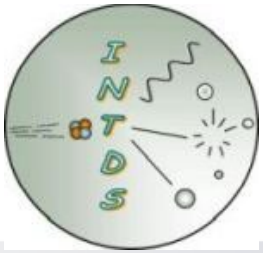
Electrodeposition of iridium for target manufacturing

Ntombizonke Kheswa
NRF-iThemba LABS, South Africa



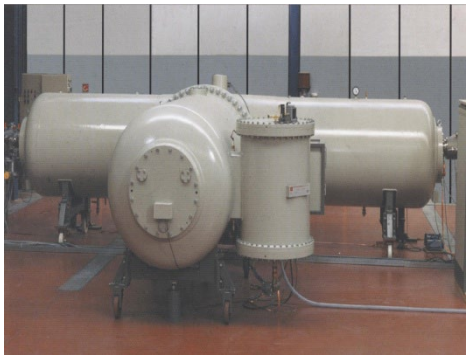
30th Conference of the International Nuclear Target Development Society (INTDS) 2022
25 – 30 September 2022, Paul Scherrer Institut, Switzerland

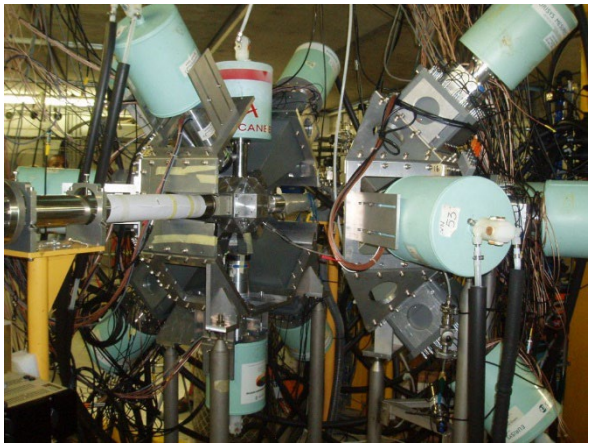
OUTLINE

- Introduction: Application of targets, target laboratory and motive to produce Ir target.
- Experimental trials: Dissolution methods for Ir, Electrodeposition set-up and deposition trials.
- Results and challenges.
- Future plans

INTRODUCTION

- Facilities where targets are used
  - AFRodite – γ array detector
  - K600 spectrometer
  - Neutron vault
  - Tandetron and Tandem-AMS facilities





Azaiez, et al (2020), iThemba LABS, Nuclear Physics News, 30:4

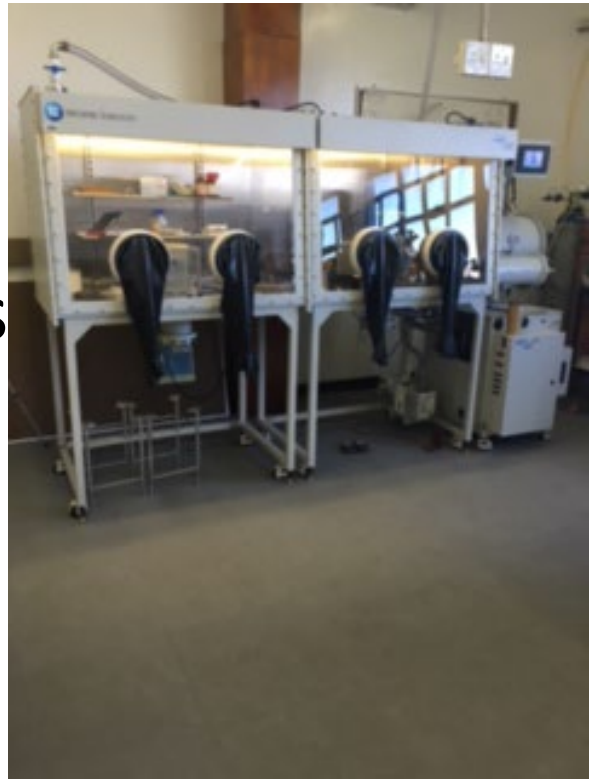
https://tlabs.ac.za/

Target laboratory

Glove Box

Supplier: Innovative technology

- Gas purifier and 2 antechambers
- $O_2 < 1$ ppm
- Moisture < 1 ppm
- Working gases: N_2 or Ar
- Housed: rolling mill and weighing balance



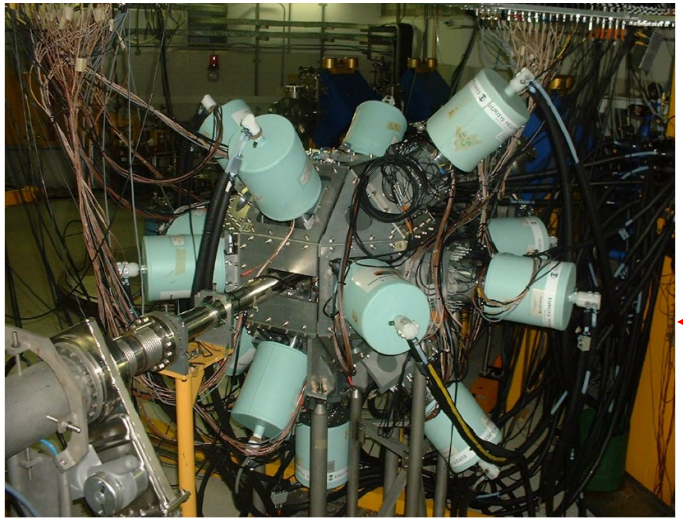
Target laboratory

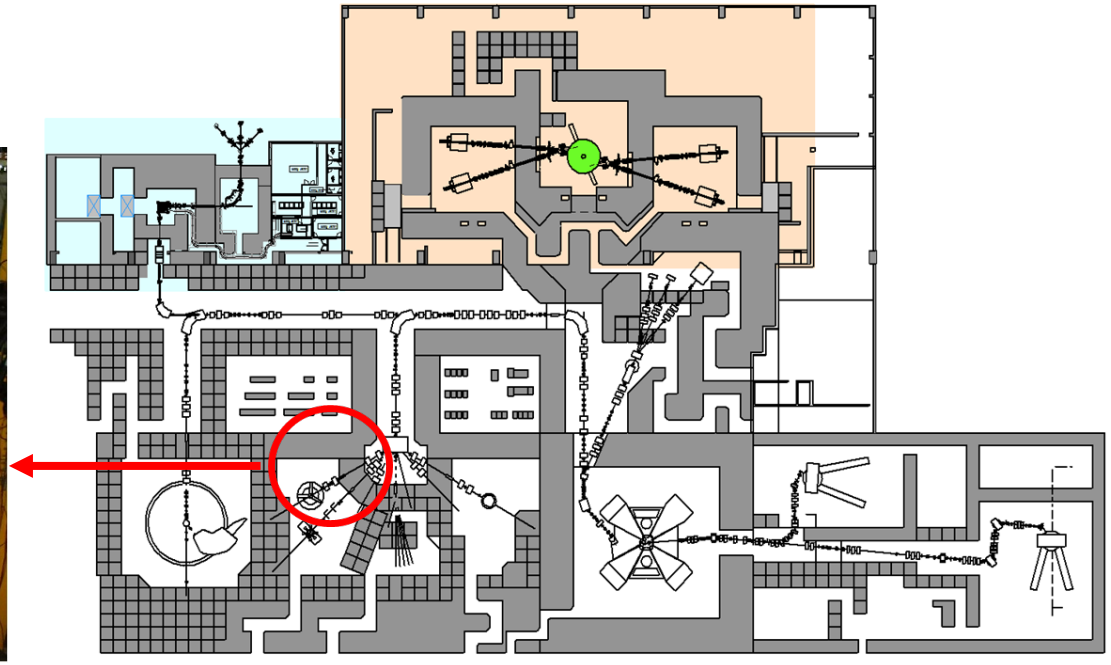
Vacuum_evaporators

Rolling mill

THE EXPERIMENT

- Irradiate a self-supported ^{193}Ir target with an α beam of 48 MeV in the F-line chamber and collect prompt gamma-rays in ^{193}Au with the upgraded AFRODITE array; **^{193}Ir(α,4n)^{193}Au reaction**
- A self-supported ^{193}Ir target with thickness of 2mg/cm^2 sufficient to stop the ^{193}Au recoils (A stack of 100 µg/cm^2).





Experimental

Aqueous Electrolyte- metal should dissolve in a solvent

Dissolution of Ir to $IrCl_2$

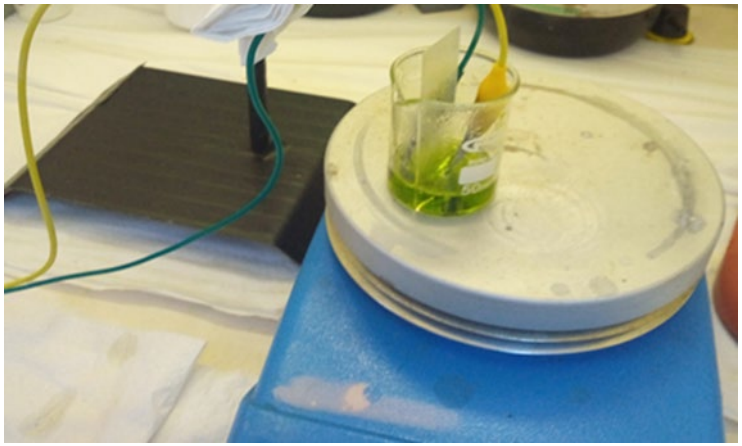
- Iridium is the second hardest, not ductile element of the PGMs [Green, *et.al*., 2020; Leddicotte, 1961].
- It is the most corrosion resistant element- not attacked by most of the acids (aqua-regia, molten metals, etc.) [MacDaniel, et.al., 1971; Leddicotte, 1961].

Experimental: conversion to Iridium complexes which will dissolve in acids to form an electrolyte.

Experimental

Three types of electrolyte: Iridium chloride, sulfamic acid and Iridium bromide ([Platinum metals rev, 1966].

- Iridium Chloride was a pre-requisite compound for all three electrolytes.
- Preparation: Mixed Iridium powder with HCl (32 %) and refluxed. The process was slowly to reach the green colour.
- Electroplating was performed using Iridium chloride as the electrolyte.



Sulfamic acid electrolyte

Due to unavailability of H_3NSO_3 in our lab, it was prepared inhouse by mixing oleum with urea (this was done during lockdown restrictions). Resulted acid was then diluted to 1 M concentration and mixed with iridium chloride.

Electroplating set-up

DC Power supply: 25 V

Cell : 5 cm^3

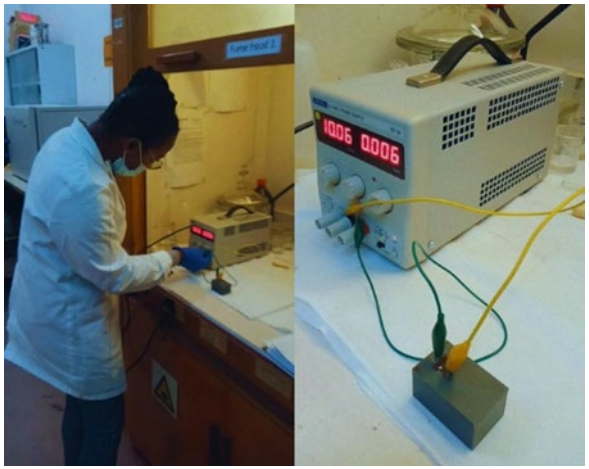
<u>Conditions</u>

Cathode: Cu (0.15 mm)

Anode : Pt (0.15 mm)

Current Densities: 500 mA

We couldn't be precise, Cu was attacked by electrolyte.



Iridium Chloride and Sulfamic Electrolyte Comparison

Iridium Chloride

- Black deposit noticed on Cu, no deposits of Ir.
- Cu attacked by electrolyte

Recommendation

AC auxiliary electrode

Sulfamic Electrolyte

- Silvery white thin broken Ir pieces were noticed on a Cu substrate.
- Cu attacked by electrolyte, had to stop the depositions
- Electrode clamps also got damaged as well

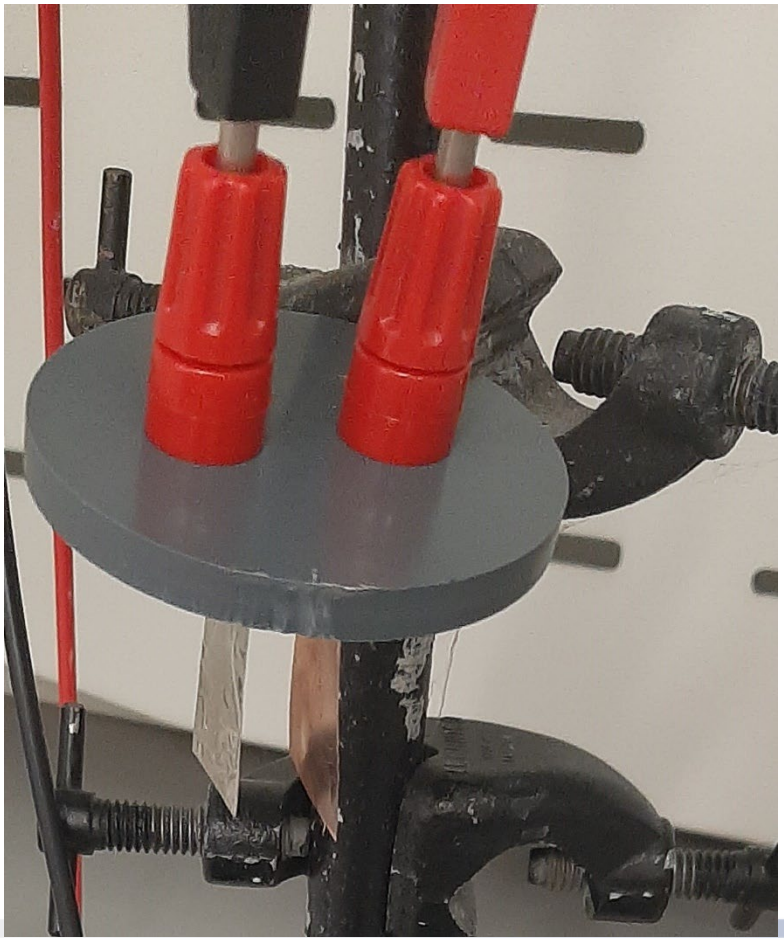
Complete Dissolution of Ir Methods

- Ir was not dissolving complete in HCl
- Digestion under vacuum using HCl and H_2O_2 solvents (No facility nearby).
- Alkali fusion with KOH and KNO_3

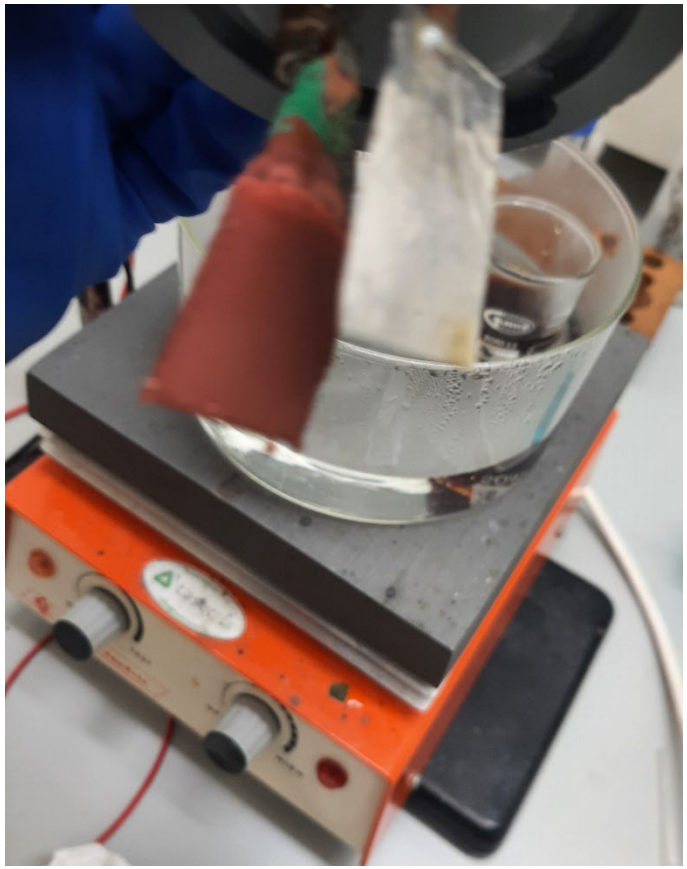
Microwave digestion – University of Stellenbosch

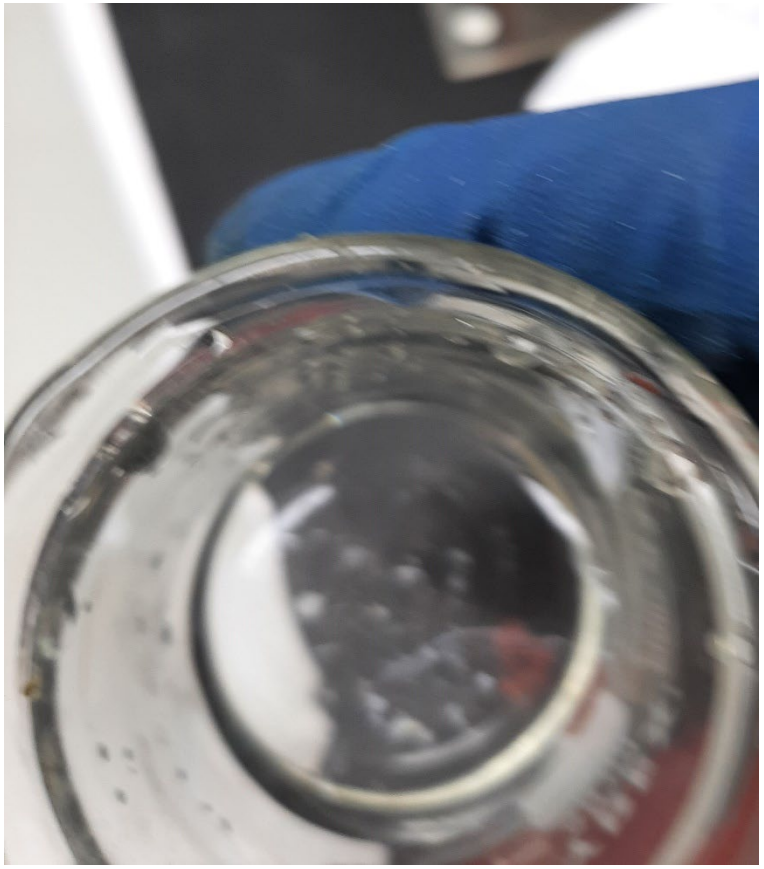
- Partially digested (solvents HNO_3 and HCl)
- Reducing metal was added- NaOH pellets
- Solution was further refluxed for days trying to improve the dissolution of Ir.
- Electroplating continued using Sulfamic electrolyte

Electroplating design modification



Results and challenges





Future Plans

- Investigate the reason on why Cu substrate is attacked by the electrolyte.
- Increase microwave digestion duration
- Use bromide electrolyte- awaiting for chemicals
- Make improvements on the setup- smaller volume cells to minimise waste.

Thank you

NGIYABONGA