BUILDING A COMPOSITES INFRASTRUCTURE (or "GETTING COMPOSITES USED - PART 1")

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SUMMARY: An apparently minor issue having a major impact on the use of composites in new areas is the ease that specifiers and purchasers can order composites to a recognised specification. This is difficult for composite materials due to a lack of international test methods and specifications standards equivalent to those existing for metals. This infrastructure will be needed to show compliance with EU Directives and other regulations.

The situation will be substantially improved by work undertaken currently by ISO (International Standards Organisation) and CEN (Comité Européen de Normalisation) to satisfy these needs. Several international test methods published recently provide the building blocks for a database standard, ISO 10350-2, and specification standards for thermosets (eg. sheet moulding compounds, SMC), thermoplastics (eg. glass mat thermoplastics, GMT) and pultruded profiles. These development will bring cost savings, provide new opportunities and confirm the increasing maturity of the composites industry.

KEYWORDS: standards, test methods, specifications, infrastructure, ISO, CEN, ASTM

INTRODUCTION

It is easiest to introduce a material when a well established infrastructure exists consisting of,

- test methods
- material specifications (eg. alloy specifications)
- validated design data
- established design procedures
- established inspection procedures
- history of use

These facets have been established over an extensive period for metals mainly by a process of evolution so that an infrastructure exists to support all involved in the design and use of engineering metals. This is important because it allows, for example, a purchaser to order easily from a general supplier a metal section in a particular alloy with a designated heat treatment. This infrastructure is not generally available for reinforced plastics/composites, although for traditional glass-fibre reinforced plastics (GRPs), some facets are available.

If we make the distinction that the term “composite materials” is often applied to the high performance end of the spectrum of the range of materials; the situation there is even less developed but perhaps having greater potential due to a higher level of standardisation of products as fibre and matrix are more often supplied as an integral product (ie a preimpregnate). This paper reviews some of the international infrastructure aspects established to underpin the development of the composites industries’ potential. It includes some aspects of national work but does not review the extensive work by others, such as trade groups (eg. SAE) that hopefully will contribute to future international standards.

PRODUCT TRACEABILITY AND APPROVAL

To demonstrate the validation of a design (ie to regulatory bodies, such as the CAA or FAA) or to meet the requirements of an EU Directive, a hierarchy of test methods and specifications are needed at several levels from constituent materials (fibres and matrices) to products. As the need to demonstrate compliance with safety orientated EU directives increase, the pyramid of substantiation often quoted for aircraft certification will become increasingly familiar in other application areas. A simplified “chain” of validation showing the alternating need for each specification or product level to be based on appropriate test methods, is shown in Fig. 1. In addition, product approval requires approved inspection and repair procedures to ensure continued safe operation of the product.

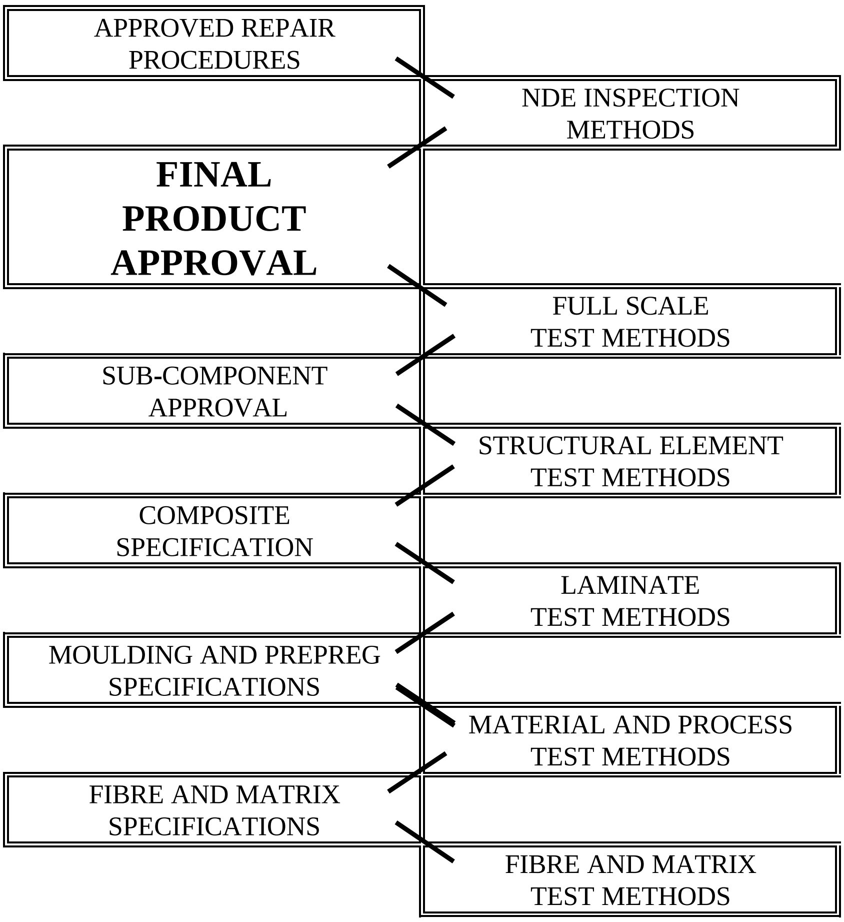

Fig. 1: Chain of validation for composite products based on alternating specification and test methods standards

FIBRES AND MATRICES - TEST METHODS AND SPECIFICATION

For glass-fibres and resins (both thermoplastic and thermoset) there has been available a fairly complete sets of ISO standard test methods and resins covering both testing and specification requirements. However, the situation was less satisfactory for carbon-fibres and aramid fibres. There was only sufficient interest initially to support work on test methods for carbon-fibres. Several work items were lead by Courtaulds, who are no longer producing carbon-fibres. This work lead to several basic test methods covering linear density, twist, diameter, tensile strength of filaments and impregnated yarns.

Following the publication of these standards, ISO TC61/SC13/WG12 has been successfully developing non-specific fibre test methods (eg. linear density) by unifying the procedures at the test method level. This avoids the long gestation period before material specific standards can be prepared for new fibres. Of course, this will not apply in all cases depending on the characteristics of the property under consideration.

These standards will be adopted for the EN General series. In addition, the European Aerospace series, being developed by AECMA (European Association of Aerospace Industries) on behalf of CEN, has adopted all the ISO carbon-fibre test methods; a useful step towards harmonisation. However, AECMA is still progressing its own version of the ISO standard, (ISO 13002 [1]), covering carbon-fibre designation, an area that illustrates very effectively the need for a single harmonised procedure. This need drove the selection in the ISO standard of Method B (based on strain) for determining the code number relating to modulus in the designation. The alternative, Method A (based on N/tex values), gives different and higher results. Both are allowed in the yarn tensile test standard (ISO 10618 [2]) as the specialist carbon-fibre committee did not originally agree on a single method. This situation can be avoided by indicating in the test standard the reference method to be used.

In addition, there are several standards covering both test methods and specification aspects for glass-fibre products such as mats, rovings, fabrics and woven rovings. These do not yet exist for carbon-fibre fabrics, although a new work item was proposed in CEN to develop standards for multiaxial reinforcements, which will apply to all fibre systems.

Table 1 Test methods for reinforcing fibres

Property	Range of applicability		
	Glass-Fibre	Carbon-Fibres	All Fibres
Density	ISO 1888	ISO 10119	
Linear Density			ISO 1889
Twist			ISO 1890
Diameter		ISO 11567	
Breaking force - single filament		ISO 11566	
Breaking force - yarn	ISO 3341	ISO 10618	
Size content	ISO 1887	ISO 10548	
Designation yarns	ISO 2078	ISO 13002	

MOULDING COMPOUNDS AND PREIMPREGNATES

Although, fibre and matrix are sourced separately in many cases (eg. hand and spray lay-up, RTM and filament winding process routes), an increasing amount is likely to be sourced as moulding compounds or preimpregnates. Moulding compounds are available with both thermoset (eg. sheet moulding compounds, SMC) and thermoplastic (glass-mat thermoplastics, GMT) matrices. These materials are compression moulded under appropriate conditions. Standards are being developed, principally within CEN, covering the specification for these materials and for the important process parameters. The area of advanced preimpregnates is not so developed, although AECMA has work items in this area (eg prEN 2833 [3]), which ideally could be adopted by the CEN General Series providing the scope can be made sufficiently wide and appropriate ISO standards referenced. Some examples of specification standards and process measurement standards are given in Table 2.

Table 2: Moulding compound/pre-impregnates - typical specification and test methods

Specifications	Test Methods
prEN 2833 Aerospace - Reinforced Plastics - Glass-fibre pre-impregnates prEN yyyyy Reinforced plastics composites - Specifications for thermoset moulding compounds (SMC, BMC, DMC) prEN yyyyy Reinforced plastics composites - Specifications for thermoplastic moulding compounds (GMT)	EN ISO 12115 - SMC Flowability, masturation and shelf life. EN 1842 Prepreg and randomly reinforced compounds - Linear shrinkage EN ISO 12114 - SMC Curing characteristics prEN yyyyy Determination of flowability

TEST PANEL FABRICATION

At the next higher level of testing, tests are conducted using coupons cut from the product or from a test panel. ISO 1268 [4] is being revised to cover all process routes for long-fibre composites (see Table 3), supported by existing ISO standards (i.e. ISO 293-295) for short fibre composites. The procedure used in prEN 3074 [5]for designation of the ply orientations, hopefully harmonised with the code given in ASTM D 5687 [6], will be offered as an annex to Part 1 of ISO 1268. ISO 1268 will form also the EN General Series standard in this area.

Only Part 4, prepared by the author, includes precision data [7] that was obtained through supplying preimpregnates to eight establishments who prepared panels at three thicknesses. The panels when evaluated at NPL for a range of physical and mechanical properties showed a good level of consistency as given by the repeatability and reproducibility precision data.

Table 3: ISO 1268 for test panel manufacture.

	Title
Part 1	General principles
Part 2	Contact and spray up moulding
Part 3	Wet compression moulding
Part 4	Moulding of preimpregnates
Part 5	Filament moulding
Part 6	Pultrusion moulding
Part 7	Resin transfer moulding
Part 8	Moulding of SMC/BMC
Part 9	Moulding of GMT/STC
Part 10	Injection moulding of BMC/DMC

An important issue not covered satisfactorily is the machining of composites and specimen preparation. Some minimal instructions are included in the test methods described below as a common annex. Some further information is given in the ASTM standard for test panel manufacture, ASTM D 5687 [6]. It is hoped that a current UK project can provide the basis of a more detailed document (cf. ISO 2818 - Machining of Plastics [8]).

HARMONISATION OF EN ISO COUPON TESTS WITH ASTM

An important new set of standards for laminate mechanical test methods, see Table 4, were published in the period 1997-1999 that were harmonised with, and in one case based on, ASTM equivalent methods. These standards should result in considerable cost savings for the composites industry and encourage new opportunities[9, 10].

Table 4: Harmonised Coupon Test Methods

Number	Title
EN ISO 527 - Part 1	*Plastics - Determination of tensile properties - General principles*
EN ISO 527 - Part 4 (ASTM D 3039)	*Determination of tensile properties - Test conditions for isotropic and orthotropic fibre-reinforced plastic composites.*
EN ISO 527 - Part 5 (ASTM D 3039)	*Plastics - Determination of tensile properties - Test conditions for unidirectional fibre-reinforced plastic composites*
EN ISO 14125 (ASTM D 790)	*Fibre-reinforced plastic composites - Determination of flexural properties*
EN ISO/FDIS 14126 (ASTM D 3410)	*Fibre-reinforced plastic composites - Determination of the in-plane compression strength*
EN ISO 14129 (ASTM D 3518)	*Fibre-reinforced plastic composites - Determination of the In-plane shear stress/shear strain, including the in-plane shear modulus and strength, by the ± 45° tension test method.*
EN ISO 14130 (ASTM D 2344)	*Fibre-reinforced plastic composites - Determination of apparent interlaminar shear strength by short-beam method.*

Other coupon tests at different stages of drafting and balloting under progress concern plate twist (ISO 15310 [11]), fatigue (ISO/CD 13003 [12]), and Mode I delamination fracture energy (ISO/DIS 15024 [13]). Work on a Mode II fracture energy test is expected to be initiated shortly following a VAMAS (Versailles Advanced Materials and Standards) round-robin programme aimed at identifying the preferred method for standardisation [14]. There is also increasing interest in physical-chemical test methods (eg. Differential Scanning Calorimetry - ISO 11357 [15] and Dynamic Mechanical Analysis - ISO 6721 [16]) related to the desire for direct measurements of cure, thermal behaviour, etc. rather than using indirect techniques (eg interlaminar shear strength). NPL has a project aimed at providing a "good practice guide" for these methods, including both additional guidance on their use and round-robin precision data.

PRELIMINARY DATABASE STANDARD FOR COMPOSITE MATERIALS

The above test methods support the database standard (ISO 10350-2 [17]), aimed at technical data sheet requirements, which provides a useful reference source for recommended test methods. ISO 10350 gives numbered entry lines for each property, identifies the standard to be used and the preferred conditions if options exist in the standard. Part 2 relating to composites is expected to be as influential as Part 1 of the standard was for plastics, encouraging the use of ISO test methods to achieve comparable data.

To develop a more design orientated database will require additional properties, such as, the through-thickness properties. NPL has researched the test methods that could be used and has prepared drafts on direct tensile and compression tests using plain or tailored blocks [18]. ASTM is developing a "right-angled" specimen that develops a maximum transverse tension stress when loaded by increasing the internal angle (ie. flattening the section). As for other specimens loaded in basically a flexure mode, the state of stress is multi-axial so that some interaction of the various stress is possible. There is concern also that the specimen will be susceptible to fabrication difficulties due to the tight radius specified (NB unless this is the radius occurring in practice).

STRUCTURAL ELEMENT TEST METHODS (OHC, CAI ETC)

At the next level, attention is focused on quantifying stress concentrating features that occur in many designs. NPL has researched test methods for pin-bearing strength (PB) open/filled hole compression (OHC) and open-hole tension (OHT). In each case, a 6 mm diameter hole in a 36 mm wide coupon is used, or an Imperial unit equivalent based on 0.25" diameter hole and a 1.5" wide coupon (ie a specimen width/hole diameter of 6/1) [19, 20].

Although the pin-bearing test is used for aerospace material, there is a need for this information for all types of composites. In fact, the first mandatory use of the pin-bearing test is likely to be in the specification standard for pultruded profiles. ASTM D 5961 [21] is similar to the pin loaded test, but uses a finger tight torqued bolt. NPL is concerned that this approach, while not attempting to represent the real level of torque applied and not withstanding the effect of stress relaxation, applies a range of torque loads that may be in a region when the performance of the test is most sensitive to bolt torque and the failure point is unclear. The preference in

NPL's view is to conduct, firstly, the pin-bearing test to determine a "material" property. Secondly, to repeat the procedure for the actual materials, plate thicknesses, bolt/rivet material and diameter, washer type and applied torque appropriate to the application, as these parameters are too wide ranging to be represented by a "standardised" version.

SPECIFICATION STANDARDISATION FOR PULTRUDED PROFILES

Sub-components, such as sandwich laminates and pultruded profiles have wide applicability. Pultrusions, fairly uniquely for composites, are available as finished product in stock sections and sizes, but also in an increasing range of special profiles. Specification standards for structural pultruded profiles (eg box section, I beams) were identified by the European pultrusion industry as an essential requirement for increased use of their products.

Many new opportunities are expected from the ability to order pultrusions to an agreed international specification with minimum properties specified for each grade. These trends will be further increased by health and safety regulations that will encourage closed open mould processing. The percentage of GRP produced by pultrusion in Europe is likely to rise from the current 3% towards the 12% found in the USA.

The new standard was undertaken by the materials committee, CEN TC249/SC2/WG6, which in part recognised the use of pultruded profiles in a wide range of applications. It was agreed that the structure used for other ISO/CEN specification was suitable in the case of pultruded profiles. By adopting the 3-part structure and approach already used for other specification standards, rapid and effective progress was made in drafting the new standard [22].

In Part 1 of the standard (prEN yyyyy Fibre Reinforced Plastics - Specifications for pultruded profiles) is given the "designation" that identifies the basic components used (eg profile shape, fibre and resin type) with the material modulus obtained in a full profile section flexural test providing the code number indicating the grade (eg. Exx). Part 2 gives information on test methods and general recommendations regarding defects etc. that are used to demonstrate compliance with the requirements given in Part 3 - Specification. Part 2 includes reference to manufacture of test panels, dimensional tolerances, visual defects and workmanship are also covered in Part 2. The property data required in the Part 3 technical specification to show compliance are obtained from the standard laminate test methods discussed above. Currently two grades are proposed, with a list of minimum mandated properties for each grade.

PRODUCT STANDARDS - DESIGN CODES

The principle level of standardisation concerns component and product specification. This is the main area of standardisation of concern in CEN due to the use of these standards in support of the safety-orientated EU Directives. This interest balances the preference in CEN for test methods to be principally prepared in ISO.

There are several committees working on product standardisation, which may be fully composites (e.g. GRP pressure vessels - CEN TC 210 and GRP piping - CEN TC155) or an area where composites can compete alongside its competitors (e.g. access engineering - CEN

TC 114 [23]). Several documents are being balloted including specialist test methods, such as, for chemical resistance testing a section of product rather than a coupon.

In addition, an ISO committee has been initiated under TC67 on off-shore GRP piping based on the document developed by the UK Offshore Operators Association [24]. Harmonisation, or complimentary action, of CEN and ISO work in the general area of GRP piping is obviously important. Other product standards, such as for access engineering, which includes ladders, walkways and handrails, are also under development. These standards are open to all materials that can meet the technical requirements (eg. maximum acceptable deflections under prescribed loads) such as steel sections or pultruded profiles.

There may be additional tests requirements to support this level such as the chemical resistance of material samples cut from the GRP vessel or tank [18] or tests on full components (eg. filament wound gas cylinders).

APPROVAL OF INSPECTION AND REPAIR PROCEDURES

At this level, it is necessary to inspect final products and to develop repair procedures. A recent NPL led project that assessed the standardisation needed for the ultrasonic C-scan technique, has resulted in three procedures [25] that have been tabled at BSI as new work items. Both national and international round-robin validation is being undertaken. In addition, work is underway on developing repair procedures. For example, within the CACRC (Commercial Aircraft Composites Repair Committee) project there are tasks forces on several aspects, including material approval, operator training etc.

CONCLUSION

The above discussion has reviewed the work being undertaken internationally to create the essential infrastructure needed to enable the composites industry to complete effectively with other materials. The work relies on a high degree of co-operation between standards bodies principally through ASTM, CEN, ISO and JIS.

The issues discussed included:

- test and specification standards for fibres and matrices,
- new EN ISO coupon test methods harmonisation with ASTM,
- development of through-thickness test methods to complete the 3D database,
- structural element test methods and in-service representations (OHC, CAI etc),
- preliminary database standard for composite materials,
- specification of standardisation of sub-elements, such as pultruded profiles,
- development of design codes (prescriptive or performance based ?),
- inspection and repair techniques.

The degree of completeness being obtained is highlighted by the typical examples shown in Table 5.

Table 5: Examples of specification and test method standards at each level

Level	Specification Standards	Test Method Standards
Constituents (fibres, matrix)	prEN 12654, Textile glass - Yarns (Specification for) ISO 3672-1 - Plastics Unsaturated polyester resins. Designation and basis of specification	ISO 3341 Textile-glass - Yarns Determination of breaking force and breaking elongation. ISO 1890 Reinforcement Yarns - Determination of twist
Moulding compound	prEN yyyyy Reinforced plastics - Specifications for thermoset moulding compounds (SMC, BMC, DMC)	EN ISO 12115 -SMC Flowability, masturation and shelf life
Laminate	prEN 2833 Aerospace - Reinforced Plastics - Glass-fibre pre-impregnates	EN ISO 14125. "Fibre reinforced plastic composites - Determination of the flexural properties" (1998).
Structural Feature	N/A	prEN Annex 4 Fibre reinforced plastic composites Determination of the pin-bearing strength ASTM D 6921, Determination of bolt bearing strength
Sub-component	prEN yyyyy Reinforced plastics composites - Specifications for pultruded profiles	prEN Annex 5 Full section bend tests
Product	ISO/D 14692 GRP Piping for off-shore use prEN 13121 GRP Tanks and vessels for above ground use	prEN 977 Underground tanks of GRP - Method for one side exposure to fluids

This infrastructure of specification and test method standards will serve the increased needs due to the maturity of the industry, and the needs for traceability for more demanding applications and to meet the requirements of the European Union directives.

REFERENCES

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2. ISO 10618, Carbon fibre - Determination of tensile properties of resin-impregnated yarn.
3. prEN 2833, Aerospace - Glass-fibre thermosetting preimpregnates - Technical Specifications.
4. ISO 1268, Fibre reinforced plastics - Test plate manufacturing methods.
5. prEN 3074, Aerospace - Drawing annotation for composite laminate structure.
6. ASTM D5687, Standard guide for preparation of flat composite panels with processing guidelines for specimen preparation.

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23. prEN 12437, "Safety of machinery - Permanent means of access to machines and industrial plant".
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26. prEN 977 Underground tanks of GRP - Method for one side exposure to fluids.

Standards, including the additional ones listed in tables, can be obtained from national standards bodies who maintain up-to-date indexes. Titles and recommendations are also available from www.npl.co.uk/cog/index.html.