

# पदार्थ प्रक्रमण में नवाचारों हेतु अभिलक्षणन Characterization for Innovations in Materials Processing

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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.  $TiB_2$ ,  $ZrB_2$ ,  $HfB_2$ ,  $EuB_6$ ,  $LaB_6$ ,  $Gd_2B_4$ . 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-precipitation 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  $\mu m$  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.

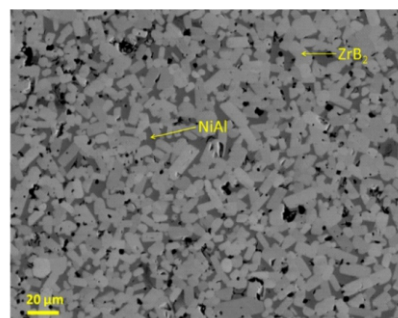


Fig. 2: Microstructure of  $ZrB_2$ -NiAl composite.

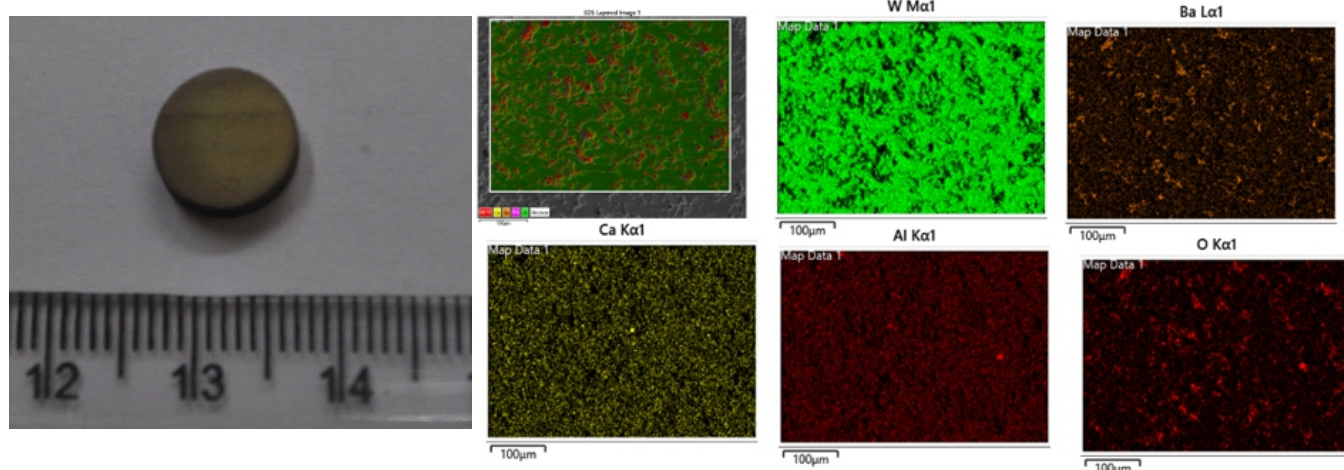


Fig.3: W-BCA composite and corresponding EDS-Mapping.

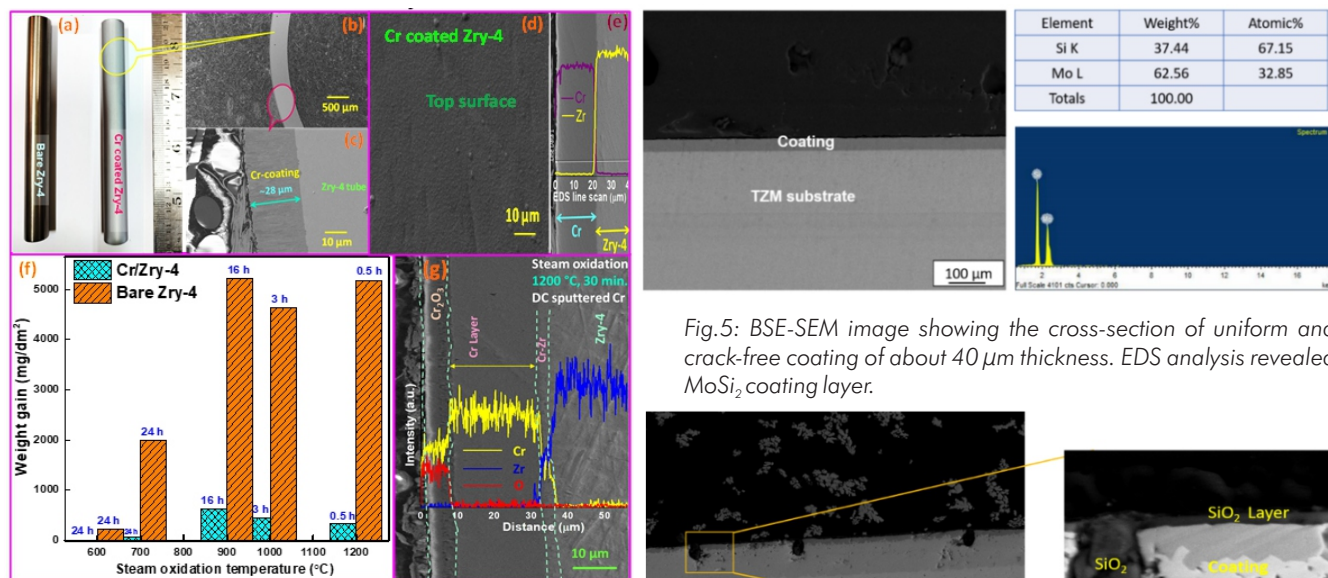


Fig.4: (a) Photographic images of bare and Cr coated Zry-4 fuel tubes, (b-c) X-section FESEM images of Cr coated tube; (d-e) FESEM surface topography and X-section image with EDS line scan of Cr coated Zry-4 coupon; (f) Wt. gain vs. steam oxidation temperature and duration; (g) FESEM X-section image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

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

Molybdenum metal based alloy TBM (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 TBM alloy forms volatile oxide vapor  $\text{MoO}_3$  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

Fig.5: BSE-SEM image showing the cross-section of uniform and crack-free coating of about 40  $\mu\text{m}$  thickness. EDS analysis revealed  $\text{MoSi}_2$  coating layer.

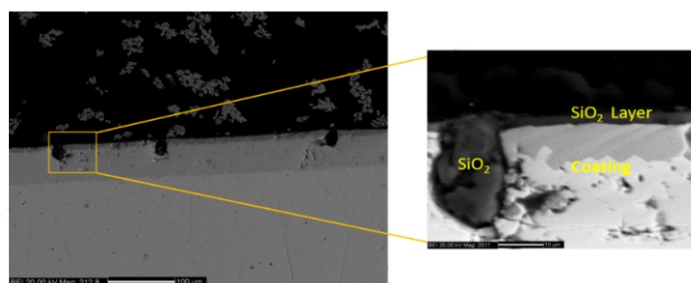


Fig.6: BBSE-SEM image showing the cross-section of oxidized sample. EDS analysis revealed protective  $\text{SiO}_2$  layer formed during the oxidation treatment.

temperature oxidation resistance of these molybdenum-based alloys.  $\text{MoSi}_2$ -based anti-oxidation coating on TBM 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 TBM alloy showing the cross-section of uniform and crack-free  $\text{MoSi}_2$  coating of about 40  $\mu\text{m}$  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  $\text{SiO}_2$  layer (Fig.6). The coating technology has matured and is now available for transfer in the BARC/DAE technology transfer website.