

# यूरेनियम निष्कर्षण प्रभाग में नाभिकीय पदार्थ प्रौद्योगिकी का विकास

## 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  $\text{UO}_3$  particles is about  $25\text{ }\mu\text{m}$  and that of  $\text{UF}_4$  particles is about  $35\text{ }\mu\text{m}$ . The representative SEM images of the  $\text{UO}_3$  and  $\text{UF}_4$  are shown in Fig.1 (a) and 1 (b) respectively.  $\text{UO}_3$  is supposed to be porous and uniform agglomerated product for assuring the complete conversion to  $\text{UO}_2$  followed by  $\text{UF}_4$ , which is quite evident in the SEM images shown in Fig.1(a). The specific surface area of  $\text{UO}_3$  is about  $10\text{ m}^2/\text{g}$ . 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  $\text{UF}_4$  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  $\beta$ -quenching and  $\alpha$ -annealing cycles to reach sizes around  $100\text{ }\mu\text{m}$  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

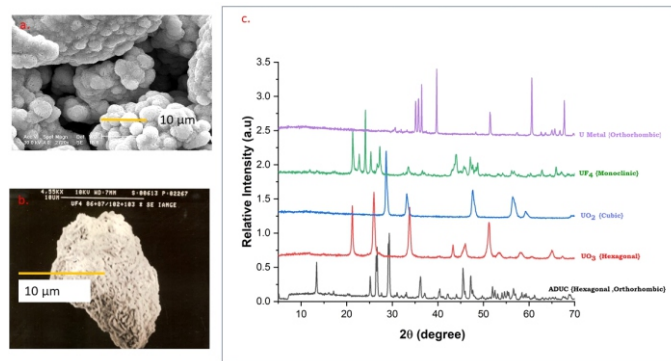


Fig. 1: SEM image of (a)  $\text{UO}_3$ , (b)  $\text{UF}_4$  and (c) XRD of the various intermediates in the uranium metal preparation.

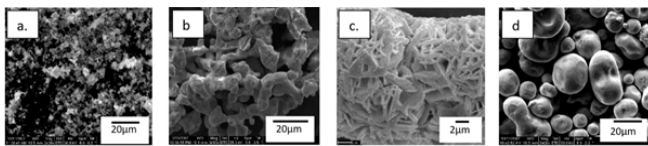


Fig. 2: The SEM images of the metal powders and the respective oxides depicting the typical morphological features.

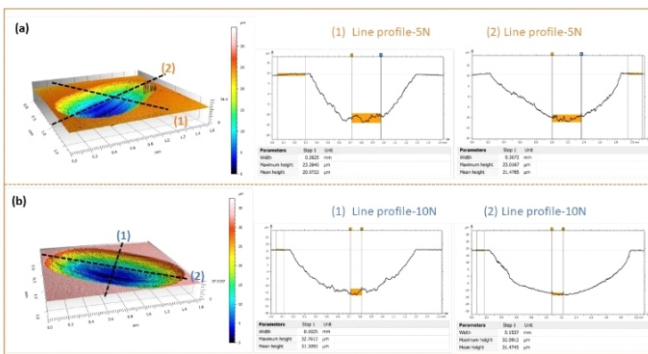


Fig. 5: U-1.5Ti alloy, 3D map of wear scar for (a) 5 N & (b) 10 N normal load and adjacent figures present the line profile of wear scar along the both major and minor axes as marked by dashed line.

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,  $\text{ThF}_4$  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  $\text{ThF}_4$  of required quality specifications in a static

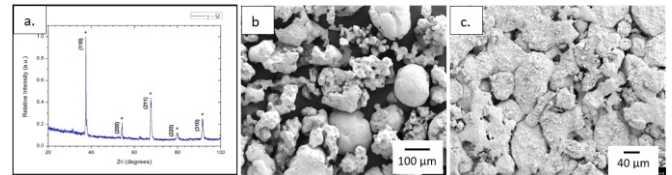


Fig. 3: (a) XRD plot shows completely stabilized  $\gamma$  phase and (b) SEM image, of U – 10 wt% Mo alloy powder produced by R-D process, (c) Microstructure of partially sintered pellet prepared using R-D U-Mo powder.

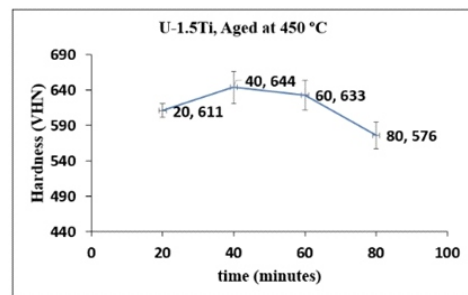


Fig. 4: Hardness vs ageing duration at 450 °C Of U-1.5Ti alloy.

bed hydro-fluorination set up.  $\text{ThF}_4$  was utilised for the various studies related to the Indian Molten salt Reactor program.

Studies on U-Ti alloys of 0.8-1.7 wt.%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 °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.  $\text{U}_2\text{Ti}$  precipitates in U-1.5 wt.%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  $1.796 \times 10^{-4} \text{ mm}^3/\text{Nm}$  and that of U-1.5Ti aged to maximum hardness was found to be  $1.6549 \times 10^{-4} \text{ mm}^3/\text{Nm}$ . 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.