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Chromium target preparation for the measurement of nuclear data for fusion technology

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Abstract

Chromium is a hard, shiny and brittle metal. Due to its brittle nature, it is very challenging to make a thin film of very low thickness. In the present work, self supporting thin films of natural chromium deposited on copper foils in the range of thickness 400 μg/cm^2-900μg/cm^2 have been prepared by thermal evaporation technique. The copper foil substrate is dissolved in nitric acid. Self-supporting chromium thin films of such thickness fulfill the requirements of the proposed surrogate nuclear reaction experiment for fusion technology.

Introduction

Self-supporting thin films of various materials are always required as targets for the experiments in nuclear physics and nuclear chemistry. The target preparation is an essential step for getting thin and uniform self supporting foils to be used in the experiment of nuclear reactions with ion beam and charged particles[1].

Purpose to prepare the thin films of natural chromium in the range of 400-900 μg/cm^2, as it is to be used as a target in the proposed surrogate nuclear reaction experiment**.** Surrogate nuclear reaction technique is an indirect way of determining cross-sections of a nuclear reaction, mainly used for the case where direct experimental measurement of cross-section is not possible[2]. During the fusion reactor operation a large number of long-lived radionuclides are produced like ^{53}Mn (t $_{½}$ = 3.74E+6 year), ^{55}Fe(t$_{½}$ = 2.73 year), ^{59}Ni (7.6E+4 year)[3-4]. So there are a large number of chances that neutron get react with these long-lived nuclides. The important helium and hydrogen production reactions of these long-lived radionuclides lead to the swelling and embrittlement of the structural and wall materials as helium and hydrogen accumulate at different locations inside the fusion reactor. Thus it is very important to study the neutron induced cross-section. Direct measurement of neutron induced cross-section of these nuclides is extremely difficult as they do not exist in nature.

Detailed study of ^{55}Fe(n,p) reaction cross-section has been performed earlier[5-6]**.** After satisfying all the conditions**,** ^{6}Li+ ^{52}Cr → d + (^{56}Fe)* reaction has been proposed as a surrogate nuclear reaction of the n + ^{55}Fe → (^{56}Fe)* → p+ ^{55}Mn reaction. Estimate of chromium target thickness is calculated by SRIM code, taking minimum energy loss (E$_{loss}$= 50 KeV) of incident Li-6 beam (E $_{Li-6}$= 25-33 MeV). Chromium is a transition metal that is a steely-gray, lustrous and hard. It is odorless, tasteless and malleable. It has a melting temperature of about 1907°C (2180K)[7]**.** Due to its high vapour pressure at the melting temperature under high vacuum condition sublimation more than a melting process takes place. ^{nat}Cr and its different isotopes ^{50}Cr(4.345%), ^{52}Cr(83%),^{53}Cr(9.5%), and ^{54}Cr(2.37%). In the present manuscript the methodology we have adopted for the preparation of self-supporting thin film of chromium of thickness 400-900 μg/cm^2 has been discussed in details.

Choice of the process

For finding the appropriate process we studied the production of chromium thin film reported in the literature. A variety of preparation techniques have been proposed to produce the thin film of Cr (self supporting/on substrate) including rolling method, electron beam deposition, electroplating, thermal evaporation technique and pulse laser deposition. However new routes with optimized process parameters for obtaining the self supporting thin film deposition (in the desired range of thickness) remain an interesting subject.

At first effort we tried to prepare the thin film of Cr by rolling method (cold roll). Rolling is very efficient method for producing thicker self supporting targets. As Cr is very brittle so at the time of rolling it was broken into pieces and was not possible to get a thin film of order (400-900 μg/cm^2) by rolling method.

Kuehn et al.[8] described the preparation of self supporting metallic foils of several chromium isotopes by electroplating. We have tried the thin film deposition of Cr by electroplating method. We deposited the Cr on nickel and copper substrate. But at the time of removal of substrate it was not possible to get a self supporting thin film as per our requirements (400-900 μg/cm^2).

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Yoshiaki Uemura et al.[9] discussed the preparation of various elements thin film by electron gun (e^- beam deposition) method and achieved the self supporting thin film of 0.1-1 mg/cm^2.In our study, we also tried with e^- beam deposition to get the thickness of Cr thin film around 400-900 µg/cm^2. We have deposited chromium on the glass micro slides using Teepol and NaCl films as a release (parting) agent. But the deposition of Cr was not uniform. We tried to float these Cr films on the water but Cr did not separate from the release agent.

Birgit K. et al. [10] recently prepared thin film of isotopic ^{50}Cr by thermal evaporation technique, in the range of 250-620 µg/Cm^2. In our work we perform complete study of natural Cr thin film deposition by thermal evaporation technique and got success to achieve the desired thickness of around 400-900 µg/cm^2. We have optimized the process parameters of thermal evaporation method, to get the desired thickness (400-900 µg/cm^2) of natural Cr self supporting thin film, which will be used as a target in the nuclear physics experiment. The flow chart for ^{nat}Cr thin film synthesis in this study is outlined in Fig.1.

Figure 1: Flow chart illustrating the procedure for the preparation of ^{nat}Cr thin film

Experimental

Deposition of Chromium on Cu Substrate

A Tantalum boat having diameter of 10 mm and height about 20 mm, was connected to two copper electrodes Fig.2. This assembly was kept in the vacuum chamber and evacuated to the pressure of about 5× 10 $^{-5}$ mbar. Thermal Evaporation set-up is shown in Fig.3. Then tantalum crucible was blank heated for 3 minutes to remove any impurities if at all present in it. After cooling the electrodes and tantalum boat, the air was released in the chamber. 99.9% pure chromium chunks of about 89 mgs were powdered by mortar and pestle. From this powder 7.3 mgs powder was put in the tantalum boat.

Commercially available Cu foil having a thickness of 37 microns was cleaned, by dilute nitric acid to dissolve the impurities and oxides present, and then by isopropyl alcohol. After that it was cold rolled to get the thickness of 27.6 microns, which was cut into a foil of about 30mm × 30 mm. The weight of Cu foil was 221.7mg. This Cu foil was kept at a distance of 16 mm from the top of the tantalum boat, on the aluminum mask having a hole of 25 mm diameter, kept on the brass stand. On this copper foil a micro-glass slide was kept to avoid the curling or folding of the Cu-foil at the time of evaporation, as shown in Fig. 4**.**

Figure 2: Tantalum boat connected between copper electrodes

Figure 3: Thermal Evaporation Set-up

Figure 4: Micro-glass slide on copper foil

The glass chamber was evacuated to about 5× 10 $^{-2}$ mbar and argon was flushed thrice into the chamber to remove air molecules. Then this chamber was again evacuated initially to 10^{-2} mbar by rotary pump and then to 5× 10 $^{-5}$ mbar by oil diffusion pump. The current of 195-200 amp was passed through the copper electrodes to a tantalum boat, through a transformer via VARIAC. The heating was continued for three minutes. After 3 mins current was stopped. The chamber was allowed to cool down for about 30

mins. Then the Cr deposited copper foil was removed and weighed. The weight of copper foil along with deposited Cr was 224.1 mg. The increase in weight shows that 2.5 mg of Cr got deposited on Copper foil. The total Cr deposited area was 4.9 Cm^2 (as shown in Fig. 5). That is the thickness of Cr deposited was 0.9 mg/cm^2.

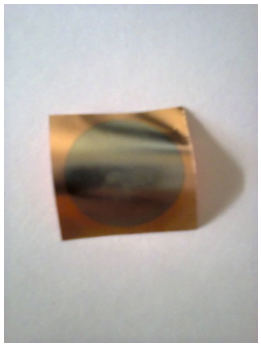


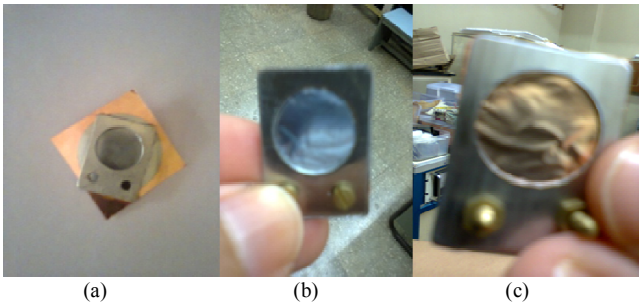
Figure 5: Chromium deposited on the copper

Dissolution of Copper

The Cr deposited Copper foil was sandwiched in between two stainless steel target frames having area of 25 mm × 18 mm and 14 mm diameter hole, (as shown in Fig.6) by the screws. These sandwiched frames were immersed in 40 % diluted nitric acid as shown in Fig 7. and kept it for approximate 2 mins. After 2 mins it was observed that all the copper was dissolved and pure chromium film was intact in between the frames.

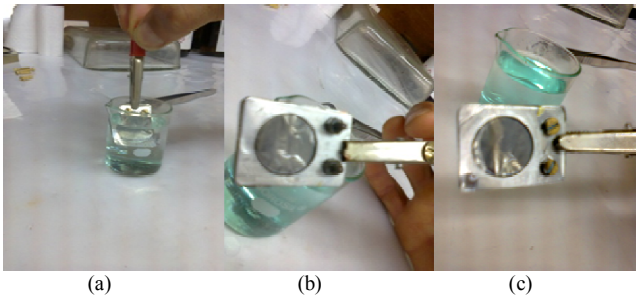
$$3Cu + 8HNO_3 \rightarrow 3Cu(NO_3)_2 + 2NO + 4H_2O \quad \text{.....(1)}$$

Using same procedure we have prepared 2 more Cr targets of thickness 0.408 $mgms/cm^2$ and 0.410$mgms/cm^2$, as shown in Fig. 8, by varying the weight of Cr powder, thickness of Cu foil, distance between source and substrate, evaporation time, conc. of HNO_3. Details are given in Table 1.



(a) (b) (c)

Figure 6: Cr deposited Copper foil, (a) sandwiched in between two stainless steel target frame (b) front part- deposited Cr (c) back part -Cu foil



(a) (b) (c)

Figure 7: (a) Sandwiched frames immersed in diluted nitric acid (b) Cu removed from the back side (c) Cr- thin film on target holder

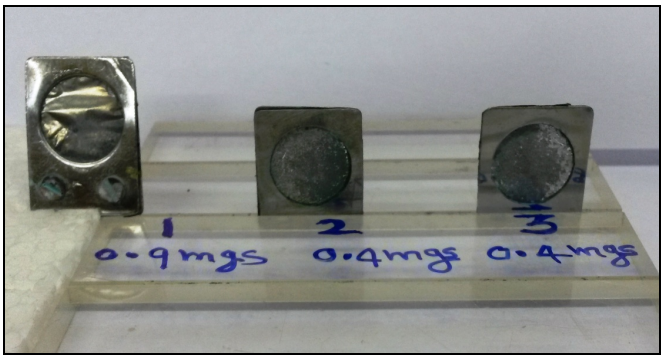


Figure 8: Self-supporting thin film of Chromium

Table 1: Optimized process parameters for self supporting Cr thin film (400-900 µg/cm^2)

Weight of Cr Powder (mgms)	Thickness of Cu Foil (microns)	Distance between Top of a boat and substrate (mm)	Evaporation time (mins)	Conc of HNO_3	Dissolution time of Cu Foil (mins)	Thickness of Cr Foil per Sq.Cm. (mgms)
7.3	27.6	16	3	40%	2	0.900
7.6	11.4	21	1	20%	40	0.408
9.2	20.36	21	2	20%	75	0.410

Conclusions

We have successfully prepared the natural chromium self supporting thin film of thickness 400-900 µg/cm^2 using thermal evaporation technique. Due to very brittle nature of Cr the self supporting thin film preparation of less than one micron is not an easy task. In the present work we have optimized the process parameters to obtain a stable self-supporting thin film of the required thickness.

The natural chromium and its different isotopes are desired as a self supporting foils (target) for nuclear research experiment, that's why it is very important to have a knowledge of optimized process parameters by which we can prepare the fresh targets at the time of nuclear physics experiment.

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