

Chapter 12

Contribution of Bhabha Atomic Research Centre to the “Metals Journey of India”



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Abstract Bhabha Atomic Research Centre (BARC) was established by Dr. Homi Jehangir Bhabha in January 1954 as Atomic Energy Establishment, Trombay (AEET) for pursuing multidisciplinary research activities to support the nuclear power programme of India. Since its inception, BARC is deeply involved in the adoption, ingestion and development of new technologies in various fields—be it electronics, computations, nuclear, radiation, chemical, biological/health/agriculture and materials. Among these fields, developments of materials have taken a centre stage right from the inception of BARC. Materials and processes required for every stage, from ore processing to extraction, alloy making, fabrication, support during operation, degradation modes to spent fuel reprocessing and waste management have been indigenously developed and successfully implemented with complete understanding. BARC is providing materials related support not only for Indian nuclear power reactors but also for heavy water plants, Indian Rare Earths Limited (IREL), Uranium Corporation of India Ltd (UCIL) and various other plants of Department of Atomic Energy (DAE).

12.1 Introduction

Genesis of the materials development programme in BARC got initiated by a request from Dr. Homi Bhabha to Prof. Brahm Prakash, then a faculty at IISc Bangalore, to take up the challenge of establishing a facility for fabrication of fuel assemblies at the BARC campus. Prof. Brahm Prakash and his team at BARC met the challenge of fabrication of fuel assemblies in a short time. One fundamental aspect in meeting this challenge pertained to beta quenching of the uranium rod. Uranium rods on rolling acquire texture but for better irradiation response, it is necessary to have a random texture, which is obtained by quenching the rolled rod from the high temperature beta phase to room temperature. For this purpose, a unique in-house induction heating

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facility was developed. Two fuel elements that were sent to Canada for qualification when subjected to irradiation at NRX reactor withstood radiation levels of 2000 MWD/t as compared to the normal level of about 1000 MWD/t. This surprised everyone who was probably not expecting a better performance from the Indian fuel elements. Project Faggots now known as Atomic Fuels Division (AFD) delivered the initial half charge as well as all the subsequent periodic replacement fuel elements for CIRUS research reactor until its recent decommissioning. This experience reinforced the drive in Prof. Brahm Prakash to set up facilities at BARC to study and understand the science required to develop components for plant applications. BARC follows this legacy and works to fully understand the science as well as develop a fundamental understanding and uses it to develop materials and processes to support the robust nuclear programme of the country.

BARC, right from its inception, build the concept of 'Atmanirbhar Bharat' in its DNA. Although Dr. Homi Bhabha knew at that time that Indian uranium ores are too lean to give a commercially viable product, yet, with the idea of India to become a sovereign nuclear state, he went ahead with the exploration and extraction of uranium from Indian mines with U_3O_8 content as low as 0.04%. The same concept was applied in the case of extraction of zirconium from zircon sand. These decisions paid off in later years when sanctions were imposed on India, but all the Indian PHWRs continued functioning with fuel made in the country. Since then the spectrum of materials activities in BARC have widened their scope and several entities flourished (e.g. Uranium Corporation of India Ltd. (UCIL), Indian Rare Earths Ltd. (IREL), Nuclear Fuel Complex (NFC) and Heavy Water Board (HWB)) using BARC developed technologies for targeted production. From the lightest metallic element beryllium (Be) to the heaviest naturally found uranium (U), nearly all the metallic elements of interest to Department of Atomic Energy (DAE) were successfully extracted and processes were developed for utilisation in BARC.

As the primary resources/ores of most of the elements present in India are very lean, the challenges to selectively take out the metal values associated with the unwanted gangue led to the development of unique extraction processes for each metal, involving various steps, like innovative physical ore beneficiation, selective chemical ore-breakdown techniques, ion-exchange, solvent extraction, halogenation, metallothermic reduction, hydrogen reduction, reduction-diffusion, molten salt electrolysis etc. Expertise was developed on related technologies for melting-consolidation and vacuum refining using arc and electron beam melting technologies, ultra-purification using thermal decomposition, iodide refining and electron beam zone refining etc. Along with these, the fabrication technologies e.g. powder metallurgy, rolling, extrusion, pilgering, welding, coating, and ceramic metallurgy were pursued vigorously and strong groups were developed that contributed significantly. The development of fundamental understanding of materials, microstructural and mechanical properties characterisation at different length scales, structure-property correlations and degradation mechanisms specifically corrosion has been a strong point of materials activities in BARC. The fundamental understanding thus developed helped to address materials related issues in plants and gave impetus to develop newer applications with a strong basis.

Described below are some salient achievements in the journey of materials activities in BARC.

12.2 Technologies Developed at BARC

The technologies developed for important materials at BARC which were later transferred to produce at industrial scale are described below:

12.2.1 *Zirconium, Hafnium and Titanium, and Their Alloys and Applications*

The principal source of zirconium is zircon (zirconium silicate ($ZrSiO_4$)) which is found in the coastal beach sand in India. This sand also contains many other valuable minerals because of which a special physical beneficiation technology for the separation of the individual minerals was first established at BARC, Trombay at a pilot plant scale and subsequently, a plant was established in Tamil Nadu by Indian Rare Earths Limited (IREL) for the processing of Manavalakurichi beach sand. The zircon contains about 2.5% Hf, an element chemically similar to Zr and it was successfully separated at BARC (Prakash and Sundaram 1955) using vapour phase dechlorination technique. A process for separation of Zr and Hf was developed by chlorination of Zircon, purification to obtain $ZrCl_4$ and $HfCl_4$ mixed salt, selective dechlorination of $ZrCl_4$ from the vapour phase. This led to the formation of pure ZrO_2 in the residue by adjusting the ratio of the flowing gas mixture composed of chlorine and oxygen at high temperatures leaving the volatiles enriched with $HfCl_4$. Solvent extraction process was developed later for a more effective and larger scale separation of the two elements. Chlorination of ZrO_2 was further optimised using static-bed reactor technology. Magnesio-thermic reduction of $ZrCl_4$ producing pure zirconium (Kroll process), was used for making zirconium sponge at BARC, Trombay and a schematic of the initially designed assembly having different heating zones used for carrying out Kroll process is shown in Fig. 12.1a (Subramanyam and Sundaram 1966). Iodide refining technology (Van Arkel-De Boer Process) was developed and demonstrated at BARC for making ultra pure zirconium from impure zirconium and zirconium alloy scraps. Figure 12.1b shows the outlook of the zirconium crystal grown by iodide refining process, and the inset of the figure represents the overall view of the crystal bar zirconium formed on U-shaped zirconium filament. The zirconium produced by Kroll process was subsequently vacuum arc melted to produce various zirconium alloys, such as Zircaloy 2, Zircaloy 4, Zr-2.5Nb, Zr-2.5Nb-0.5Cu, for structural components in nuclear reactors. A dedicated plant was further established in 1971 for the production of Zr and its alloys, and for the fabrication of different reactor components at NFC, Hyderabad.

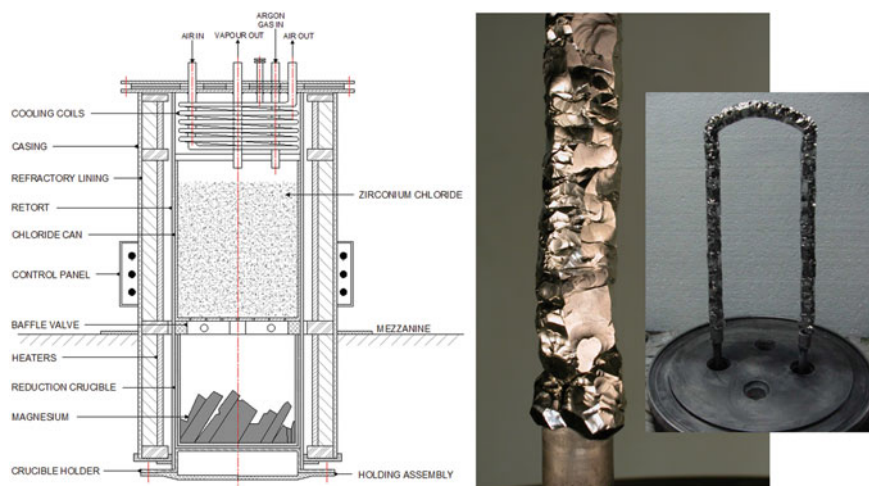


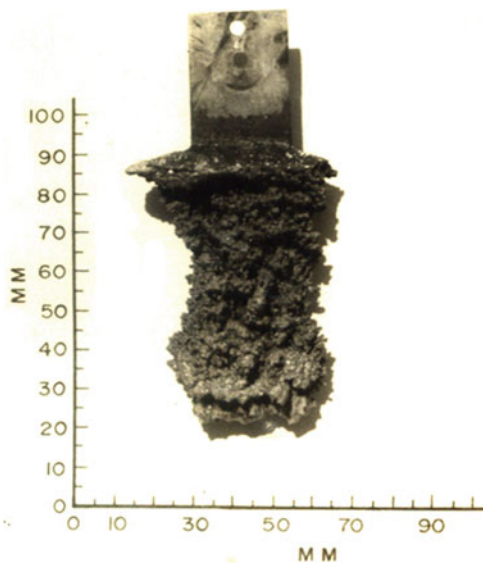
Fig. 12.1 **a** Schematic of the Kroll reduction assembly for zirconium sponge production designed at Bhabha Atomic Research Centre, Trombay, **b** Zirconium crystal bar grown by iodide refining technique at BARC campus and the inset shows the crystal bar zirconium formed on U-shaped zirconium filament

India adopted a different route involving extrusion and pilgering based forming processes for Zr alloys to produce various tubular products required for various structural applications in Indian Pressurised Heavy Water Reactors (IPHWR). For this purpose, detailed deformation processing maps were developed. Detailed studies on the evolution of phases, texture, microstructure, irradiation effects and corrosion behaviour were carried out. Various processing routes were tried and eventually, an optimised route giving products with uniform and desirable properties has been adopted. The basic research carried out in BARC solved many production issues faced by NFC when the expected strength of zircaloy tubes was not achieved initially. Subsequently, a major role in establishing the flow sheet for the production of Zr–Nb tubes was successfully executed with the help from BARC material scientists.

The major drawbacks of Zr based alloys are nodular corrosion and hydride induced embrittlement which have been studied under various conditions. Reactor conditions were simulated and the basics of nodular corrosion and hydride embrittlement have been studied. The oxidation behaviour, corrosion resistance and irradiation response of Zr alloys under various conditions have been studied. BARC provided support in monitoring the healthiness of the materials in service. From hot conditioning during the start-up of a reactor to establishing root cause analysis for components that showed degradation during its service are a few of the services rendered by BARC engineers and scientists.

Hafnium metal was mainly prepared by molten salt electrolysis at 800–950 °C using NaCl–KCl–HfCl₄ salt. Figure 12.2 shows the as-deposited hafnium flakes on molybdenum cathode plate. Iodide refining process was used for making crystal bar hafnium. The technology for the production of titanium metal was developed

Fig. 12.2 Hafnium metal produced by molten salt electrolysis



and demonstrated (Sridhar Rao et al. 1969) using Kroll reduction and pyro-vacuum distillation processes as used for Zr. TiCl_4 being liquid is fed from a separate chamber, and the MgCl_2 formed after magnesio-thermic reduction is progressively removed from the reduction crucible. This technology was demonstrated in pilot plant scale at NFC, Hyderabad, and subsequently, the technical know-how was given to Defence Metallurgical Research Laboratory (DMRL), Hyderabad.

12.2.2 Materials for Fuel: Uranium, Plutonium and Thorium

The detailed flow sheet for the preparation of metallic uranium starting from the lean grade ores was developed at BARC, Trombay. Depending upon the chemistry, the ore was leached with acid or alkali followed by ion-exchange and solvent extraction to separate impurities and to produce uranyl nitrate solution. For the uranium ore at Thummapalle, BARC developed an alkali process and it has been used for the first time in the country. The ammonium diuranate precipitated out from the uranyl nitrate solution was calcined to produce UO_3 . UO_3 was further converted to UO_2 by hydrogen reduction followed by hydrofluorination to form UF_4 . Metallothermic reduction of UF_4 using calcium or magnesium has been successfully demonstrated to prepare uranium metal. Figure 12.3 shows the reactor used for metallothermic reduction of uranium metal and an example of the uranium ingot produced at BARC.

Plutonium is a man-made element produced during nuclear reaction between neutron and uranium. The detailed flow sheet was developed for reprocessing of

Fig. 12.3 Reactor used for metallothermic reduction of UF₄ for making uranium metal (inset: U metal ingot)



nuclear waste for separation and extraction of plutonium metal. The solvent extraction process was developed to separate plutonium from other radio-nuclides in the form of pure plutonium nitrate which was converted to plutonium oxide or peroxide. Halogenation studies for preparing plutonium fluoride and plutonium trichloride were conducted. Metallic plutonium was produced by calico-thermic reduction of plutonium halides and oxide and subsequently by vacuum induction melt consolidation (Roy and Mahajan 1975). Normally, the metal is produced in small quantities (known as ‘micro-metallurgy’ for microgram level production) to avoid the uncontrolled nuclear chain reaction inside the plutonium matrix.

Thorium metal extraction technology was developed starting from the Monazite ore processed through several stages of solvent extraction, precipitation and calcination to pure thorium oxide. The process of metallothermic reduction of thorium oxide by calcium producing thorium metal powder was standardised at a pilot plant scale.

Development of metallic or ceramic based fuels for nuclear reactors was a challenge as strict restrictions and imposed embargoes did not allow sharing of any information. Without any support, entire laboratories, materials and technologies were developed from scratch at BARC. Being radioactive materials, the fuels for nuclear reactors needed additional precautions and remote handling technologies. In spite of such restrictions, optimising sintering parameters for the (U, Pu)-oxide fuels, development of (U, Pu)-carbide fuels and other fuels were successfully carried out. In this connection, development of carbide fuel needs special mention as the composition of carbon in the materials has to be very strictly monitored. Excess carbon would carburize the wrapping structural material, whereas hypo-stoichiometric carbon leads to the formation of low melting eutectic of U-Fe-Pu. Such a tight control over carbon

chemistry has been mastered at BARC and is not an easy task to achieve in commercially produced products. The fuel and clad developed with BARC technologies are produced today to provide the required fuel assemblies for all the Indian nuclear power and research reactors.

12.2.3 Refractory Metals: Niobium, Tantalum, Vanadium, Molybdenum and Tungsten

The technology for aluminothermic reduction of the respective oxides (Nb_2O_5 , Ta_2O_5) to produce massive forms of niobium and tantalum metals was developed at BARC. The metal thus produced by the thermit reaction is further refined and consolidated using electron beam melting in which purification is done by vacuum degassing, carbon de-oxidation and sacrificial de-oxidation mechanisms (Gupta and Jena 1968). Technology for the production of capacitor grade tantalum powder by reducing K_2TaF_7 with sodium was established (Jain et al. 1969). All these BARC developed technologies were transferred to NFC, Hyderabad for plant scale production. Aluminothermic reduction reactor was specially designed for making thermit vanadium with low amount of nitrogen, and subsequent electron beam melt refining. These metals were also produced by molten salt electro-extraction using metal carbide as anode feed. The process flow sheet has also been established on laboratory scale at BARC to recover molybdenum from low grade indigenous sources and various secondary sources. The molten salt electro-metallurgical processes for producing pure molybdenum from its carbides and sulphides are also reported earlier (Suri et al. 1974; Mukherjee and Gupta 1974). The technology for the production of molybdenum metal powder by hydrogen reduction of molybdenum trioxide has also been demonstrated (Majumdar et al. 2008a). The flow sheet for low grade wolframite concentrate was developed with the objective of recovery of tungsten and other valuable associates. Process know-how was established for producing tungsten metal powder by hydrogen reduction of WO_3 (Majumdar et al. 2016).

12.2.4 Cobalt

There are no primary resources of cobalt in India. The secondary resources were explored to recover cobalt. BARC has established a flow sheet for producing cobalt oxalate intermediate starting from spent ammonia cracker catalyst (SACC) by hydro-metallurgical route followed by thermal decomposition of cobalt oxalate to produce cobalt metal powder (Majumdar et al. 2008b) in kg scale batches. The technology for recovering cobalt from the alloy scraps containing cobalt has been successfully developed and demonstrated recently at BARC. Further, the conversion of cobalt powder into required shapes of cobalt slugs and cobalt pellets, used for cancer therapy

Fig. 12.4 Neodymium metal ingot produced by calcio-thermic reduction of its fluoride (NdF_3)



and other applications, was successfully demonstrated through powder metallurgical route by optimising various process parameters.

12.2.5 Rare Earth Metals

Technologies for the separation and purification of rare earth (RE) oxides (Sm, Ce, La, Pr, Nd etc.) from monazite were developed at BARC and transferred to IREL which is currently producing and supplying various rare earth oxides and compounds. Presently, the technologies are being developed for the separation of rare earths from xenotime, from newly discovered ore deposits and also from various secondary resources. The technologies for the preparation of some of the rare earth metals such as Ce, La, Nd (Fig. 12.4) using molten salt electrolysis and metallothermic reduction of RE-fluoride or oxide and their alloys e.g. SmCo_5 using reduction-diffusion technique have been developed. In order to make the country self-reliant in the production of high purity heavy rare earth oxides, rare earth metals, alloys and phosphors, an LoU has been signed between BARC and IREL to develop and transfer the technologies for demonstration at pilot plant scale at the upcoming IREL Theme Park, Bhopal. A plant to produce SmCo_5 and $\text{Sm}_2\text{Co}_{17}$ permanent magnets is under construction at Vizag campus of BARC.

12.2.6 Alkali Metals: Lithium, Magnesium and Calcium

Process flow sheets were developed for the preparation of various lithium metal compounds such as carbonate, hydroxide and chloride, from the lithium bearing ore Spodumene, Lepidolite etc. Technology for the preparation of lithium metal from lithium chloride using fused salt electrolysis process was established. The same technique was also employed to produce magnesium from MgCl_2 , a by-product produced

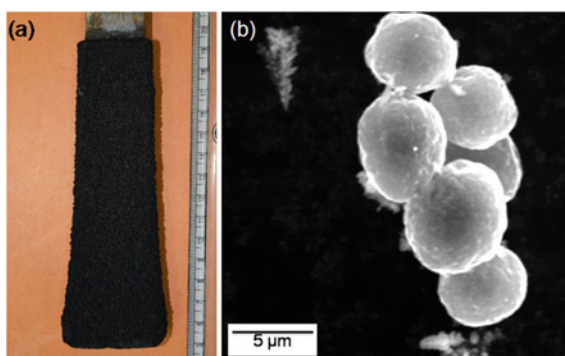
in the Kroll process. The know-how for magnesium production was provided to DMRL. The Central Electrochemical Research Institute, Karaikudi, developed electrolytic processes for the extraction of crude magnesium and crude calcium. The pyro-vacuum distillation of electrolytic crude calcium was done at BARC to produce pure calcium metal.

12.2.7 Light Elements: Beryllium and Boron

Technologies to extract beryllium from the Indian Beryl ore to produce the intermediates such as oxide and fluoride were developed. Magnesium-thermic reduction technology was established for producing beryllium metal from its fluoride, and also for making Cu-Be alloys. A plant was set up at Navi Mumbai for the production and fabrication of Cu-Be alloy, beryllium metal and BeO for meeting the requirements of space and nuclear industries.

The technology for the preparation of elemental boron from potassium fluoborate (KBF_4) by molten salt electrolysis using KCl-KF-KBF_4 electrolyte was developed and transferred to Indira Gandhi Centre for Atomic Research. The fused salt electro-winning technology is currently being used at Heavy Water Plant, Manuguru for the production of elemental enriched boron. Molten salt electro-extraction technology (Majumdar 2020) using boron carbide as soluble anode was also established for making elemental boron and is shown in Fig. 12.5a, b presenting the particle size and morphology of the electro-extracted boron produced from B4C scraps. This technology was transferred to Ordinance Factory at Khamaria. Figure 12.6 shows a representative image of the molten salt electro-extraction cell designed and fabricated at BARC for making molybdenum, tantalum, vanadium and boron using their carbides as anode feed.

Fig. 12.5 **a** Outlook of the electro-extracted boron deposited on cathode plate and **b** Scanning electron microscopic image showing the morphology of individual boron particles



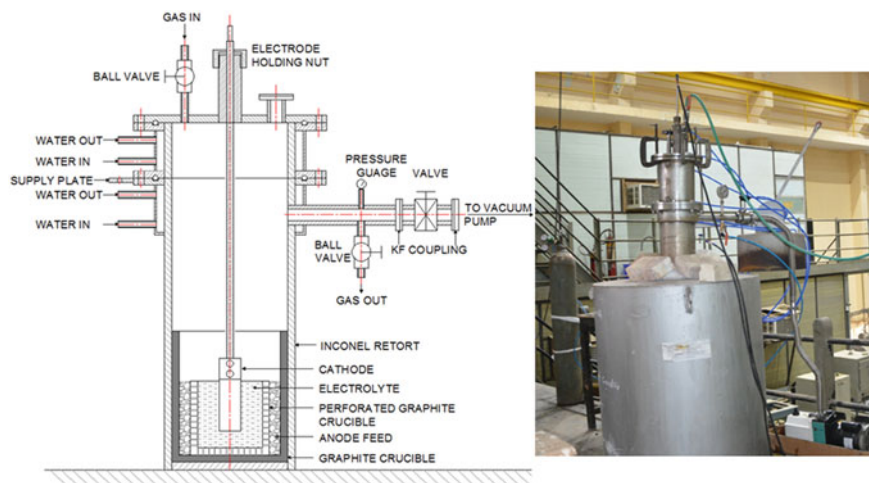


Fig. 12.6 Schematic of the molten salt electro-extraction cell and the set up developed at BARC

Technologies for the synthesis of borides of various reactive and rare metals such as ZrB_2 , TiB_2 , EuB_6 , NdB_6 etc. using carbo-thermic and boro-carbo-thermic reduction at high temperature and high vacuum were developed at BARC. These borides are produced in larger quantities and used for departmental (DAE) applications.

12.2.8 Ferroalloys

Aluminothermic co-reduction technology for the preparation of low carbon ferroalloys such as ferrochromium (Majumdar et al. 2020), ferroniobium, ferromolybdenum, ferrovandium, ferroboration was developed and transferred to private industries.

12.3 Alloy Development Programme: Evaluation of Physical, Mechanical and Corrosion Properties

In addition to various technological developments, BARC has paid special attention to fundamental scientific research work on structure-processing route-property correlations. Considerable amount of work has been done in understanding phase transformations occurring in various alloy systems (Dey et al. 2004; Tewari et al. 2008, 1999), deciphering the underlying mechanisms of degradation of materials and thermodynamic studies of phase and phase formation reactions. These studies paved

the way for better understanding of the performance of materials under various conditions. Detailed diffusion studies on various systems not only generated enormous data on diffusion of species which helped in correcting few of the phase diagrams, but also helped in evolving a new diffusion bonding technique for joining dissimilar materials. Detailed crystallographic work led to the understanding of texture evolution in U, Zr and their alloys, understanding the martensitic phase transformations in Zr (Banerjee and Krishnan 1971), Ni and Fe based alloys which successfully led to tailor the microstructures for specific applications. Among various alloy development work, development of shape memory Ni–Ti alloys (Madangopal et al. 1994) needs special mention, as the coupling produced using this alloy became an integral part of ‘Light combat aircraft’.

The physical metallurgy of superalloys and evolution of various phases in this alloy system solved many issues related to ammonia cracker tubes in the production of heavy water. The study of formation of precipitates in various generations of superalloys addressed very long term issues in these alloys. The detailed characterisation studies of various alloys with respect to phase transformation and mechanical properties (Chakravarty et al. 1995) including tensile, fracture, fatigue, creep, embrittlement effects due to hydrogen pick up (Singh et al. 2004), and material degradation behaviour in different corrosive and oxidative environments paved the way for the most optimum use of various alloys in operating plants. Understanding the structure-processing path property including corrosion behaviour remained always an integral part to develop fabrication process flow sheets for many alloys. The materials science activities were extended to understand the physical, mechanical and corrosion properties of nickel based alloys (Inconel 690, Alloy 718, SuperNi etc.), D9 and RPV steels, refractory metal based alloys (Nb-1Zr-0.1C, Mo-0.5Ti-0.1Zr-0.02C, Mo-Ti-Si etc. (Majumdar et al. 2021; Majumdar et al. 2018)) etc. Corrosion behaviour of stainless steels, Zr based, and Ni based alloys has been established at BARC and used to guide the successful deployment of these alloys to ensure safety and plant availability in nuclear power and research plants as well as other plants of DAE. The key strength of BARC is in the fields of stress corrosion cracking (Kain et al. 2002), flow accelerated corrosion (Kain et al. 2008) and various localised forms of corrosion and the developed knowledge and expertise is used to provide metallurgical and corrosion related support to operating plants. Environmental barrier coatings were developed by treating the metal or alloy surfaces using different chemical/physical vapour deposition processes. The powder metallurgical processes are developed to synthesise and consolidate the ceramic materials for instance boron carbide, oxides, carbides and borides of reactive and rare metals.

12.4 Future Perspectives

BARC is currently extending its research and development activities on developing various processes for the preparation and fabrication of metals and alloys. The Centre is extending its support for plant scale production of different rare earths

and rare metals technologies for being taken up by public sector companies and private entrepreneurs beyond the family of Atomic Energy. New alloys and coatings are being developed for high temperature and advanced technological applications. Needs for materials in new fields including composites, meta-materials, bio-materials and electronic materials are being addressed for sensor, functional and structural applications. BARC is poised to take up research on newer projects of DAE e.g. molten salt reactors, pressurised water reactors and also on advanced fabrication processes e.g. 3-D printing.

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