भाभा परमाणु अनुसंधान केंद्र का आधिकारिक द्विमासिक प्रकाशन
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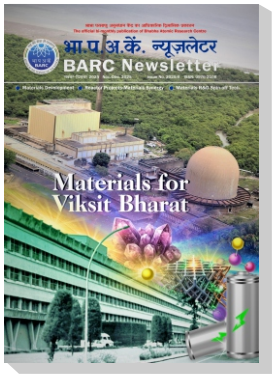
- Materials Development
- Reactor Projects-Materials Synergy
- Materials R&D Spin-off Tech

Materials for Viksit Bharat



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बेहतर भविष्य हेतु पदार्थ

पदार्थ विज्ञान ने दीर्घ-काल से परमाणु ऊर्जा विभाग के अनुसंधान एवं रिएक्टर विकास कार्यक्रमों के लिए एक निर्णायक नींव के रूप में कार्य किया है। पदार्थ वर्ग समाचार पत्र के इस विशेष अंक में वैज्ञानिक प्रगति को रेखांकित करने वाले लेखों के विविध रचनाओं को समेकित किया गया है, जिसमें - प्रगत मिश्रधातु विकास एवं उच्च-तापमान वाले चीनी मिट्टी से लेकर आधुनिक इलेक्ट्रॉन सूक्ष्मदर्शी, अत्याधुनिक यांत्रिक गुण-धर्म अभिलक्षणन का वर्णन, योजक निर्माण, उत्प्रेरक, नाभिकीय पदार्थ प्रक्रमण और सामरिक खनिज निष्कर्षण तक शामिल हैं। प्रत्येक लेख इस बात पर प्रकाश डालता है कि कैसे मौलिक समझ, निरंतर प्रयोग और स्वदेशी प्रौद्योगिकी विकास नाभिकीय क्षेत्र की वैज्ञानिक एवं अभियांत्रिक चुनौतियों का सामना करने के लिए एकजुट रहे हैं और नाभिकीय क्षेत्र से परे समाज एवं उद्योग के लिए स्पिन-ऑफ लाभ प्रदान करने में अग्रणी भूमिका अदा कर सकते हैं।

इस अंक के लेखों में, पदार्थ वर्ग के कई प्रभागों द्वारा अर्जित महत्वपूर्ण प्रगति को दर्शाया गया है। इसके अलावा, पाठकों को पदार्थ वर्ग, भापअ केंद्र के पदार्थ विज्ञान से संबंधित चतुर्दिक गतिविधियों का रसास्वाद करवाने हेतु भापअ केंद्र, पऊवि की विभिन्न इकाइयों और शैक्षणिक संस्थानों के अनुसंधान समूहों के साथ सफल सहयोग के चुनिंदा विषयों को भी शामिल किया गया है। इनमें, रिएक्टर दाबा पात्र हेतु बैनेटिक स्टील्स में हुई प्रगति, द्रविभूत-लवण प्रौद्योगिकियों के लिए Ni–Cr–Mo मिश्रधातु; विभिन्न आयाम एवं अवधि के पैमाने पर पदार्थ परीक्षण में विकास; चुंबकीय पदार्थ; बोराइड-आधारित अवशोषक; ऑक्सीकरण-प्रतिरोधी लेपन; उच्च प्रदर्शन वाले शीशे, चीनी मिट्टी और यौगिकों; हाइड्रोजन उत्पादन के लिए छिद्रयुक्त चीनी मिट्टी के पात्र; उभरते रिएक्टर अवधारणाओं के लिए संरचनात्मक पदार्थ; और iDPC-STEM और PIGE सहित नवीन विश्लेषणात्मक क्षमताओं पर आधारित लेख शामिल हैं। इनमें लेसर योजक निर्माण, नैनोपार्टिकल संश्लेषण के माध्यम से ई-अपशिष्ट पुनश्चक्रण, बृहत पैमाने पर नैनोट्यूब उत्पादन और उद्योग को हस्तांतरित प्रौद्योगिकियों का एक व्यापक पोर्टफोलियो जैसी उपलब्धियों के विषय भी शामिल हैं जोकि देश की आत्मनिर्भरता को सुदृढ़ करते हैं। समग्र रूप से, ये विकास अगली पीढ़ी की रिएक्टर प्रणालियों को सक्षम बनाने, सुरक्षा मार्जिन का विस्तार करने और देश के स्वदेशी प्रौद्योगिकी आधार को मज़बूत करने में पदार्थ वर्ग की केंद्रीय भूमिका को रेखांकित करते हैं।

यह अंक, डॉ. राघवेंद्र तिवारी, विशिष्ट वैज्ञानिक एवं निदेशक, पदार्थ वर्ग को समर्पित है, जिन्होंने नाभिकीय पदार्थ अनुसंधान में अतुलनीय वैज्ञानिक कार्यों के माध्यम से स्थायी योगदान किया है। यहाँ प्रस्तुत लेखों में से फेज़ कायांतरण, इलेक्ट्रॉन सूक्ष्मदर्शी, जर्कोनियम धातु विज्ञान एवं संरचनात्मक पदार्थ आदि उनके योगदान के माध्यम से ही साकार हो सके हैं। उनका नेतृत्व सहयोगात्मक समस्या-समाधान, कठोर लक्षण वर्णन पद्धतियों और वैज्ञानिक अनुशासन की संस्कृति को पोषित करने में भी समान रूप से महत्वपूर्ण रहा है। इस अंक में शामिल शोध का विस्तार उस वैज्ञानिक पारिस्थितिकी तंत्र को दर्शाता है जिसे बनाने में उन्होंने मदद की और भाभा परमाणु अनुसंधान केंद्र, नाभिकीय ईंधन सम्मिश्र, राजा रमन्ना प्रगत प्रौद्योगिकी केंद्र, इंदिरा गांधी परमाणु अनुसंधान केंद्र तथा शैक्षणिक संस्थानों में उनकी साझेदारी को बढ़ावा दिया।

हम, आशा करते हैं कि यह विशेष अंक हाल में प्राप्त उपलब्धियों के अभिलेख तथा पदार्थ अनुसंधान, विकास एवं प्रौद्योगिकी रूपांतरण में निरंतर नवाचार के लिए एक प्रेरक का कार्य करेगा। हम, सभी योगदानकर्ताओं द्वारा किए गए प्रयासों सहित पदार्थ वर्ग की वैज्ञानिक गतिविधियों के साथ उनके निरंतर जुड़ाव के लिए पाठकों के प्रति हार्दिक आभार प्रकट करते हैं।

डॉ. राम निवास सिंह
उत्कृष्ट वैज्ञानिक एवं प्रमुख, यांत्रिक धातुकर्म प्रभाग
पदार्थ वर्ग, भाभा परमाणु अनुसंधान केंद्र

foreword

Materials for a better tomorrow

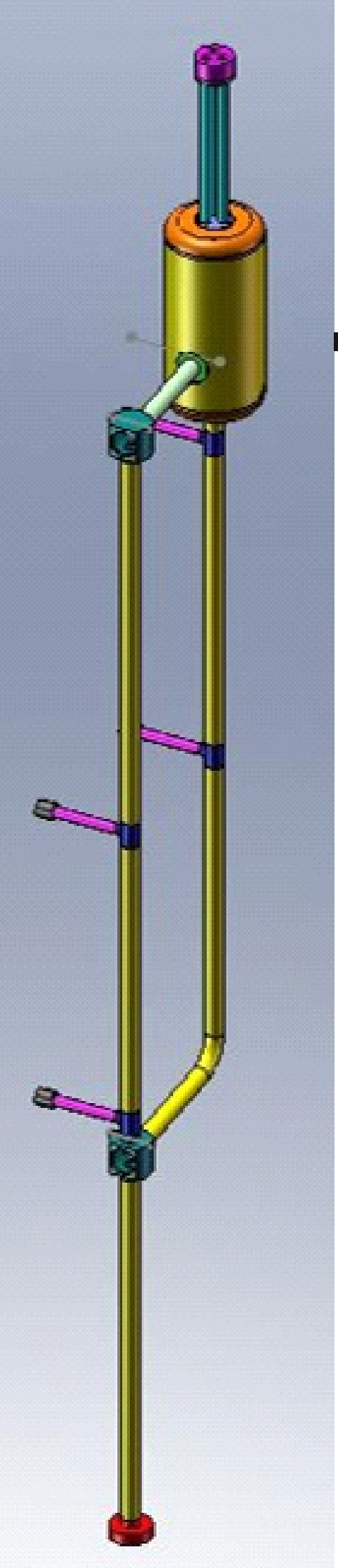
Materials science has long served as a decisive foundation for the Department of Atomic Energy's research and reactor development programmes. This special issue of the Materials Group Newsletter brings together a diverse set of articles reflecting this scientific breadth—from advanced alloy development and high-temperature ceramics to modern electron microscopy, state-of-the-art mechanical property characterization, additive manufacturing, catalysis, nuclear materials processing, and strategic mineral extraction. Each contribution highlights how fundamental understanding, sustained experimentation, and indigenous technology development converge to meet the scientific and engineering challenges of the nuclear sector and can lead to spin-off benefits to society and industry beyond the nuclear realm.

The articles in this issue illustrate the significant progress achieved across multiple divisions of the Materials Group. In addition, selected cases of successful collaborations with other research groups from BARC, other DAE units and research groups from academic institutions have also been included to give readers a flavour of the breadth and depth of the materials related activities of Materials Group, BARC. They document advances in bainitic steels for reactor pressure vessels; Ni–Cr–Mo alloys for molten-salt technologies; developments in materials testing at various length and time scales; magnetic materials; boride-based absorbers; oxidation-resistant coatings; high-performance glasses, ceramics and composites; porous ceramics for hydrogen production; structural materials for emerging reactor concepts; and innovative analytical capabilities including iDPC-STEM and PIGE. Also featured are achievements in laser additive manufacturing, e-waste recycling through nanoparticle synthesis, large-scale nanotube production, and a broad a portfolio of technologies transferred to industry—each reinforcing national self-reliance. Together, these developments underscore the Materials Group's central role in enabling next-generation reactor systems, expanding safety margins, and strengthening the country's indigenous technology base.

This issue is published in honour of Dr. Raghvendra Tewari, Distinguished Scientist & Director, Materials Group, whose distinguished scientific career has made enduring contributions to nuclear materials research. His work in phase transformations, electron microscopy, zirconium metallurgy, and structural materials has shaped many of the themes represented here. Equally significant has been his leadership in nurturing collaborative problem-solving, rigorous characterization methodologies, and a culture of scientific discipline. The breadth of research showcased in this volume reflects the scientific ecosystem he helped build and the partnerships he fostered across BARC, NFC, RRCAT, IGCAR, and academic institutions.

We hope this special issue serves both as a record of recent accomplishments and as an inspiration for continued innovation in materials research, development and technology translation. We thank all contributors for their efforts and the readership for their continued engagement with the scientific activities of the Materials Group.

Dr. Ram Niwas Singh
Outstanding Scientist &
Head, Mechanical Metallurgy Division
Materials Group, BARC



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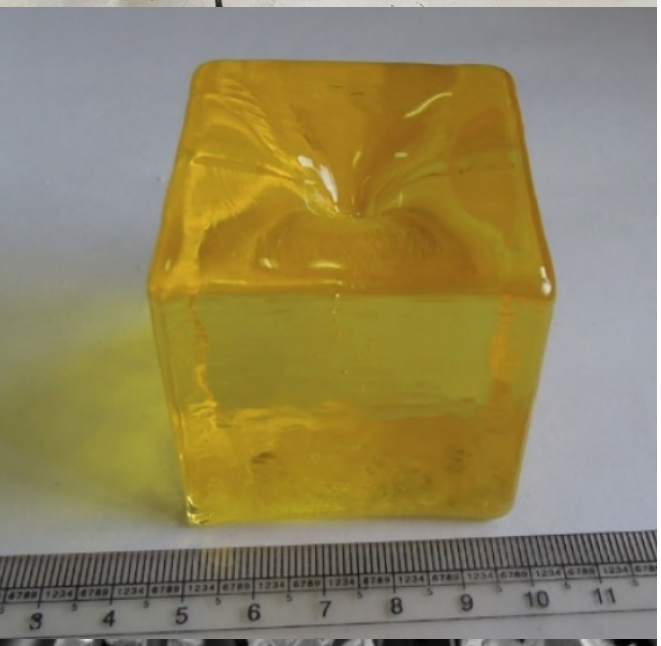

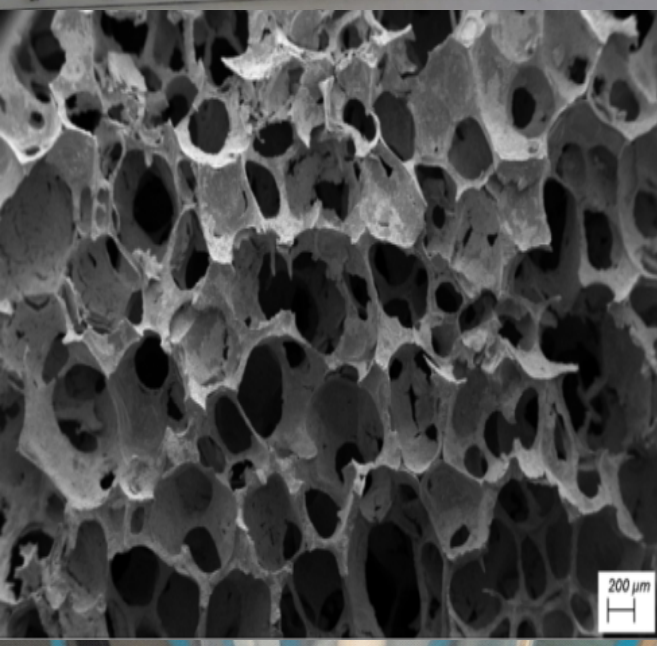

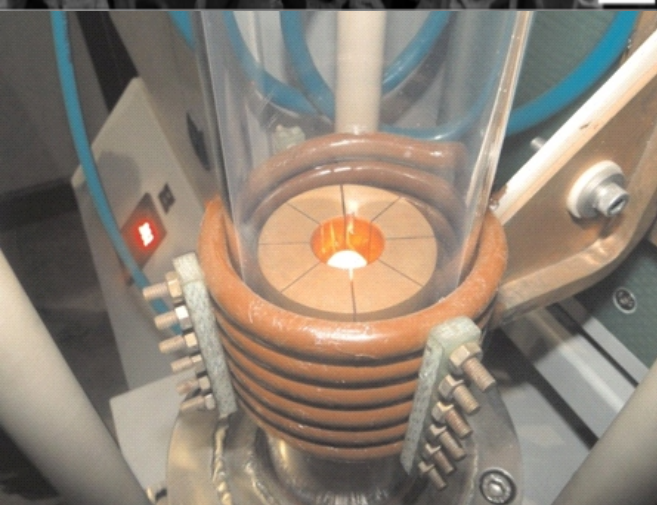

Materials Science
Research & Development

driving multidisciplinary nuclear energy expansion

पदार्थ विज्ञान प्रभाग, भाभा परमाणु अनुसंधान केंद्र में पदार्थ विकास गतिविधियाँ

1

Materials Development Activities at Materials Science Division in BARC

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Materials being at the forefront of any societal advancement, Materials Science Division at BARC is actively engaged in the pursuit of development of materials with improved properties and performance for applications, spanning across in-core and out-of-core structural materials for nuclear reactors to materials to be used as magnets. This article presents a glimpse of this journey for three specific materials: (1) Bainitic steel for reactor pressure vessel (RPV) applications; (2) Ni-Cr-Mo alloy for molten salt breeder reactor (MSBR) applications; and (3) Fe-Co and Co–Zr based alloys for use as soft magnets and permanent magnets.

Development of Low-carbon Carbide-free Bulk Nanostructured Bainitic Steel

Carbide-free bainitic steels consisting of a fine network of bainitic ferrite platelets interspersed by thin retained austenite films could emerge as a material of choice for applications demanding high strength with satisfactory fracture toughness, like RPV. Achievement of uniformity of microstructure and properties across thick sections (~ 400-800 mm) is paramount for RPV. This poses challenges to fabrication routes relying on rapid heating/cooling to generate specific microstructural features. In view of this, Materials Science Division designed a low-carbon (0.23 wt.%) carbide-free bainitic steel composition, which would enable the development of a nanobainitic microstructure in bulk without resorting to faster heating/cooling cycles, together with exhibiting better microstructural uniformity, high strength and toughness, lower residual stresses, better fabricability, and good weldability. It was possible to demonstrate in the designed steel composition, following melting and forging, the evolution of a carbide-free bainitic microstructure through both isothermal holding (Fig.1) as well as via continuous cooling at very slow rates. The carbide-free fine bainitic microstructure displayed a tensile strength of ~ 1.5 GPa, tensile elongation of ~ 20% and fracture toughness of ~ 65 MPa.$m^{1/2}$.

Development of Ni-Cr-Mo Alloy

Ni-based alloys are suitable for MSBR applications due to their excellent molten salt corrosion resistance, high-temperature strength, and good weldability. To further improve these properties, the composition of the alloy was optimized based on thermodynamic calculations and laboratory scale melting (Table 1). Compression tests were carried out to develop processing map (Fig.2a), which identified 1150-1200^{0}C and 10^{-3}-10^{-2} s^{-1} as temperature and strain rate windows, respectively, to achieve dynamic recrystallization. The optimized processing conditions helped in successful forging of a large-scale ingots (~ 2 tons) without cracking. Heat treatment experiments revealed 1080 ^{0}C/30 mins as recrystallization parameters to generate strain-free grains (Fig.2b). In collaboration with NFC, several tubes were fabricated from the forged billets with properties superior to the design requirements. Aging treatments were performed up to 6500 h at 470 ^{0}C to study microstructural changes during operation. Fig.2c shows a high-resolution electron micrograph of the aged

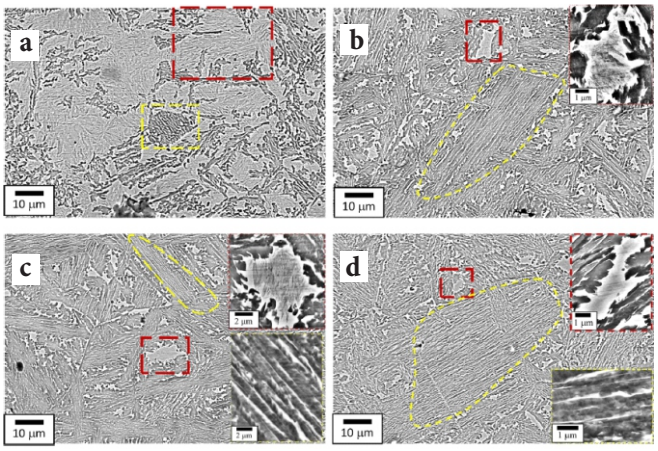

Fig.1: BSE-SEM micrographs of bainitic microstructure after isothermal treatment at (a) 450 ^{0}C, (b) 425 ^{0}C, 400 ^{0}C and (d) 375 ^{0}C. The regions marked in red and yellow, and their magnified insets, highlight martensite-austenite islands and film austenite, respectively.

Table 1: Optimized composition of the Ni-Cr-Mo alloy

Elements	Ni	Cr	C	Mo	Ti
Composition (wt.%)	78.8	7.65	0.07	12.28	1.2

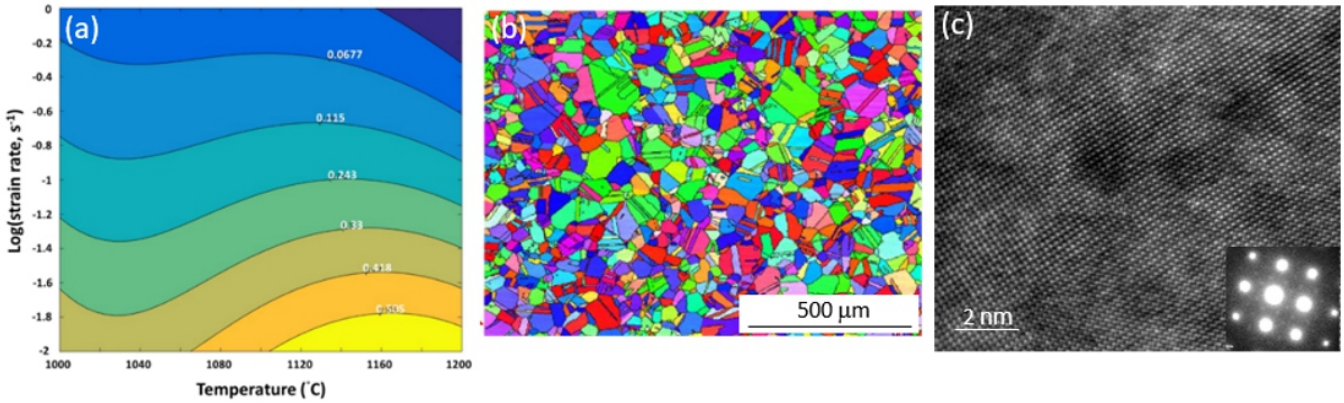

Fig.2: (a) processing map; (b) recrystallized microstructure; (c) high resolution electron micrograph (inset SAED pattern) of the aged Ni-Cr-Mo alloy.

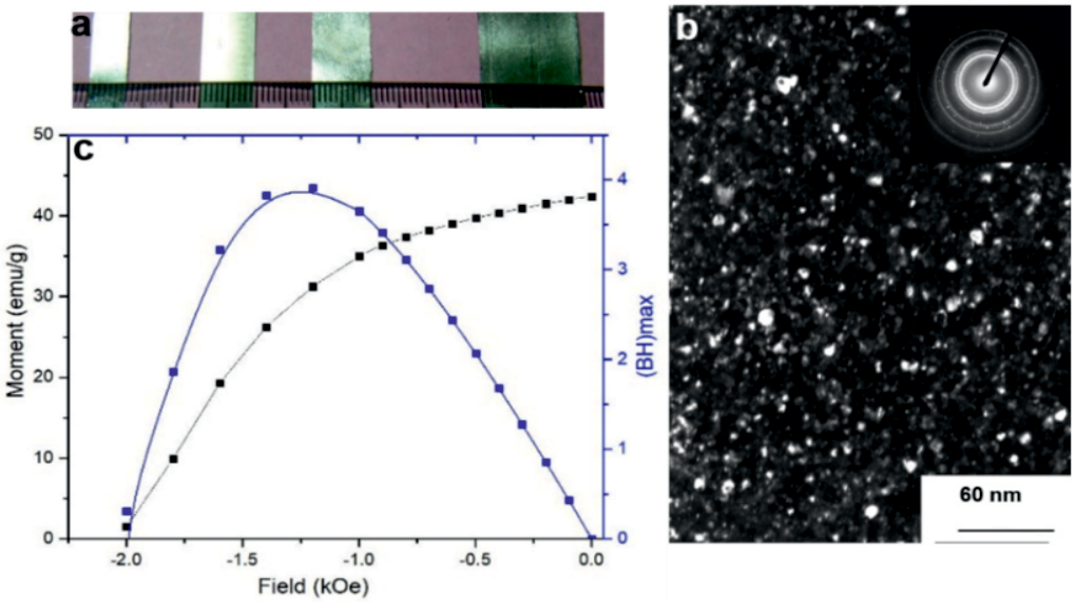

Fig.3: (a) Soft magnetic ribbons of varying width; (b) TEM micrograph showing a typical microstructure required for extremely soft magnets; (c) Magnetic moment and energy product of Zr-Co based metallic ribbon.

sample, revealing the formation of short-range ordered domains. The alloy exhibited weight loss comparable to that of commercial Hastelloy-N in molten salt corrosion experiments at 700 ^{0}C in FLiNaK salt. Welding trials using GTAW, electron beam welding, and laser welding were conducted. The weld joints satisfied ASME tensile and bend test requirements and exhibited better corrosion resistance than base material.

Development of Fe-Co and Co–Zr Based Alloys

Materials Science Division has developed the know-how for producing Fe- and Co- based soft magnetic metallic glasses in ribbon form, with widths ranging from 3 mm to 25 mm. Fig.3a shows soft magnetic ribbons of varying widths fabricated from the same alloy. These materials can exhibit magnetic inductions in the range of 1.6 – 2.1 Tesla, along with high permeability and elevated Curie temperatures. A minimum coercivity as low as 1 A/m can be achieved, and it can further be tuned to desired values through nanoscale microstructural tailoring. A representative microstructure corresponding to such optimized magnetic properties is shown in Fig. 3b.

In parallel, Materials Science Division is also engaged in the development of rare-earth-free permanent magnets with an energy product exceeding 10 MGOe. This goal is being pursued through nanoscale microstructural engineering to integrate soft and hard magnetic phases within nanocomposite materials. A Zr-Co based alloy has been identified as a promising candidate due to its high magnetic anisotropy and Curie temperature. The magnetic ribbon prepared from this alloy has demonstrated an energy product of 4 MGOe (Fig.3c).

***List of Co-Workers:** Donthula Harish, Vishal Singh, Naveen Kumar N., B. Vishwanadh, Nilabja K. Sarkar, Poulami Chakraborty, Sanjay Saini, A.P. Srivastava, S. Neogy & A. Biswas. All are affiliated to Materials Group, BARC.

2

बहु पैमानों पर नाभिकीय संरचनात्मक पदार्थों का यांत्रिक परीक्षण

Mechanical Testing of Nuclear Structural Materials at Multiple Scales

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Understanding the mechanical behavior of structural materials used in nuclear systems requires comprehensive testing over a wide range of time, length, and temperature scales. In the Mechanical Metallurgy Division (MMD), extensive experimental programs are conducted to characterize materials such as zirconium alloys, austenitic and ferritic steels, refractory alloys, and nickel-based alloys. The generated database covers tensile, compression, creep, fatigue, and fracture properties across conditions simulating the extreme environments experienced in nuclear reactors. This multi-scale approach ensures that materials are qualified for safe, reliable, and long-term operation.

Mechanical Testing Across Time Scales

Mechanical behavior is highly dependent on the time scale of deformation, which is governed by the applied strain rate or frequency of loading. In MMD, tests span an exceptional range of strain rates over 11 orders of magnitude to study both quasi-static and dynamic deformation mechanisms. At the slowest rates, creep testing is performed at strain rates as low as 10^{-8} s^{-1}, capturing time-dependent plastic deformation over thousands of hours. Such long-duration experiments help assess the creep life and microstructural stability of zirconium alloys and nickel-based superalloys used in high-temperature reactor components. At intermediate rates (10^{-4}–10^{-2} s^{-1}), tensile and compression tests are carried out under controlled strain rates using servo-hydraulic and electromechanical testing machines. These tests provide baseline mechanical properties such as yield strength, ultimate tensile strength, and ductility at different temperatures. For high strain-rate behavior, the Split Hopkinson Pressure Bar (SHPB) facility enables testing up to 10^{3} s^{-1}, simulating transient loading conditions such as impact or seismic events that can occur in reactor systems. These studies are crucial for safety assessment under accident scenarios. In cyclic loading, fatigue testing is conducted over a wide frequency range—from 0.1 Hz in low-cycle fatigue (LCF) testing, where large plastic strains dominate, to 100 Hz in fatigue crack growth rate (FCGR) experiments, where small cyclic stresses drive subcritical crack propagation. Together, these tests provide a complete picture of time-dependent material degradation mechanisms.

Mechanical Testing Across Length Scales

Mechanical properties also depend on the length scale at which they are measured, from the microstructural to the component level. The MMD carries out testing from the nano- to macro-scale to correlate intrinsic material behavior with engineering performance. At the microscale, nanoindentation is employed to determine local hardness and modulus, enabling property mapping across grains, phases, and irradiated zones. This is particularly valuable for studying radiation-induced hardening in zirconium and steel cladding materials. Progressing to the mesoscale, miniature tensile testing is performed on small specimens machined from irradiated or limited-volume materials. These tests, often with gauge lengths of a few millimeters, allow evaluation of mechanical properties without compromising radioactive safety or consuming large quantities of valuable materials. At the macroscale, standard tensile, compression, and fracture toughness testing is performed as per ASTM standards to provide design-relevant mechanical data. The results serve as benchmarks for validating predictive models and supporting structural integrity assessments. Finally, component-level tests are performed to simulate actual service conditions. Examples include burst testing and pull-out testing of one-meter-long zirconium alloy tubes to evaluate deformation and failure under internal pressurization and mechanical loading. These large-scale experiments bridge the gap between laboratory-scale measurements and reactor component performance.

Mechanical Testing Across Temperature Scales

Temperature exerts a profound influence on the deformation and fracture behavior of materials. In nuclear reactors, components experience extreme thermal environments ranging from cryogenic to high-temperature conditions, and MMD's facilities are equipped to cover this entire spectrum. At low temperatures, Charpy impact and fracture toughness tests are conducted down to –150 °C to assess the ductile-to-brittle transition behavior of steels and weldments. Such data are critical for evaluating reactor pressure vessel steels and ensuring their toughness during start-up or shutdown conditions. At ambient and intermediate temperatures (25–400 °C), conventional tensile, compression

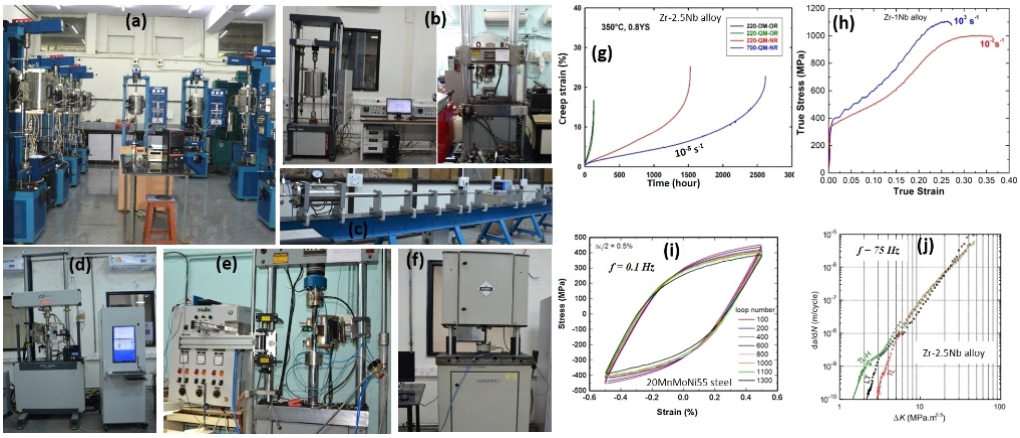

Fig.1: Representative facilities for time-scale testing: (a) Creep testing machines for testing at strain rate range of 10^{-8} to 10^{-4} s^{-1}. (b) Universal testing machines for testing at strain rates 10^{-5} to 10^{2} s^{-1}. (c) Split Hopkinson Pressure Bar for testing at strain rates 10^{2} to $8x10^{3}$ s^{-1}. (d) Servo Electric low cycle fatigue machine for cyclic testing in the range of 0.1 to 2 Hz (e) Servo hydraulic universal testing machine for cyclic testing in the range 0.1 to 20 Hz (f)Resonant fatigue machine for cyclic testing in the range of 1 to 100 Hz (g) Creep data of Zr2.5Nb pressure tube material- strain rate ~ 10^{-8} s^{-1} (h) Compression data of Zr-2Nb alloy at strain rate of 10^{-2} and 10^{3} s^{-1}(i) LCF data of 20MnMoNi55 steel at frequency 0.1 Hz and (j) FCGR data of Zr2.5Nb alloy at frequency 75 Hz.

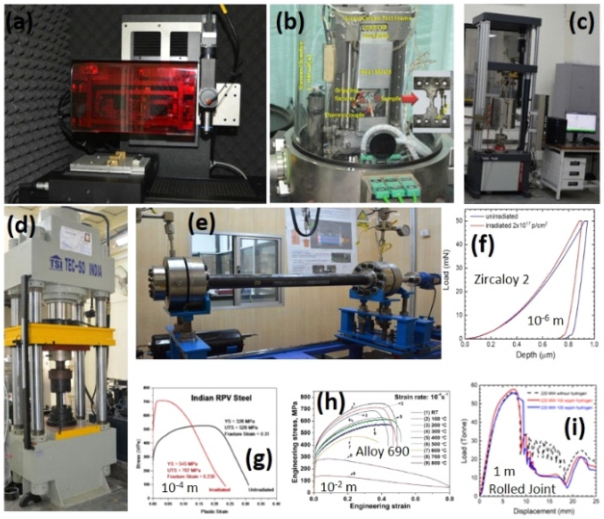

Fig.2: Examples of tests across length scales: (a) Nanoindentation setup, (b) Miniature tensile testing frame (c) Standard size specimen universal testing machine (d) 300 Ton press for component testing (e) Tube internal pressurization facility (f) Representative nanoindentation load-displacement plots for irradiated Zircaloy 2 at length scale of 10^{-6} m (g) Miniature tensile test data of irradiated RPV steel at length scale of 10^{-4} m (h) Tensile data of alloy 690 at length scale of 10^{-2} m (i) Zr2.5Nb PT-SS403 End fitting rolled joint pull out data at length scale of 1 m.

and fatigue tests are performed to evaluate in-service mechanical performance. These conditions correspond to normal reactor operation and transient loading scenarios. At high temperatures, creep and tensile/compression testing is conducted up to 1200 °C using vacuum or inert-gas furnaces. These experiments help in understanding creep rupture behavior, grain boundary sliding, and oxidation effects in nickel-based alloys and refractory materials designed for advanced reactor systems. This wide thermal testing range ensures reliable prediction of material performance across operating and

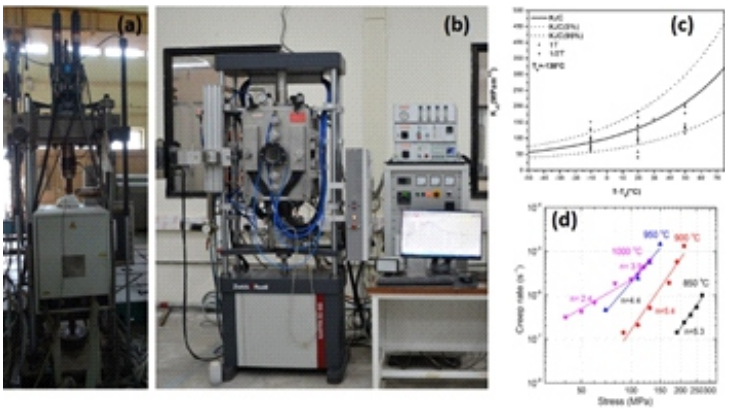

Fig.3: Mechanical testing across temperature scales: (a) Low-temperature testing chamber, (b) Elevated-temperature creep testing setup, (c) Master curve of reactor pressure vessel steel generated fracture toughness testing at temperature scale of -15^{0}C (d) high temperature creep data generated for a refractory alloy at temperature scale of 100 ^{0}C.

accident scenarios, supporting both design and life-extension of nuclear components.

Conclusion

The Mechanical Metallurgy Division of BARC has developed an extensive experimental framework for characterizing nuclear structural materials across time, length, and temperature scales. The integration of nanoindentation, miniature testing, high strain-rate experiments, and elevated-temperature creep studies provides a comprehensive understanding of material behavior under service-like conditions. The resulting multi-scale mechanical property database serves as a cornerstone for materials design, safety evaluation, and structural integrity assessment of critical nuclear components, contributing to the reliability and advancement of India's nuclear energy program.

पदार्थ प्रक्रमण में नवाचारों हेतु अभिलक्षणन

Characterization for Innovations in Materials Processing

3

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Development of Boride Based Neutron Absorber Materials

Borides have emerged as potential candidate materials for control rod applications in nuclear reactors owing to their favourable neutron absorption characteristics. A comprehensive processing route has been developed for the synthesis and consolidation of selected borides, e.g. TiB_2, ZrB_2, HfB_2, EuB_6, LaB_6, GdB_4. Cylindrical disks (Fig.1) of different borides have been prepared by hot pressing. The hot-pressing temperature and applied pressure were effectively reduced through the incorporation of suitable sintering additives, such as silicides and aluminides. The characterization techniques have been extensively employed for process optimization. For example, the microstructure of the ZrB_2–NiAl composite (Fig.2), prepared by hot pressing at 1750 °C, reveals that the NiAl phase acts as a binder, effectively holding the ZrB_2 grains together. The ZrB_2 grains exhibit rounded corners, which are characteristic of liquid-phase sintering and indicate the occurrence of solution–reprecipitation mechanisms during the process. The addition of the aluminide phase results in a reduction in hardness from 23 GPa to 16 GPa, accompanied by an increase in fracture toughness from 3.5 $MPa{\cdot}m^{1/2}$ to 6.8 $MPa{\cdot}m^{1/2}$. The enhancement in fracture toughness can be attributed to mechanisms such as crack bridging and crack arrest at the NiAl grains.

Development and Characterization of W-BCA Composites for Dispenser Cathode Applications

Tungsten (W) dispenser cathodes are widely used in high-power, high frequency vacuum electronic devices such as klystrons, because of the inherent ability to emit electrons with high current density. Porous tungsten matrix impregnated with ceramic oxide materials composed of barium oxide (BaO), calcium oxide (CaO) and aluminium oxide (Al_2O_3), known as BCA, are used in dispenser cathodes. The presence of barium is essential to obtain a low work function in the W-cathode materials. The W-BCA composite, with a diameter of 10 mm, has been successfully synthesized by innovatively preparing W-Cu composite followed by impregnation of a BCA compound at high temperatures. Fig.3 presents the SEM-EDS analysis of the W-BCA composite showing a uniform distribution of Ba, O, Al, and Ca atoms within the W matrix.

Development of Thick Cr coating on Zircalloy-4 by Magnetron Sputtering for Accident Tolerant Fuel (ATF) Applications

Post Fukushima Nuclear Accident in the year 2011, worldwide nuclear community has devoted extensive research to finding an alternate clad material to prevent hydrogen explosion related accident under LOCA conditions. Among various possibilities, Cr coated Zry-4 has shown the most promising alternative near-term solution with limited compromise of nuclear reactivity and power output. A 15-30 μm thick, highly adherent compact Cr coating on Zry-4 has been developed for the first time in India. Fig.4(a-c) shows Cr coating on 100 mm long Zry-4 fuel tube and corresponding cross-section images depicting integrity of the coating with the substrate. Fig.4(d-e) show Cr coating surface topography and corresponding cross-section images. High temperature steam oxidation of the Cr/Zry-4 samples showed excellent oxidation resistance behaviour compared to bare Zry-4 [Fig.4(f)] and under LOCA condition (1200 °C), Cr_2O_3 layer growth was limited and fully protected the underlying Zry-4 as shown in Fig.4(g).

Fig.1: Hot pressed boride discs.

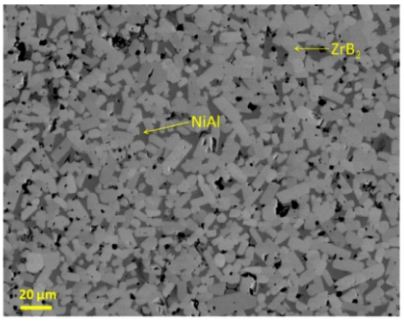


Fig.2: Microstructure of ZrB_2- NiAl composite.

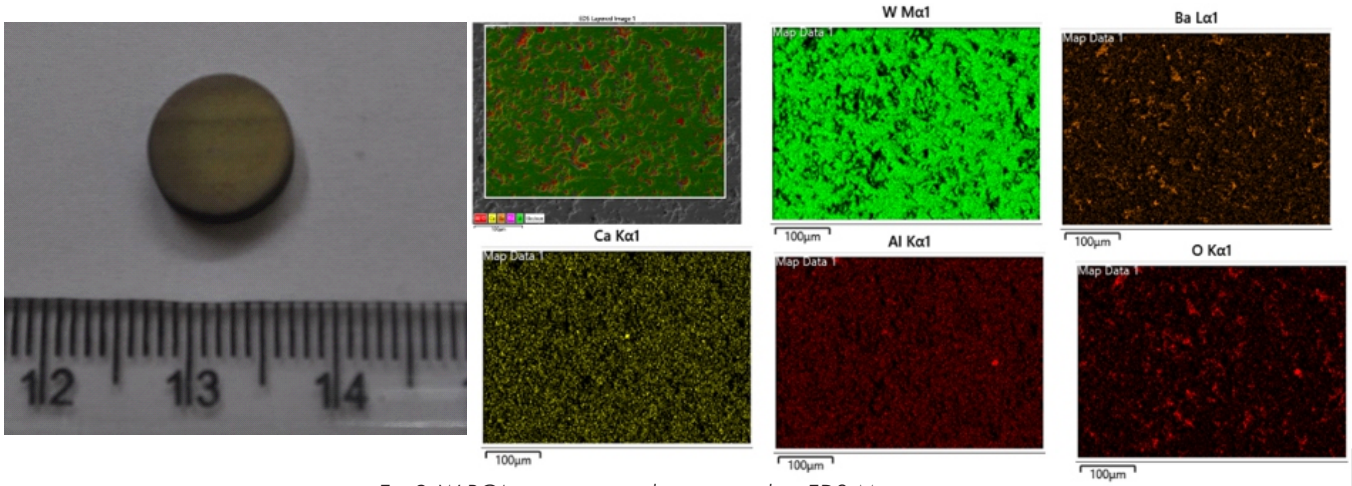


Fig.3: W-BCA composite and corresponding EDS-Mapping.

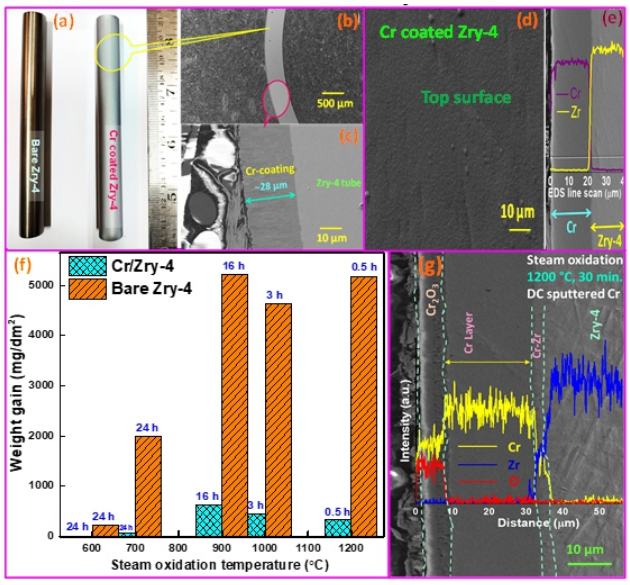
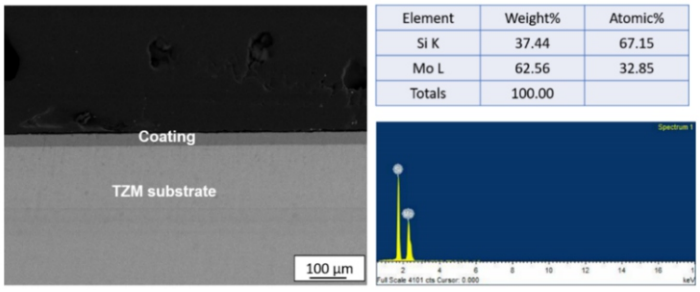


Fig.4: (a) Photographic images of bare and Cr coated Zry-4 fuel tubes, (b-c) X-section FESEM images of Cr coated tube; (d-e) FESEM surface topography and X-section image with EDS line scan of Cr coated Zry-4 coupon; (f) Wt. gain vs. steam oxidation temperature and duration; (g) FESEM X-section image of steam oxidised Cr coated Zry-4 at 1200 °C for 30 min.



Element	Weight%	Atomic%
Si K	37.44	67.15
Mo L	62.56	32.85
Totals	100.00	

Fig.5: BSE-SEM image showing the cross-section of uniform and crack-free coating of about 40 µm thickness. EDS analysis revealed $MoSi_2$ coating layer.

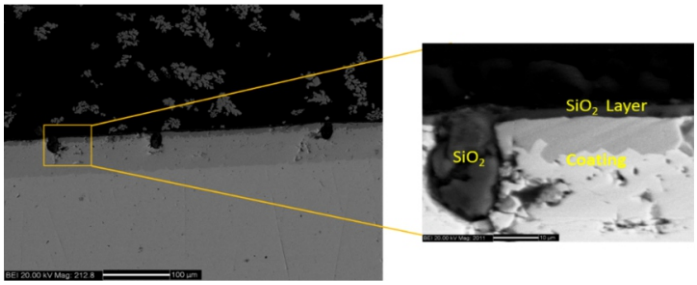


Fig.6: BBSE-SEM image showing the cross-section of oxidized sample. EDS analysis revealed protective SiO_2 layer formed during the oxidation treatment.

Development of Anti-oxidation Coating on Molybdenum-Based Alloy (TZM, Mo–0.5Ti–0.1Zr–0.02C) Fasteners

Molybdenum metal based alloy TZM (Mo–0.5Ti–0.1Zr–0.02C) is a potential material for high temperature applications such as nuclear reactor, hypersonic vehicle, space, etc, as it offers high strength and creep properties at higher temperatures (~1500 °C), excellent corrosion resistance and good resistance against irradiation embrittlement beyond 800 °C. Mo in the TZM alloy forms volatile oxide vapor MoO_3 at higher temperatures (beyond 700°C) in oxidizing environments, which leads to loss of material during application. Development of a suitable protective coating is necessary for improving the high temperature oxidation resistance of these molybdenum-based alloys. $MoSi_2$-based anti-oxidation coating on TZM alloy fasteners for hypersonic glide vehicle was prepared by pack siliconizing technique. Fig.5 represents the SEM image and EDS data of the as-coated TZM alloy showing the cross-section of uniform and crack-free $MoSi_2$ coating of about 40 µm thickness. Oxidation tests conducted at 1450°C for 2.5 h in air, indicate that the coating could survive the oxidizing environment at high temperature for longer duration owing to the formation of a protective SiO_2 layer (Fig.6). The coating technology has matured and is now available for transfer in the BARC/DAE technology transfer website.

कांच, सिरेमिक एवं कार्बन-आधारित प्रगत पदार्थ का विकास

4

Development of Glass, Ceramics and Carbon-based Advanced Materials

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Glass & Advanced Materials Division (G&AMD) has been actively involved in Development of glass, ceramics and carbon-based advanced materials for nuclear, defence, energy and biomedical applications. Some of the important contributions are:

Development of Yttrium-aluminosilicate Glass Microspheres (Bhabhasphere) for Radiotherapy Applications

Radiation therapy is used as one of the most effective treatments for cancers. Glass microspheres containing therapeutic radionuclides are effective for delivering high radiation doses to deep-seated tumors, especially liver cancers. Conventional external radiation often damages healthy tissue and is less effective for deep tumors, so radionuclide-loaded microspheres are used to provide targeted internal radiotherapy. β-emitting radionuclides such as Y-90, Ho-166, Lu-177, and Re-188 are suitable for treating primary and metastatic liver malignancies. Up until now an imported material 'Therasphere®' was being used for treatment of liver cancer. A similar material, Bhabhasphere, was developed by G&AMD, BARC at affordable costing one tenth of the imported cost. Yttrium-aluminosilicate glass microspheres (Bhabhasphere) having composition 40 Y_2O_3 – 20 Al_2O_3–40 SiO_2 (Bhabha sphere) were synthesised. Clinical trials of Bhabhasphere have been concluded and more than 25 doses have already been delivered. The Bhabhasphere was found to be on par or better than Therasphere® in terms of all properties. The glass microspheres were synthesized by melt-quench method, followed by grinding of glass frits to prepare feed particle for flame spheroidization. Microspheres of 20–35 μm diameter, suitable for arterial injection, were formed through flame sherardization and later neutron-activated to produce therapeutic radioactivity.

The Ho-166 and Lu-177 loaded glass microspheres, which are in an advanced stage of development, offer additional advantages: higher neutron absorption cross-sections and weak gamma emissions useful for diagnostics. Ho-166 has a 26.8 h half-life, while Lu-177 emits low-energy beta particles (0.4 MeV) with a longer 6.7-day half-life.

Carbon Technologies for Nuclear Energy Applications

Carbon is indeed a versatile material with a wide range of structures and properties that can be tailored to suit various applications, including those in the nuclear field.

Nuclear-grade graphite is used as a moderator and reflector in high-temperature nuclear reactors and molten salt breeder reactors. Being import restricted, indigenous nuclear-grade graphite has been developed using meso carbon microbeads (Fig.1).

Carbon nanotubes (CNTs) and graphene, as carbon nanomaterials, find applications in various aspects of the nuclear fuel cycle. Functionalized CNTs or graphene are used for the selective recovery or separation of lanthanides and actinides, which are important components in nuclear fuel processing.

CNTs and graphene with controlled porosity have the potential for use in nuclear desalination, where they can be used to purify water through advanced filtration methods. The high specific surface areas of CNTs make them useful for the adsorption of gases or vapours, which is relevant in various nuclear applications, such as gas storage and separation. CNTs and graphene can be incorporated into polymer and ceramic matrix composites to improve mechanical properties. These composites have applications in the nuclear field for structural and functional components.

A significant challenge lies in translating the unique properties of carbon nanomaterials from the nano-scale to the engineering scale. Technologies have been developed for CNT fibres (Fig.2), CNT sheets, and graphene oxide-based high compressive strength concrete, to address this challenge.

In summary, carbon-based materials play critical roles in various aspects of nuclear technology, from reactor components to fuel cycle management. Materials Group, BARC is actively engaged in research and development efforts to harness the potential of these materials for nuclear applications.

Development of SiC for Nuclear Applications

Silicon carbide (SiC), owing to its thermal stability, radiation tolerance, mechanical strength, and corrosion resistance, is widely regarded as a leading material for advanced nuclear systems. Its potential applications span fuel cladding, core structural components, and accident-tolerant

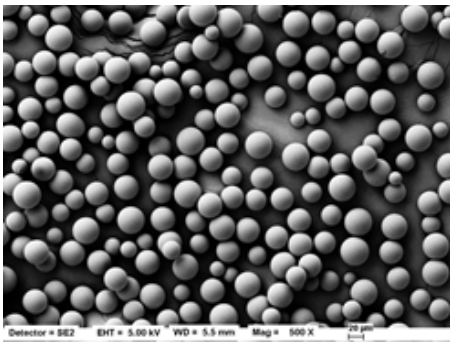

Fig.1: Y-89 aluminosilicate glass microspheres.

Fig.2: Indigenous nuclear graphite.

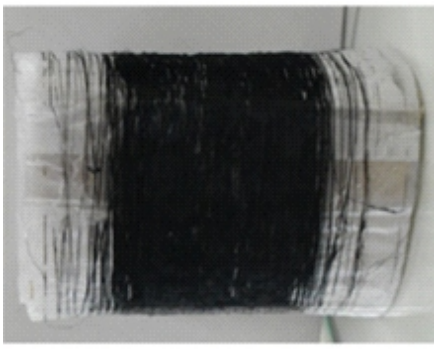

Fig.3: Continuously spun CNT fibre.

Fig.4: SiC coated Carbon fibre. Inset shows the micrograph of the fibre.

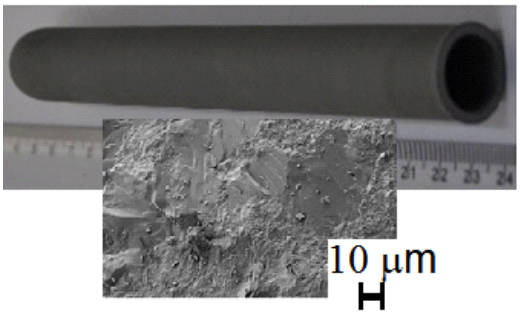

Fig.5: SRBSiC tube having >93% density. The micrographs revealed the dense structure present.

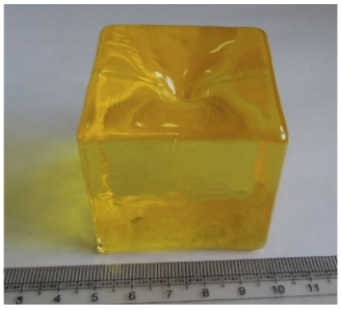

Fig.6: RSW glass.

fuel concepts in both fission and fusion reactors. However, the low fracture toughness of SiC necessitates the development of SiC fiber-reinforced composites to enhance toughness, damage tolerance, and reliability. Glass and Advanced Materials Division is actively engaged in the development of SiC fibre (fully converted and SiC coated C fibre) (Fig.4), monolithic and particulate-particulate composite of SiC (Fig.5), and SiC-based fibre–matrix composites using multiple processing routes. SiC fibres were synthesized by reacting SiO gas with carbon fibres at temperatures exceeding 1500 °C, where SiC formation initiates at the fibre surface and progresses inward. Dense SiC particulate composite (containing both α and β SiC) with >97% relative density, and <6 vol% free silicon, was successfully produced through the reaction-bonding (RB) method. In this process, a preform containing coarse α-SiC and fine carbon was infiltrated with molten silicon at temperatures above 1450 °C. The infiltrated Si reacts with the carbon in the preform to form in-situ SiC, which bonds the existing SiC particles, thereby increasing densification and enhancing mechanical strength. The optimized process was successfully applied to make more than170 mm long and 22 mm dia (wall thickness 2 mm) RBSiC tube (Fig. ACS-2). Preparation of SiC fibre matrix composite through RB method is under progress.

Preparation and Characterization of Lead Silicate Glass

Radiation shielding window (RSW) glasses are optically transparent shielding materials required for safe monitoring and handling of radioactive materials. RSW glasses required for BARC are mostly imported. The purpose of this work is in-house development of radiation shielding glasses so as an import substitute for RSW requirements in India. Composition optimization of CeO_2-BaO-PbO-K_2O-B_2O_3-SiO_2 glass system done with varying components to achieve density 4.5 gm/cc and having desired optical and radiation shielding properties. Successfully prepared glass blocks of dimensions 50 x 50 x 50 mm^3 and 100 x 100 x 40 mm^3 without casting defects. Then glass discs were prepared to study post irradiation changes in optical, thermal, mechanical and radiation shielding properties. Linear attenuation coefficient remained the same pre and post gamma irradiation. Glasses doped with CeO_2 showed radiation stability against browning up to 100 kGy in Co-60 gamma chamber. The post irradiation changes in physical properties are correlated with structural changes probed using Raman spectroscopy and positron annihilation lifetime spectroscopy. Radiation induced colors can be efficiently annealed by heating of the samples up to 300 °C temperature. However, heating of large glasses is not recommended because of the risk of braking. Possibility of UV annealing of color centers was studied post gamma irradiation. After 8 hours of UV treatment, considerable was improvement in transparency found in the glass. Though complete recovery of transmission was not observed however, it was sufficient to see the objects clearly through the glass.

5

हाइड्रोजन उत्पादन प्रौद्योगिकी हेतु छिद्रयुक्त पदार्थों का विकास

Development of Porous Materials for Hydrogen Production Technology

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Introduction

Porous materials are a class of materials in which pores of desired size range and volume fraction are introduced intentionally through a suitable powder metallurgy technique. Presence of pores makes significant changes in material characteristics, such as, decrease in thermal conductivity and increase in specific surface area. With increase in interconnected pore fraction permeability for liquid and gas increases significantly in porous materials. These distinctive features of porous materials make them suitable for numerous advanced applications, such as, thermal insulation, catalyst, filtration of hot gas and liquid etc. Many porous ceramics have excellent temperature and corrosion resistance enabling their use in severe conditions.

There are numerous ways to produce porous materials. These include partial sintering, replica methods, sacrificial pore formers, direct foaming, gel-casting and additive manufacturing. Suitability of a process depends on the material, desired pore structure and application. Some porous materials developed at Powder Metallurgy Division of Materials Group, BARC applications in hydrogen programme of DAE programmes are reported here.

Alumina Ceramic Foam

Ceramic foams are high porosity materials with interconnected pores. A process has been developed for making α-alumina based ceramic foam by polymeric sponge replication method. In this process, commercial polymeric foam (template) is dipped into specially formulated ceramic slurry followed by drying and sintering to yield a replica of the original polymeric foam. The properties of ceramic foam can be adjusted by varying the polymeric foam characteristics, viscosity of the ceramic slurry and sintering schedule. The process is capable to making ceramic foam up to 90% porosity. Typical characteristics of the developed alumina ceramic foam are shown in Table 1. Physical appearance of alumina foam and its SEM micrograph is shown in Fig.1. α-alumina based foam can be used at high temperature upto 1200 °C. The alumina foam can be used as catalyst support for various applications, such as catalytic converter, waste water treatment.

γ-Alumina Coated Alumina Ceramic Foam Catalyst for I-S Process

Alpha alumina foam has been used to support Pt catalyst for its application to decompose HI in Iodine-Sulphur process of hydrogen production at Chemical Technology Division, BARC. For this application α-alumina macro-porous support is usually coated with γ-alumina to enhance the surface area. Finally, the ceramic foam is impregnated with platinum salt solution followed by heat treatment resulting in formation of nano-particles of catalytically active phase. SEM micrograph of the Pt catalyst loaded foam and Pt distribution in it is shown in Fig.2.

Iron Oxide Based Foam Catalyst for I-S Process

$(Fe,Cr)_2O_3$ is an efficient catalyst material for sulphuric acid decomposition reaction in I-S process for generation of hydrogen. As in the case of alumina foam preparation polymeric sponge replication method has been used for making foam type $(Fe,Cr)_2O_3$ catalyst. Physical appearance of the foam catalyst and its SEM micrograph is shown in Fig.3. The pores formed in the foam catalyst remain stable after its use at the operating temperature of 900 °C.

Porous Electrodes for Solid Oxide Cell

Solid oxide cells are ceramic electrochemical devices based on oxygen ion conducting electrolyte. These cells can be used in fuel cell mode (SOFC) for direct conversion of chemical energy of fuel (e.g. hydrogen) into electrical energy. In reverse mode of operation with the supply of electrical energy the cell can be used for electrolysis of steam. High temperature operation improves process efficiency. Ni-YSZ cermet electrode supported cell is the most common configuration. Processes

Table 1: Characteristics of alumina foam

Property	Typical value
Density	0.7 – 0.9 g/cm^3
Surface area	>35 m^2/g
Compressive strength	2 – 3MPa

Fig.1(a): α-alumina foam.

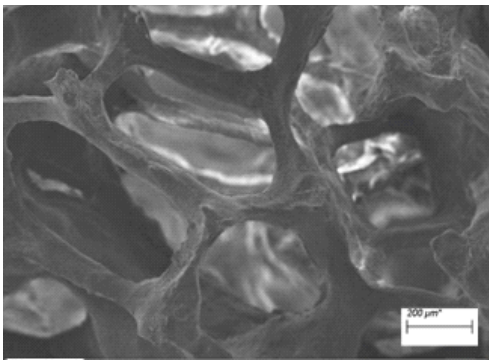

Fig.1(b): SEM micrograph of the foam.

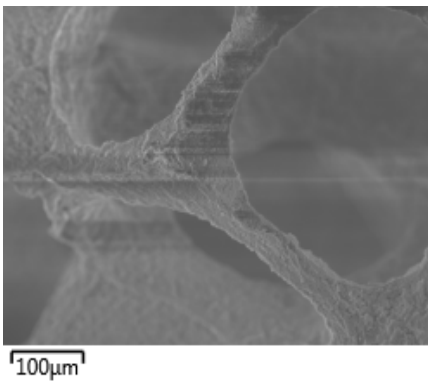

Fig.3(a): $(Fe,Cr)_2O_3$ foam catalyst.

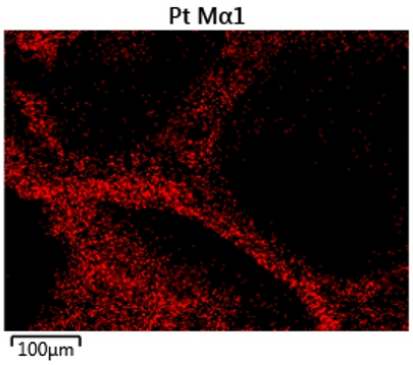

Fig.3(b): SEM photomicrograph of $(Fe,Cr)_2O_3$

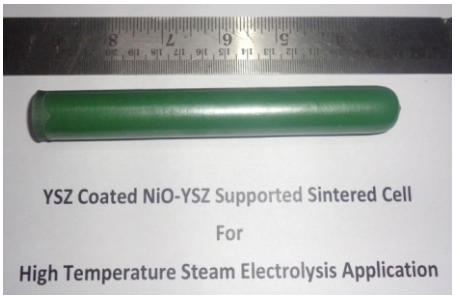

Fig.4(a): NiO-YSZ support tube with dense 8YSZ electrolyte.

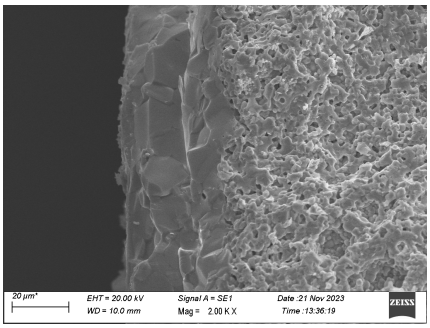

Fig.4(b): SEM micrograph showing bilayer (porous Ni-YSZ electrode with thin and dense 8YSZ electrolyte).

have been established to make porous NiO–YSZ supported cells with dense 8YSZ electrolyte. NiO-YSZ tubes are made by cold isostatic pressing and the green tube is coated with 8YSZ ceramic slurry. The 8YSZ slurry coated NiO-YSZ tube is sintered at around 1350 °C to get dense 8YSZ coating. To make a complete cell Sr-doped lanthanum manganite slurry with suitable pore former is coated over the dense electrolyte and fired at around 1100 °C.'. Ni-YSZ cermet with interconnected pore structure is developed when hydrogen is passed through the tube at high temperature at the start of operation. Typical microstructure of bilayer (porous Ni-YSZ electrode with thin and dense 8YSZ electrolyte) is shown in Fig.4. The cells have been operated at 800 °C in electrolyser mode for generation of hydrogen. Steady performance of single tubular cell for more than 150 h with hydrogen production rate of 4 Nlph has been demonstrated. Work has been initiated to develop porous metal supported cell for better electrical contact.

यूरेनियम निष्कर्षण प्रभाग में नाभिकीय पदार्थ प्रौद्योगिकी का विकास

6

Development of Nuclear Material Technology at Uranium Extraction Division

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Natural uranium and thorium are key materials used for energy production and strategic applications. Uranium metal is used as nuclear fuel due to its high fissile density and excellent thermal conductivity. Thorium is primarily used as a fertile material in breeder reactors, where metallic thorium offers a shorter doubling time compared to other forms. Metallic fuel is also being considered for use in next-generation fast reactors. Uranium metal is derived from various impure materials through a combination of hydro- and pyro-metallurgical processes. Similarly, the production of thorium metal from its ores involves a series of hydro- and pyro-metallurgical steps. Each process step requires stringent monitoring of both quality and quantity related parameters. Physio-metallurgical characterization, alongside chemical purity assessments, is critical for the various intermediate products. Specific surface area, O/U ratio, flowability, tap density, morphology (SEM), phase composition (XRD), mean particle size and particle size distribution are such important characteristics. These characteristics play a pivotal role in the processing of intermediates and in ensuring that the final uranium metal product meets stringent specifications. The chemical purity specifications of uranium metal produced at UED, BARC, in fact exceeds the standards set by ASTM C1462-21.

Higher specific surface area of the intermediates is desirable for their better conversion to the respective products. O/U ratio of the oxides determines the extent of their conversion to the desired oxide and the relative composition of the various oxides. Higher tap density is desirable, so as to accommodate more quantity in a given volume resulting in higher productivity. The specific surface area and tap density are in turn dictated to a large extent by the morphology of the intermediates. Spherical particles are likely to have more flowability and tap density compared to particles with irregular and acicular morphology of the same material. Morphological features e.g. particle shape, size, porosity, roughness etc. affects the specific surface area. Particle size distribution also plays a key role in the obtainable tap density. A wider particle size distribution results in particles of different sizes, and smaller particles may fit into the voids between larger particles, leading to more efficient packing and, consequently, a higher tap density.

The average particle size of the UO_3 particles is about 25 μm and that of UF4 particles is about 35 μm. The representative SEM images of the UO_3 and UF_4 are shown in Fig.1(a) and 1(b) respectively. UO_3 is supposed to be porous and uniform agglomerated product for assuring the complete conversion to UO_2 followed by UF_4, which is quite evident in the SEM images shown in Fig.1(a). The specific surface area of UO_3 is about $10 m^2/g$. The XRD pattern of the various solid intermediates indicating the various phases present in the preparation of the uranium metal ingot has been presented in Fig.1(c) .

Monitoring and controlling of critical process parameters have led to consistent quality of UF_4 with about more than 98% assay, which in turn ensures the desired yield of the uranium metal ingot. Uranium ingot having orthorhombic crystal structure typically has a very coarse grain size, often in the range of several millimeters, due to slow cooling rates. The final grain size is highly dependent on the cooling rate during casting and can be refined through subsequent heat treatments like multiple β-quenching and α-annealing cycles to reach sizes around 100 μm or smaller.

Uranium and thorium metal powder is prepared by the calciothermic reduction of the respective oxides followed by removal of slag by chemical processing using acetic acid. Uranium and thorium metal powders have been utilised for various departmental activities. The SEM images of the metal powders and the respective oxides are shown in Fig.2 depicting the typical morphological features. The difference in particle morphologies between thorium and uranium particles can be attributed to the melting of uranium, in contrast to thorium, which remains in a solid state after the reaction due to its higher melting point.

A novel reduction diffusion process has been developed for the synthesis of U-10wt%Mo alloy in powder form. The desired gamma phase of uranium could be stabilized at room temperature as shown in the XRD plot in Fig.3 (a). The SEM micrograph of the powder depicting the size, shape and morphology is presented in Fig.3(b). U-Mo based fuel prepared by powder metallurgy route is likely to have more porosity to accommodate the fission products. Preliminary studies on the compaction and sintering have been done on the U-10Mo

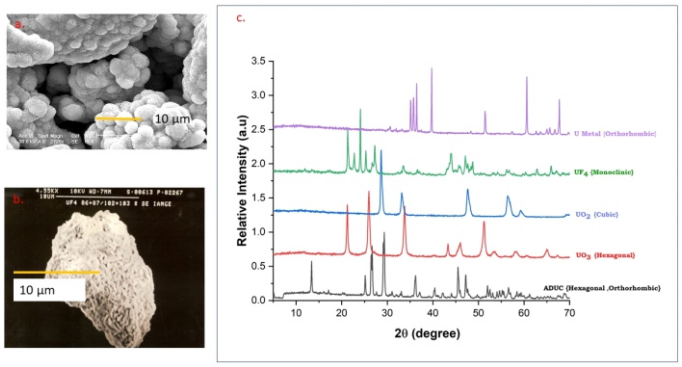

Fig.1: SEM image of (a) UO_3, (b) UF_4 and (c) XRD of the various intermediates in the uranium metal preparation.

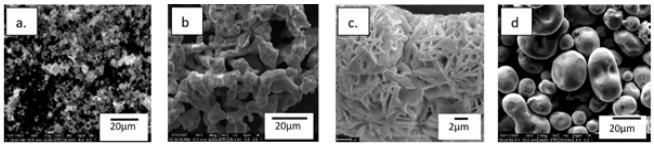

Fig.2: The SEM images of the metal powders and the respective oxides depicting the typical morphological features.

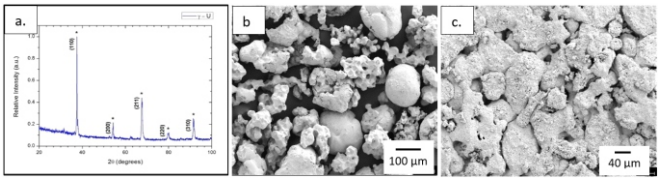

Fig.3: (a) XRD plot shows completely stabilized γ phase and (b) SEM image, of U −10 wt% Mo alloy powder produced by R-D process, (c) Microstructure of partially sintered pellet prepared using R-D U-Mo powder.

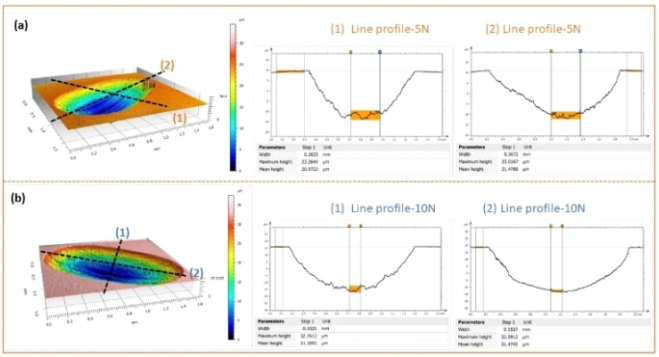

Fig.5: U-1.5Ti alloy, 3D map of wear scar for (a) 5 N & (b) 10 N normal load and adjacent figures present the line profile of wear scar along the both major and minor axes as marked by dashed line.

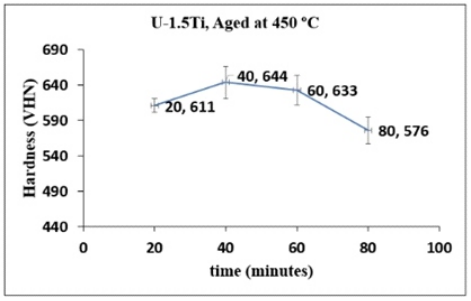

Fig.4: Hardness vs ageing duration at 450 °C Of U-1.5Ti alloy.

powder and the microstructure of the partially sintered pellet has been presented in Fig.3(c) showing the partial consolidation of the powder particles and necking and particle fusion is evident from the image. Parameters for the sintering need to be further optimized to get desired sintered density.

Thorium based metallic fuels are the promising fuel candidates for various advanced high temperature and breeder reactors. Extensive studies on Th-U based binary and ternary alloys viz. Th-U-Mo, Th-U-Zr and Th-U-Nb have been carried out. Th-U alloys containing less than 20 wt% U have been found to be suitable for fuel applications, as confirmed by irradiation studies conducted worldwide. To enhance the characteristics of Th-U alloys with higher uranium loading, ternary additions of Mo have been found to be suitable, as Mo stabilizes the isotropic gamma phase of uranium at room temperature.

Thorium Fluoride, ThF_4 is used for the preparation of Th metal and it is one of the salts used for the molten salt breeder reactor. The process parameters viz. bed depth, temperature, time and excess HF requirement were optimized for the preparation of ThF_4 of required quality specifications in a static bed hydro-fluorination set up. ThF_4 was utilised for the various studies related to the Indian Molten salt Reactor program.

Studies on U-Ti alloys of 0.8-1.7wt.%Ti were done for potential application for grinding of uranium ores. Optimum composition and heat treatment scheme for desired microstructure and hardness has been established. It has been found that ageing of U-1.5Ti alloy at 450 ^{0}C for 40 minutes results in peak hardness of 644 VHN (Fig.4). Microstructure of U-1.5Ti homogenised alloy consists of alpha-U and TiC precipitates. U_2Ti precipitates in U-1.5wt%Ti aged alloy increases the hardness. From the results of wear test, it was found that wear resistance of aged U-1.5%Ti alloy at 10N normal load was slightly better than U-metal. The specific wear rate of pure uranium at 10N load was found to be 1.796×10^{-4} mm^3/Nm and that of U-T1.5Ti aged to maximum hardness was found to be 1.6549×10^{-4} mm^3/Nm. Existing grinding media i.e. chrome steel showed better wear resistance and hardness than U-1.5%Ti aged alloy, when tested against the hardened steel counter body. So, U-1.5%Ti alloy needs to be surface hardened to further improve the hardness as well as wear properties.

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लेपिडोलाइट अयस्क से लिथियम का उपयोग - एक स्वच्छ ऊर्जा भविष्य के लिए भाभा परमाणु अनुसंधान केंद्र की स्वदेशी प्रक्रिया

Harnessing Lithium from Lepidolite Ores - BARC's Indigenous Process for a Clean Energy Future

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Applications of Lithium

Lithium (Li) has become one of the world's most important elements because it powers almost everything - from mobile phones and laptops to electric vehicles and renewable-energy storage systems. Global demand is rising rapidly, and India, too, aims to build a strong battery manufacturing ecosystem for clean mobility and green energy missions. An equally important use of lithium, especially for the Department of Atomic Energy, is its application in nuclear technology viz. lithium compounds are used as reactor coolants, in fusion-related research and other strategic usages. Although India does not have large lithium deposits like South America or Australia, it does possess significant lithium-bearing pegmatites in the forms of mineral spodumene in Karnataka, as lepidolite in the mica belts of Bihar and Rajasthan. Lepidolite is a lilac-coloured mica mineral that contains Li along with other valuable alkali metals such as rubidium and caesium (Fig.1). To utilise these resources effectively, BARC has successfully developed an indigenous process flowsheet for recovery of lithium compounds of high purity.

A Closer Look at the Rock That Holds India's Lithium

The first step towards lithium extraction is to understand the nature of the ore. Ore Samples from Bihar mica belt was studied using instrumental and chemical methods. The characterisation information confirmed that the ore contains about 3.15% Li_2O, mainly hosted in lepidolite. Quartz and feldspar are the major gangue. Photomicrographs showed that the lepidolite occurs as fine lilac coloured flakes, while XRD patterns clearly identified lepidolite, muscovite, K-feldspar, albite and quartz (Fig.2). Presence of rubidium, caesium and potassium which are valuable by-products were identified in the chemical analysis of the sample that can be recovered later in the process. The characterisation studies helped in design and development of recovery flowsheet.

Exploring Multiple Metallurgical Routes to Unlock Lithium from the Ore

To identify a robust method for recovery of lithium values from lepidolite, three alternating processing routes were examined in detail. The comparative plot on the leachability of Li by the three alternative routes are shown in Fig.3. The first approach, whole ore direct acid roasting, involved heating the ore with sulfuric acid, but this proved ineffective for the Bihar lepidolite. Even with fine grinding and high acid consumption, only about 36% of the lithium could be extracted, as the mineral structure restricted the reaction. A second route combined high-temperature calcination at 1000°C with subsequent acid roasting. Breaking of the mineral structure before acid treatment improved leaching of Li significantly, about 94%. However, this method created challenges during removal of impurity, leading to loss of Li values in the sludge thereby reducing the overall recovery. A third approach involving salt roasting -aqueous leaching, provided the most effective recovery (97%). In this method, the ore was mixed with Na_2SO_4 and $CaSO_4$ and roasted at 1000°C to convert Li into water-soluble Li_2SO_4 with simultaneous dissolution of Rb, Cs and K in the aqueous phase. This process gives the advantage of stabilization of fluorine as CaF_2 which is otherwise release as a toxic gas during roasting, thus enhancing the environmental safety. In view of the high leachability of Li values with relatively purer leach liquor quality this salt roasting route was selected for further optimisation of downstream process.

A Clean Route to High-Purity Lithium Hydroxide Monohydrate ($LiOH.H_2O$)

After selecting the salt-roasting method, the next step was to convert the lithium extracted from the ore into high-grade $LiOH.H_2O$. Once the roasted material was leached with water, the lithium-rich solution was treated with soda ash to remove calcium and other impurities without losing Li. This produced a cleaner solution which was further concentrated through

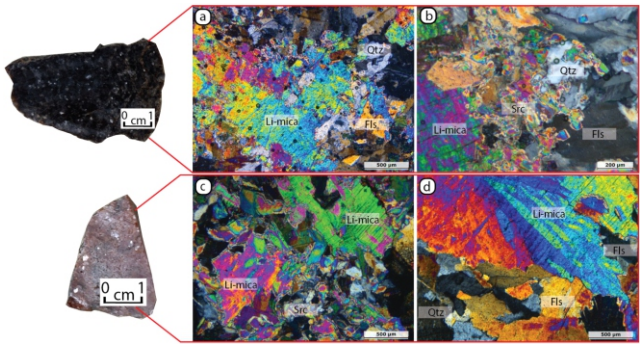

Fig.1: Photomicrographs of lepidolite showing pink/lilac mica flakes.

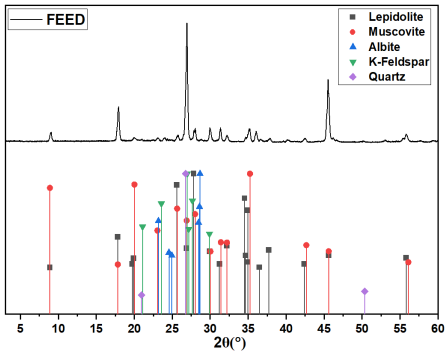

Fig.2: XRD pattern of the feed ore.

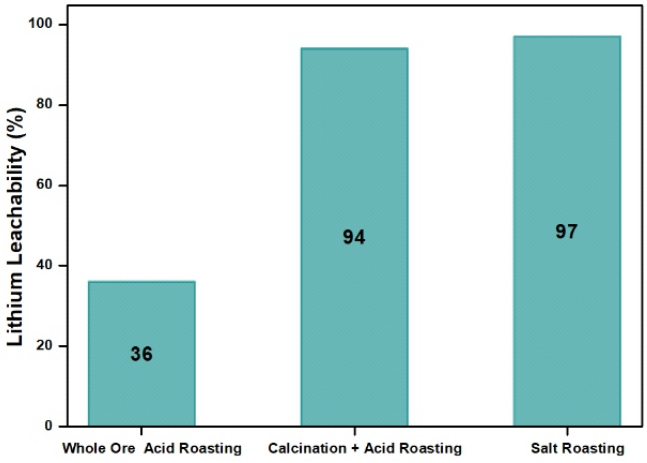

Fig.3: Comparison of leachability of lithium from lepidolite.

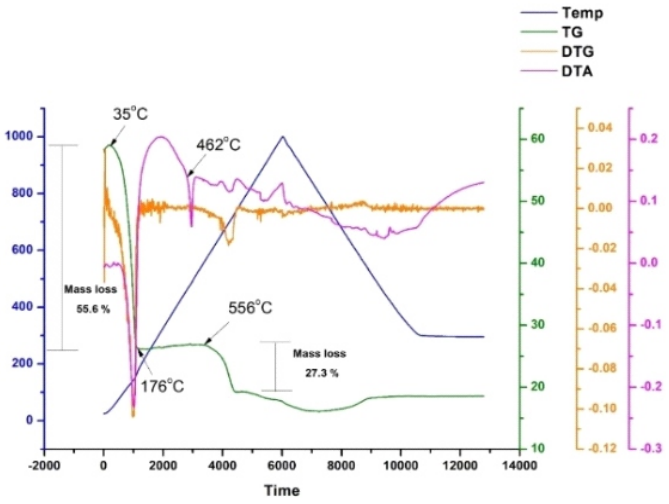

Fig.4: TG-DTA curve of $LiOH{\cdot}H_2O$.

controlled evaporation. The Li values in the concentrated solution after evaporation were precipitated as Li_2CO_3 by adding sodium carbonate. The soluble impurities (Na, K) in crude Li_2CO_3 were minimised by multiple washing of the crude precipitate. The purity of the washed precipitate was about 93%. The purified Li_2CO_3 was reacted with lime slurry at elevated temperature to produce a solution of LiOH free from major impurities. To obtain high-purity $LiOH.H_2O$, this solution was further concentrated under vacuum, which prevented carbon dioxide from entering and ensured that only lithium hydroxide monohydrate crystallized out. The thermal behaviour of the final crystals is illustrated in Fig.4, confirmed the quality of the product, $LiOH.H_2O$. The final crystals were of purity >99%. The product achieved through this flowsheet meets the purity requirements for nuclear applications.

Significance and Way Forward

The work demonstrates that high-purity lithium compounds can be produced from Indian lepidolite while safely stabilizing fluorine and allowing recovery of other alkali metals. The next phase will focus on pilot-scale validation and further refinement of process steps, including improved by-product recovery and extension to other lithium-bearing deposits. Overall, this work strengthens the country's progress toward long-term self-reliance in critical minerals and clean-energy materials.

Acknowledgment

The authors gratefully acknowledge Mr. Dheeraj Pandey, Director, AMD, for providing the ore samples and entrusting this assignment. Sincere thanks are also extended to Dr. Raghvendra Tewari, Director, Materials Group, BARC, and to Dr. T.S.R.C. Murthy, Head, MinD BARC for their guidance and encouragement. The author also wishes to thank Dr. Abhishek Mukherjee, Head, P&MTS, MPD, BARC for assistance with TG-DTA analysis; Mr. Mohit Rattanpal, SO 'D', MSD, BARC for support with XRD measurements; Mr. Anuj Singh, SO 'C', MinD, for optical microscopy, and Mr. Ajay Kumar, SA 'D', MinD, for chemical analysis. Appreciation is further extended to all colleagues of the MinD for their continued cooperation.

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बोरॉन सिरेमिक में कुल बी की अविनाशी परिमाणन और इसकी समस्थानिक संरचना के लिए पीआईजीई विधियों का विकास एवं अनुप्रयोग

Development and Applications of PIGE Methods for Non-Destructive Quantification of Total B and its Isotopic Composition in Boron Ceramics

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Chemical characterization of materials is the most important step in chemical quality control (CQC) exercises and it involves the determination of elemental and/or isotopic contents at major, minor and trace levels with adequate accuracy and precision for ensuring the quality of the material's finished product as per the specified compositions. Routinely used analytical methods are mostly wet chemical and atomic and mass spectroscopic techniques, which are destructive in nature. When the materials of interest are of complex matrix samples like glass, ceramics, alloys, carbides, oxides and high purity materials, there is a need of simpler non-destructive methods for CQC. Among these samples, boron based ceramic/refractory compounds such as B_4C, transition metal di-borides, and rare earth hexa-borides are key functional materials in nuclear power technology due to higher neutron absorption cross section over a wide range of energy due to the presence of ^{10}B isotope (thermal neutron absorption cross section, 3837 barn). These materials are widely used in control rod, shutoff rod, neutron shielding and neutron sensor applications. As a part of CQC, it is necessary to quantify total B as well as its isotopic composition ($^{10}B/^{11}B$ atom ratio) by suitable analytical technique(s), to know whether the material is natural or enriched with respect to ^{10}B and the total B contents are within specified limits or not. Routinely used techniques for isotopic composition of B is Mass Spectrometry techniques like TIMS and ICP-MS, whereas for total B is determined by techniques like titrimetry, IC, ICP-OES & ID-MS. These techniques yield high sensitive results with very good precision but are destructive in nature. There was a need/scope to analyze these ceramics/refractory samples by non-destructive techniques like Nuclear Analytical Techniques (NATs). Prompt Gamma-ray Neutron Activation Analysis (PGNAA) using n-beam and Particle Induced Gamma-ray Emission (PIGE) using p-beam are two such methods capable of isotopic and total concentration determination. PGNAA using thermal n-beam is a very sensitive method to determine ^{10}B isotope and total B utilizing 478 keV from $^{10}B(n,\alpha)^{7*}Li$, but it is not sensitive for ^{11}B and also it suffers from neutron self-shielding effect at high concentration level. On the other hand, PIGE using low energy proton beam (3-5 MeV) is capable of determining low Z elements (like Li, B, F, Si, Al etc) including IC of B from low to high concentration level [1-3]. It involves measurement of prompt gamma-rays of 429, 718 and 2125 keV from $^{10}B(p,\alpha\gamma)^{7}Be$, $^{10}B(p,p'\gamma)^{10}B$ and $^{11}B(p,p'\gamma)^{11}B$ reactions, respectively.

Development of PIGE Facilities at FOTIA, BARC for Low Z Elements & Isotopic Composition of B

At FOlded Tandem Ion Accelerator (FOTIA), IADD, BARC, vacuum chamber PIGE facility (4 MeV proton beam, with 10-25 nA current) with *in situ* current normalization method utilizing F or Li was developed for low Z elements including B via pellet method using cellulose matrix [1-3]. In order to have rapid analysis of "as received powder" and direct glass/ceramic/alloy samples, an external PIGE facility was setup at FOTIA by extracting the proton beam using a thin 25 μm Ta window, which is a First Of A Kind facility [4]. Samples are wrapped in thin Mylar film and analyzed using external current normalizer either Ta (135 and 165 keV of $^{181}Ta(p,p'\gamma)^{181}Ta$) or N from air (2313 keV of $^{14}N(p,p'\gamma)^{14}N$) for total B in natural and enriched B_4C and other ceramics like di & hexa borides. PIGE facilities and typical prompt gamma-ray spectra are given in Figures 1a-1d. All calculations are done by relative method taking natural B

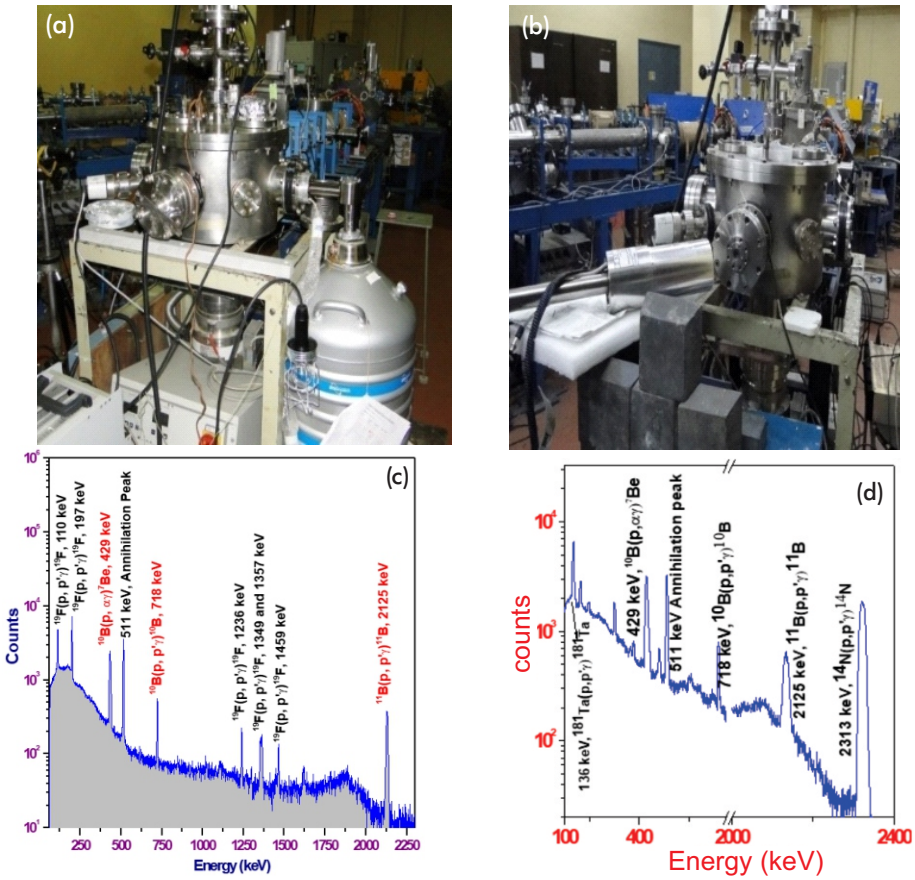

Fig.1: FOTIA PIGE Facilities: (a) Vacuum Chamber, (b) External (in air) with Ta window; vPIGE spectra of B_4C, (c) Conv. PIGE with in situ current normalizer F & (d) Ext-PIGE with Ta & N γ-peaks.

compound. Advantage of PIGE method is that determination of IC of B and 10B isotope content does not need current and mass of sample as input. All these R&D & applied works have been carried out in collaboration with MPD, Materials Group of BARC.

Lab to Industry Applications of Conventional and External PIGE Methods for IC of B and Its Total Content in Boron Based Ceramics

An in situ current normalized vacuum chamber PIGE method using a 4 MeV proton beam to determine total B and its IC in boron-based ceramic and refractory neutron absorbers/ shielding materials (natural and enriched B4C and borides of Ti, Zr, La, Ce etc) using sample pellets in cellulose matrix [1,2]. Results were compared with TIMS and PGNAA for IC or B-10 and with titrimetry and ICP-OES for total B. For Ti & Ti-Cr based ceramics PIGE is found to be suitable method for IC compared TIMS [2]. External (in air) PIGE using 3.5 MeV proton beam on target was utilized for rapid analysis of "as received" samples of B_4C & other ceramic samples for IC determination [4]. This innovative method is very simple, rapid and non-destructive in nature and sample can be returned back after assay as there is no radioactivity. By utilizing this PIGE method, simultaneous quantification of mass fractions of Fe and B in the ferroboron alloys was carried out non-destructively [5]. External PIGE method was employed to prepare in-house reference materials (IRMs) for five isotopic compositions of B from IC of 0.247 (natural, 19.8 atom% ^{10}B) to 2.03 (enriched, 67 atom% of ^{10}B) in B_4C matrices using direct powder and pellets samples. In August 2023, external PIGE method at FOTIA was utilized for commercial application, for the first time, for ^{10}B isotope content certification in industrial natural B_4C samples relevant for Indian power reactors [6]. Results were report with QA/QC of PIGE methods. For single sample analysis, the % uncertainty on obtained results of IC of B is about 1%, whereas for replicate samples (N=5 of higher) both IC of B and total B, the %RSD is about 0.5% or lower. Developed external PIGE method is very suitable for quantifying low Z elements including IC of B as well as total B contents in a simpler and faster way by analyzing "as received" samples compared to conventional vacuum chamber PIGE method.

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पदार्थ अनुसंधान एवं विकास के लिए प्रेरण लेविटेशन गालक

9

Induction Levitation Melter for Material Research and Development

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Introduction

Induction Levitation Melter (ILM) is an advanced technology designed to produce ultrapure specimens of highly reactive metals and alloys with exceptional homogeneity. This method employs a water-cooled segmented copper crucible in an inert Argon or vacuum environment, allowing the magnetic field to penetrate and melt the metal charge. The process generates strong Lorentz forces, causing vigorous stirring in the melt and preventing contamination by levitating the molten mass away from the crucible walls. These features ensure uniform temperature distribution, higher achievable superheat, and minimized contamination, making ILM especially valuable for preparing samples used for development of new alloys.

Development

The laboratory-scale ILM system (Fig.1) developed by Nuclear Recycle Group offered reliable performance in melting and homogenizing various alloys, producing small specimens (up to 25 g) for material characterization. Its high temperature availability, efficient vacuum operations, and the use of high-purity Argon ensured the preparation of ultrapure alloy buttons with excellent homogenization and utility for novel alloy development. The ILM has been subsequently used to produce specimens of various alloys including Ni – Ti – Fe alloy, Zr – Cu alloy, Ti – Al– V alloy, Zr – Nb alloy etc. for Materials Group R&D activities. Recognizing these advantages, the Materials Group, BARC engaged with Nuclear Recycle Group for a high-capacity ILM suitable for producing samples up to 100 g.

Induction Levitation Melter for Materials Group

Nuclear Recycle Group has taken up the design of ILM of higher capacity, considering the requirement raised by Materials group. The main features of the design included -

Crucible Design: The new ILM features a 12-segment water-cooled copper crucible, with an internal cavity diameter of 50 mm and height of 45 mm. This allows for larger batch processing of up to 100 g charge and enhanced electromagnetic field distribution for optimal levitation and homogenization.

Vacuum and Atmospheric Control: A robust vacuum delivery system achieves up to $1x10^{-3}$ mbar within minutes, with integrated Argon purging, digital vacuum monitoring, and automatic safety interlocks for process stability and contamination avoidance.

Power Supply and Coil: The melter is powered by a 75 kW induction heating unit, providing variable frequency and efficient energy delivery for melting larger samples. Safety features include load-matching, surge protection, automatic shutdown, and continuous water cooling for both the induction coil and crucible.

Cooling Arrangement: A dual-loop, closed water cooling system ensures reliable operation under continuous industrial duty, with precise control over flow, pressure, and temperature for all subsystems. The Primary and secondary water circuit maintain system integrity and safe operating temperatures.

Instrumentation and Control: Centralized PLC-based automation provides real-time parameter display, touchscreen operation, integrated safety logic, fault monitoring, and rapid emergency response. Fiber-optic signal channels and noise-resistant architecture support consistent and reproducible melting operations.

Based on the above design features an Induction Levitation Melter (Fig.3) of 75kW capacity has been manufactured, installed and commissioned for Materials Group at Mod Labs, BARC Trombay. The system has been tested for melting and homogenization of SS, Al and Zr –SS alloys during commissioning trials.

Summary

The non-contact levitation technology eliminates crucible contamination, crucial for high purity metals and research-grade alloys. The enhanced batch size of this new ILM enables various characterization studies and material property evaluations, which were not possible with small samples. The deployment of the new high-capacity ILM for Materials Group underscores its value for advanced alloy development, offering Materials Group and Nuclear Recycle Group a platform for collaboration in materials research. The equipment can provide necessary foundation for future process scale-up, alloy exploration, and fundamental metallurgical studies.

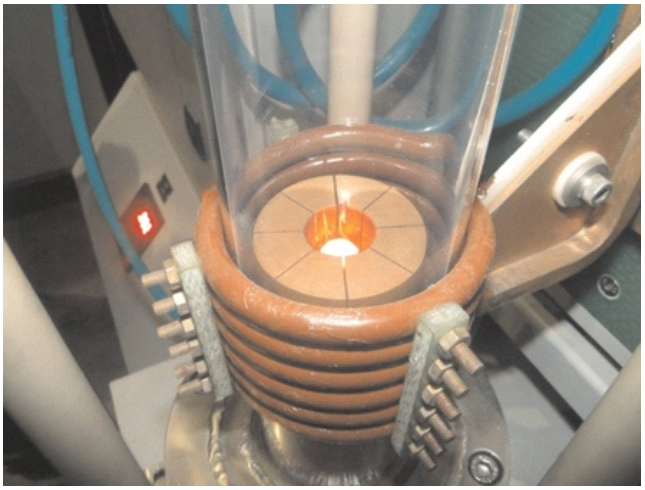

Fig.1: Induction Levitation Melter produced in ILM.

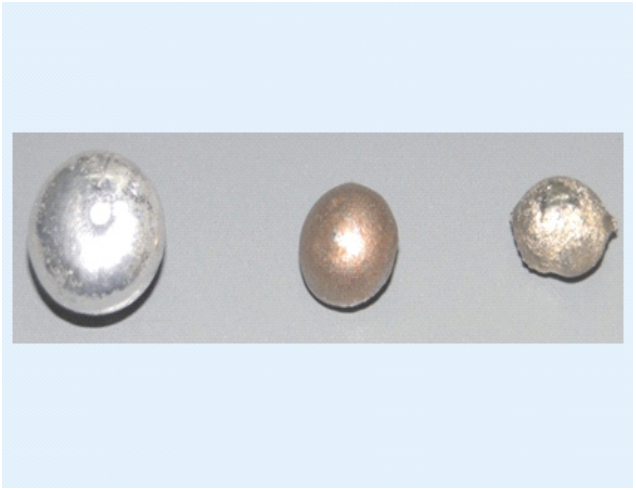

Fig.2: Various Alloy samples.

Fig.3: Induction Levitation Melter installed at Modular Laboratory, BARC.

रिएक्टर परियोजना वर्ग के साथ उच्च प्रभाव वाले पदार्थ का तालमेल

10

High Impact Materials Synergy with Reactor Projects Group

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The Reactor Projects Group, BARC is engaged in design, development, construction and operation of pressurised water reactor based plants. Indigenisation and self-reliance have been our core philosophies and our development efforts invariably emanated from these. Our resolute partnership with Materials Group ensured excellent outcome in all these efforts.

Recently, we had completed development of technology for industrial scale production of special grade low alloy steel forgings (christened APURVA) meant for manufacturing of reactor pressure vessel (RPV) for the Indian PWR programme viz. APURVA was the result of a decade-long collective multidisciplinary collaborative R&D efforts undertaken at BARC in partnership with a domestic industrial partner. Qualification of the final forgings and evolving of the manufacturing technology were the major milestones of this task. As part of the acceptance committee of the material developed, Dr R. Tiwari's thorough review of the test results of APURVA lead to its eventual certification for pressure vessel applications. During the development of APURVA we were faced with the challenge of bringing out its performance under irradiation environment. Towards this Dr. Tewari initiated a test program involving proton irradiation of APURVA samples at VECC Kolkata followed by post irradiation examination. Results of the above studies on APURVA samples revealed its remarkable properties in comparison with a similar imported popular low alloy steel grade. Dr. Tiwari's untiring support and guidance were crucial in reinforcing confidence in APURVA as an indigenous alternative for pressure vessel steel.

Dr. Raghvendra Tewari has also been guiding the team, with members from MG, PG, RDDG and RPG, which undertook development of stainless steel–titanium alloy diffusion bonded tubular adaptors. Under his leadership, the metallurgical team successfully executed a pilot comprehensive development programme involving 50 trial joints, culminating in the establishment of optimized bonding specifications. He supervised various metallurgical evaluations including determination of high-temperature mechanical properties of the alloys through hot-compression testing, metallographic examination of the Ti–SS interface, optical micrographs for groove-filling assessment, characterization of intermetallics and diffusion zones, SEM-based estimation of intermetallic layer thickness, EDS elemental mapping, EPMA diffusion profiling, and microhardness measurements across the interface to benchmark joint quality. His technical guidance during the review process played a pivotal role in finalizing the diffusion bonding parameters. Following successful pilot development, the process was scaled-up for bulk production. Dr. Tewari continued to mentor and guide the metallurgical activities, enabling timely batch-wise microstructural evaluation and microhardness qualification to ensure consistent production quality in this phase as well.

RPG was tasked with development of nickel-based clad material (with a composition of 42%Cr, 56%Ni,1% Mo) capable of superior corrosion resistance and high residual irradiation ductility. This task was realised with the involvement of MG and Nuclear Fuels Complex Hyderabad. MG under the guidance of Dr. Tewari optimised extrusion parameters such as strain-rate and temperature through dilatometry studies and process parameters w.r.t annealing and study of micro-structure by SEM, TEM, precipitate sizes, density of precipitates, grain size etc. as well. Extensive comparison of the final micro-structure with the reference samples was carried out by MG resulting in its eventual certification for reactor use.

Titanium alloy tubes and structural materials are the invariable choice in tubular heat exchangers subjected to aggressive seawater environment. Indigenous production of these tubes was essential to facilitate expansion of the domestic PWR programme. Hence, development of titan-24 (typical composition Al 1.8-2.5%, Zr 2-3%) was progressed with RPG with MG's deep involvement in association with MIDHANI and NFC. MG was responsible for optimization alloy melting techniques to yield desired composition with tight control of impurity levels at MIDHANI. Also crucial were the evolution of pass schedules, annealing temperature range, micro-structure assessment by SEM, TEM etc. This was followed by an extensive corrosion study in autoclave to quantify oxidation kinetics and hydrogen pick-up. Comparison of the final micro-structure with the reference samples and certification of the material developed for the intended application was accomplished by colleagues at MG under Dr. Tewari's mentorship.

In order to ensure longer service life of shut-off rods of the reactor MG developed a special boron alloy capable of withstanding high temperatures and irradiation environment. Dr. Tewari established an in-house production facility for this material at MG to ensure availability of this material in line with the project schedule.

With the recent launching of indigenous small modular reactor programme by the Department, it was recognised that in-core structures including control rods guide tubes need to be designed with Zr-Sn-Nb-Fe which is capable of providing longer service life with minimum distortion in neutron irradiation environment. Development of this material was co-ordinated by NFC in association with RPG and MG. During this optimization of parameters for extrusion, beta quenching, annealing and pass schedules followed by study of micro-structure by techniques such as optical microscopy, SEM, TEM, EBSD XRF ascertain microstructure properties like composition, grain size, phase distribution, texture, hydride orientation, secondary phase properties (composition, size distribution, particle density) were scrupulously carried out MG team. This material was recently launched for industrial scale production by NFC.

Dr. Tewari leaves behind a strong legacy of scientific discipline, mentoring, and leadership that has materially resulted in the realization of several technologies for multiple projects of national importance. RPG stands deeply indebted to him.

अगली पीढ़ी के संदीप्त पदार्थ

Next Generation Luminescent Materials

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Lanthanide ions doped in nanoparticles/ nanomaterials of different inorganic hosts are considered to be next generation luminescent materials and have potential applications in the area of sustainable lighting, bio-labelling and up-conversion luminescence based technologies [1]. Synthesizing such nano-materials in different sizes and shapes and understanding their photo-physical properties constitutes one of the frontline research activities for the past few years in our group. Representative systems investigated are described briefly in this manuscript.

Luminescent properties of inorganic nano-phosphates

$CePO^4$, $CePO^4$:Tb and $CePO^4$:Dy nano-rods with monoclinic structure were prepared at a low temperature of 140°C in ethylene glycol medium. These nanorods are found to be dispersible in solvents like methanol and water. High Resolution Transmission Electron Microscope (HRTEM) and Selected Area Electron Diffraction (SAED) images reveal that the nano-rods are highly crystalline with very high degree of homogeneity. These nano-rods are crystallo-graphically aligned to each other. Strong Tb^{3+} and Dy^{3+} emission has been observed on direct excitation of the $Ce3^+$ levels in the $CePO_4$ host, due to energy transfer from the host to Tb^{3+}/Dy^{3+} ions. These nano-rods were dispersed in sol-gel films and such materials exhibited improved photo-physical properties.

Bi^{3+} is considered to be an ideal substitute for lanthanide ions due to its comparable ionic radii and coordination characteristics. Keeping this in mind hexagonal and monoclinic forms of $BiPO_4$ nanomaterials were prepared based on the reaction of Bi^{3+} and PO_4^{3-} ions in ethylene glycol medium at 100 and 185°C respectively. From the differential thermal analysis (DTA) studies it is confirmed that the difference in the nucleation mechanism rather than the phase transition is responsible for the monoclinic phase formation at low temperatures. Monoclinic $BiPO_4$ is quite stable and forms random solid solutions with lanthanide phosphates having both monoclinic (monazite) and tetragonal (xenotime) structures as confirmed by XRD, FTIR and ^{31}P solid state nuclear magnetic resonance studies. On excitation corresponding to the $^1S_0 \rightarrow {}^3P_1$ transition of Bi^{3+} in $BiPO_4$:Ln nanomaterials, energy transfer from host to lanthanide ions takes place. Upon doping Eu^{3+} ions, monoclinic form of $BiPO_4$, bright luminescence is observed (Fig.1). The studies are quite relevant as there is a growing interest all over the world in replacing lanthanide based hosts used for different applications with easily available, cost effective main group elements such as Sb, Bi etc., or such hosts.

Luminescent properties of oxide based hosts

Nano-crystalline $ZnGa_2O_4$ doped with varying concentrations of lanthanide and Ge^{4+} ions were prepared and

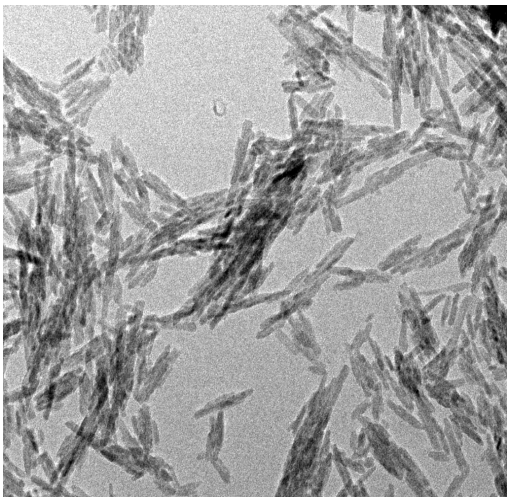


Fig.1: $CePO_4$: Dy nano-rods.

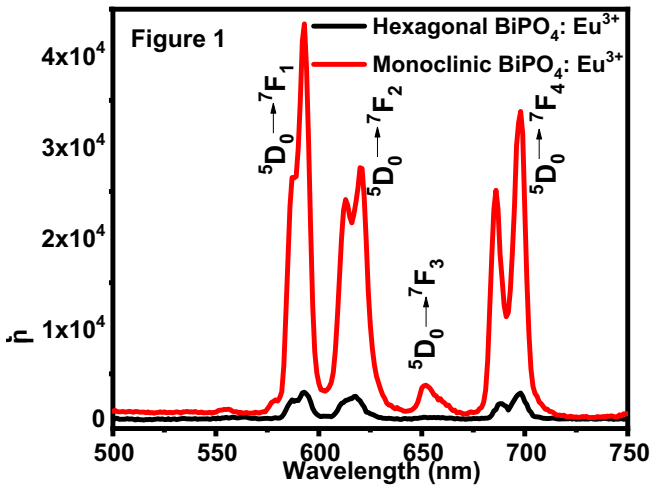


Fig.2: Upon doping Eu^{3+} ions, monoclinic form of $BiPO_4$ exhibits bright luminescence.

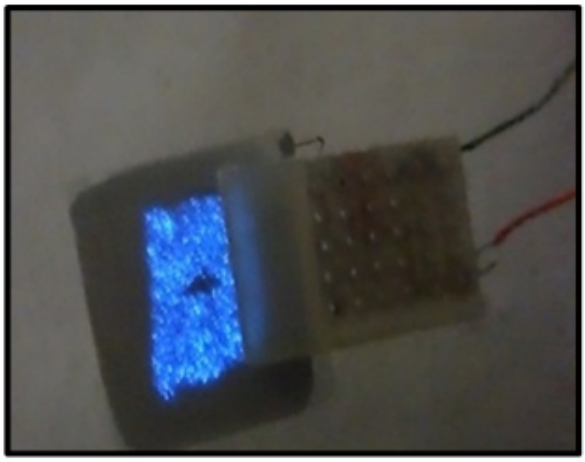


Fig.3: Electro-luminescence from $ZnGa_2O_4$:1%Ge.

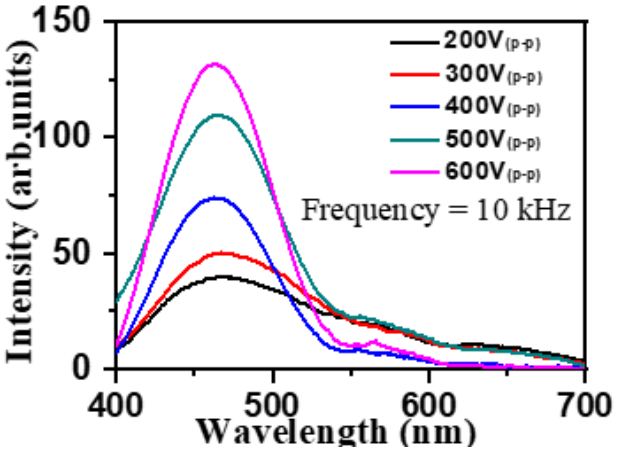


Fig.4: Electro-luminescence spectra.

their photo- and electroluminescence properties were investigated in detail before and after annealing at 900°C. X-ray diffraction (XRD) studies confirmed that Ge^{4+} doping even to a level of 0.5 at.% at the expense of Ga^{3+} in $ZnGa_2O_4$, leads to incorporation of significant extent Ga^{3+} ions (~29%) at Zn^{2+} site (tetrahedral site) in $ZnGa_2O_4$ lattice thereby increasing relative extent of distorted gallium-oxygen (GaO_x) structural units. XRD, UV-Visible optical reflectance and lifetime measurements confirmed that a maximum of 0.65 at.% Ge^{4+} is doped in nanocrystalline $ZnGa_2O_4$. Bright blue electro-luminescence has been observed from Ge^{4+} doped $ZnGa_2O_4$ nanoparticles upon application of AC voltages (shown above). Lack of oxygen vacancies in Ge^{4+} doped annealed samples facilitate selective excitation of regular GaO_6 and GaO_x structural units, upon application of AC voltages, leading to around 50% reduction in line width of electroluminescence peak from doped sample compared to undoped one. Variation in electroluminescence properties between doped and undoped samples is due to the difference in nature of defects generated in the lattice.

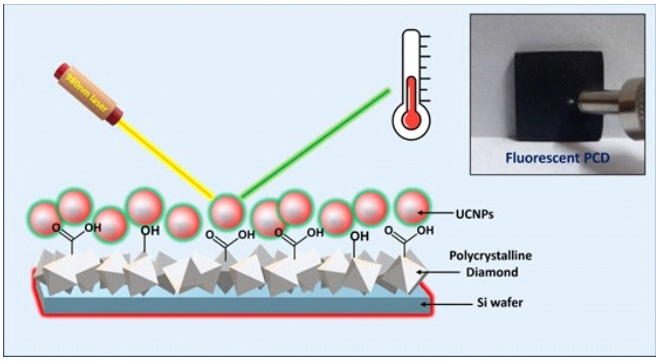
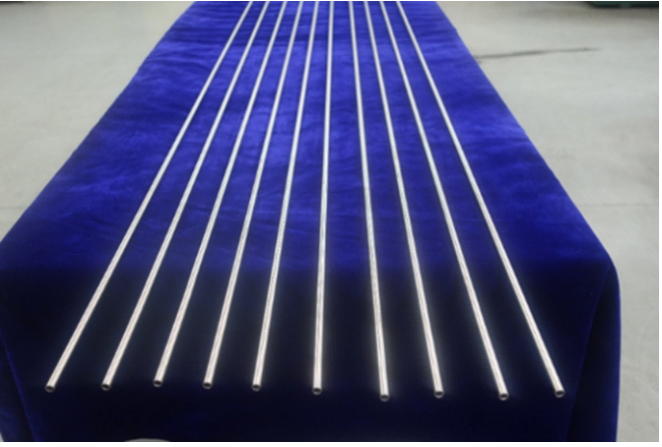
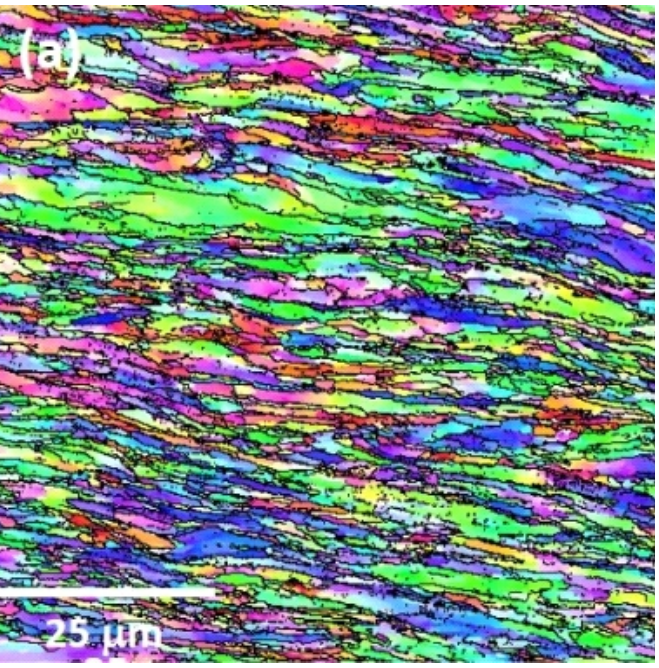


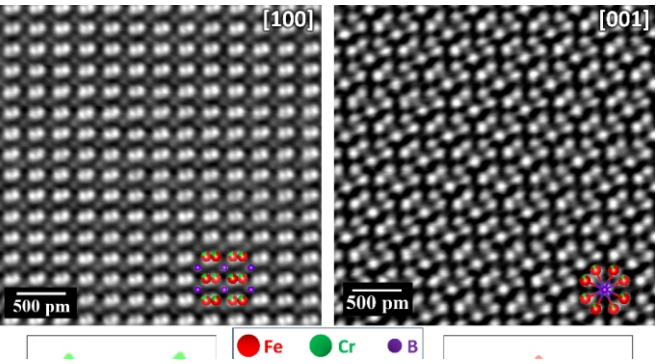
Fig.5: PCD films functionalized with $NaYF_4$:Er,Yb upconversion nanoparticles.

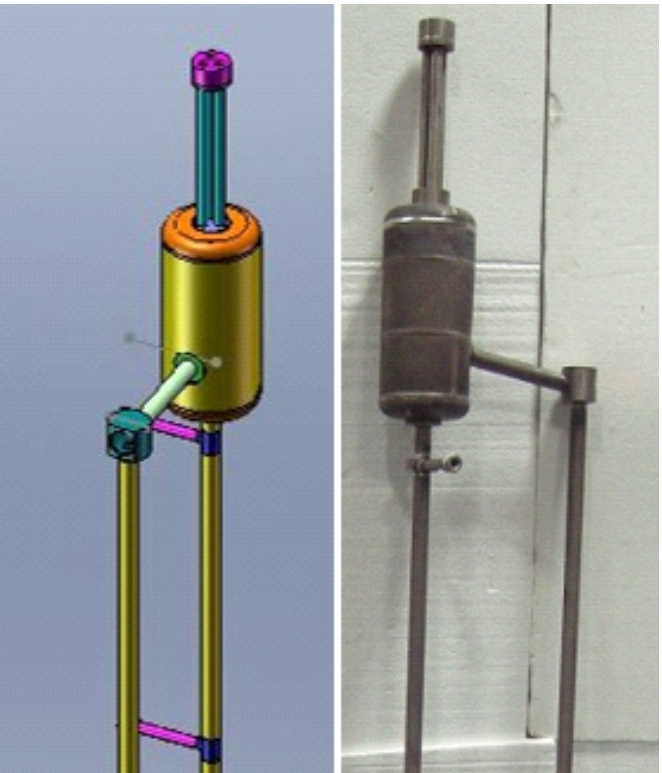
Luminescence properties of upconversion nanoparticles functionalised on lab grown diamond (LGD) films for non-contact temperature measurements

Preparation and characterization of lab grown diamond (LGD) films with luminescent nanoparticles on the surface, is the subject matter of this study. Polycrystalline diamond (PCD) films functionalized with NaYF4:Er,Yb upconversion nanoparticles (UCNPs) were developed and their temperature sensitivity of luminescence characteristics has been evaluated. UCNPs functionalized on surface treated PCD films (UCNPs – TPCD) exhibited superior temperature sensitivity (Sr = 1.08% at 300 K) compared to as prepared UCNPs and UCNPs coated on as grown PCD films (UCNPs – PCD). Surface oxidation of PCD films upon treatment and uniform distribution of UCNPs on PCD films were confirmed by upconversion luminescence, X-ray Photoelectron, IR and Raman spectroscopic techniques along with Scanning Electron Microscopic (SEM) and Atomic Force Microscopic (AFM) techniques. Increase in temperature of diamond lattice due to 980 nm laser irradiation leads to increase in population of 2H11/2 level of Er3+ and associated increase in the fluorescence intensity ratio and improved temperature sensitivity of UCNPs functionalized on treated diamond surface. Treatment of diamond surface led to surface oxidation and uniform functionalization of the nanoparticles on PCD film









Materials Science
Research & Development

expanding nuclear energy into newer horizons

नाभिकीय अनुप्रयोग के लिए संरचनात्मक पदार्थ विकास - एक संक्षिप्त अवलोकन

12

Structural Materials Development for Nuclear Application-A Brief Overview

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Indigenous development of Ni-Mo-Cr alloy seamless Tubes

Nuclear energy is proven as an inevitable option across the globe, for sustainability and commitment towards climate control. Government of India laid an ambitious roadmap towards significant enhancement in share of nuclear power in the energy matrix and contribute towards net zero emission. This not only entails accelerated growth in the proven Pressurized Heavy Water Reactors (PHWRs), but requires simultaneous development of advanced reactor technologies. In this regard, structural material development plays a pivotal role for realizations of the milestones. Severe operating conditions prevails in nuclear reactors impose enormous challenges on material performance. Sustainability under higher operating temperature and aggressive corrosive environment, better fuel efficiency and accident tolerant designs, are additional requirement in new generation reactors. Therefore, continual improvement in materials and associated processing techniques remain one of the key factor. Zirconium alloys (zircaloys and Zr-Nb alloys) remains the work horse for core structural in water cooled reactors. However, new reactor designs and adoption of advance reactor technologies require enhanced material performance of the conventional alloys, as well, demand development of several alloys viz. special grades stainless steels, superalloys, titanium based and niobium based alloys etc.; in order to sustain high operating temperature (upto 1000°C) and severe corrosive environment.

Nuclear Fuel Complex (NFC), has been in the forefront in transformative contributions towards indigenous development and manufacturing of structural materials for critical applications in nuclear power programme, as a testament towards 'Atmanirbhar Bharat'[1]. The development of these manufacturing technology involves establishing series of thermo-mechanical operations with combination of hot extrusion, forging, cold pilgering, heat treatment along with surface finishing operations. Design aspirations of structural materials have been accomplished at NFC through combination of two approaches viz. (a) improved metallurgical characteristics engineered through advancement in processing technologies, (b) indigenous development of 'high performance alloys'. This article gives a brief overview of indigenous development of important nuclear structural materials at NFC, in strong collaboration with BARC. Indeed, article emphasizes on the fact that collaborative approach has a profound impact and remains the prime strengthening mechanism in the entire saga of materials development.

Development of Nb-1%Zr-0.1%C alloy test loop

Nb-1%Zr-0.1%C alloy has been considered as structural material for the Compact High Temperature Reactor (CHTR), which is envisaged to operate at high temperature (~1000°C) containing molten Pb-Bi as coolant [2]. Refractory alloys remain the obvious choice for such combination of operating temperature and aggressive environment. Thermal hydraulic behaviour of the molten Pb-Bi eutectic alloy for CHTR, required a test loop with Nb-1%Zr-0.1%C as material of construction. However, refractory alloys like Nb-1%Zr-0.1%C, exhibit poor fabricability, was hardly investigated with respect to alloy preparation, establishing thermo-mechanical processing aspect. NFC, in collaboration with BARC team led by Dr. R. Tewari, shouldered the task of fabricating the test loop with difficult to process refractory alloy. Alloy melting has been successfully achieved by electron beam melting under high vaccum and technology development for forming the material in various shapes has been accomplished in record time. Rich experience of Dr. Tewari in the frontier of material research and characterization, has contributed extensively in bringing insights on material behavior during hot deformation processing and thermo-mechanical operations. Fig.1 illustrates the designed vis-a-vis as-fabricated test loop of CHTR, developed utilizing tubular and sheet products successfully manufactured through complex combination of thermo-mechanical processing and optimization of electron beam welding technique for several weld configurations.

Indigenous development of Ni-Mo-Cr alloy seamless Tubes

Molten Salt Breeder Reactors (MSBRs) technology is a promising advanced reactor technology mainly because of the liquid form of the fuel in the reactor. One key activity of this programme is the fabrication of MSBR loop to study the thermal hydraulics and corrosion attack from the liquid fuel and structural integrity up to service temperature of 650 °C. Again, the challenge boils down to materials development in high

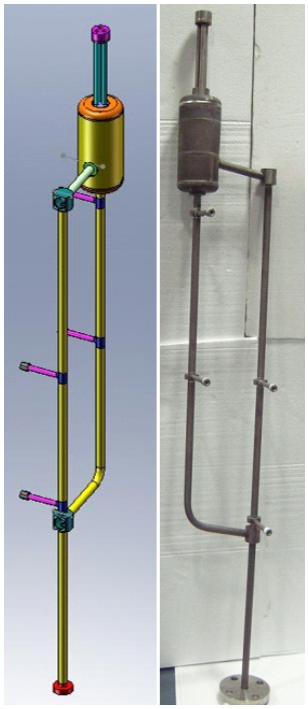

Fig.1: Designed and as-fabricated test loop for CHTR.

temperature corrosive fluid, which envisaged Ni-Mo-Cr alloy as material of construction owing to its excellent corrosion resistance in fluoride environment. Strength of collaborative effort of Materials Group, Reactor Design and Development Group, BARC and Nuclear Fuel Complex, Hyderabad jointly established the process route and produced 'difficult-to-process' Ni-Mo-Cr-Ti alloy seamless tubes (Fig.2), first time in the country. This collaborative effort in research and development mark a significant milestone in the advancement of Ni-Mo-Cr alloys, which are crucial for achieving self-reliance in molten salt breeder technologies.

Materials development for Small and Modular Reactor

Development of Small and Modular Reactor (SMR) technologies require development of structural material for longer in-reactor residence time. In this respect, the task of

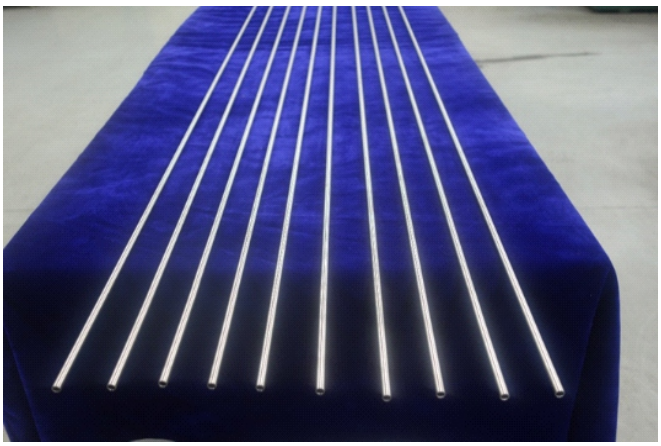

Fig.2: Ni-Mo-Cr alloy seamless tube developed for MSBR.

indigenous alloy development (Zr-Nb-Sn-Fe quaternary alloy for in-core and Alloy 690TT for steam generator aplication) and establishment of thermo-mechanical process flow sheet has been bestowed. Another milestone accomplished through successful establishment of thermo-mechanical process route and development of these seamless tubes. The expertise of Dr. Tewari and Dr. R.N. Singh, Materials Group, BARC has been the major support in deep explorations on various aspect of microstructural evolution, phase transformation, precipitate characterization etc.

Above are the few development activities in past 1-2 years to showcase the strong association of NFC and Materials Group, BARC.

Materials development for Small and Modular Reactor

The large thrust in nuclear energy and high order design aspirations, will entails enormous demand in materials development, in near future. This reminds the importance of collaborations, as resonated by Late Dr. Srikumar Banerjee, Dr. Raghavendra Tewari, and several others in bringing illustrious success stories for the department. With profound knowledge in the realm of material science and materials characterization, Dr. Tewari inspired, in several programme, as an esteemed member of NFC Board and other advisory committees.

अनुसंधान एवं विकास तथा औद्योगिक अनुप्रयोगों के लिए स्वदेशी लेसर योजक विनिर्माण में अग्रणी

13

Pioneering Indigenous Laser Additive Manufacturing for R&D and Industrial Applications

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Laser Additive Manufacturing Activity at RRCAT

Additive Manufacturing (AM) is shaping the future of production by offering unprecedented design flexibility, faster product development cycles, localized manufacturing, and improved resource efficiency. Around the world, industries and research institutions are investing in AM to overcome the limitations of traditional manufacturing and advancing sustainable industrial growth.

In India, RRCAT has taken a leadership role in developing indigenous Laser Additive Manufacturing (LAM) systems, establishing itself as a national pioneer in additive technology. The centre has successfully designed and deployed Laser Directed Energy Deposition (LAM-DED) and Laser Powder Bed Fusion (LAM-PBF) systems, complemented by Wire-Arc Additive Manufacturing (WAAM) and Wire-Laser Additive Manufacturing (W-LAM) platforms to cater to a range of component sizes and material types.

These state-of-the-art systems are employed in cutting-edge research across the Department of Atomic Energy (DAE) laboratories and through collaborations with leading academic institutions and industries. Applications span complex functional structures, graded materials, and multi-metal interfaces that are otherwise unachievable through conventional manufacturing. Through its technology development and technology translation initiatives, RRCAT has strengthened India's additive manufacturing ecosystem—directly contributing to the vision of Atmanirbhar Bharat and the national mission of technological self-reliance.

Collaborations with Materials Group at BARC

A major success story of RRCAT's innovation is its long-term collaboration with the Materials Group, BARC, for developing advanced materials vital to nuclear and aerospace technologies. Under the collaborative work with Dr. R. Tewari, Director, Materials Group, BARC, the teams have developed novel material systems and joining methods using the LAM-DED process.

One of our remarkable achievements is the creation of functionally graded transition joints between titanium and steel—a critical innovation for high-performance applications that demand both low weight and high strength. Traditional welding or brazing methods often form brittle intermetallic layers between these metals due to large differences in thermal and physical properties. To overcome this challenge, the RRCAT–BARC team engineered a graded transition zone using vanadium (V) and chromium (Cr) interlayers using LAM-DED technology. The LAM-DED process offered the spatial control necessary to sequentially deposit these interlayers, ensuring gradual compositional transitions from stainless steel to titanium without disrupting metallurgical continuity. Comprehensive microstructural and phase-evolution studies were carried out under the guidance of Dr. Tewari using advanced scanning and transmission electron microscopy (SEM/TEM). These investigations revealed diffusion-controlled solid-solution formation with minimal intermetallic precipitation. The developed joint at optimized process parameter is found to have excellent mechanical strength developed with functionally graded approach.

This collaborative research not only provided a fundamental understanding of solidification dynamics, diffusion phenomena, and phase transformations in multicomponent alloy systems but also showcased the potential of laser additive manufacturing for designing site-specific materials architectures. The results have significant implications for fabricating dissimilar metal joints in nuclear reactors, aerospace propulsion systems, and high-temperature structural components. The success of this effort underscores the importance of cross-institutional cooperation that integrates laser processing technology with materials characterisation.

Indigenous Development of Laser Powder Bed Fusion System for MG, BARC

In a further step toward technology localization, RRCAT has developed a compact LAM-PBF system for MG, BARC, featuring a 500W fiber laser with a build volume of

Fig.1: Indigenously developed LAM-PBF System.

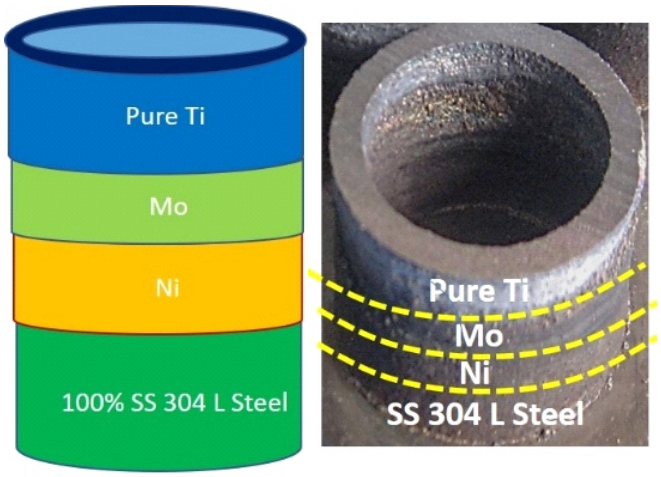

Fig.2: LAM built SS-Ti Transition Joint.

150mm×150mm×150mm to accelerate the development and qualification of advanced materials for future nuclear technologies. The system is designed for controlled experimentation with advanced materials, providing high process reproducibility while minimizing powder handling.

Such a platform provides an ideal balance between laboratory-scale throughput and process control, enabling fabrication of representative test coupons, graded specimens and parameter matrices while minimizing powder inventory and radiological handling footprint. The developed system is presently under trial testing, marking a significant step toward deploying this advanced manufacturing technology for nuclear material development and related applications.

Perspective and Outlook

The growing convergence of laser additive manufacturing and advanced materials characterization is reshaping how materials are designed, processed, and applied. The collaborative efforts with Dr R. Tewari illustrate this synergy, where deep insights into phase transformations, diffusion mechanisms, and microstructural control enable the creation of tailor-made materials with superior properties.

Looking ahead, future research is expected to focus on developing different materials using LAM-DED and LAM-PBF technologies with enhanced properties for various applications. There is also growing interest in exploring multi-material printing, functionally graded coatings, and repair of high-value components for critical sectors.

The sustained collaboration between RRCAT and BARC stands as a model of multidisciplinary synergy—bridging laser technology, metallurgy, and materials characterization. This partnership continues to inspire innovations that address challenges in nuclear materials development, aerospace structures, and advanced manufacturing contributing to India's advancement in strategic materials research.

एक बहु-घटक प्रणाली के विपथन संशोधित STEM-iDPC कंट्रास्ट में स्पष्ट विसंगतियों की व्याख्या

14

Understanding Apparent Anomalies in Aberration Corrected STEM-iDPC contrast of a Multicomponent System

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Integrated Differential Phase Contrast (iDPC) has emerged as one of the most useful detectors in recent times, owing to its ability to simultaneously image both high- and low-Z elements. It is an advanced STEM technique that creates potential maps of thin samples by integrating DPC signals from segmented detectors [1]. The method typically uses a quadrant detector (or multi-sector detector) to measure the center-of-mass (COM) of the convergent beam electron diffraction pattern, which is then integrated to produce a scalar phase image. iDPC provides linear phase contrast imaging with low dose efficiency, making it particularly suitable for beam-sensitive materials. The technique excels at imaging light elements next to heavy ones, even at low electron doses. Unlike ABF, iDPC produces images where light elements appear with bright contrast against a dark background. Advanced implementations use multi-sector detectors (8, 12, or even 48 segments) instead of simple quadrant detectors to achieve more isotropic contrast transfer functions and improve image quality. This enhancement reduce anisotropy artifacts and provide better contrast and resolution, especially for thick samples. iDPC-STEM produces an image contrast that is proportional to the projected electrostatic potential [2], enabling high-resolution visualization of both light and heavy elements, where the contrast is directly proportional to the atomic number Z [3]. However, this narrative is only valid under certain conditions, as will be demonstrated here.

Fig.1 shows the STEM-iDPC images acquired along the [100] and [001] ZA in (Fe,Cr)2B of a melt-cast ferrite–boride composite [4 – 6]. It is interesting to note that the Fe/Cr and B atomic columns appeared with nearly equal intensities along [001] in contrast to [100], where the intensity was proportional to the Z of the atom. The approach that iDPC intensities are proportional to Z implicitly assumes that iDPC intensity differences directly reflect projected atomic potential. This treatment leaves a key ambiguity: why did the Fe-B contrast in [001] differ so markedly from the [100]. The ambiguity arises because of the influence of imaging conditions, such as specimen thickness, defocus, and zone-axis-dependent dynamical scattering, as discussed below.

The iDPC contrast transfer function (CTF), derived in detail by Lazic et al. [7] and evaluated in Liang et al. [8], governs how different spatial frequencies are transferred to the image and is extremely sensitive to defocus, specimen thickness, accelerating voltage, convergence angle, collection angle, sample tilt, and electron dose. For thin samples, the iDPC signal is approximately proportional to the projected potential, making the intensity roughly proportional to atomic number. For thicker samples, however, multiple scattering, dechanneling, and the anisotropy of the CTF can drastically alter contrast, even reversing the relative intensities of atomic species. The experimental thickness for the region studied here, determined from zero-loss EELS analysis, was approximately 20 nm and 30 nm for [100] and [001] ZA samples, respectively. The iDPC images were acquired at a nominal defocus of -4 nm. Multislice simulations were carried out using Dr. Probe software to interpret the experimental contrast behavior. The simulation series varied defocus from -10 nm to +4 nm in 2 nm steps and thickness from ~0.5 nm to ~50 nm. Fig.2 shows the simulated iDPC contrast variation for (a) [100] and (b) [001], respectively, across the full defocus-thickness space.

Along the [100] direction, the channeling of the probe along Fe/Cr columns is less perturbed by neighboring B columns. In the simulated contrast maps, shown in (a), even at t=20 nm, the Fe/Cr-B intensity difference remains appreciable over a wide range of defocus values. This thinner sample remains closer to the single-scattering regime, so iDPC intensities retain more of their monotonic Z-dependence. Along the [001] direction, the projection geometry places B columns in closer lateral proximity to Fe/Cr columns, thereby increasing the likelihood of dynamical scattering between them. This effect is pronounced at intermediate thicknesses (20-40 nm), as shown in Fig.2(b), where multiple scattering redistributes intensity between columns. In Fig.2(b), the contrast maps reveal a plateau in the defocus-thickness space where the Fe/Cr and B intensities converge.

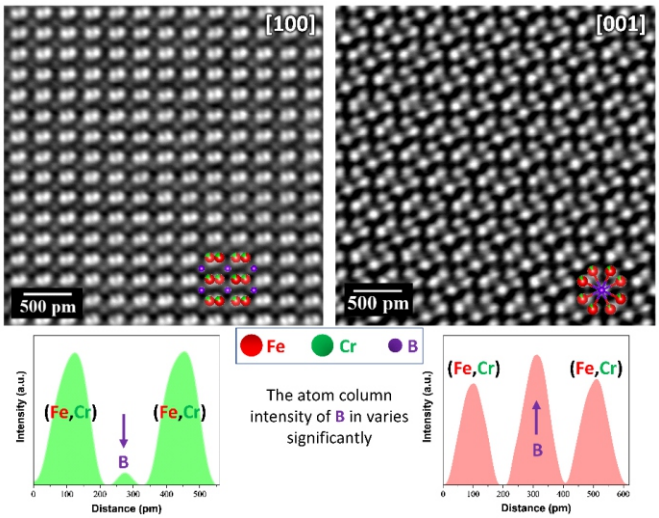

Fig.1: Quantitative comparison of iDPC-STEM contrast between Fe/Cr and B atom columns for (Fe,Cr)2B along [100] and [001].

The experimental defocus (-4 nm) and thickness (~30 nm) sit in this plateau, explaining the observed similarity in intensity.

Therefore, it is clear that an intriguing interplay between sample thickness and defocus in iDPC imaging is responsible for relative intensities of higher vis-à-vis lower Z elements in a multicomponent system.

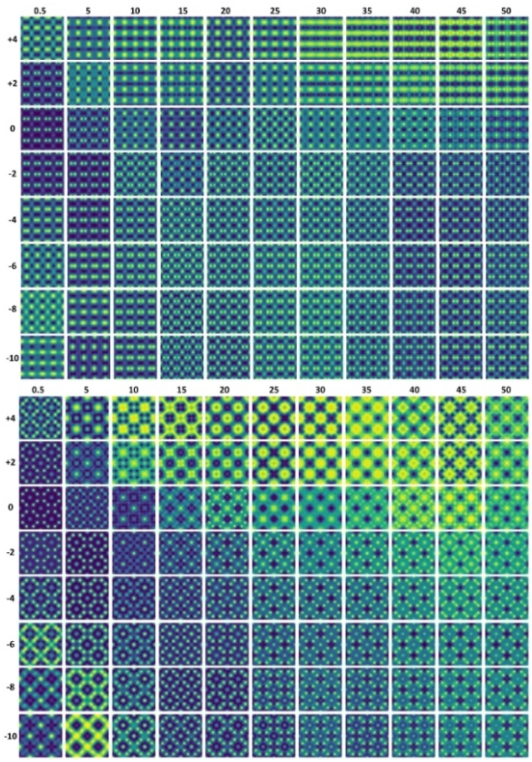

Fig.2: Simulated iDPC-STEM contrast for $(Fe,Cr)_2B$ along (left) [100], and (right) [001] as a function of defocus (vertical axis, nm) and specimen thickness (horizontal axis, nm).

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15

Electron Back-Scattered Diffraction in Materials Engineering

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Electron Back-scattered Diffraction (EBSD) is an effective and efficient tool for point-specific measurements of crystallographic orientation in polycrystalline materials. EBSD has become a foundational characterization tool in modern materials science because it uniquely combines high spatial resolution with quantitative, crystallographic information across microstructural fields. Where transmission techniques (e.g., TEM) give atomic-scale detail at single points, and X-ray/neutron diffraction gives bulk averages, EBSD fills the crucial mesoscale niche: mapping grain orientations, boundary character, local texture, phase distributions and strain-related misorientations across areas from microns to millimetres. That capability underpins advances in processing–structure–property understanding and has been widely exploited to link deformation/processing routes to mechanical and functional behaviour [1].

One of the clearest contributions of EBSD is its ability to quantify texture and its evolution during plastic deformation and annealing processes. EBSD maps give orientation distributions and allow computation of pole figures, inverse pole figures, and orientation distribution functions (ODFs) at spatial resolution-enabling researchers to separate global texture from local, heterogenous orientation patterns caused by, for example, shear bands or retained sub-grains. EBSD has been routinely used to study texture evolution in metals processed by severe plastic deformation techniques such as equal channel angular extrusion/pressing (ECAE/ECAP), accumulative roll bonding (ARB), and extrusion, showing how processing route, strain path and temperature determine final crystallographic textures that strongly influence ductility and anisotropy. These EBSD-based texture studies have guided interpretations of why certain processing paths produce better formability or strength in Mg, Al, Ti alloys and steels.

EBSD also excels at characterizing grain boundary character and misorientation distributions quantities that control recrystallization, grain-boundary sliding, intergranular fracture, and many diffusion-controlled processes. Quantitative misorientation analysis (low-angle vs. high-angle boundaries, special coincidence site lattice boundaries) is routinely extracted from EBSD maps and used to predict and tailor mechanical response. A notable example is the study of misorientation scaling in heavily deformed metals, where detailed EBSD-derived misorientation distributions supported the discovery of general scaling laws across materials and deformation conditions - an insight that helps unify mesoscale deformation behaviour across crystal systems [1, 2].

Another powerful application is correlating microstructure with local mechanical behaviour. EBSD, when combined with indentation, in-situ deformation, or site-specific testing, allows one to connect an individual grain's orientation, boundary neighbours, and local misorientation gradient with its observed deformation mode (slip, twinning) or propensity to damage. EBSD has been used alongside TEM and mechanical testing to relate texture and local microstructure to macroscopic responses for example, explaining differences in ductility and strain hardening in Mg alloys and Ti alloys by mapping lattice rotations and twin activity across regions exposed to different processing histories. This multi-technique approach with EBSD providing the spatial orientation/strain map has been central to designing processing strategies for better ductility/strength combinations [3].

Phase identification and phase-specific orientation relationships are a further area where EBSD contributes uniquely. Modern EBSD indexing can distinguish crystallographic phases in multiphase alloys and determine orientation relationships at phase interfaces data that are critical for understanding martensitic transformations, precipitation textures, and phase-transformation driven property changes. For instance, investigations of boron-modified Ti-6Al-4V using EBSD to follow $\beta \rightarrow \alpha$phase evolution and the associated texture changes during thermomechanical processing, related the processing schemes to final phase morphology and mechanical performance [4].

EBSD's utility also extends to nanoscale and ultrafine-grained materials when combined with careful sample preparation and complementary techniques. Fig.1 is an example of the Nb alloy Nb-1Zr-0.1C alloy subjected to severe plastic deformation by high pressure torsion to the strain levels 4.5 and 70. Grain-by-grain EBSD mapping in severely deformed or nanocrystalline materials, corroborated with TEM has allowed to reveal sub-grain formation, recovery/recrystallization pathways, and the role of low-angle boundaries in stabilizing refined microstructures. Such analyses were essential to explain mechanical behaviour after large

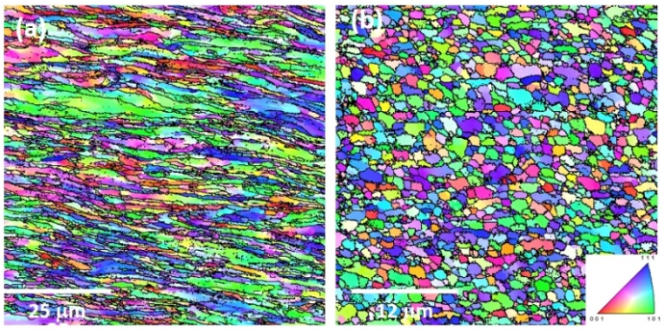

Fig.1: EBSD generated inverse pole figure maps of pure Nb subjected to high pressure torsion subjected to strain (a) 4.5 and (b) 70.

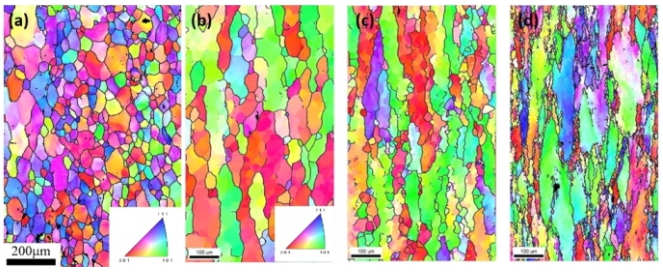

Fig.2: EBSD generated inverse pole figure map for the Nb-1Zr-0.1C alloy as the (a) starting material, materials deformed at 1500°C at the strain rates (b) 0.001 s^{-1}, (c) 0.01 s^{-1}, and (d) 0.1 s^{-1}.

strain processing and annealing, and to design post-processing heat treatments to control texture and grain size [5].

Practically, EBSD also supports industrial and applied research objectives optimizing additive manufacturing parameters, tailoring nanocomposites, and evaluating failure surfaces. SEM-EBSD stands among the core characterization tools used for both fundamental texture studies and applied projects, for example, texture-property studies in functional materials like thermo-electrics or in structural materials like selective laser melted alloys. By providing spatial maps of phase, orientation and boundary character, EBSD helps engineers identify process windows that minimize deleterious textures or adverse phase distributions.

Methodological advances tied to EBSD have also influenced broader practice. Improvements in indexing algorithms, higher-speed detectors, and better software for strain and grain-boundary analytics mean EBSD datasets can now be used quantitatively for input to crystal plasticity models and mesoscale simulations. EBSD data are not only descriptive but increasingly predictive when integrated with simulations, enabling process design rather than just post-mortem analysis [1].

EBSD-based characterization of niobium and molybdenum alloys has significantly advanced the understanding of deformation, recovery, and recrystallization behaviour in body-centred cubic (BCC) refractory metals (Fig. 2). Using high-resolution EBSD orientation mapping, texture evolution, grain boundary character, and intragranular misorientation development resulting from thermomechanical processing such as rolling, forging, and annealing have been quantified. EBSD analyses have been particularly effective in revealing the heterogeneity of stored deformation, sub-grain formation, and the role of low and high angle grain boundaries in controlling recrystallization kinetics in Nb and Mo based alloys. By correlating EBSD-derived microstructural metrics with mechanical response at elevated temperatures, critical insights have been developed into processing microstructure property relationships essential for the design and optimization of refractory alloys for high-temperature structural and nuclear applications [6].

EBSD has led to the understanding of radiation-induced microstructural evolution and its consequences for mechanical behaviour in structural alloys used in nuclear environments. EBSD in conjunction with advanced transmission electron microscopy (TEM-OIM) has been very effective to characterize irradiation-induced defects such as dislocation loops, defect clusters, grain boundary modification, and changes in local crystallographic orientation. By systematically comparing unirradiated and irradiated states, the role of irradiation on deformation mechanisms, strain localization, grain boundary stability and damage accumulation has been deciphered. These insights have contributed to a deeper understanding of irradiation tolerance and degradation pathways, providing a microstructure based framework for designing materials with improved performance and reliability under prolonged radiation exposure [7].

In summary, EBSD has transformed materials engineering research by providing spatially resolved, quantitative crystallographic information across length scales directly relevant to mechanical and functional properties. EBSD maps have been used to quantify texture evolution after plastic deformation and annealing, to measure misorientation statistics that led to general scaling insights, to resolve phase-specific orientation relationships in engineering alloys, and to tie local microstructural features to macroscopic mechanical outcomes. Together with complementary tools (TEM, XRD, neutron/synchrotron methods and modelling), EBSD forms a central pillar of contemporary materials characterization one that continues to enable both fundamental discoveries and targeted materials engineering.

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16

Role of Microstructure and Texture in Nuclear Materials

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Microstructure and texture together form the underlying architecture that governs the performance of structural materials. While microstructure encompasses grain morphology, phase distribution, second-phase particles and defect configurations, texture describes the collective orientation of these grains and the anisotropy that arises from it. In metals with hexagonal close-packed crystal structures, such as zirconium and titanium, this anisotropy exerts a particularly strong influence on deformation pathways, irradiation response and dimensional stability. Thus, in any engineered component intended to operate reliably for decades, microstructure sets the intrinsic framework of strength and stability, and texture determines how this framework interacts with stress, temperature, hydrogen, radiation damage and time.

These considerations become central in nuclear materials, where zirconium alloys serve as the backbone of water-cooled nuclear reactor technology. Their low neutron absorption enables their use in fuel cladding and pressure tubes, but their performance is inseparably linked to their microstructure–texture state. This microstructure and texture evolves through complex series of thermo mechanical processing steps involved in the manufacturing of nuclear components and continues to evolve during the service period of these in-core components.

Over the course of thermomechanical processing—processing steps like β-quenching, extrusion, cold pilgering and carefully controlled annealing-Zircaloy-4 evolves from a Widmanstätten α morphology to a refined, partially recrystallized structure with a strong preferred orientation. Early steps such as β-quenching provide a fine basket-weave template; hot extrusion elongates α grains, strengthens prism-plane alignment and introduces the first major texture components; pilgering heavily fragments grains and sharpens the orientation distribution; and annealing restores selected orientations while stabilizing those which provide resistance to irradiation creep and growth. In pressure-tube alloys such as Zr-2.5Nb, the coexistence of metastable β-phase adds an additional layer of complexity. Depending on processing temperature and strain path, β may persist as continuous films, lamellae or globular regions, each influencing deformation behaviour and hydride nucleation sites. The α grains inherit part of their orientation from the β→α transformation, meaning that texture is simultaneously shaped by deformation, variant selection and recrystallization.

Such engineered textures have concrete consequences in service. The distribution of basal poles strongly influences axial and circumferential irradiation growth, the ease of cross-slip or twinning, and the propensity for hydride reorientation under applied stress. A high radial Kearns factor in Zircaloy-4 cladding suppresses radial hydrides that can compromise integrity, while specific prism-plane alignments in Zr-2.5Nb pressure tubes are associated with improved creep performance and dimensional stability. Microstructural details including second-phase particles, their stability under irradiation, and the redistribution of alloying elements further shape long-term behaviour. Irradiation-induced amorphization of precipitates in Zircaloy-4 or the spheroidization of β-Nb in Zr-2.5Nb alters defect sink strengths, influences corrosion behaviour and modifies hydride precipitation patterns. Thus, what appears as a static structure is in fact a dynamic system continually evolving under neutron flux, temperature gradients and hydrogen ingress.

Looking ahead, several emerging developments promise to further refine our understanding and control of these alloys. Nanoscale tools such as atom probe tomography, precession electron diffraction, high-angular-resolution EBSD and Transmission Kikuchi Diffraction, Electron channelling contrast imaging etc are enabling unprecedented resolution in characterising dislocation loops, near boundary gradient zones, precipitate grain boundary interactions in these complex systems. Computational methods from crystal plasticity to phase-field simulations are beginning to predict texture evolution, irradiation-induced damage and hydride behaviour with high fidelity. Future progress will depend on integrating these advanced characterization and modelling approaches into processing design, allowing microstructure and texture to be deliberately engineered for next-generation reactors, accident-tolerant fuel systems and high-temperature zirconium alloys for emerging reactor concepts.

In closing, it is appropriate to acknowledge that much of the progress in this field has been shaped by decades of

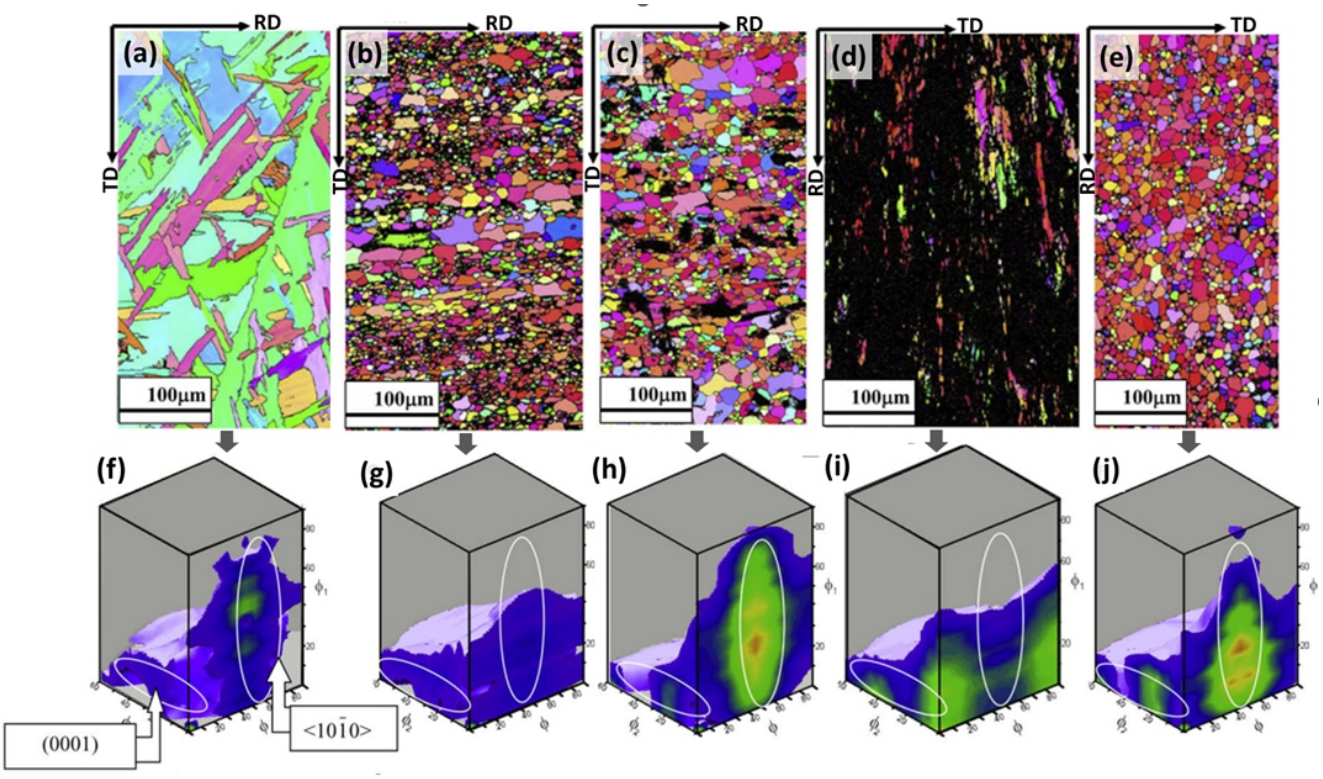
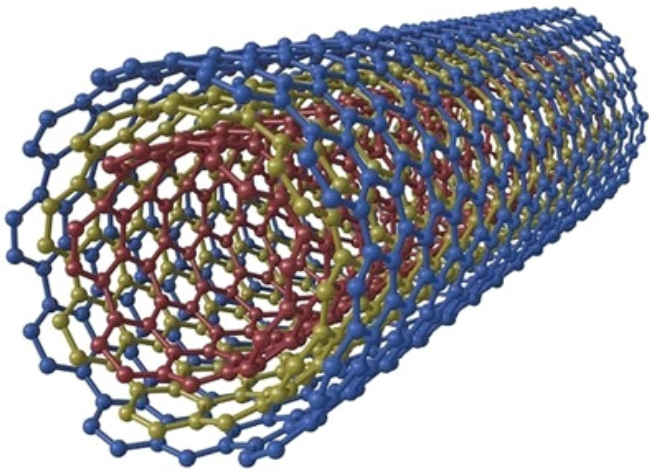


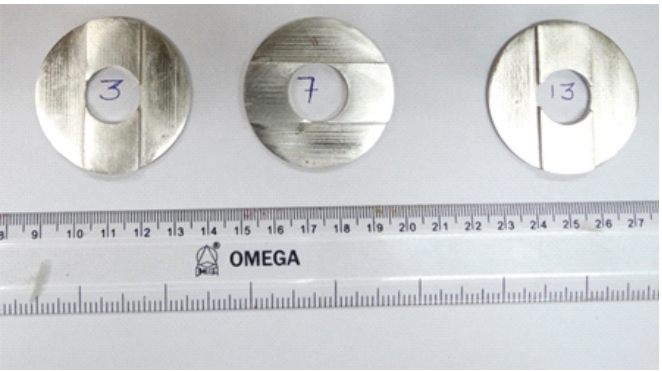
Fig.1: Evolution of microstructure (a–e) and texture (f–j) in Zircaloy-4 during TMP, illustrating refinement, deformation effects and strengthening of major orientation components.

scientific collaboration between BARC and our research group at IIT Bombay. The understanding of microstructure–texture interdependence in zirconium alloy owes greatly to these collective efforts. Among them, the contributions of Dr. R. Tewari, in whose honour this volume is assembled, stand out for their depth, clarity and enduring influence. His work in phase transformations, microscopy and zirconium metallurgy has been instrumental in shaping up of some of the outstanding results presented here.



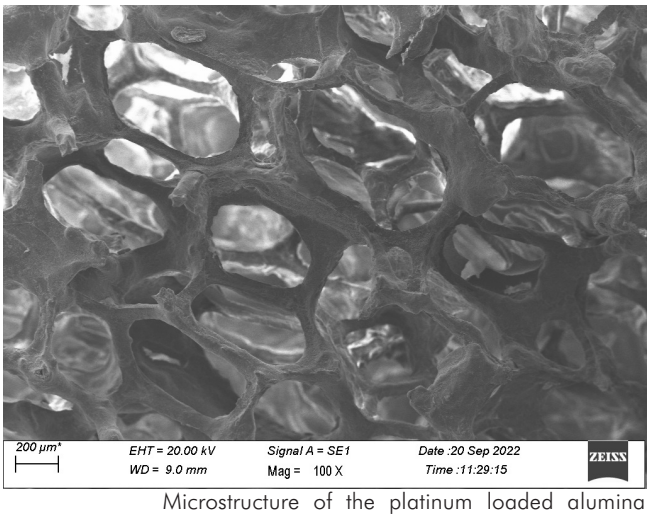
Materials Science
Research & Development

translating into new technologies benefiting Indian industry



Transfer of Technologies
Materials Group

The Bhabha Atomic Research Centre (BARC) is a premier multi-disciplinary nuclear research centre of India with expertise spanning the entire spectrum of nuclear science and engineering and related areas. Within BARC, the Materials Group undertakes fundamental and applied R&D to develop advanced materials, processes and technologies that underpin national programmes and industrial growth. Over the past few years, this group has translated laboratory innovations into transfer-ready technologies that help domestic industries produce strategic materials, advanced composites, ceramic products, catalysts and nano-enabled devices. The following sections present brief summaries of a few recent technology transfers from the Materials Group, offering a snapshot of the diverse solutions developed for societal needs and industrial benefits. Due to space limitations in this newsletter, only a select set of technologies is highlighted here. Readers may refer to the official BARC website for the complete list of technologies transferred or currently available for transfer.



Microstructure of the platinum loaded alumina ceramic foam.

1 Platinum-Loaded Alumina Ceramic Foam

Platinum-loaded alumina ceramic foam is an advanced catalyst support that combines the open-cell structure of α-alumina foam with high-surface-area γ-alumina and finely dispersed platinum nanoparticles. The foam is fabricated by a replica method in which a polyurethane sponge is coated with α-alumina slurry, squeezed to remove excess material, dried and sintered to produce a ceramic replica. Because α-alumina alone has insufficient surface area for catalysis, the macroporous support is subsequently coated with γ-alumina before being impregnated with a platinum salt and heat-treated to generate catalytic Pt particles. The resulting ceramic foam exhibits porosity of 80–90 %, density around 0.7–0.9 g cm^{-3} and compressive strength ≥ 2 MPa, with an effective surface area ≥ 35 $m^2 g^{-1}$.

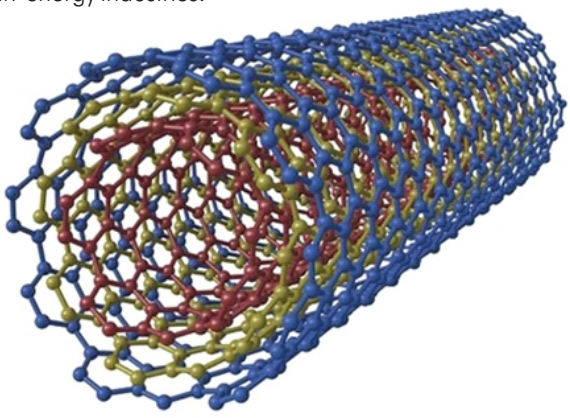
Microstructure of the platinum loaded alumina ceramic foam

These macroporous foams are lightweight, chemically stable and highly refractory, offering excellent heat and mass transfer properties. These are inexpensive compared with other ceramics and can withstand working temperatures up to about 500 °C. The high porosity and tortuosity make them suitable as catalyst supports for steam reforming, hydrogen-iodine decomposition and other heat- and mass-transfer-limited processes. Beyond catalysis, platinum-loaded alumina foams are used in molten metal and hot-gas filters, diesel exhaust treatment, biomaterials, thermal insulation and lightweight structural components in aerospace and high-temperature industries.

2 Large Scale Synthesis of Carbon Nanotubes

The large-scale synthesis technology for multi-walled carbon nanotubes (CNTs) produces high-purity (≈95 %) nanotubes with diameters of 20–50 nm and lengths of 5–10 µm. These CNTs exhibit exceptional tensile strength, high modulus, low density, and excellent thermal and electrical conductivity, making them a versatile nano-building block.

The material's high aspect ratio and porosity enable applications across diverse sectors: high-strength structural composites, electrodes for energy storage and conversion (supercapacitors, batteries), electromagnetic interference shielding, transparent conductive films, field emission displays and thermal interface materials. CNTs are also used as catalyst supports, membranes for water purification, adsorbents for gas storage, drug delivery vehicles and biosensors. By offering a reliable domestic process for bulk CNT production, this technology supports advanced manufacturing, electronics and clean-energy industries.

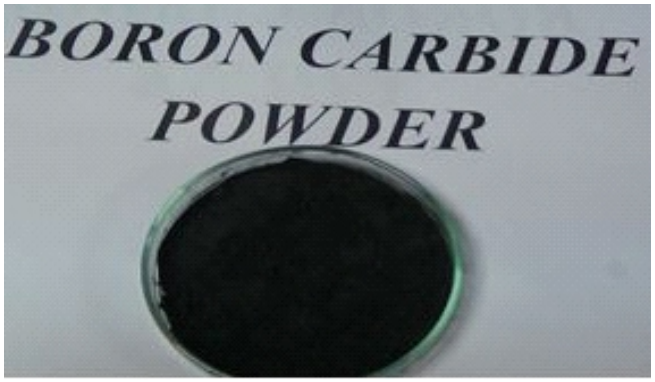


Schematic of molecular structure of CNT made at BARC.

3 Abrasive Grade Boron Carbide Powder

Boron carbide is the third hardest material after diamond and cubic boron nitride, and BARC's abrasive grade powder exploits this extreme hardness along with low density and high melting point. The powder, produced in multi-tonne quantities, is used as a high-performance abrasive for lapping and polishing gemstones, ceramics and metals, and for manufacturing sand-blasting nozzles and ceramic bearings.

Fully dense boron carbide shapes derived from the powder serve as lightweight armour plate and neutron-absorbing components in nuclear shielding. Because of its chemical stability and high elastic modulus, boron carbide is also incorporated into mortars, pestles, refractory linings and rocket propellants. The technology thus enables domestic supply of a strategic ceramic material used in defence, aerospace, nuclear power and precision finishing industries.



The boron coarbide synthesized at BARC

4 Titanium Diboride Powder and Dense Shapes

Titanium diboride (TiB_2) is a hard, refractory ceramic with a high melting point and excellent electrical and thermal conductivity. BARC synthesises high-purity TiB_2 powder by solid-state carbothermic reduction and converts it into near-theoretical-density shapes using hot pressing. The process uses inexpensive raw materials and yields >99 % pure powder and dense discs or pellets close to theoretical density.

Illistration of the TiB_2 powder and dense component made from it at BARC

Owing to its high hardness and chemical stability, TiB_2 is used in impact-resistant armour, cutting tool inserts, nozzles for molten metal handling, wear-resistant coatings and neutron-absorbing components. The availability of indigenous TiB_2 powder and densification technology supports defence, aerospace and metallurgical industries by reducing reliance on imported ultra-high-temperature ceramics and enabling new high-performance applications.

5 Zirconium Diboride Powder and High Density Shapes

Zirconium diboride (ZrB_2) is an ultra-high-temperature ceramic known for its high melting point, strong thermal and electrical conductivity, and chemical stability. BARC's process synthesises ZrB_2 powder (>99 % purity) through carbothermic reduction of zirconia with boron carbide and carbon, followed by milling and hot pressing to produce shapes approaching 99 % of theoretical density.

The material's properties—high temperature strength, low thermal expansion and resistance to extreme environments—make it attractive for thermal protection systems in hypersonic and re-entry vehicles, turbine shrouds, rocket motor components and electrodes. ZrB_2 also serves in wear-resistant coatings, molten metal crucibles and as a neutron absorber. Developing this technology domestically is strategically important for aerospace and defence sectors and supports industries requiring materials that can withstand very high temperatures.

6 Nano-Nickel Coating on Difficult-to-Plate Metals/Alloys

Metals containing aluminium, titanium, zirconium and niobium form passive oxide layers that hinder electroplating. BARC's nano-nickel coating technology removes the oxide layer in-situ using chemical/electrochemical conditioning and deposits a nano-structured nickel interlayer before conventional plating. The resulting nano-Ni layer exhibits microhardness in the range 700–900 HV and significantly improves corrosion, erosion and wear resistance.

This scalable process achieves uniform coverage on complex shapes and enhances adhesion of subsequent coatings. It finds applications in the automobile, aerospace and nuclear sectors and in metal finishing industries where protection of lightweight or reactive alloys is critical. By enabling reliable plating on otherwise difficult substrates, the technology contributes to longer component lifetimes and improved performance.

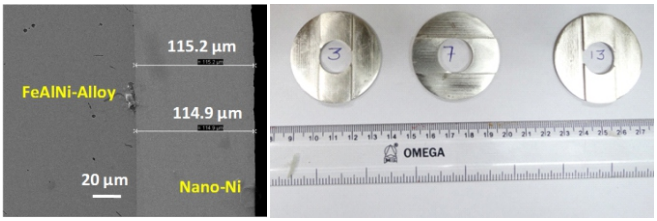


Illustration of the interface microstructure of Nano-Ni coating on a FeAlNi alloy and actual components coated with Nano-Ni using BARC technology.

7 Extracting High-Purity Copper Oxide Nanoparticles from E-Waste

BARC developed a hydrometallurgical process to extract copper from discarded printed circuit boards (PCBs) and convert it into high-purity copper oxide nanoparticles. The process leaches PCBs to produce copper-rich solutions, then uses novel copper-selective polymeric resins that remove tin and other impurities; after scrubbing and elution, the copper is precipitated and calcined to yield CuO nanoparticles. By-products such as tin oxide (SnO) and lead sulfide (PbS) are also recovered.

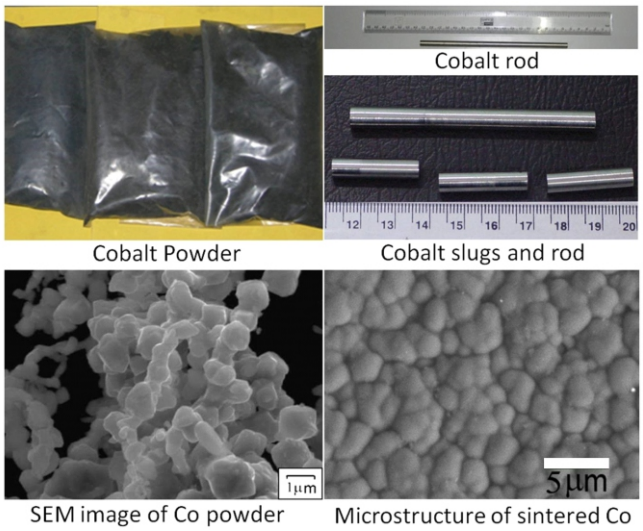
The technology produces >99.9 % pure CuO nanoparticles in a scalable, environmentally friendly manner and demonstrates a value-added pathway for recycling electronic waste. High-purity CuO nanoparticles find use in electronics, sensors, solar cells, catalysis, antimicrobial coatings and other nano-enabled applications, highlighting both industrial relevance and societal benefits of sustainable e-waste management.

Infographic demonstrating the conversion of e-Waste into wealth through production of high purity CuO nano particles.

8 Making Cobalt Metal Powder and Shapes

Cobalt is a strategic metal used in permanent magnets, superalloys, radiation sources and batteries. BARC's technology prepares cobalt metal powder by thermal decomposition of cobalt oxalate and shapes the powder through compaction and sintering. The process uses simple equipment and minimal manpower, making it suitable for small-scale manufacturing.

Fine cobalt powders and rod-shaped products produced via this route serve as feedstock for Sm–Co and AlNiCo magnets, carbide tool materials, high-temperature cobalt-based superalloys and cobalt-60 radiation sources. Domestic production of cobalt metal shapes supports strategic sectors such as energy, aerospace and medical technology.



Production of Co powder and shapes from it.

9 Making Tungsten Metal Powder and Heavy Alloys

Tungsten possesses the highest melting point among metals, high density and exceptional mechanical strength. BARC produces tungsten powder via hydrogen reduction of tungsten oxide and fabricates tungsten metal and heavy alloys (W–Ni–Fe, W–Ni–Cu) using hot pressing and liquid-phase sintering. Tungsten heavy alloys have superior radiation-shielding capability and mechanical robustness, while W–Cu composites offer good thermal and electrical conductivity.

Applications include cemented carbide tools and high-speed steels, plasma-facing components for fusion reactors (e.g., ITER divertor plates), W–Cu electrical contacts, heavy-alloy penetrators, collimators and medical radiation shields. Access to domestic tungsten powder and heavy alloy fabrication supports aerospace, defence, energy and medical engineering industries and provides materials for emerging programmes like BHABHATRON cancer therapy units.

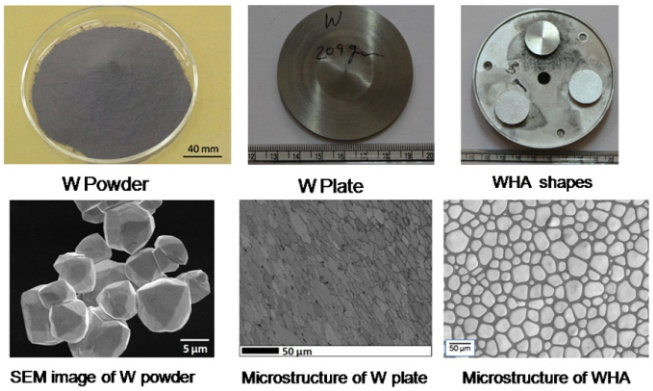
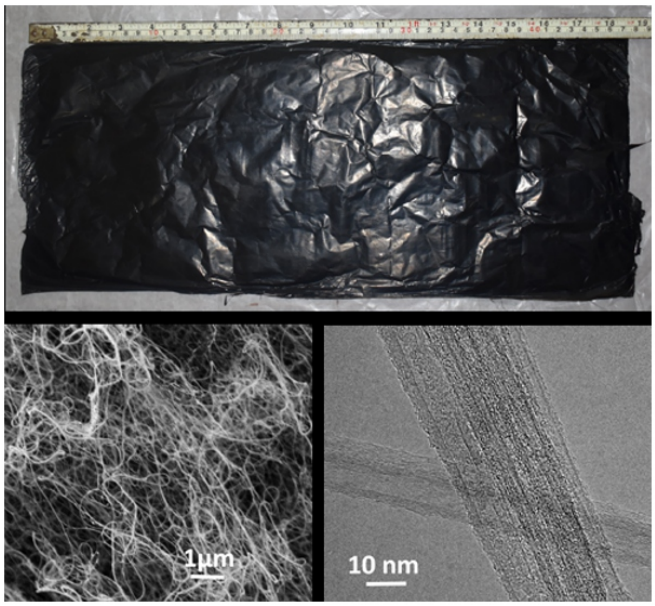


Illustration of tungsten powder and various shapes and their microstructures produced using BARC technology.

10 Making Freestanding Carbon Nanotube Sheet

Freestanding CNT sheets are produced via a floating catalyst chemical vapour deposition (FC-CVD) process where an aligned CNT aerogel is collected on a rotating drum and converted into a uniform sheet. The material consists of multi-walled CNTs with diameters of about 10–25 nm and purity >95 %. The sheets exhibit exceptional mechanical strength, low density and high electrical and thermal conductivity.

Because the sheets translate nanoscale properties into a macroscopic form, they find applications in high-strength composites, electromagnetic shielding, supercapacitors and battery electrodes, catalysts and drug delivery, energy storage and conversion devices, transparent conducting films, field emission displays and porous membranes. The scalable FC-CVD process therefore delivers a versatile nanomaterial platform for advanced energy, aerospace and biomedical technologies.



The freestanding CNT sheet produced using BARC technology and its microstructure.

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7 **Harnessing Lithium from India's Lepidolite Ores - BARC's Indigenous Process for a Clean Energy Future**

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Industry

BARC's Nuclear

By Technology Transfer & Collaboration Division and SIRD Newsletter Editorial Team

Technology Transfers

Twenty-two nuclear spin-off technologies have been transferred to 29 pan-India companies by BARC through inking of 37 technology transfer agreements by the Centre's Technology Transfer and Collaboration Division during the last quarter of the calendar year 2025. A brief update on these technologies spanning Metals and Metallurgical processes, Farming, Water Purification, Waste Management, Food Processing, Nutrition is presented below.

The technologies transferred to the industry include 10 kW, 15 kV Electron Beam Melting (EBM) Machine Technology; Air Plasma Incinerator; Hybrid Granular SBR For Wastewater treatment; Arsenic Removal from Drinking Water by physio-chemical process; Membrane Assisted Defluoridation Process for Safe Drinking Water; Fluoride Remediation from Water; Soil Organic Carbon Detection & Testing Kit (SOCDK); NISARGRUNA Biogas Plant for Processing Biodegradable Waste; rapid composting technology for decomposition of dry leaves, kitchen waste and temple waste; Superabsorbent BARC-Hydrogel; VOC-Free Radiation Indicator for Gamma Radiation Processing Plant for Medical Sterilization; Microfine Neem Biopesticide; Micropropogation of Banana; BLDC Motor Based 5HP Solar Water Pump to mention but a few. Detailed description alongside procedure for acquiring the licenses to BARC technologies being offered to the industry is listed on the dedicated webpage https://barc.gov.in/technologies/

New Technologies

BARC streamlines new technologies in the public domain for the benefit of the industry. During the review period, ten technologies with immediate applications in sectors including farming, advanced technology, metals and metallurgy, QA, health and nutrition were introduced.. These technologies alongside their Codenames is summarised here.

Compact Laser based OSL Reader (RT25RPAD). USB-powered TeleECG Machine (MD32ED). Portable Raman Spectroscopy System for Oral Cancer Detection (MD33DRHR). An indigenous, standalone solid-state RF power amplifier (SSRFPA) system (13.56 MHz, 200W) along with impedance matching network (AI41APCS). Technology for preparation of anti-oxidation coating on TZM alloy shapes by pack siliconizing (CH49MPD). Nutri- Shakti: Innovative Plant-Based Protein technology (AB64FTD). Trombay-Actino C3: Formulation for plant defence and growth in vegetable crops (AB65NABTD). Copper Electrode 100kW Hollow Cathode Thermal Air Plasma Torch (EG49L&PTD). Proportional counter for soft X ray/gamma ray energy Spectroscopy (RT26SSPD). Production of O-18 enriched water (H218O) of isotopic purity above 96% 18O using vacuum distillation of water.

Renewal of Technology Licenses

BARC shares the know-how of its technologies to the industry.

Inking of an MoU between AIC Anushakti and HBNI to formalize broader collaboration.

Licenses to these technologies are renewed continuously on the basis of demand and merit considerations. During the review period licenses Handheld 12 Channel Tele-ECG Instrument Technology renewed (1st Renewal) - DNA Microrrayer System - Production of $CaSO_4$: Dy embedded Teflon Discs and TLD Cards - Production Of Dysprosium Doped Calcium Sulphate Thermoluminescence Dosimetry Phosphor Powder were renewed.

Public Awareness in BARC Technologies

The Technology Transfer and Collaboration Division in coordination with various scientific R&D, engineering and technology development teams in BARC organises public awareness programs for the benefit of industry, young entrepreneurs and general public. At a related event held on 14th November 2025 at Training School guest house in Anushakti Nagar, Mumbai , around 40 industry delegates and mentors from various divisions of BARC participated. The event encompassed lectures on various BARC technologies, knowledge management programmes and incubation programmes, among others. A licensee feedback session was also organized. Members of BARC Trombay Council and TSC participated in the event.

AIC ANUSHAKTI Activities

The AIC BARC Anushakti Foundation, a deep-tech incubation centre, established here and supported by Atal Innovation Mission (AIM), continued to strengthen its initiatives toward fostering technology translation, innovation-driven entrepreneurship, and industry engagement. Some of the important activities it conducted during the review period included:

MoU with Homi Bhabha National Institute (HBNI) to formalize collaboration in areas such as Entrepreneurship development, Technology translation and Student training and innovation programs. The MoU was signed by Dr. A.K. Tyagi, Dean, HBNI, and Mr. Martin Mascarenhas, Director, Beam Technology Development Group, BARC & Chairman & Director Board of Directors, AIC Anushakti.

An outreach and awareness session at the Haffkine Institute, Mumbai, engaged postgraduate students, researchers, and faculty members

beckons

Spin-off Technologies

interested in scientific innovation and translational research. The session introduced participants to Deep-tech startup pathways, Opportunities for commercializing laboratory research and Support mechanisms available through AIC Anushakti.

The team, in collaboration with HBNI, delivered a dedicated session titled "Funding Pathways for Scientific Innovators & Entrepreneurs" for JRFs, research scholars and students. The session, led by Mr. Aadesh Suryarao, CEO of AIC Anushakti, provided an in-depth overview of Govt. of India-funded grant schemes (through DST, DBT, BIRAC, SERB, AIM).

Participated in the India Food Forum 2025, India Food Forum 2025, considered India's premier platform for food processing, retail and the FMCG ecosystem. The AIC Anushakti team leveraged the platform to interact with multiple stakeholders - MSMEs, technology providers, and retail chains to assess industry interest in BARC's irradiation-based food preservation technologies and value-added food products developed by BARC scientists, and opportunities for market deployment through startup-led interventions, among others.

At the Medicall 2025 exhibit at NESCO, Goregaon, Mumbai, during 12–14 December 2025, various medical technologies of BARC were demonstrated to the public. Technologies like DEAP: A Device to Ease Apnea Problem, DNA Microrrayer System, Extra-Cellular Acidity Analyzer (ECAA), Spot Picker Robot for Proteomics, Deep Brain Stimulator (DBS), Multi-contaminant Exposure Respiratory Cartridge (MERC) for Face-Mask/Respirator, Portable Raman Spectroscopy System for Oral Cancer Detection were demonstrated.

Outreach programmes covered students of St. Francis Institute of Technology, Thakur College of Engineering, and Thadomal Shahani Engineering College at their respective campuses.

AKRUTI

AKRUTI Kendras engage in display, dissemination, demonstration and training on rural-oriented technologies with a focus on promoting rural entrepreneurship and sustainable development through BARC technologies. In October, an agreement was signed with Gangadhar Meher University, Sambalpur, Odisha to establish India's 12th AKRUTI Kendra.

Inking of an agreement with Gangadhar Meher University at Sambalpur, Odisha for the establishment of a new AKRUTI Kendra. October 13, 2025.

AIC Anushakti conducted an outreach and awareness session at the Haffkine Institute campus in Mumbai.

Notable Developments at AKRUTI

AKRUTI Kendra Tarapur launched a sales outlet at Boisar to market food products made using BARC technologies; organised a technology exhibition for AECS-2 school students at BARC Colony, Tarapur; and conducted hands-on training, demonstrations and field visits on low-cost societal technologies for Jai Hind College (TYBSc Physics Biotechnology) with hands-on, demo and field visits on low-cost societal technologies.

Additionally, familiarisation visits were hosted for MG University, Kottayam (14 October); Karmayogi Institute of Technology (16 October); Jai Hind College AKRUTI Kendra, Mumbai; and Dandekar College, Palghar (7 November).

The AKRUTI Kendra – MGU, Kottayam, conducted a development programme on food and agriculture, with demonstrations by the AKRUTI group leader and the BARC Division (DMTD) during 10–14 September; participants included entrepreneurs, SHGs, rural representatives and members.

AKRUTI Kendra – Jai Hind College (JHC), Mumbai, organised an educational visit (six faculty, seventeen BSc and two PhD students) on 17 October. The visit aimed to provide academic exposure to technology implementation.

Five faculty members from AKRUTI Kendra–GITAM attended familiarisation and training on up to 20 BARC technologies at BARC Tarapur to study their functioning and impact on development.

Tech Transfers from AKRUTI

Two technology transfer agreements via AKRUTI were signed: one with M/s. Pure N Care Agro Pvt. Ltd., Palghar, for a self-stable, preservative-free natural multigrain premix, and the second with another company for a similar product.

Inaugural Day event of the Supervisory Training Programme on Backend of Fuel Cycle.

Capacity Building for Backend of Fuel Cycle

Nuclear Recycle Group conducts Supervisory Training Programme

Nuclear Recycle Group, BARC organized a four-day 'Supervisory Training Programme on Backend of Fuel Cycle' which commenced on September 10th at SCF Auditorium in Trombay. The programme was designed to equip the nuclear energy workforce with requisite skills to oversee operations associated with spent fuel reprocessing and radioactive waste management.

The programme was inaugurated by Clement C. Verghese, Chairman, BARC Safety Council. He emphasised that safe, secure, and efficient management of spent nuclear fuel and radioactive wastes is integral to backend of fuel cycle. In the welcome address, Neelima Singh Tomar, Programme Convenor and Head of PMT&SS (NRG) presented a brief outline of the training programme. Dr. G. Sugilal, Associate Director, NRG delivered the keynote address and Probal Chaudhury, Associate Director, HS&EG addressed the participants during the inaugural function.

The training programme brought together 80 participants and covered best practices, lessons learned from decades of operational experience, and the evolving landscape of science and technology in the backend of the fuel cycle. As a result, participants gained a deeper understanding of the complexities of spent fuel and waste management, enhanced their ability to supervise diverse teams, and reinforced their commitment to nuclear safety and environmental protection.

The valedictory function (on Sept. 13th) was graced by P.D. Maniyar, Head, NRG Projects and Y.C. Shivakumar, Head, TDD as Chief Guest and Guest of Honour, respectively. Meanwhile, Dr. G. Sugilal, Associate Director, NRG encouraged participants to share honest feedback on the training, highlighting that their inputs would help organizers further improve the programme.

Valedictory function.

Participants and Organizers pose for a Group photograph.

The conference Abstract book and Souvenir inaugurated by dignitaries from IIT Roorkee and BARC, Mumbai. Left to right: Prof. R. Chandra, Dr. D. Bhattacharya, Prof. V.K. Malik, Dr. D.V. Udupa, Prof. K.K. Pant, Dr. S.L. Chaplot, Dr. G.D. Varma, Dr. V.K. Aswal, Dr. Himal Bhatt, Dr. N.S. Ramgir

69th Solid State Physics Symposium

The annual edition of Department of Atomic Energy's Solid State Physics Symposium (SSPS) was held at the Indian Institute of Technology (Roorkee, Uttarakhand) during December 19-23, 2025. The symposium saw attendance from nearly 800 participants from various parts of the country and few foreign delegates. The symposium comprised 4 plenary lectures, 35 invited talks, 3 evening talks, 6 best PhD thesis talks and 6 Young Achiever talks in addition to 680 poster presentations and 16 oral presentations by students. Director, IIT Roorkee, Prof. K.K. Pant, inaugurated the symposium, highlighting the importance of synergy and strong partnerships between premier science & engineering institutions and national laboratories for sustainable growth and development towards Viksit Bharat (Developed India) goal. The keynote lecture was by delivered by Dr. S.L. Chaplot, former Director of Physics Group, BARC, who emphasized upon the need of basic research to achieve innovative and challenging targets for applications and the history of this legacy national symposium which started in 1956 after the commissioning of first nuclear reactor in Asia, namely Apsara, which provided neutrons for condensed matter research. Among those who were present at the symposium, Dr. Sakura Pascarelli, from the European XFEL, spoke on the simultaneous evolution of another Mega Facility for solid state physics research, the synchrotron radiation sources, which was complemented with talks by Prof. Olof Karis from MAX-IV another European Centre and Prof. S. Rai on Indian 3rd generation synchrotron facility being set up by DAE in India.


Group photograph of participants to DAE-SSPS 2025 taken after conclusion of inaugural day program.

हिंदी दिवस 2025

@भाभा परमाणु अनुसंधान केंद्र

Hindi Day 2025 at BARC was celebrated with grandeur and substance through a month-long affair starting September 14. These events promoted awareness, growth, and propagation of Hindi alongside other Indian languages. Exclusive competitions, talks, presentations, workshops, and a book exhibition highlighted the vital role of languages. Pictures showcasing various activities from the celebrations are featured here.

Celebrating Dr. Homi Bhabha's Enduring Legacy

Clockwise from top: Mr. Manoj Singh, Head of SIRD, and Dr. Adarsh Kumar Dureja, Associate Director of KMG, welcome Chief Guest Mr. K.T. Shenoy, Director of Chemical Engineering Group, on the exhibition's inaugural day. Mr. Manoj Singh guides attendees with a brief presentation. Attendees explore the Dr. Bhabha collection on display. Group photograph of exhibition attendees.

The Scientific Information Resource Division (SIRD) of the Knowledge Management Group (KMG) at BARC organized a week-long exhibition at the Central Library coinciding with the 116th Birth Anniversary of Dr. Homi J. Bhabha. It showcased the professional career, achievements, and pivotal moments in India's science and technology landscape during the period of Dr. Bhabha's active life.

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