



TECHNOLOGY टेक्नोलॉजी फोकस FOCUS

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Material Development and Characterisation

From the Desk of Guest Editor



Dear Readers,

It gives me immense pleasure in contributing to this knowledge sharing platform amongst DRDO fraternity and civil society. Design and development of cost-effective guided weapon systems come with their own complex challenges of assuring quality and reliability. In our consistent efforts of meeting relevant standards and delivery schedules, analysis and characterisation of employed materials at every stage is indeed a crucial task. It therefore gives me sheer delight in introducing the Materials Development and Characterisation facility at Defence Research Development Laboratory (DRDL) for materials characterisation throughout Dr APJ Abdul Kalam Missile Complex to you all. The Centre has proven its technical competence by obtaining accreditation to NABL for its test facilities as per ISO/IEC 17025: 2005. This first-of-its-kind achievement at organisational level decrees it to be amongst elite service providers throughout the world.

This issue of Technology Focus with its crisp design, has laid ample emphasis upon technical and managerial capabilities and ensured comprehensive detailing; to create awareness throughout DRDO fraternity and civil society about available technologies and invite users to avail the infrastructural facilities.

Though an intricate task, an attempt has been made to compact such facilities in a nutshell through this issue, which is apt as a benchmark; all available technologies/facilities have been broadly listed; future plans have been briefly described. We have tried to showcase the transition of such dexterity from taking baby-steps of growth to leaps of expansion to achieve technical and technological self-reliance.

I sincerely hope that this issue of Technology Focus on Material Development and Characterisation will promote better solidarity in the process of empowerment of the nation.

JAI HIND..!!

M. S. R. Prasad
Distinguished Scientist & Director, DRDL

Material Development and Characterisation

Defence Research and Development Laboratory (DRDL), one of the constituent laboratory working under Defence Research & Development Organisation (DRDO) is a multi-disciplinary missile system laboratory with thrust on design, development and flight evaluation of various types of missile systems for armed forces.

Right from conceptualisation of the weapon system to production and induction into services, the laboratory has capabilities and the state-of-the-art infrastructure in terms of facilities and human resource. The laboratory is ISO 9001:2015 Certified. It design, develop and lead to production of cost-effective guided weapon systems of assured quality and reliability with continual improvement, meeting the safety standards and time schedules.

The laboratory is equipped with material development and characterisation facility. Materials Development Division (MDD) of DRDL is capable of catering to users' requirements by employing available world-class equipments while conforming to stringent aerospace quality and standards. Expert guidance from Defence Metallurgical Research Laboratory (DMRL) and availability of requisite equipment has nourished MDD to a stage that it provides round-the-clock services through its routine activities to all technology and

project directorates in Dr APJ Abdul Kalam Missile Complex. The facility has matured to a stage and various laboratories like Defence Metallurgical Research Laboratory (DMRL), Regional Centre for Military Airworthiness (RCMA), Combat Vehicle Research & Development Establishment (CVRDE), Bharat Dynamics Limited (BDL), Hindustan Aeronautics Limited (HAL) and other institutions like Missile System Quality Assurance Agency (MSQAA) and Aeronautical Development Agency (ADA) show faith by getting their jobs done in MDD. State-of-the-art technologies and sophisticated/custom-built facilities established by experts are indeed a boon to its productivity. Every machine, no matter how small or big, is being utilised and maintained continually and geared up to accomplish challenging tasks.

With grant of NABL accreditation to its test facilities in mechanical, chemical and metallography disciplines, DRDL has got yet another feather added in the cap. This unique achievement at organisational level has recognised technical competence and efforts to deliver.

The vision of MDD is:

- ◇ To be a pioneer centre of development of new materials and processes for application in missile systems
- ◇ To outshine in the area of

material characterisation by employing latest technology advancements

- ◇ To deliver quality output within stipulated time-frame and meet user requirements

Its mission is

- ◇ To be a unique centre for material characterisation in the missile complex
- ◇ To solve technical issues related to material testing and characterisation
- ◇ To interact with core R&D institutes and gain technical expertise in relevant fields
- ◇ To be up-to-date with latest technology advancements
- ◇ To meet delivery schedules of users
- ◇ To develop new materials and processes to cater to all projects

MDD has been broadly categorised under three laboratories based on the area/nature of work:

- ◇ Chemical Analysis
- ◇ Mechanical Testing
- ◇ Metallography

All equipment are state-of-the-art and fully capable of meeting user requirements.

Facilities Available

Optical Emission Spectrometer (DV-4)

DV-4 spectrometer model was procured way back in 1987 and upgraded to MC-20 read-out system in 2009. Further, in 2012, it was upgraded with extra elemental channels of Niobium and Titanium to facilitate analysis of PH-steels and Ti-base alloys.

The DV-4 spectrometer has an incorporated optics module with greatest sensitivity to even trace elements like Carbon (C), Sulphur (S), Lead (Pb), Boron (B), Stannum (Sn) and Potassium (K).

- ◇ Excitation source: KH-3 with an upgraded MC-20 read-out system
- ◇ Specimen size: Flat sample of \varnothing 6 mm–50 mm and thickness 6 mm–25 mm
- ◇ Purging media: Argon gas of 99.96 % purity

Optical Emission Spectrometer (DV-6)

High Energy Pre-burn Spark (HEPS) system of this model homogenises the sample, leading to accuracy comparable to x-ray spectroscopic techniques. DV-6 spectrometer has versatility in analysis of diverse ferrous and non-ferrous alloys.

In 2013, this spectrometer was upgraded with latest version of analysis software and re-calibrated for Iron (Fe), Aluminium (Al), Copper (Cu), Titanium (Ti), Nickel (Ni) base alloys.

- ◇ Excitation source: HR-400 with MC-20 read-out system
- ◇ Specimen size: Flat sample of \varnothing 15 mm–50 mm and thickness 6 mm–25 mm
- ◇ Purging media: 99.96 % pure Argon

Portable x-ray Fluorescence Analyser

This equipment is based on energy dispersive x-ray fluorescence principle. In addition to being portable, it is weather-proof and dust-proof. Fast and precise non-destructive identification of individual elements in diverse ferrous and non-ferrous alloys is however limited to only heavier metals as trace elements cannot be detected.

- ◇ Excitation source: x-ray tube, W-anode, 10-40 KeV, 5-80 μ A, five-filter positions
- ◇ Detector source: Si PIN diode, thermo-electrically cooled, with high resolution



Optical Emission Spectrometer (DV-4)



Optical Emission Spectrometer (DV-6)

Electric Arc Re-melt Furnace

This equipment is utilised to prepare samples for spectrometers out of drillings, chips, turnings, pins and wires. The re-melt furnace has a pre-installed vacuum pump to avoid contamination of melt. Violence in generated arc creates turbulence and stirring of melt delivering homogenous sample. It has versatility in re-melting all ferrous and non-ferrous alloys and provision of cooling of crucible through re-circulating DM water.

- ◇ Heat source: Electric arc between W-anode and raw-material cathode
- ◇ Maximum temperature: 1900 °C
- ◇ Purging media: 99.96 % pure Argon



Direct Reading Optical Emission Spectrometer

This equipment boasts of accurate and reliable analytical performance with high sensitivity to even low Carbon and Nitrogen alloy grades. In-built vacuum optics reduces contamination and Argon consumption.

- ◇ Wavelength range: 130 nm–800 nm using high resolution multi-CCD optics
- ◇ Specimen size: Surface area of 1 mm² onwards
- ◇ Other features: High energy pre-spark, digital plasma generator, Argon purge



Simultaneous ONH Analyser

This equipment is designed for simultaneous detection of Oxygen, Nitrogen and Hydrogen content in steels, refractory materials and other inorganic materials. The detection system comprises both Non Dispersive Infra Red (NDIR) and Thermal Conductivity (TC) detectors which deliver quality results with high precision.

- ◇ Furnace: up to 3000 °C
- ◇ Measurement Range:
Oxygen-1 ppm to 5 %
- ◇ Nitrogen-1 ppm to 3 %
- ◇ Hydrogen-0.100 ppm to 0.25 %
- ◇ Cycle time: Less than 2 min

250 kN Electromechanical Testing Machine

The 250 kN Universal Testing Machine (UTM) is fully loaded to address all kinds of mechanical tests. Features like compression testing, three-point bend testing and temperature range of the equipment make it a state-of-the-art facility

- ◇ Load range: 25 kg-25000 kg
- ◇ Accuracy: ± 5 %
- ◇ Temperature range: -150 °C-+1100 °C
- ◇ Extensometers: 10/25/50 mm

Axial Spring Testing Machine

The 1 kN UTM was primarily meant for axial compression testing of springs for stiffness measurements. However, with requisite fixtures, it now caters to testing of wires and polymers.

- ◇ Load range: 5 N-1000 N
- ◇ Accuracy: ±1 %
- ◇ Maximum stroke: 600 mm
- ◇ Cross-head speed: Up to 1000 mm/min



25 kN Servo Hydraulic UTM

Project LRSAM had projected critical and urgent requirement of inhouse testing of composite solid rocket propellant material in combinations like very low (0.005 mm/min) and very high (4000 mm/min) cross-head speeds and assorted temperatures. Based on those requirements, MDD formulated installation of this UTM which is now a unique facility throughout India and has not only helped save organisational funds but also sped up the testing activity.

- ◇ Load capacity: 25 kN
- ◇ Stroke: 150 mm
- ◇ Speed: 0.1–5000 mm/min
- ◇ Test environment: -70 °C to +100 °C



100 kN Servo Hydraulic UTM

This testing system is equipped with wide range of grips, fixtures and accessories to cover almost entire spectrum of mechanical testing. In-built Low Cycle Fatigue (LCF) and fracture mechanics (K1C, J1C) applications ensure versatility in testing of materials.

- ◇ Load capacity: 100 kN
- ◇ Stroke: 100 mm
- ◇ Sample size: 0–19 mm thick flat, Ø 2.5–16 mm round



200 kN/400 kN Hydraulic UTM

These robust machines offer reasonable results within the load range defined. Both machines are driven hydraulically and an attached control panel ensures operation in both automatic and manual mode. Both machines were reconditioned from dilapidated condition and made functional.

- ◇ Load capacity: 200 kN/400 kN
- ◇ Stroke: 500/750 mm
- ◇ Sample size: Assorted



Universal Pendulum Impact Testing Machine

This equipment has also been refurbished to its full functionality and is being duly calibrated. Charpy and Izod impact tests can be carried out on these equipment, conforming to IS standards.

Test Type	Max. Impact Energy of Pendulum	Min. Value of Scale Gradation
Charpy test	300 J	2 J
Izod test	168 J	2 J



Digital Universal Hardness Testing

It is proclaimed to be a single-stop solution for all kinds of indentation hardness measurements. This machine has versatility across all scales state-of-the-art and can efficiently handle jobs of any size owing to its test-height and throat-depth.

- ◇ Test height: 450 mm
- ◇ Throat depth: 250 mm
- ◇ Load range: 0.5–250 Kgf
- ◇ Measurements: Rockwell, Knoop Brinell, Vickers,



Automatic Abrasive Cut-off Machine

This heavy duty machine is capable of cutting \varnothing 80 mm solid steel bar within 10 minutes and is electro-hydraulic. The cutting parameters can be manipulated as per the requirements

as the equipment is programmable and fully automatic. A wide range of materials can be cut with ease, beginning from pure Aluminum to high strength steels owing to its high capacity motor.

S a m p l e s extracted from cutting are subjected to subsequent sample preparation procedures.



Sample Mounting Machine

The mounting machine is fully automatic and programmable to mount small samples to facilitate subsequent polishing procedures. It is simple, robust and vital equipment for metallography and sample preparation.

- ◇ Maximum temperature: 200 °C
- ◇ Mount sizes: Ø 26 mm, 28 mm, 32 mm and 50 mm respectively



Automatic Dual Disc Sample Polishing

With automation, this machine is capable of polishing eight samples of Ø 26 mm in one go. An attached magnetic stirrer ensures uniform suspension of powdered Alumina used for lapping.

- ◇ Polishing disc: Ø 200 mm
- ◇ RPM of disc: 50–800 RPM
- ◇ RPM of specimen-holding head: 60 RPM



Portable Residual Stress Analyser

Imported, computer-controlled, fully automatic and portable equipment used for measuring surface and sub-surface residual stresses in a material.

The equipment is based on the principle of Bragg's law of diffraction and evaluates changes in the inter-planar spacing of specimen's crystal lattice due to inherent stresses in the material.



Muffle Furnace

This furnace was primarily meant for inhouse R&D activities and small-scale heat-treatment operations.

However, owing to its high temperature capabilities, the muffle furnace often serves to various projects in their routine activities.

- ◇ Temperature: RT-1200 °C
- ◇ Maximum heating rate: 10 °C/min
- ◇ Chamber size: 350X350X350 (mm)



Stereo Microscope

Primarily used for weld qualifications, coating thickness measurements and failure analysis, Stereo microscope is a vital tool for a metallurgist to accomplish one's activities. With ring-light and dual-LED gooseneck lights, ample lighting is ensured to analyse even dull and pale fracture surfaces and observe crack initiation and propagation features. Magnification ranges from 6.3 X to 95.4 X



Metallurgical Microscope

With features like reflected bright-field/dark-field/DIC observations, MDD is capable of doing image analysis and microstructural characterisation of all metallic materials. The associated software has diverse features like image processing, interactive image measurements and phase analysis.

- ◇ Light intensity range: continuously variable from 1.0–12.0 v DC
- ◇ Focusing system: 25 mm max. with 0.1 mm fine and 17.8 mm coarse stroke per rotation
- ◇ Magnification range: 50X–1000X



Metallurgical Microscope

Digital Micro Hardness Tester

Primarily used for analysis of welds and surface coatings, this equipment is versatile for micro-hardness measurements on diverse metallic and non-metallic materials.

Features like fully automated load-hold-release mechanism and multi-line/random measurements are accompanied with data accuracy as per ASTM standards.

- ◇ Measuring modes: HV and HK
- ◇ Load range: 5 gf–1000 gf
- ◇ Magnifications: 100X and 400X
- ◇ Test-table size: 100 mm X 100 mm (with a max stroke of 25 mm along X and Y axis)
- ◇ Dwell time: Variable from 5–99 sec (with 1 sec interval)



Digital Micro Hardness Tester

Electrolytic Polishing and Etching

This table-top equipment is programmable, automatic and has provision of electrolytic polishing and etching of diverse varieties of metallic materials in a matter of just few minutes thereby cutting down the time of sample preparation drastically. Integrated chiller maintains the bath at $-10\text{ }^{\circ}\text{C}$.



Experimental Set-up for Determination of Fiber Volume Fraction for Composite Samples

This test method determines constituent content of composite material which consists of matrix (resin) and reinforcement (C-fibre). The matrix is removed by hot acid digestion leaving the reinforcement essentially unaffected and the weight percent of the reinforcement is calculated. If the densities of both composite and reinforcement are known, the volume per cent or volume fraction is calculated. This is one of the parameters for the characterisation of composite materials.



Determination of Open Porosity of Graphite Samples

Refractory materials are used for demanding conditions and need to be sound, robust and capable. Material properties like density, porosity, permeability are indication of manufactured quality and during the service these properties influence strength, slag resistance, flow of gases and liquids, thermal conductivity and thermal shock resistance of the refractory. Understanding these properties and their interactions will help users to understand the performance of these materials better.

Open porosity is the percentage of the total volume of the material occupied by both open and closed pores. Pore size and distribution in the carbon system are the keys to its performance particularly in a re-entry environment.



Scanning Electron Microscope

A state-of-the-art equipment with Tungsten heated cathode for low/high/extended vacuum operations that are best suited for materials science, metallography, failure analysis of metallic materials. It features flicker-free digital imaging of superior resolution and user-friendly interface that is easy to comprehend. Specimen stage is fully motorised for movement along five axes.

Technical Specifications:

- ◇ Accelerating voltage: 200 V to 30 kV
- ◇ Magnification: 20X to 10,00,000X
- ◇ Resolution: 3 nm at 30 kV (SED) and 4 nm at 30 kV (BSED)
- ◇ Specimen size: Max. Ø 50 mm and Max. 50 mm
- ◇ EDS: Oxford make with resolution 131 eV



Differential Scanning Calorimeter

The equipment caters to measurement of transition temperatures, specific heat, heat of transition, etc. and is applicable to polymeric and composite materials.

Technical Specifications:

- ◇ Type: Heat flux/Power compensation
- ◇ Temperature range: -150 °C to +700 °C
- ◇ Heating rate: ≥ 100 °C/min
- ◇ Atmosphere: inert (Ar/N/O), Vacuum (10^{-4})



Thermo Mechanical Analyser

This equipment serves to the requirements of measurement of deformation characteristics of solid polymers, films, fibers, thin films, coatings, viscous fluids and gels. The analyser measures linear coefficient of expansion, glass transition, dimensional stability, creep and tensile modulus can also be derived.

Technical Specifications:

- ◇ Temperature: -150 °C to +1000 °C
- ◇ Sensitivity: 20 nm
- ◇ Force range: 0.01 to 2 N (Resolution: 0.001 N)
- ◇ Atmosphere: inert (Ar/N/O), Vacuum (10^{-4})



Thermo Gravimetric Analyser facility is also available from room temperature to 1000 °C.

Torsion Testing Machine

Torsion testing machine is compact, table top, fully automatic and computer controlled that features repeatability and accuracy on angle within $\pm 2^\circ$. The equipment is extensively used for testing of springs under torsion loads.

Technical Specifications:

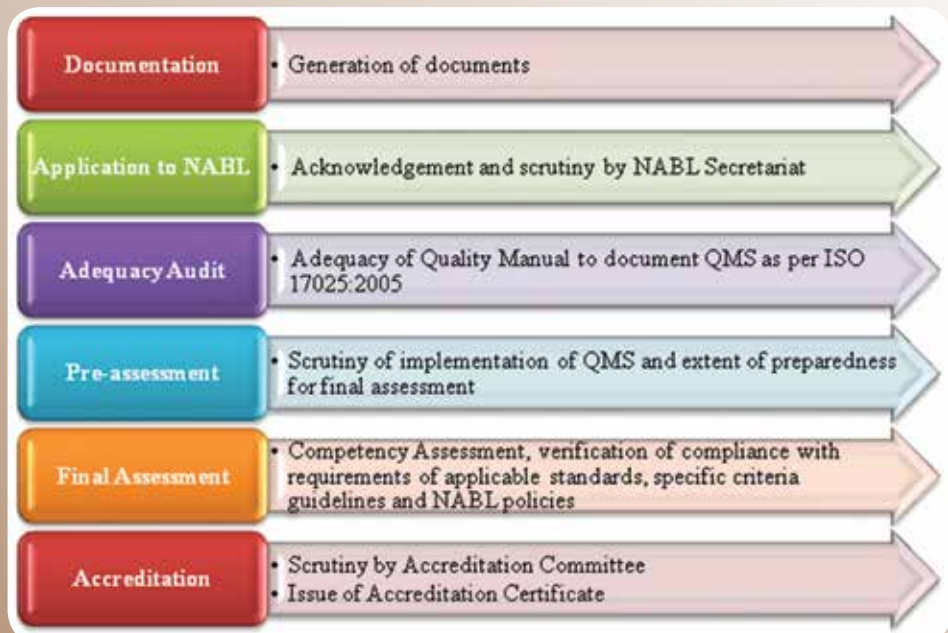
- ◇ Torque range: 0.2 NM–20 NM
- ◇ Spring inner diameter: 2.4 mm–20 mm
- ◇ Max. spring length: 300 mm
- ◇ Torque resolution: 0.0001 NM



NABL ACCREDITATION TO TEST FACILITIES

Obtaining certification from National Accreditation Board for Testing & Calibration Laboratories (NABL) for all test facilities of MDD was commenced in September 2015. In spite of an existing framework of well established management system of DRDL certified to ISO 9001:2015, accreditation of MDD test facilities to NABL was indeed a tedious task with its own challenges.

As evident, activities pertaining to NABL are consistent and diligent. Even miniscule aspects like environmental conditions of testing, standardisation procedures, personnel training and safety apparatus demand attention and meticulous monitoring.





National Accreditation Board for Testing and Calibration Laboratories
SCOPE OF ACCREDITATION

Specific Test Parameters	Test Method Specifications	Range of Testing/Level of Detection
Chemical Analysis	ASTM E299	0.1% to 100%
Control	ASME B79.10.1	0.1% to 100%
Leak Detection	ASTM E524	0.1% to 100%
Strength	ASTM E8	0.1% to 100%
Welding	ASME B79.10.1	0.1% to 100%

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National Accreditation Board for Testing and Calibration Laboratories
CERTIFICATE OF ACCREDITATION

**MATERIALS DEVELOPMENT DIVISION
DIRECTORATE OF ADVANCED MATERIALS & PROCESSES, DRDO**
has been assessed and accredited in accordance with the standard
ISO/IEC 17025:2015
'General Requirements for the Competence of Testing & Calibration Laboratories'
for its facilities at
Kanchanbagh P.O., Hyderabad, Telangana
in the field of
TESTING

Certificate Number: TC-007
Issue Date: 14/05/2017

This certificate remains valid for the Scope of Accreditation as specified in the statement subject to continued satisfactory compliance to the above standard & the relevant requirements of ISIRI.

Signed By and in Behalf of: **N. Venkateswaram** Program Director

Signed By: **Ajit Patil** Chief Executive Officer

Roadmap for Future

MDD has been consistently striving to do better in all aspects with sole aim of exceeding user satisfaction. MDD has set up few activities on the roadmap to achieve its ultimate aim.

Establishment of New Test Facilities

MDD had initiated a few procurement activities for establishment of new test-systems.

Instrumented Impact Testing Machine

The maximum capacity of equipment is 450 J with a resolution better than 0.1 J and maximum velocity of 5.5 m/s. The equipment conforms to all relevant national/international standards. Accessories like specimen tempering unit and notching machine make it a single-stop solution for impact testing of metallic materials.

Portable Liquid Nitrogen Plant

To address to minimal requirements of equipment installed in MDD, this portable plant is procured with a capacity of 20 litres per day with minimum purity of 99 per cent.

Materials Development Division (MDD), Defence Research and Development Laboratory (DRDL) urges DRDO fraternity to give an opportunity for material testing and characterisation requirements

Please feel free to contact



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