FINE FINISH NEWS VOL -21

INSIDE

Editor's Note

It is a pleasure to introduce our 21st edition of Fine Finish News. We are proud to bring to you our new products, new test facilities and capabilities, upcoming proficiency testing programs, technical training programs and technical news from around the world.

India just celebrated her 72nd Independence Day. In our Prime Minister Narendra Modi's words- 'India is a land of reform, perform & transform. Not only will India emerge as a great nation but will also inspire others'

Fine Finish is proud to play a small role in India's space research program, defence and aerospace sector and contribute its bit towards reforming the testing capabilities of this country. Our analytical laboratory complies with the requirements of various ASTM, ISO and IEC standards. Our proficiency testing division has had participants from all across Europe, Central and South America, China and Africa helping Indian laboratories rank themselves against the world players.

In our quest to customer service, we are introducing a topic of problems & remedies in the shop floor. In this issue, we are bringing problems & remedies for casting problems in epoxy resins.

- **Dr. G.S. Prabhu**

NEW PRODUCTS

Finehard 1618 - Modified Cycloaliphatic Amine Hardener

Finehard 1618 is a cycloaliphatic amine hardener suitable for cold or warm curing of epoxy resins. This gives high gloss films that are resistant to variety of chemicals. These properties make it ideal for floorings, maintenance coatings, tank linings and secondary containment linings. It has a characteristic good color stability making it ideal for clear and pastel shade coatings.

Finehard 1618 can be used for manufacturing high solid coatings. It is an excellent coating for self-levelling and pebble finish flooring. It is used in chemically resistant tank linings, mortars and grouts. It can be also used in carbon fiber composites and has excellent heat resistance above 150^0C. It also has very good chemical resistance and electrical insulation.

100 parts by weight of Epofine 250, 60 parts by weight of Finehard 1618 and 500 parts by weight of Silica are mixed and the system is cured for 2 to 7 days at 25^0C to 35^0C. End Loading Compressive Strength is in the range of 80 to 120 MPa

- **Kishore Prabhu**

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Fineset 3560 AD- High Performance Electrical Grade Modified Epoxy Resin

Fineset 3560 AD is a high-performance electrical grade modified epoxy resin-based adhesive with pre-mixed hardener. The high temperature adhesive system has excellent wetting action to composites. This resin system provides electrical, chemical and mechanical properties. The long shelf life of this system makes it ideal for large electrical equipment to be processed at high ambient temperatures.

The strength and durability of an adhesive joint to a large extent is decided by pre-treatment of the substrate. Strongest joints are made by mechanically abrading or chemically etching. The mechanically abraded surfaces shall be further cleaned with a solvent. Minimum treatment that is required is cleaning by an oil free solvent like trichloroethylene or environmentally friendly degreaser such as Fineclean EC.

The resin hardener mix is generally applied using a clean spatula to the pre-treated and dry substrate. An adhesive layer of 0.10-0.20 mm thickness generally imparts the greatest lap shear strength to the adhesive joints of composites. As soon as adhesive is applied, joints are clamped with extra pressure.

The following advantages make it ideal for impregnation of electrical machines:

1. This is a single component system with shelf life of over 6 months @ 25° C.
2. Very slight odor.
3. Decrease in viscosity on heating, which ensures proper wetting.
4. Excellent continuous heat resistance up to 250° C and short time temperature up to 300° C.
5. Very Good chemical resistance and electrical insulation.

- **Kishore Prabhu**

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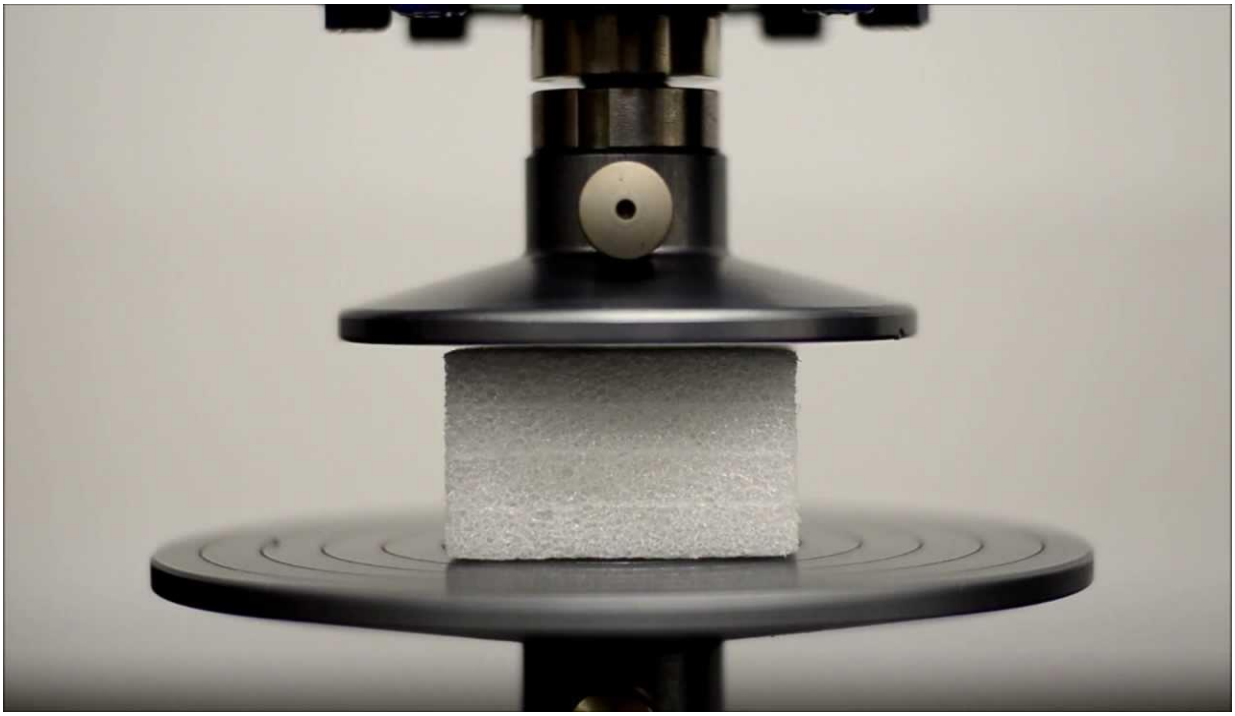
NEW TEST FACILITIES

Compressive Properties of Rigid Cellular Plastics (Foam/Core)

ASTM D 1621 test method provides information regarding the behavior of cellular materials under compressive loads. Deformation data can be obtained, and from a complete load deformation curve it is possible to compute the compressive stress at any load (such as compressive stress at proportional limit load or compressive strength at maximum load) and to compute the effective modulus of elasticity.

Compression tests provide a standard method of obtaining data for research and development, quality control, acceptance or rejection under specifications, and special purposes. The tests cannot be considered significant for engineering design in applications differing widely from the load - time scale of the standard test. Such applications require additional tests such as impact, creep, and fatigue.



- **Vishakha Patil**

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Shear Test Methods: IOSIPESCU vs. V-Notched Rail

The Iosipescu Shear and the V-Notched Rail Shear test methods are ASTM standards, ASTM D 5379 and D 7078, respectively. Both are used primarily for in-plane shear testing, but each can be used for interlaminar shear testing as well.

The Iosipescu shear test, developed initially in the 1960s for use with homogeneous, isotropic structural materials such as metals, has been adapted during the past ten years for use with composite materials. For generating accurate shear properties design data, the Iosipescu Shear and V-Notched Rail Shear test methods are the obvious choices."

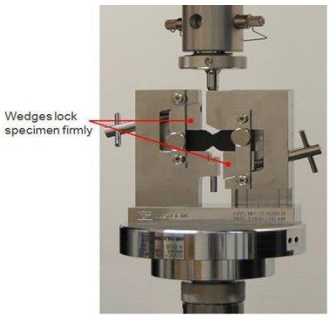

Fig. 1: Iosipescu shear test fixture (ASTM D 5379).

The V- notched beam testing method (Iosipescu method) is widely used in the composite field for testing in-plane shear. This test method applies an asymmetrical four- point compression load to a sample with V-notches, which enables the application f only shear stress on the evaluation area. This test method can be used with various CFRP laminate materials, including unidirectional materials, orthogonally laminated materials and materials with discontinuous fibres.

The older Iosipescu Shear method was standardized by ASTM in 1993 but has been used extensively by the composites testing community since the mid-1980s and it has proven to be very accurate and reliable. The test fixture is shown in Fig. 1.

Standardized by ASTM in 2005, shortly after its development, the V-Notched Rail Shear test has a relatively short history, but its use has grown rapidly. The test fixture is shown in Fig. 2.

Fig. 2: V-notched rail shear test fixture (ASTM D 7078).

Test method designers deliberately increased the width of the V-Notched Rail Shear specimen relative to that of the Iosipescu Shear specimen to provide a larger zone of uniform shear stress between the notches. A similar width increase is not possible for the Iosipescu Shear specimen unless the specimen length is increased proportionally because the concentrated loadings on the specimen edges would become unacceptably high, risking edge crushing. For the V-Notched Rail Shear test, a specimen width increase with no increase in length is possible because face loading is much more efficient than edge loading.

In addition, the shear stress levels that can be achieved in the V-Notched Rail Shear specimen are typically three to four times higher. This might not be very important when materials with lower shear strengths (<140 MPa) are tested. This includes typical unidirectional and cross-ply composites and similar materials. The Iosipescu Shear test method will typically accommodate such materials, but it might be necessary to tab the specimens (to increase the load-bearing area of the edges and thus avoid crushing) as the upper limit is approached. However, laminates that have increasing percentages of off-axis plies (e.g., ±45°) tend to exhibit much higher shear strengths and cannot be tested successfully using edge loading. In these cases, the face loading used in the V-Notched Rail Shear test method is mandatory. For example, with ±45° laminates, shear stresses as high as 500 MPa/75 ksi have been achieved using this test method. The limit is reached when the specimen slips in the grip plates despite the application of maximum allowable torque on the bolts.

Fig. 3: Combined loading shear (CLS) test fixture

Thus, the question arises *How can even higher shear forces be transferred into the specimen?* One possibility is to combine the edge and face loadings into a new test fixture design, resulting in a Combined Loading Shear (CLS) test method. One fixture design that has been proposed is shown in Fig. 3. Note that the clamping plates run the full length of the holders. Thus, the holders exhibit elastic deflection symmetrically when the screws are tightened, providing more uniform clamping force.

Fig. 4: BCLS fixture (left) and ASTM D 7078 fixture (right).

This new fixture concept is compared to the standard ASTM D 7078 fixture in Fig. 4. The concept fixture is sturdier, and the four clamping screws are larger than the three screws used in the ASTM standard fixture. Also, the widths of the cut-out and the clamping plates are equal and are the same as the specimen width, 56 mm/2.2 inch. The space between grips is 25 mm/1 inch, the same as for the V-Notched Rail Shear test fixture. The fixture shown in Fig. 4 will accommodate a specimen up to150 mm/6 inches long, although users have tended to use shorter specimens as well.

Because a close fit is maintained between the specimen and the fixture, the specimen edges take the load when the specimen tends to slip under an applied force, just as for the Iosipescu Shear test fixture.

- **Vishakha Patil**

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TECHNICAL ARTICLE

Using DSC for Cure Kinetics - Is your FRP structure optimally cured?

Now a days, large and complicated composite structures are used in various applications like aerospace, manufacturing of windmill blades, boats etc. Monitoring the cure of these epoxy resin matrix composites during the manufacturing or molding process of these composites structures is of utmost importance for maintaining the quality and reliability of the final product. The degree of cure can affect the strength and modulus and if a composite is over cured, it may become brittle due to excessive cross-linking. To prevent under-curing and premature demolding manufacturers have to determine the optimal cure time. However, most of the cure times are larger than required to prevent the worst-case scenarios and this leads to increase in the manufacturing cycle times, over- curing and increase in cost of the components.

Different analytical methods for determining degree of cure exist such as Differential Scanning Calorimeter (DSC), Raman Spectroscopy, Dielectric Analysis (DEA) etc. Most composite manufacturers prefer the DEA technique as it can measure the degree of cure in situ. However, DEA technique is difficult as it involves complicated calculation for determination of cure information from dissipation factor of the resin.

Differential Scanning Calorimeter (DSC) is a sensitive instrument that is used to study the thermal behavior of different polymers. DSC is used to measure the heat flow into or out of a very tiny sample as it is exposed to a controlled thermal profile. DSC provides both qualitative & quantitative information about material transitions such as glass transition, crystallization, curing, melting, and decomposition.

ASTM E2160 is a standard test method for determination of Heat of Reaction of Thermally Reactive Materials by DSC. The total heat of the curing reaction is measured on uncured material. This test method is useful in determining the fraction of reaction that has been completed in the sample prior to testing, that is the degree of cure. If any reaction heat (exotherm, delta H, in joules per gram) can be measured on a an already cured sample then it was not fully cured. The percentage of cure can be calculated by dividing the residual delta H by the total delta H.

Testing standards, ASTM E 698 and ASTM E2041 are used to estimate more kinetic parameters. ASTM E 698 is a standard test method that covers the determination of kinetic parameters of thermally unstable materials using DSC and Flynn/ Wall/ Ozawa Method. This technique is applicable to those reactions that follow the Arrhenius equation and the general rate law. ASTM E2041 is a standard test method that covers the estimation of kinetic parameters using DSC by the Borchardt and Daniels Method. This test method is applicable to temperature range of -100ºC to 600ºC in smooth exothermic reactions with no shoulders, discontinuous changes or shifts in the baseline.

Both the standards are useful for rapid automatic calculation of reaction order (n and/ or m), Activation energy (Ea), pre-exponential factor (Z) and rate constant (k). The Arrhenius parameters combined with the general rate law and the reaction enthalpy can be used for the determination of thermal explosion hazards. Kinetic values obtained from the peak temperature- heating rate relationship are used to predict a reaction half-life at a selected temperature.

- **Karishma Prabhu**

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PROBLEMS & REMEDIES

Casting Process- Faults, Causes & Solutions

Epoxy resin casting involves filling molds with liquid thermosetting epoxy resins containing fillers. The most preferred casting resins are epoxy resin systems since polyester resins shrink due to large exotherm generated during the reaction.

Now although, epoxies are relatively simple to use, there are some basic concerns that need to be addressed:

1. Design of the mold
2. Properties of the final product
3. Viscosity of the selected epoxy resin system
4. Cure kinetics of the resin system
5. Thermal stability
6. Expansion & shrinkage characteristics

The possible faults that may occur in an epoxy casting and the reasons for these faults are listed in this table.

Some of the possible solutions for these faults are:

1. Select the right epoxy resin system and fillers
2. Completely drying the fillers
3. Full Vacuum
4. Longer cure schedules
5. Correct demolding

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1st Sep 2018

Fault Identification & Possible Reasons

Sl. No.	Fault Identification	Possible Reasons													
		Materials		Inserts, Moulds, Armatures			Preparation			Processing					
		Unsuitable Products	Wet Filler	Poor Design	Cleaning, Pre-treatment	Material Not Compatible	Mixing ratio wrong	Mixture not homogeneous	Vacuum not enough	Poor Casting Process	Curing temp. too low	Curing temp. too high	Curing time too short	Difficult Environment	Poor Design
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Tg too low		■				■				■		■		
2	Casting brittle										■	■	■		
3	Flow-out of liquid					■		■			■		■		
4	Soft surface patches					■		■			■		■		
5	Sticky surface	■					■				■			■	
6	Surface bubbles		■						■						
7	Inside bubbles		■			■			■			■			
8	Inside cracks	■										■			■
9	Surface cracks	■					■	■				■			■
10	Discolouration	■										■			
11	Shrinkage	■		■						■		■			
12	Cracks at Inserts			■	■										■
13	Partial Discharges				■			■	■	■					■
14	Thermal breakdown	■					■	■							
15	Erosion Breakdown				■		■	■	■	■					■
16	Tracking	■												■	■

- **Karishma Prabhu**

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PROFICIENCY TESTING

In Proficiency testing, PT items are sent to a number of participating laboratories by a PT provider accredited as per ISO/IEC 17043. After the testing is completed, the laboratory sends the results to their PT provider and the PT provider grades the test results using statistical calculations as per ISO/IEC 13528 and sends the laboratory their z - scores.

Proficiency testing determines the performance of individual laboratories for specific tests or measurements and is used to monitor laboratories' continuing performance. Routine reviews of PT reports by the laboratory staff and the laboratory director will alert them to areas of testing that are not performing as expected as well as indicate subtle shifts and trends that, over time, would affect their patient results.

Scheduled PT Programs

1. Abrasion Resistance of Rubbers:

Abrasion resistance is the ability of a material to resist mechanical action such as rubbing, scraping, or erosion that tends progressively to remove material from its surface. When a product has abrasion resistance, it will resist erosion caused by scraping, rubbing, and other types of mechanical wear. This allows the material to retain its integrity and hold its form. This can be important when the form of a material is critical to its function, as seen when moving parts are carefully machined for maximum efficiency. Abrasion resistant materials can be used for both moving and fixed parts in settings where wearing could become an issue.

Test Method: ISO 4649

2. Charpy Impact of Plastics:

Charpy Impact is a single point **test** that measures a materials resistance to **impact** from a swinging pendulum. **Charpy impact** is **defined** as the kinetic energy needed to initiate fracture and continue the fracture until the specimen is broken. Impact testing reveals how “tough” a material is. Toughness can be defined as the ability of a material to absorb energy without breaking. High molecular weight favors high toughness. Crystallinity gives higher strength, yet lower toughness; unless the material can transfer the energy through its intermolecular structure. For example, nylon is crystalline and is tough due to the molecular strength of its backbone. Toughness is often considered to be the most critical mechanical property of thermoplastics because it relates to the service life of the part and influences product safety and liability. This test simulates a high-speed flexural test (three-point bend) of the material. Charpy impact is particularly valuable in measuring the effect of micro cracking, flow and weld lines on the parts toughness.

Test Method: ISO 179 / ASTM D 6110

3. Ash Content of Plastics:

The filler content of a material needs to be measured either for quality control purposes or as part of a troubleshooting exercise where verification of a specification for filler content is needed.

Test Method: ASTM D 5630

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- **Prathamesh Phansekar**

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CERTIFIED REFERENCE MATERIALS

Fine Finish RMP Division is accredited as per requirements of ISO Guide 34:2009. CRM (Certified Reference Material) is a reference material which is homogeneous and stable with respect to one or more specified properties and is characterized by a metrologically valid procedure, accompanied by a certificate that states the value of the specified property, its associated uncertainty, and a statement of metrological traceability.

Certified Reference Materials (CRMs) are standards used to check the quality and metrological traceability of products, to validate analytical measurement methods, or for the calibration of instruments. A certified reference material is a particular form of measurement standard.

Reference materials are particularly important for analysis. Since most analytical instrumentation is comparative, it requires a sample of known composition (reference material) for accurate calibration. These reference materials are produced under stringent manufacturing procedures and differ from laboratory reagents in their certification and the traceability of the data provided.

List of Available Certified Reference Materials

1. **Melt Flow Index - ASTM D1238**
2. **Tensile Testing of Composites - ASTM D 3039**
3. **Density of Plastics - ASTM D 792**
4. **Chemical Composition of Metals by Spectrometer (LAS) - ASTM E 415**
5. **Tensile Strength of Rubber - ASTM D 412**
6. **Tensile Strength (Metals) - ASTM E 8**

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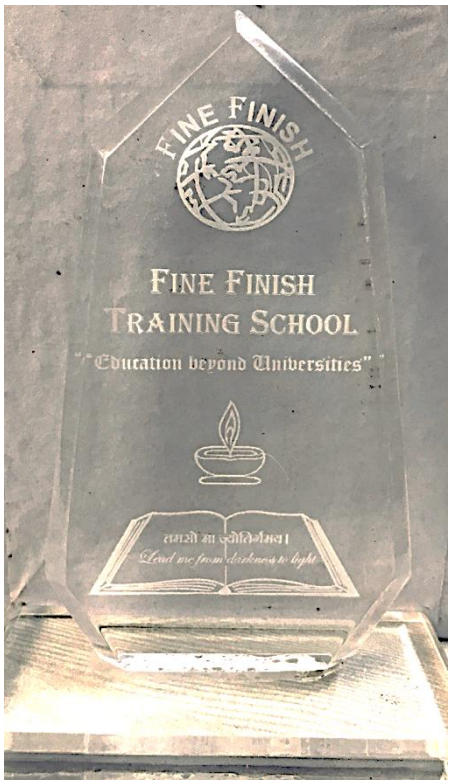
TRAINING & CONSULTANCY

Training is a vital aspect for all industries and hence all our customers are deeply involved with our entire team. Our training school has been imparting training to prospective candidates since last four years. Fine Finish Training School is accredited by National Board for Quality Promotion to conduct training program as per requirements of ISO/IEC 17025. National Board for Quality Promotion is under the aegis of Quality Council of India. Fine Finish Training School has been involved in training participants working in various testing and calibration labs and primarily covered topics are ISO/IEC 17025:2005, ISO/IEC 17025:2017, method validation, measurement uncertainty, proficiency testing as per ISO/IEC 17043:2010, reference material production as per ISO/IEC 17034:2016, ISO 15189:2012 for medical testing labs etc. We have trained close to 300 personnel in various fields and we aspire to encompass various training topics that are prevalent in many industries.

Practical demonstration in Fine Finish Analytical Laboratory

We recently conducted 1-day training in mechanical testing of composites in which we trained lab personnel from various composite manufacturing and testing industries located in Maharashtra, Goa, Andhra Pradesh and Karnataka. We are also organizing 2-day training program in motorette testing for electrical motor manufacturers and rewinders. Motorette testing will be helpful in unravelling quagmire of thermal performance of electrical insulating materials and systems. We are also planning to organize training program in corrosion protection of steel and various other techniques.



Forthcoming Training Programs

Sr. No.	Title	Date	Fees	
			Non-residential	Residential
1	Laboratory Management System and Internal Audit as per ISO/IEC 17025:2017	24 to 27 September, 2018	₹. 14,000/-	₹. 21,000/-
2	1 Day Awareness Training Programme in ISO/IEC 17043:2010.	8 October, 2018	₹. 8,000/-	₹. 10,000/-
3	1 Day Awareness Training Programme as per ISO 9001:2015 for testing & Calibration Laboratories	11 October, 2018	₹. 8,000/-	₹. 10,000/-
4	2 Day Training Programme in Uncertainty of Measurement	15 to 16 October, 2018	₹. 10,000/-	₹. 13,500/-
5	4 Day Training Programme as per requirements of ISO 15189:2012.	22 to 25 October, 2018	₹. 14,000/-	₹. 21,000/-

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