^{50}Ti for a High-Intensity Heavy-Ion Beam

Bettina Lommel[1], Andreas Beusch[1], Willi Hartmann[1], Annett Hübner[1], Birgit Kindler[1], Jutta Steiner[1], Vera Yakusheva[2,1]

[1]GSI, Darmstadt, Germany; [2] Helmholtz-Institut Mainz, 55099 Mainz, Germany

The semi-magic ^{50}Ti has a closed neutron shell with N = 28 and is the most neutron rich of the stable Ti-isotopes. This makes the isotope especially interesting as a beam projectile for heavy-element synthesis. For the production of a high intensity ion beam over long time periods, the metallic form of the enriched isotope is preferable for both, ECR and PIG ion sources.

Highly enriched ^{50}Ti is delivered either as the dioxide or as the tetrachloride. The task was to establish a process to reduce the compound to the metal with a high efficiency and a high chemical purity.

Process

For the reduction process we preferred the solid TiO_2 to the liquid $TiCl_4$ since handling gaseous $TiCl_4$ at higher temperatures needed for the reduction process involves a higher risk of losing material. For the TiO_2 there are in principle two common routes for reducing it to the metal either with metallic Ca or with CaH_2 as reducing agent [1]. Since for the affectivity of the reduction the purity of the Ca is crucial we decided to use the CaH_2 in the process. The Ca vapours are generated within the mixture via thermal decomposition of the hydride and can react immediately with the adjacent TiO_2.

The process chosen [2] is described by the following chemical equation:

$$TiO_2 + 2\ CaH_2 \rightarrow Ti + 2\ CaO + 2\ H_2 \uparrow \qquad (1)$$

For the reduction we prepared a mixture of the two components in an agate mortar with a 40% access of CaH_2 to the stoichiometric composition described by the reaction equation (1) and pressed to tablets with a hydraulic press. The tablets are heated in a molybdenum boat inside a stainless steel tube at 950°C for an hour in a constant flow of dry argon. After the reduction the tablets now containing Ti-metal, CaO and rest of Ca are cooled down to room temperature, dissolved in diluted acetic acid; then the precipitate is washed and dried, obtaining a fine powder of metallic Ti.

Analysis

At first the setup and the process were tested with different batches of natural TiO_2 where a yield of 95 % was achieved. For the application in the ion sources ten different batches of $^{50}TiO_2$ from three different producers were reduced. The starting material obviously differed in colour, in grain size and in softness leading to quite different behaviour by pressing tablets for the reduction and by melting metallic Ti, as well as a significantly variation in yield during reduction.

To get an understanding of the impurities in the starting material and their behaviour during the reduction process, energy-dispersive x-ray analysis (EDX) of all batches before and after the reduction was applied. In the natural material no impurities were detectable before the reduction, after the reduction a Ca-content of ~ 0.3 – 0.6 % was observed. Several batches of the enriched $^{50}TiO_2$ showed no impurities before and only the expected amount of calcium after the reduction. But a number of batches had impurities of Cl, Si or Sn or a combination of those. All impurities were in in the range of 1.5 % up to 5 %. The Cl vanished completely after the reduction, whereas the Si and the Sn stayed in the same amount.

The different impurities caused different behaviour in the further processing especially by melting tablet of Ti powder to beads.

Results

With the new setup we are able convert between 0.5 g up to several grams of highly enriched ^{50}Ti in one run with yields between 90 % and 98 %. Different impurities in the starting material influence the melting behaviour and therefore the final yield significantly. We obtain enriched metallic titanium for application in the ion source or as a starting material for target production. The reduced material was used at UNILAC for production of ^{50}Ti-beam with intensities of about 1 particleµA at a target for experiments which lasted several month in 2011 and 2012 [3,4,5].

References

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