

Procedure for ThO₂ ablation target pressing and sintering

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General notes:

- Radiation meter and probes must be tested against a calibrated source before use
- HEPA filter must be running continuously
- The following protective clothes are required inside the tent whenever thorium is in use until cleanup is complete:
 - long pants
 - closed-toed shoes
 - labcoat
 - double nitrile or latex gloves
 - dosimeter
 - long hair must be tied back

When handling exposed Th or ThO₂ outside the glovebox:

- P100 particle masks
- When loading items into the glovebox, only one airlock door may be open at a time; the other door must be secured by at least two wingnuts
- The large glovebox door must remain closed and the valves must be closed or have a carbon filter placed in front of them unless the glovebox has been thoroughly cleaned and radiation tested
- Anything removed from the glovebox must be wipe-tested

Procedure for target preparation (general outline):

1. Pulverize approximately 4 parts niobia, 1 part distilled H₂O in crucible
2. Dry at 100 degrees C for 1 hour in small oven
3. Crush again and mesh through the nylon mesh (14 mesh), 80x80 mesh, then 325x325 mesh
4. Mix 2.52 parts Nb₂O₅ by weight with 1,000 parts by weight of ThO₂ (0.25 mol % of Nb₂O₅).
5. Pre-compact at 90 MPa ~ 12,600 psi in 3/4 in diameter die (2.228mV load cell readout)
6. Break up the precompact
7. Rub through nylon mesh and dry 1 hour at 75 degrees C
8. Press slowly to 180 MPa ~ 26.1 kpsi in 3/4 in die (4.612mV load cell readout)
9. Sinter 3 hours at 1,150 degrees C, ramping to target temp at a rate of 300 degrees C/hour

Procedure for target preparation including radiation protocol:

Have the following things inside the glovebox:

- nylon mesh (14 mesh)
- mortar and pestle
- kimwipes
- beaker
- funnel

- forceps
 - scoop
 - spatula
 - isopropanol
 - scales
 - dies and half-cylinder for removing pellets
 - distilled water
 - ziplock bags
 - trashbag (use one of the ziplock bags)
 - masking tape
 - pen
- Outside the glovebox:
- radiation meter calibrated and ready outside the glovebox (on the other table)
 - box for storage of pellets

1. Outside the glovebox

1. Pulverize approximately 4 parts niobia, 1 part distilled H₂O in crucible
2. Dry at 100 degrees C for 1 hour in small oven
3. Crush again and mesh through the nylon mesh (14 mesh), 80x80 mesh, then 325x325 mesh
2. Pass ThO₂ powder and prepared niobia powder inside the glovebox through airlock door
3. Mix 2.52 parts by weight of Nb₂O₅ with 1,000 parts of ThO₂ (0.25 mol % of Nb₂O₅)
4. Load the die with the mixture
5. Put the die with the powder, the necessary pushing rods, and the split cylinder for removing the pellet into one ziplock bag
6. Pass the bag outside through the airlock door and put another "clean" ziplock bag on
7. Pre-compact at 90 MPa ~ 12,600 psi in 3/4 in diameter die (2.306mV)
8. Pass the bag with the precompact back inside the glovebox along with the crucible (with cover)
9. Remove the precompact from the bags
10. Break up the precompact
11. Rub mixture through nylon mesh
12. Load the die with the mixture
13. Put the die with the powder and necessary dies along with half-cylinder for removing the pellet inside one ziplock bag
14. Pass the bag outside through the airlock door and put another "clean" ziplock bag on
15. Check with the radiation meter
16. Press slowly to 180 MPa ~ 26.1 kpsi in 3/4 in die (4.612mV)
17. Pass the bag back inside the glovebox
18. Put the pellet inside the crucible, cover and tape the cover
19. Wipe the outside of the crucible with isopropanol to clean it
20. Pass the crucible outside and check with the radiation meter
21. Sinter 3 hours at 1,150 degrees C, ramping to target temp at a rate of 300 degrees/hour
22. After the sintering is done tape the cover to the crucible and handle with the double gloves
23. Check exterior of crucible and interior of furnace for radioactivity
24. Pass the crucible back inside the glovebox
25. Remove the pellet and place it inside two ziplock bags
26. Pass the pellet outside and place one more ziplock bag
27. Store in the appropriate box inside the locked cabinet

Cleanup procedure

1. Clean the inside of the glovebox and all the equipment used for ThO₂ handling with dampened paper towels and kimwipes to pick up obvious powder spills
2. Do a final wipe of glovebox interior and equipment with isopropanol
3. Dispose of cleaning supplies and other glovebox trash in the glovebox trash bag
4. Test the following items outside the glovebox with the pancake probe. Document results and clean if counts above background are observed:
 - lab coat
 - gloves
 - clothing
 - dust mask
 - skin and hair
5. Perform wipe test of the following items outside the glovebox. Document results and clean if counts above background are observed:
 - target pressing equipment (press, load cell, etc.)
 - glovebox exterior near airlock
 - lab benches on which target equipment was handled
6. Perform fortnightly radiation tests on the following items. Document results and clean if counts above background are observed:
 - Wipe test
 - tent walls
 - tent floor
 - cabinets
 - furnace bench and furnace exterior
 - peripheral target pressing equipment (cutting tools, calculator, boxes of gloves, and anything else that might be inadvertently exposed to radioactivity during target preparation)
 - radiation meter and probes
 - Pancake probe scan
 - HEPA filter
7. Dispose of dust mask once per week or whenever it shows counts above background
8. Dispose of all radioactive waste in labeled double ziplock bags in the radiation disposal area. Keep track of the amount of material disposed of, and label the bag of accumulated waste with a radioactive waste label, available from the Radiation Safety Office at Harvard.
9. Wash hands thoroughly with soap

Emergency cleanup

1. Call Amar Vutha (857)284-2869 or John Doyle (617)495-3201
2. Wipe affected area with damp paper towels, working from the outside in. Use cleaning solution if needed.
3. Test cleaned area, clothing, and persons for radiation.
4. Repeat if necessary.
5. Call Harvard EHS if spill is very large or difficult to contain. Emergency number: (617)495-5560
Radiation Office: (617)496-5560