in the composite skin such as voids and the presence of cracks. Defects in the bond and/or in the fiber reinforced structure present failure initiation sites that could propagate to catastrophic failures in-service.

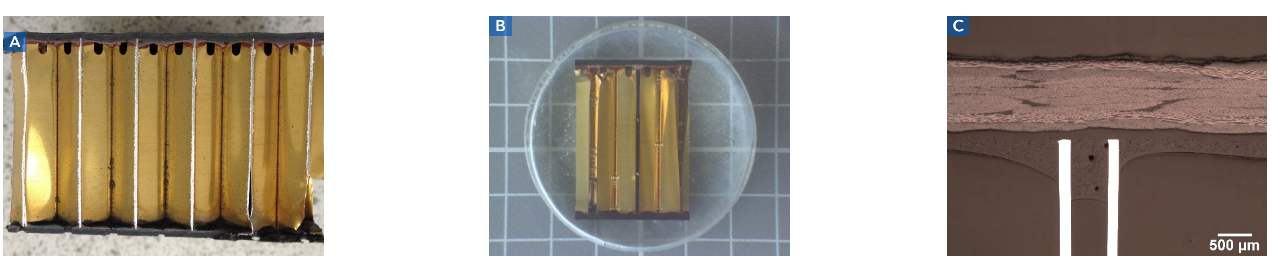

Figure 5 shows (a) sandwich composite structure, (b) mounted in EpoThin LT and (c) after grinding and polishing procedure illustrating the CFRP outer skin adhesively bonded to aluminium honeycomb core

Carbon fiber reinforced composite titanium laminate

These types of laminates, also referred to as hybrid composites, are increasingly being used in aerospace and aircraft structures due to their light weight capability. They are joined to other aircraft structures through mechanical fastening, which offers best reliability and allows easy detachment for inspection [4]. The addition of metallic inserts provides additional strength that would otherwise be lacking for a fiber reinforced structure.

To mechanically fasten the laminate, holes have to be drilled through the hybrid structure, Figure 6. This presents another challenge of ensuring no cracks or significant damage occurs at the interface between the CFRP and titanium inserts and can be validated through metallographic testing. If the titanium microstructure is of interest when investigating the drilled holes, an attack polish using hydrogen peroxide combined with polarised light microscopy will show general grain structure. Figure 6(b) shows the fiber reinforced composite (top) and titanium substrate (bottom) laminate structure after final polishing. Figure 6(c) illustrates the CFRP as polished with the layered structure illustrating different fiber layer orientations.

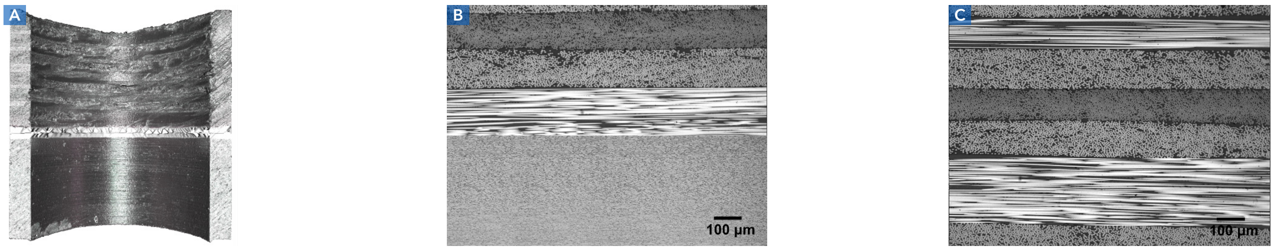

Figure 6 (a) shows a drilled hole on carbon fiber reinforced composite with titanium Ti6Al4V metal insert after semi-automatic preparation. The method shown in table 1 was modified by changing the last step using MasterMet colloidal silica for 5minutes on ChemoMet cloth surface.

Summary

This article has provided an overview of metallographic preparation of fiber reinforced components by providing best practices during sectioning, mounting and grinding/polishing stages in a preparation sequence.

It has also highlighted different preparation procedures for carbon and glass fiber reinforced composites for both manual and semi-automatic preparation and modified procedures for laminates containing metallic inserts.

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