ABSTRACT

This report reviews progress on standardisation of test methods and material specifications and product standards during the period April 2001 to March 2002. The report only considers those materials containing “long” fibres. Long fibre composites are now defined in ISO standards as those containing fibres with a length greater than 7.5 mm in the starting material or compound. This is before processing whereas some processes, such as, injection moulding can reduce the fibre length; and includes all continuous fibre formats (eg swirled mat, fabrics and unidirectional plies. There are several levels of increasing complexity that contribute to the traceability of any final product. Progress is reviewed against the different areas listed below,

- Constituent material specifications and test methods
- Compound specification and test methods
- Coupon level test methods
- Composite material database standards
- Structural element test methods
- Sub-component specifications
- Product approval standards.

This review will be produced annually as part of Project MMS1 on “implementation of UK work under the materials measurement programmes as international standards”. The outputs suitable from standardisation have arisen from the prior Performance of Adhesive Joints (PAJ) and Composites Performance and Design (CPD) programmes, and will be followed by output from the current Measurement for Materials Systems (MMS) programme. The MMS1 project includes round-robin validation of test methods developed in CPD and PAJ, convenorship of two CEN working groups and the project leadership of several “UK owned” international standards, as detailed on the NPL Composites Group web site [www.npl.co.uk/cogi/index.html]. Enquiries can be sent to coil@npl.co.uk.

The report was prepared as part of the research undertaken at NPL for the Department of Trade and Industry’s Measurements for Material Systems programme.
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APPENDIX A STANDARDS FOR FIBRE REINFORCED PLASTICS 14
1. INTRODUCTION

This report reviews progress on standardisation of test methods and material specifications and product standards during the period April 2001 to March 2002. The report only considers those materials containing “long” fibres. Long fibre composites are now defined in ISO standards by the NPL (GDS) proposed wording “as those containing fibres with a length greater than 7.5 mm in the starting material or compound”. This is therefore before processing when some processes, such as, injection moulding can reduce the fibre length and includes all continuous fibre formats (e.g., swirled mat, fabrics and unidirectional plies). Composites containing “short” fibres are treated for both test method and database purposes alongside unfilled and filled (e.g., particulate) plastics. However, material with discontinuous fibres, say, between 7.5 mm and 12 mm long will be tested in specimen designs similar to “short” fibre systems. In Section 2 the overall requirements for an infrastructure of standards and codes is briefly reviewed. There are several levels of increasing complexity that contribute to the traceability of any final product. Progress is reviewed in Section 3 against the different levels, as listed below,

- Constituent material specifications and test methods
- Compound specification and test methods
- Coupon level test methods
- Composite material database standards
- Structural element test methods
- Sub-component specifications
- Product approval standards.

The work is identified via the lead body, although most test work in ISO is eventually published as triple numbered BS EN ISO standards. The main bodies responsible for standardisation in this field are ISO, CEN – general, CEN Aerospace, ASTM, JIS and other national bodies. In this first annual review the standardisation bodies and standards ballot approval procedures are reviewed in Section 4. A major issue discussed is the effect on standardisation work of the increased financial pressure on standardisation organisations, such as AFNOR.

This review will be produced annually as part of Project MMS1 on “implementation of UK work under the materials measurement programmes as international standards”. The outputs suitable from standardisation have arisen from the prior Performance of Adhesive Joints (PAJ) and Composites Performance and Design (CPD) programmes, and will be followed by output from the current MeasurementS for Materials Systems (MMS) programme. The MMS1 project includes round-robin validation of test methods developed in CPD and PAJ, convenership of two CEN working groups and the project leadership of several “UK owned” international standards, as detailed in the NPL Composites Group web site [www.npl.co.uk/cogi/index.html]. Further details enquiries can be sent to coil@npl.co.uk.
2. **INFRASTRUCTURE REQUIREMENTS**

As the harmonisation of practices within the EU and the need to demonstrate compliance with safety orientated EU directives develops, there is a need for a supporting infrastructure at all levels (i.e. from constituents to final application, including in-service maintenance and repair), so that the pyramid of substantiation often quoted for aircraft certification will become increasingly familiar in other application areas. A “validation chain” can also be used to show, see figure 1, the alternating dependence of specification and test methods standards at each level. In this report a cross-section of documents representing different approaches and different applications are reviewed.

![Diagram of validation chain for composites products](image)

**Figure 1: Chain of validation for composites products**

Standardisation was seen as one of the major, and longest threatening issue limiting the advance of the industry in a recent DTI survey conducted by NPL [1].

3. **PROGRESS IN STANDARDISATION**

3.1 **CONSTITUENT MATERIAL SPECIFICATIONS AND TEST METHODS**

The supply of the constituent materials is covered by a fairly complete set of ISO standards for fibres and resins covering both testing and specification requirements. These standards will be adopted and extended for the EN General series. There was additional work during the last year to complete the carbon fibre test methods, covering carbon fibre density (ISO
10119) and size content (ISO 10549) (see Table 1). Some revisions to the tensile strength test for carbon fibres is also underway (ISO 10618).

The most significant new work in this area is the CEN projects on specification standards for fibre secondary products, as listed in Table 1. The standard for non-crimp fabrics (NCF) shows earlier recognition of the increasing importance of this reinforcement format [1]. The main problem is to find a new project leader for prEN 13002, following the retirement of the previous project leader, for the carbon fibre yarn specification, which is important for the industry to illustrate to potential users that fibres are available to a consistent specification, and from more than one supplier.

Table 1: Constituent material specifications and test method standards

<table>
<thead>
<tr>
<th>Standard Number</th>
<th>Brief Title</th>
<th>Status</th>
</tr>
</thead>
<tbody>
<tr>
<td>ISO 1818</td>
<td>Reinforcement fibre Sampling plan applicable to received batches</td>
<td>Withdrawn, replaced by ISO 2856 and 3951</td>
</tr>
<tr>
<td>ISO 3616</td>
<td>Textile glass chopped strand and continuous filament-mats – Determination of average thickness under load and recovery after compression</td>
<td>Published</td>
</tr>
<tr>
<td>ISO/DIS 10119</td>
<td>Carbon-fibre – Determination of density</td>
<td>Forwarded for publication</td>
</tr>
<tr>
<td>ISO/DIS 10549</td>
<td>Carbon-fibre – Determination of size content</td>
<td>Ready for publication</td>
</tr>
<tr>
<td>ISO/CD 10618</td>
<td>Carbon-fibre – Determination of tensile properties</td>
<td>Minor revision to a published standard</td>
</tr>
<tr>
<td>prEN 13002 Parts 3</td>
<td>Specification for carbon fibre yarns. Part 3 Technical specification</td>
<td>On stand-by awaiting new project manager</td>
</tr>
<tr>
<td>prEN 13417 Parts 1-3</td>
<td>Specification for woven fabrics</td>
<td>Sent to CEN for formal ballot</td>
</tr>
<tr>
<td>prEN 13473 Parts 1-3</td>
<td>Specification for multi-axial multi-ply fabrics (NCFs)</td>
<td>Formal vote completed</td>
</tr>
<tr>
<td>prEN 14020 Parts 1-3</td>
<td>Specification for textile glass rovings</td>
<td>Enquiry completed, to prepare for formal vote</td>
</tr>
<tr>
<td>CEN Project Nos. 419-21</td>
<td>Specification for textile glass mats</td>
<td>Sent to CEN for formal ballot</td>
</tr>
</tbody>
</table>

3.2 COMPOUND SPECIFICATION AND TEST METHODS

Work is also underway in CEN on the specification of moulding compounds, both thermoset (SMC/BMC) and thermoplastic (GMT), as indicated in Table 2. The GMT documents are most advanced, under the convenorship of Andrew Downey (UK). The SMC documents have been delayed by the unexpected death of the project leader.

Table 2: Compound specification and test methods

<table>
<thead>
<tr>
<th>Standard Number</th>
<th>Title</th>
<th>Status</th>
</tr>
</thead>
<tbody>
<tr>
<td>CEN Project Nos. 240-2</td>
<td>SMC/DMC Specifications</td>
<td>Awaiting texts</td>
</tr>
<tr>
<td>prEN 13421</td>
<td>SMC/BMC – determination of anisotropy</td>
<td>Text being prepared for formal ballot</td>
</tr>
<tr>
<td>prEN 13677 Parts 1-3</td>
<td>GMT - Specifications</td>
<td>To be sent to CEN for formal vote</td>
</tr>
<tr>
<td>prEN 13706</td>
<td>Pultruded Profiles - Specifications</td>
<td>Sent to CEN for formal ballot</td>
</tr>
</tbody>
</table>
3.3 COUPON LEVEL TEST METHODS

At the next higher level of certification, laminate properties, it is necessary to undertake the test using coupon cut from the product or, more usually from a test plate/panel. An existing standard ISO 1268 has completed its revision to cover the manufacture of test plates by all process routes for long-fibre composites (see Table 3), supported by existing ISO standards (i.e. ISO 293-295) for short fibre composites. All these parts were published recently, were at formal ballot (FDIS) or already sent for publication. As new processes outside the existing scopes are commercialised, further parts will be added to the standard.

Table 3: Parts of (EN) ISO 1268 for test plate manufacture

<table>
<thead>
<tr>
<th>No.</th>
<th>Title</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>General principles</td>
</tr>
<tr>
<td>2</td>
<td>Contact and spray-up moulding</td>
</tr>
<tr>
<td>3</td>
<td>Wet compression moulding</td>
</tr>
<tr>
<td>4</td>
<td>Moulding of preimpregnates</td>
</tr>
<tr>
<td>5</td>
<td>Filament moulding</td>
</tr>
<tr>
<td>6</td>
<td>Pultrusion moulding</td>
</tr>
<tr>
<td>7</td>
<td>Resin transfer moulding</td>
</tr>
<tr>
<td>8</td>
<td>Moulding of SMC/BMC</td>
</tr>
<tr>
<td>9</td>
<td>Moulding of GMT/STC</td>
</tr>
<tr>
<td>10</td>
<td>Injection moulding of BMC/DMC</td>
</tr>
</tbody>
</table>

Part 4 on pre-pregs, drafted by NPL(GDS), references a designation code for ply orientations that can be generally applied and is the only part that includes precision data. The data are based on the results of a NPL round-robin that showed participants could prepare panels to a consistent and high standard [2]. The Part 4 annex contains a description of the stacking sequence definitions and coding. It is important that this coding is standardised to ensure that designer, fabricator and user are using the same coding system and therefore have the same panel under consideration. Confusion over the lay-up could lead to dangerous errors through inadequate stiffness or strength components.

An important issue not yet covered satisfactorily is the machining of composites and specimen preparation. Some minimal instructions are included in the mechanical test methods, described below, as a common annex. Some further information is given in the ASTM standard (D 5687) for test panel manufacture. It is hoped that the recent NPL Measurement Good Practice Guide No. 38 [3] on “Machining of Composites and Specimen Preparation” can provide the basis of a future standard equivalent to ISO 2818 - “Machining of Plastics”. The traceability of the inspection of the test panel by ultrasonic C-scanning inspection techniques will be greatly improved through EN standards based on the procedures developed in a prior NPL/Quinetiq research programme [4]. Delays in this case have been caused through the re-structuring of AECMA by cancelling working groups and using a project leader approach. These documents were both produced with DTI support. Airbus has agreed to sponsor these documents through the new AECMA process.

Laminate test methods are still principally defined in the ISO work programme. Current work includes “Fatigue Testing of Composites – General Principles (ISO 13003)”. The project has a French project Leader but NPL(GDS) has been responsible for technical lead and final master English text. The standard is approved for publication and will be sent to ISO (Geneva) during 2002 following the final editing of the English text. Other work has
involved the revision of the four-part standard, ISO 3597, covering the properties of fibre-reinforced rods, which has been approved for publication.

Table 4: Composite material test methods

<table>
<thead>
<tr>
<th>Standard Number</th>
<th>Title</th>
<th>Status</th>
</tr>
</thead>
<tbody>
<tr>
<td>ISO 3597 Part 1-4 (Revision)</td>
<td>Textile glass reinforced plastics - Determination of mechanical properties on rods made of roving reinforced resin (Preparation of rods, flexure, tension and shear strengths)</td>
<td>Forwarded for publication</td>
</tr>
<tr>
<td>ISO 13003</td>
<td>Fatigue - General principles</td>
<td>Forwarded for publication</td>
</tr>
<tr>
<td>ISO/DIS 14127</td>
<td>Carbon-fibre laminates - Determination of resin, fibre and void content,</td>
<td>Needs to be re-submitted as outside 7 years allowed.</td>
</tr>
<tr>
<td>ISO 15310</td>
<td>Determination of in-plane shear modulus by the plate twist test method</td>
<td>Published</td>
</tr>
<tr>
<td>ISO NWI N565</td>
<td>Glass reinforced products - Determination of fibre length</td>
<td>Forwarded to CD stage</td>
</tr>
<tr>
<td>ASTM 6671</td>
<td>Mixed Mode 1-Mode II Interlaminar fracture toughness of unidirectional fibre reinforced polymer matrix composites.</td>
<td>Published</td>
</tr>
<tr>
<td>ASTM 6641</td>
<td>Determining the compressive properties of polymer matrix composite materials using a combined loading compression (CLC) test fixture.</td>
<td>Published</td>
</tr>
</tbody>
</table>

The work conducted by NPL over the last few years led to the publication of six standards covering laminate test methods in the period 1997 to 1999. These six standards, shown in Table 5, provide the basic coupon tests used for design (in-plane tension, shear and compression) and QA (i.e. ILSS, flexure). These tests were heavily harmonised with CRAG and ASTM, with one (i.e. BS EN ISO 14129) based on the equivalent ASTM test. These standards are automatically reviewed for confirmation or revision five-years after publication.

NPL(GDS) as the project leader would be grateful for user feedback on these methods. Particularly, if there is experimental or theoretical evidence suggesting that a revision, or minor amendments, are required. The ASTM standard on compression by a combined loading fixture (ASTM D6641) duplicates the Method B for tabbed specimens in EN ISO 14126. The new ASTM standard has several cross-references and similar procedures to ASTM D3410. However, there are several points of concern including damage from end-grip clamping and the non-standard specimen design. Having achieved harmonisation between EN ISO 14126 and ASTM D 3410 with a 10 mm and 25 mm wide specimen, the D 6641 introduces a 12 mm wide specimen. The torque of 2.5-3 N.mm is similar to that used in EN ISO 14126 for clamping.

Table 5: Harmonised BS EN ISO Test Methods

<table>
<thead>
<tr>
<th>Property</th>
<th>International Standard</th>
<th>ASTM/CRAG Methods</th>
<th>Review Year</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tension - “Isotropic” (nominally)</td>
<td>BS EN ISO 527-4</td>
<td>D 3930/CRAG 300</td>
<td>2002</td>
</tr>
<tr>
<td>Tension - Unidirectional (anisotropic)</td>
<td>BS EN ISO 527-5</td>
<td>D 3930/CRAG 300</td>
<td>2002</td>
</tr>
<tr>
<td>Flexure</td>
<td>BS EN ISO 14125</td>
<td>D 695/CRAG 200</td>
<td>2003</td>
</tr>
<tr>
<td>Compression</td>
<td>BS EN ISO 14126</td>
<td>D 3410/CRAG 400</td>
<td>2004</td>
</tr>
<tr>
<td>Shear - ± 45° Tension</td>
<td>BS EN ISO 14129</td>
<td>D 3815/CRAG 101</td>
<td>2003</td>
</tr>
<tr>
<td>Shear - interlaminar by short beam flexure</td>
<td>BS EN ISO 14130</td>
<td>D 2344/CRAG 100</td>
<td>2003</td>
</tr>
</tbody>
</table>
Other ISO coupon test methods recently published are:
- in-plane shear modulus by plate twist (ISO 15310) and
- Mode I delamination fracture toughness (ISO 15024).

It is noted that other test methods covering, for instance, the thermal and chemical response of the composite material and/or the structure are required. These are dealt with as they arise for individual products, but also more generic methods are being developed. For example, NPL is the project leader on ISO 6721-11 on measurement of Tg (glass transition) using dynamic mechanical analysis (DMA) methods. This will be followed by Part 12 on calibration of DMA equipment, with a likelihood of it being based on a new NPL developed reference sample. NPL has also been responsible for the addendum to Part 1 of the DSC (Differential Scanning Calorimetry) standard (ISO 11357-1). These methods have been researched and precision data obtained in NPL/industry studio projects. This work has led to a Measurement Good Practice Guide on thermal analysis methods [5], which included the working draft for ISO 6721-11. Further work to be undertaken by the UK relates to use of temperature modulated DSC for thermal conductivity measurements, including a Part 1 General Principles document. These methods were studied in the second phase of the recent thermal analysis studio project, with a planned third phase under consideration.

Round-robin validation of tension, compression and shear methods [6] for through-thickness are currently underway in MMSI with the intention of offering these draft procedures and the resultant precision data via BSI for international standardisation (see Section 5). Through-thickness properties are of increasing interest as thicker materials are used, and as new material formats (e.g. stitched NCFs, Z-pins and thermoplastic matrices) are proposed.

Work on a Mode II fracture toughness test is expected shortly, following a VAMAS (Versailles Project on Advanced Materials and Standards) TWA5 round-robin programme aimed at identifying the preferred method for standardisation [7]. VAMAS is a G7/G8 project for pre-normalisation activities to attain early harmonisation of test methods, databases, etc., and TWA 5 covers Polymer Composites activities. (see Section 4). A new work item (NWI) will be submitted shortly covering the 4-point ENF (end-notched flexure (cf AITM) recommended by the VAMAS work, and possibly the ELS (edge longitudinal split) method favoured by ESIS. ASTM has recently published a mixed mode I-II test. ASTM were supportive of the VAMAS initiative on Mode II until progress was blocked.

3.4 COMPOSITE MATERIAL DATABASE STANDARDS

The standard ISO 10350-2, published in 2002, was prepared by NPL (GDS) as a composite materials version of the Part 1 covering plastics that has been extremely successful in encouraging the use of ISO test methods to achieve comparable data. If there are options in the reference test methods, then a particular version will be identified by the database standard. However, the “default” method will be increasingly identified in the test methods itself. To support the Single Market needs in trade or liability matters there is a need to reduce unnecessary confusion and complexity in underpinning standards. This database standard may provide a starting point for an extended database covering design data.

NPL has started a new project (MMS2) to develop a standard qualification plan (SQP), for future standardisation, that is aimed at considerably reducing the cost of qualifying new materials by allowing standard data to be provided by the supplier suitable for materials selection and preliminary design. It is widely recognised that major costs are involved in
bringing a new material to the market. For example, it cost one supplier 15 times the cost of a single qualification for undertaking it ten times to (slightly?) different user specifications with both the supplier and the ten users incurring the additional costs. The SOP will also minimise product design costs through the immediate availability of the data, rather than awaiting the completion of multiple bilateral qualification programmes between a supplier and each of its customers independently. The SOP is being prepared in conjunction with material suppliers, aerospace manufacturers, F1 manufacturers and the CAA, while taking account of other international work (e.g FAA). A series of round-robin validations will be undertaken to generate confidence in the selected methods and each others’ data. The SOP will also cover batch testing requirements. An extended qualification plan (EQP) will contain less frequently required data requirements.

3.5 STRUCTURAL ELEMENT TEST METHODS

At the next level of validation there is concern to understand and measure the effect of stress concentrations. The pin-bearing test is one of three methods using a common geometry being balloted as ISO NWI (New Work Items) following a BSI submission based on NPL prepared drafts and NPL organised round-robin (RR) experiments for precision data. The other two methods are open/filled hole compression (OHC) and open-hole tension (OHT). In every case, a 6 mm diameter hole in a 36 mm wide coupon is used, or an English unit equivalent based on 0.25” diameter hole and a 1.5” wide coupon (i.e. a 1:6 ratio).

Although the pin-bearing test is used for aerospace material, there is a need for this information for all types of composites, such as GRP roofing sheets. In fact, the first mandatory use of the pin-bearing test will be in the specification standard for pultruded profiles (i.e. prEN 13706) in recognition of the frequent use of bolting for assembly structures from these profiles (c.f. alternative of bonding).

Currently, in MMS1 the RR validation is being repeated for the pin-bearing test as in some case in the previous RR [8] due to insufficient clearance in the jig, erroneously high values were obtained at some sites.

If these methods are compared with ASTM equivalents it is found that for the OHT, ASTM D 5766 is equivalent (i.e a 6/1 ratio of plate width to hole diameter based on either a 6 mm or 0.25 inch hole) as also in EN 6035. The OHC standard - ASTM D 6484 is different in using a larger thinner plate requiring a solid support jig, compared to the shorter, thicker unsupported specimen proposed by NPL, which is similar to the Airbus based method (EN 6036). ASTM D 5961 uses a bolted joint rather than a plain pin preferred by Airbus (EN 6037). The bolted joint test method has difficulties associated with a less-well defined failure point, un-quantified bolt torque and relaxation issues; and recommended torques covering a range (ie 1-3 Nm) known to give variations in the test result.

3.6 SUB-COMPONENT SPECIFICATIONS

At the sub-component level specification, standards are being developed in CEN for pultruded profiles, prEN 13706 with NPL project leadership and convenorship. Pultruded profiles are one of the few cases where composites are available in a final cured form for immediate use, normally as bolted or bonded assemblies.
Part 1 contains the technical specification, including a coding rather similar in basis to that found for metal products (e.g. "Pultruded Profile, EN 13706, BGV, IF, E23, which is a pultruded profile, conforming to EN 13706, consisting of a box section: glass-fibre reinforced: with surface veil: and isophthalic polyester: fire retardent resin: with a section material modulus of 23 GPa"). Part 2 gives the test methods for mandated properties and other required properties, together with requirements for test plate production, acceptable tolerances, defect levels and additional test methods. The annexes contain three NPL draft test methods including the pin-bearing test described above. The materials modulus forming the designation code (e.g. E23) is obtained from a long beam test on a full section of the pultrusion (see Annex D of EN 13706). An alternative test requiring repeat tests at different loading spans in flexure and torsion has also been drafted to give the full section flexure, shear and torsion properties (see Annex G of EN 13706). In addition, recommended tests are given for all other material properties including further mechanical properties (e.g. impact), electrical, thermal, environmental and fire properties. These methods are normally in the ISO or EN series.

Part 3 of the standard gives the required levels for mandated properties, together with tolerance, defects etc requirement, in order to show compliance with the standard, for the two grades initially defined (i.e. E17 and E23).

Further work has been initiated on specialist aspects, such as chemical or fire resistant. This has enabled NPL to study in some detail the new CEN fire classification, which has implications for all composites and indeed other construction materials. NPL has negotiated a modification to the single-burning item test (SBI – EN to make it applicable to pultruded profiles (i.e. a linear rather than a sheet product). This is very important, as this is one of a limited number of tests governing fire classification under the Construction Products Directorate.

3.7 PRODUCT APPROVAL STANDARDS

Several bodies are involved in developing product specification and approvals. Within Europe the main source will be from CEN in support of European Directives. There are several committees working on product standardisation, which may be for a composites product only (e.g. GRP pressure vessels - CEN/TC 210, piping – ISO TC 138/ CEN TC 155) with equivalent standards covering use of other materials in the same application, or an area where a single standard covers all competing materials (e.g. access engineering (i.e. ladders, walkways and handrails – prEN14122 by CEN/TC 114). In addition, a standard for off-shore GRP piping has been prepared under ISO/TC67 based on the guidance document developed by the UK Offshore Operators Association. Harmonisation, or complimentary action, of CEN and ISO work in the general area of GRP piping is obviously desirable. In some specialised areas, such as civil aircraft and marine, other bodies have the regulatory responsibility (i.e. Civil Aircraft Authority (CAA) and Lloyd’s Register (LR), respectively).

Product standards, as for access engineering, open to all materials that can meet the technical requirements (e.g. maximum acceptable deflections under prescribed loads) are preferred. These performance based standards are preferred to the more prescriptive standards that either prevent completely the use of composites or inhibit the design freedom necessary for the most cost effective solution. It is important in these cases, that the standards do not include any requirement that prevents unnecessarily a composite material based design solution.
Progress is being made with several documents under ballot. However, as the developments in the test methods and the product standards are often concurrent there are often delays in implementing new test methods in the product standards unless there is good liaison between the responsible committees. These standards have been recently reviewed [9], with particular emphasis on the traceability of material properties, determination of service temperature etc. It is of interest in encouraging the greater use of composite materials by engineers that there is consistency in the design approach used (e.g. the choice of performance or prescriptive based approaches). The standards currently under development are listed in Table 6.

The most significant new standard is perhaps ISO/DIS 14692 - A working group under ISO 67 formed initially for a standard on GRP piping for off-shore use has seen the opportunity to increase the coverage, eventually, to on-shore, chemical and marine uses. The standard, ISO/DIS 14692, which will reach the last formal ballot in 2002 draws heavily from the prior document produced by UKOOA, the UK Off-shore Oil Operators Association. This document was well accepted and had been the basis of company documents, in particular for Shell where the scope was first extended to cover on-shore and chemical uses. The working group as well as having worldwide representations, enabled contact with the ASTM committee dealing with GRP marine piping. The standard is likely to have a long term significance for the future acceptance of GRP piping, and composites in general.

### Table 6: Composite material product standards

<table>
<thead>
<tr>
<th>Product</th>
<th>Standard Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>GRP pressure vessels</td>
<td>prEN 13121 (c.f BSI 4994)</td>
</tr>
<tr>
<td>GRP piping offshore</td>
<td>ISO/DIS 14692*</td>
</tr>
<tr>
<td>GRP Water piping</td>
<td>EN 1115 (c.f. BS 7159/6464)</td>
</tr>
<tr>
<td>GRP Water tanks</td>
<td>BS EN 13280 (BSI)</td>
</tr>
<tr>
<td>FRP Lighting Columns</td>
<td>prEN 40-7</td>
</tr>
<tr>
<td>Wrapped Gas Cylinders</td>
<td>prEN 12445/prEN12447/ISO 11119 (3 parts)</td>
</tr>
</tbody>
</table>

Several other are of interest as replacing existing BSI standards, eg BS 4994. Slow progress is being made in some areas due to the need to harmonise different national standards (e.g. mainly BSI and DIN for pressure vessels). This highlights the need for pre-normalisation work as in VAMAS to obtain early, and optimum, technical agreement

### 4 STANDARDISATION ISSUES

#### 4.1 STANDARDISATION BODIES

Many different bodies are involved in standardisation activities. These include the international and national standards organisations (e.g. ANSI, AFNOR, BSI, DIN, JIS), regulatory bodies such as the CAA and FAA, trade groupings and societies (e.g. ASTM). ISO has the largest country membership with 167 countries. The CEN standards covering Europe, including countries outside the European Union, has a higher “legal” profile and is encouraging increased attention to precision statements. It is important that the repeatability and reproducibility of the test method are known for both free trade and liability uses of the standard. The Vienna agreement between ISO and CEN ensures that work is not duplicated and allows a fast approval by CEN of existing ISO standards (UAP ballot - Unique Approval Procedure - YES/NO without comment).
Standards approved by CEN must be published by national committees and national standards of the same scope withdrawn. In the UK they will be dual numbered BS EN or triple numbered BS EN ISO, if accepted as an ISO standard as well and similarly numbered, NF EN ISO and DIN EN ISO in France and Germany, respectively.

Other bodies are developing standards in different application areas such as AECMA for EN Aerospace – often with input from Airbus Industries Test Methods (AITM) and ASTM principally in the USA. ASTM has had a specialised group, D-30, for many years and made strong input into international standardisation. ASTM is developing with ISO a similar “Vienna” relationship as exists for CEN so that ASTM standards can be fast-tracked into ISO standards. JIS also makes significant inputs particularly, on carbon-fibre test methods.

Table 7: International and National Standards Organisations

<table>
<thead>
<tr>
<th>ISO</th>
<th>International Standards Organisations</th>
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<tbody>
<tr>
<td>CEN</td>
<td>Comité Européen de Normalisation</td>
</tr>
<tr>
<td>AFNOR</td>
<td>Association Française de Normalisation</td>
</tr>
<tr>
<td>ANSI</td>
<td>American National Standards Institute</td>
</tr>
<tr>
<td>DIN</td>
<td>Deutsches Institut für Normung</td>
</tr>
<tr>
<td>AECMA</td>
<td>Aerospace Series of CEN standards prepared by European Aerospace Trade Federation, (n.b. 6000 series based on Airbus industry Test Methods (AITMs)).</td>
</tr>
<tr>
<td>ASTM</td>
<td>American Society for Testing and Materials</td>
</tr>
<tr>
<td>BSI</td>
<td>British Standards Institution</td>
</tr>
<tr>
<td>JIS</td>
<td>Japanese Industrial Standards</td>
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</tbody>
</table>

4.2 VAMAS PRE-NORMALISATION RESEARCH

The Versailles Project on Advanced Materials and Standards (VAMAS) aims to aid the introduction and use of advanced materials through international pre-normalisation research. Research can cover different aspects, such as, databases, reviews, specification coding, best practice guidance, new test methods, calibration methods and revision or validation of existing test methods. In all cases the aim is to get technical agreement on the optimum practices for implementation standards through the normal procedures (national NWI proposals).

Table 8: VAMAS projects, completed, approved and proposed new work areas

<table>
<thead>
<tr>
<th>Completed Projects</th>
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<tbody>
<tr>
<td>(a) Mode II fracture energies</td>
</tr>
<tr>
<td>(b) Fibre matrix interface strength by fragmentation test</td>
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</table>

<table>
<thead>
<tr>
<th>Approved Projects</th>
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<tr>
<td>(c) Damage Tolerance</td>
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<table>
<thead>
<tr>
<th>Proposed new work areas</th>
</tr>
</thead>
<tbody>
<tr>
<td>(d) Through-thickness test methods (tension, shear, compression)</td>
</tr>
<tr>
<td>(e) Thermal analysis techniques for composites (DSC, DMA),</td>
</tr>
<tr>
<td>(f) Stress concentration tests (open/filled holes in tension and compression, pin bearing),</td>
</tr>
<tr>
<td>(g) Extended and consolidated international durability database,</td>
</tr>
<tr>
<td>(h) Procedures for composites repair and strengthening of civil infrastructures,</td>
</tr>
<tr>
<td>(i) Test methods for the processing properties of composites,</td>
</tr>
<tr>
<td>(j) Qualification and databases for composites</td>
</tr>
</tbody>
</table>
The VAMAS initiative is led by G7 countries (eg Canada, France, Germany, Italy, Japan, UK, USA and European Union), with other countries encouraged to participate in individual projects. VAMAS has high level agreements with all standards development bodies. Some past, current and planned projects are listed in Table 8.

4.3 STANDARDS APPROVAL PROCEDURES

ISO documents are progressed through CD (committee draft – 3 months), DIS (Draft International Standard – 5 months) and FDIS (Formal DIS – 2 months), with a target development time of 44 months (i.e no allowance for re-balloting at any stage). The CD is the main stage for technical comment. Well written NPL drafted test methods, complete with precision data (repeatability and reproducibility) from experimental round-robin exercises, enable rapid progress to be made including missing the FDIS ballot through 100% approval at the DIS ballot stage (e.g. ISO 15310).

Within CEN, the “prEN” ballot is the main public enquiry stage and lasts 6 months, followed by a 2-month formal ballot. It is a better system as it allows adequate time for the draft standard to be “noticed” outside the specialist committees and for comment to be obtained at the main technical review stage. In contrast, the DIS ballot is five/six months for ISO but this is when all major technical changes should have already been made (i.e at CD ballot stage). For joint ballots under the Vienna rules, the timetable is closer to that used by CEN.

4.4 FINANCIAL SUPPORT OF STANDARDISATION BODIES

Currently, standards development bodies are under increasing financial pressure as government and industry support are reduced. Switzerland has given up the Thermal Methods secretariat (SC5) and the actions to be taken on existing projects (including the DSC and TMDSC projects lead by NPL) are unclear. In addition, AFNOR who have the secretariat for both ISO and CEN composite standardisation (i.e. excludes final product standards) have for some time required financial support for individual standards. To the extent of threatening to block the Mode II fracture energy standard developed specifically by VAMAS at the request of ISO TC61/SC13, and voting against the structural element tests, including the pin-bearing test needed by EN 13706 (Pultruded Profiles), while 20 other countries voted positive and France had a volunteer as a national expert. The Pultruded Profiles standard is only progressed because European pultruders pay AFNOR for this standard. Although, not in compliance with ISO rules, there is concern that this approach will become more general with other Secretariats adopting this approach.

Work on Compression-after-impact, an area to be studied in MMS13 on “defect criticality”, is expected to progress through support from Japanese industry. It is important that the UK is able to input into this process. The topic is also listed within the VAMAS projects listed in Table 9.
5. CONCLUDING COMMENTS

5.1 FUTURE REQUIREMENTS

Some requirements are identified during the programme development phase and solutions to these needs have been developed over the past programmes (e.g. CPD etc) and forwarded for international standardisation. Currently, as described below, several through-thickness tests and thick adherend tests developed in prior programmes are being validated. In the current MMS programme, new test methods can be expected for damage tolerance assessments and surface adhesion characterisation. The major requirement of durability testing and life prediction is only being treated in a minor project related to civil engineering use.

5.2 STANDARDISATION INPUTS FROM UK COMPANIES

NPL as the UK primary delegate to ISO and CEN, with membership and links to ASTM and JIS, is keen to hear from UK companies and research establishments on new requirements, revisions of existing standards or proposals for deleting obsolete standards. In particular, inputs are requested on the standards listed in table 5, at their 5-year review points.

5.3 ROUND-ROBIN ACTIVITIES

Several test methods were developed in prior programmes that require experimental validation via round-robin exercises to generate precision data (repeatability and reproducibility) prior to submission as new work item proposals for international standardisation. The project aims to undertake round-robin exercises on the following tests methods:

- through-thickness tension/compression,
- through-thickness shear,
- pin-bearing (repeat with modified jig),
- thick adherend adhesive test,

NPL is keen to involve other establishments in these validation exercises, through material supply, plate and or specimen manufacture and through testing. Further details of these round-robins and other standardisation issues are available from Dr. Graham D Sims [graham.sims@npl.co.uk] or via the NPL Composites Group web site [www.npl.co.uk/cogi/index.html].
REFERENCES

1. Sims G D and Bishop G, “ UK Polymer Composites Sector, Competitiveness Analysis and Foresight Study”, NPL Report MATC (A) 81


APPENDIX A  STANDARDS FOR FIBRE REINFORCED PLASTICS

Reinforcing fibres and fibre products - Test methods and specifications

ISO 1887: Glass fibre – Determination of combustible matter content (size)
ISO 1888: Textile glass – Staple fibres or filaments - Determination of average diameter
ISO 1889: Reinforcement yarns – Determination of density
ISO 1890: Reinforcement yarns – Determination of twist
ISO 2078: Textile glass - Yarns - Designation
ISO 3341: Textile glass - Yarns - Determination of breaking force and breaking elongation
ISO 9291: Textile glass reinforced plastics – rovings – preparation of unidirectional plats by winding. (being replaced by ISO 1268)
ISO 10119: Carbon fibre – Determination of density
ISO 10548: Carbon fibre – Determination of size content
ISO 11566: Carbon fibre – Determination of tensile properties of single filament specimens
ISO 11567: Carbon fibre – Determination of filament diameter and cross-sectional area
ISO 10618: Carbon fibre – Determination of tensile properties of resin-impregnated yarn
ISO 13002: Carbon fibre – Designation system for filament yarns.

Moulding compound / pre-impregnates – test methods and specifications

prEN yyyyy: Reinforced plastics composites – Specifications for thermoset moulding compounds (SMC, BMC, DMC)
prEN yyyyy: Determination of flowability
prEN 2833: Aerospace: Reinforced Plastics – Glass fibre pre-impregnates
ISO 9782. Plastics; reinforced moulding compounds and prepregs; Determination of apparent volatile matter content.
ISO 10352. Fibre reinforced plastics – Moulding compounds and prepregs – Determination of mass per unit area.

ISO 12115. Fibre reinforced plastics – thermosetting moulding compounds and prepregs – determination of flowability, maturation and shelf life.

ISO 12114 – Fibre reinforced plastics – thermosetting moulding compounds and prepregs – determination of cure characteristics


prEN 13677: Reinforced Plastics Composites – specifications for thermoplastic moulding compounds (GMT)

ISO 15034: Composites - Prepregs – Determination of resin flow

ISO 15040: Composites - Prepregs – Determination of gel time

Resin systems


Many other standards for unreinforced thermoplastic and thermoset resins applicable (www.bsi-global.com)

Fibre reinforced plastics (or polymer matrix composites)


Laminated materials – Mechanical property tests


BS EN ISO 527 – Part 1: Plastics – Determination of tensile properties – General principles

BS EN ISO 527 – Part 4: Determination of tensile properties – Test conditions for isotropic and orthotropic fibre-reinforced plastic composites

BS EN ISO 527-5: Plastics. Determination of tensile properties Test conditions for unidirectional fibre-reinforced plastic composites

ISO 1172. Textile glass reinforced plastics; Prepregs, moulding compounds and laminates – Determination of the textile-glass and mineral-filler content - Calcination methods (determination of loss on ignition)

ISO 1268: Fibre reinforced plastics – test plate manufacturing methods

ISO 2818: Plastics – Preparation of specimens by machining

ISO 3597. Textile glass reinforced plastics; Determination of mechanical properties on rods made of roving reinforced resin (Parts 1-4)

ISO 4899 Textile glass reinforced thermosetting plastics; properties and test methods

ISO 10350-2: Plastics – acquisition and presentation of comparable single-point data – Part 2: long fibre reinforced plastics
ISO/FDIS 13003: Fibre reinforced plastic composites – determination of fatigue properties under cyclic loading

BS EN ISO 14125: Fibre-reinforced plastics composites – determination of flexural properties

BS EN ISO 14126: Fibre reinforced plastic composites – determination of the in-plane compression strength

ISO/DIS 14127. Composites – determination of resin, fibre and void content of composites reinforced with carbon fibre

BS EN ISO 14129: Fibre reinforced plastic composites – determination of apparent interlaminar shear strength by short-beam method

ISO 15024: standard test method for Mode Interlaminar fracture toughness G_le of unidirectional fibre reinforced polymer matrix composites

ISO 15310: Fibre reinforced plastic composites – determination of in-plane shear modulus by plate twist

Thermal analysis test methods

ISO 6721: Plastics – Determination of dynamic mechanical properties

ISO 11357: Plastics - Differential scanning calorimetry

Final Products –Test method and Product Standards


BS 5480:1990. Specification for glass fibre reinforced plastics (GRP) pipes and fittings for water supply or sewerage.

ISO 7370. Glass fibre reinforced thermosetting plastics (GRP) pipes and fittings; nominal diameters, specified diameters and standard lengths.

ISO/FDIS 7432 Glass reinforced thermosetting plastics (GRP) pipes and fittings-test methods to prove the design of locked socket and spigot joints

BS 7491. Glass fibre reinforced plastics cisterns for cold water storage

ISO/DIS 7509 Plastics piping systems – glass reinforced thermosetting plastics (GRP) pipes – determination of time to failure under sustained internal pressure

ISO 7510. Plastics piping systems- glass reinforced plastics (GRP) components

ISO 7511. Plastics piping systems – glass reinforced thermosetting plastics (GRP) pipes and fittings – test methods to prove the leak tightness of the wall under short-term internal pressure.


ISO 7685. Plastics piping systems - glass reinforced thermosetting plastics (GRP) pipes – determination of initial specific ring stiffness.
ISO/DIS 8483: Glass reinforced thermosetting plastics (GRP) pipes and fittings — test methods to prove the design of bolted flange joints.

ISO/DIS 8513: Plastics piping systems - glass reinforced thermosetting plastics (GRP) pipes — determination of initial longitudinal tensile properties.

ISO 8521: Glass reinforced thermosetting plastics (GRP) pipes — determination of the apparent initial circumferential tensile strength.

ISO/DIS 8533: Glass reinforced thermosetting plastics (GRP) pipes and fittings — test methods to prove the design of cemented or wrapped joints.

ISO/DIS 8639: Glass reinforced thermosetting plastics (GRP) — test methods for leaktightness and resistance to damage of flexible and reduced-articulation joints.

ISO 10466: Plastics piping systems - glass reinforced thermosetting plastics (GRP) pipes — test method to prove the resistance to initial ring deflection.

ISO/DIS 10467: Plastics piping systems for pressure and non-pressure sewerage — glass reinforced thermosetting plastics (GRP) based on unsaturated polyester (UP) resin.

ISO/DIS 10468: Plastics piping systems - glass reinforced thermosetting plastics (GRP) pipes — determination of the long-term specific creep stiffness under wet conditions and calculation of the wet creep factor.

ISO/DIS 10471: Plastics piping systems - glass reinforced thermosetting plastics (GRP) pipes — determination of the long-term ultimate bending strain and the long-term ultimate relative ring deflection under wet conditions.

ISO/DIS 10639: Plastics piping systems for water supply with or without pressure — glass reinforced plastics (GRP) based on unsaturated polyester (UP) resin.


ISO 10952: Plastics piping systems - glass reinforced thermosetting plastics (GRP) pipes and fittings determination of the resistance to chemical attack from the inside of a section in a deflected condition.


prEN 13923: Filament-wound GRP pressure vessels. Materials, design, manufacture and testing.

ISO/DIS 14692: Petroleum and natural gas industries - GRP piping (4 Parts).

ISO/DIS 14828: Plastics piping systems - glass reinforced thermosetting plastics (GRP) pipes — determination of the long-term specific ring relaxation stiffness under wet conditioned and calculation of the wet relaxation factor.

Further information available from:

www.npl.co.uk/cog/index.html
www.bsi-global.com
www.iso.ch