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TARGET DEVELOPMENT SOCIETY

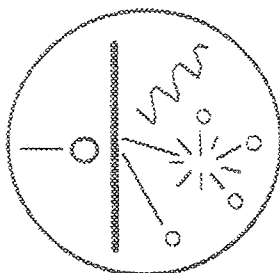# NEWSLETTER
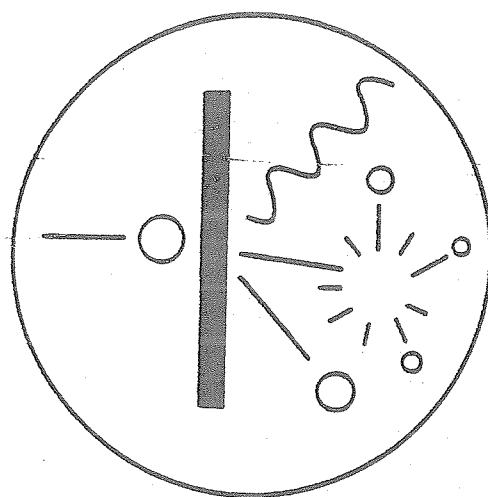
International Nuclear Target Development Society

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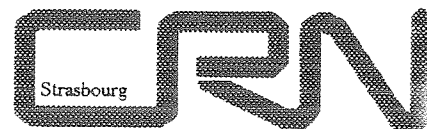
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CENTRE DE RECHERCHES NUCLEAIRES

Strasbourg CRN

18th World Conference of the INTDS

Nuclear Targets
Theory, Practice, Behavior, Studies
Strasbourg, France, 7-11 October 1996

Final call for papers

The 18th World Conference of the **International Nuclear Target Development Society** will be held in Strasbourg at the Centre de Recherches Nucléaires. The topics will cover the preparation and the characterization of targets for low, medium and high energy accelerator experiments.

The subjects will include :
A. Stable and radioactive isotope targets
    Strippers, neutralizer and window foils
    Internal targets
    Beams from stable and radioactive isotopes
B. Target chemistry
    Geological, biological and medical samples
C. Micrometallurgy : vacuum deposition, electrodeposition, isotopic materials
    Thin film technology
            Elaboration
            Equipment
D. Characterization
            Methods
            Influence of target and sample parameters in experiments
            Target and sample degradation studies

The Conference proceedings will be published in Nuclear Instruments and Methods A.

The Conference fees are : 1800 FF for INTDS members; 2100 FF for non-members

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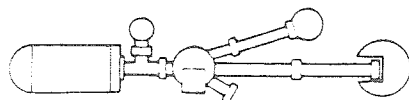
International
Conference on the Application of
Accelerators in Research and Industry
Department of Physics
University of North Texas
P.O. Box 5368
Denton, Texas 76203-0368

FIRST ANNOUNCEMENT

FOURTEENTH INTERNATIONAL CONFERENCE
on the
APPLICATION OF ACCELERATORS IN RESEARCH AND INDUSTRY
set for
November 6, 7, 8, 9, 1996 (Wednesday through Saturday)
University of North Texas, Denton, Texas USA

The Fourteenth International Conference on the Application of Accelerators in Research and Industry will be held at the University of North Texas in Denton, Texas, November 6-9, 1996 (Wednesday through Saturday). Please note that this conference is four (4) days in length. The proceedings of the conference will be published in Nuclear Instruments and Methods in April 1997. The editors of these proceedings will be Jerome L. Duggan, The University of North Texas and I. L. Morgan, IDM, Inc. The Abstracts of the manuscripts will be published in September, 1996 by the University of North Texas Press. The conference is being organized by The University of North Texas.

The purpose of the conference is to review research, and the wealth of industrial applications that are in progress with accelerators throughout the world. The conference is composed of two symposia which run in parallel. These are the Research Symposium and the Industrial Applications of Accelerators. Some of the sessions which are of general interest will be held common to both groups. Participants can easily interchange between the two symposia. Five hundred invited papers will be given at these symposia and contributed papers will be accepted in the following areas: Atomic Physics and Related Phenomena, Trace & Surface Analysis with Ion Beams, Electron Beam Processing, Nuclear Physics, CTR & Related Phenomena, Neutron Activation Analysis & Bulk Analysis with Accelerators, Radiological Safety Aspects of Accelerators, Ion Implantation with Particular emphasis on Semiconductors & Metallurgical Applications, Geosciences & Related Phenomena, Charged Particle Microprobes, Super SIMS, Carbon Dating, Computed Tomography, Synchrotron Light Source Experiments, Insitu Beams, Radiation Interaction with Ion Beams, Accelerator & Component Design & Automation, Targetry, Detectors & Electronics, Medical Applications with Accelerators, Biological & Chemical Applications, Material Analysis with Ion Beams, Channeling, Stopping Power & Radiation Effects.

Most of the contributed papers will be presented in poster sessions. An abstract (invited and contributed) in APS format is required by 1 July, 1996. The deadline for receipt of all manuscripts is October 1, 1996. Designated times will be assigned for the participants to be present at their poster station.

Further information, application blanks, poster information, manuscript materials, Airlines/Car rental discount travel, and other conference information can be obtained by contacting:

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Preparation of metal targets with a thickness of a few atom layers by noble gas ion sputtering.

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1.   INTRODUCTION

This article describes the preparation of very thin metal layers which ought to be suitable for the calibration of a surface barrier detector. The metal layers are prepared by sputtering onto a carbon backing in a 10 keV ion beam sputtering unit, type Sletten and Knudsen [1]. It is possible to prepare metal layers with a reproducible thickness of a few atom layers by sputtering. We have also an evaporation unit but it is not possible to use it for the preparation of metal layers with a reproducible thickness of a few atom layers.

The above mentioned surface barrier detector is used in our laboratory for depth profile measurements of catalytic active samples. A good absolute calibration is necessary to obtain reliable data for the transposition of energy profiles to depth profiles. Our surface barrier detector is calibrated for this aim on four metals, which are regularly spread in the available mass area, namely Al, Fe, Ag and Au.

The peak widths in the He-RBS spectra for very thin layers are (well) known from literature [2]. The energy value, at which these peaks appear can be calculated from the binary collision law. As different deviations of this law are possible, it is desirable that one has always calibration samples at one's disposal.

The layers of the above mentioned metals are prepared by sputtering with Ne^+ and He^+ ions. This procedure is described in section 2. The prepared targets are RBS analyzed with 2 MeV He^+ ions. The measured thicknesses are given in tables in sections 3 and 4.

The collection efficiencies are calculated from the used beam fluences and from well known theoretical and experimental literature concerning the sputter ratios. The calculated results are compared with the measured values and discussed critically.

The aim of this article is to present a (fast) method for the reproducible preparation of very thin calibration targets in behalf of RBS measurements and, if needed, other thin layer experiments.

2.   Procedure

The metals Aluminium (Al), Iron (Fe), Silver (Ag) and Gold (Au), which were chosen as calibration elements, were sputtered onto backings made from spectrographic pure graphite. We applied these backings, because for RBS analysis a light element as backing is preferred. These circular backings, which had a thickness of 0.5 mm and a diameter of 10 mm, were cleaned thoroughly in very pure ethanol in an ultrasonic bath, before they were mounted. The thin layers were deposited on the backings by means of 9 keV Ne^+ ions bombarding thick foils in our

sputtering unit, type Sletten and Knudsen [1]. The different foils were glued on carbon beam stops, which were machined at 45° with the incident beam as shown in ref.[3]. The diameter of the glued metal foils (20 mm) was large enough to prevent carbon sputtering during the focussing procedure. This is especially important for the preparation of very thin layers. The distance between the backing and the beam stop was 20 mm.

Two different types of targets were prepared, namely one layer targets and four layer targets. Our four layer target was prepared by subsequent sputtering of Al, Fe, Ag and Au.

After sputtering of each element, the sputtering unit had to be opened to mount a beam stop with the next metal.

The used Ne^+ beam was not mass selected. There may be some Ne^{2+} ions in the ion beam, but following measurements of Folger et al [4] for Ar, the amount will be negligible when 0.5 A arc current is used, as was done by us.

3.   Analysis of Ne^+ sputtered targets

The amount of atoms sputtered onto the targets was measured by Rutherford Backscattering (RBS) of 2.0 MeV He^+ ions from our 3 MV Van de Graaffaccelerator. The results for 10 different targets are given in table I. We recalculated the number of atoms to a number of atom layers (for an ideal bulk structure). This number is given in the last column of table I. The results given for O refer to the measured amount of oxygen found in the relevant targets.

The oxygen found in the Al and Fe targets was not only caused by the oxidation of the Al and Fe layers in air, but also by sputtering of the partly oxidized Al and Fe foils. According to Sigmund [6] the majority of the sputtered particles originates from a surface layer of a thickness of $\approx$ 5 Å.

Al and Fe surfaces are covered by oxide layers with a thickness of 15 Å or more ([7] and [8]). One can show by calculation and extrapolation that the fresh Al layers consist of Al_2O_3 + Al. For Fe-oxides are the sputter ratios unknown. As sputtering of oxides leads to a surface deficit of oxygen ([9] and [10]), a reliable approximation of the amount of sputtered metal and metaloxide is impossible.

Table 2 gives a survey of the collection efficiencies of atoms of the four utilized elements, as measured by RBS analysis in the central area of the target, deduced from the Ne^+ fluences as given in table 1.

Table 3 gives in the second column the sputtering ratios for ion incidence normal to the surface (Y_0) derived from the graphs of Matsunami et al [11]. The columns 3 and 5 give the sputtering ratios at 45° incidence (Y_{45}) calculated with the Sigmund formula for heavy ions (formula 13 at page 5 of [12]) and calculated according the example on page 22 of [12], for which the Nagoya parameters were used. The Sigmund sputter values are about 10% above the Nagoya values. The columns 3 and 5 give the Y_{45}/Y_0 values for the Sigmund and Nagoya method (Y_{45} S resp. Y_{45}N).

Table 4 gives the collection efficiencies calculated from the total amount of sputtered atoms. The RBS analyzing spot on the target is a few mm^2. The measured collection efficiencies are given in this table in atoms/cm^2 as this quantity is commonly used as a measure of a real density. The collection efficiencies have been calculated following the two methods of Sigmund and Nagoya. An example of such a calculation is given below for the Ag target number 3, obtained by Ag sputtering by 21 mC Ne^+. 21 mC represents 131 x $10^5 Ne^+$ ions. The sputtering ratios of Sigmund (5.97) and Nagoya (5.46) from table 3 give a total amount of atoms, sputtered from the beam stop equal to 78 and 72 x 10^{16} atoms respectively.

The measured number of Ag atoms on targets is 107,2 x 10^{15}. The collection efficiency is 0.137 (Sigmund) or 0.149 (Nagoya).

The collection efficiencies given in table 4 show higher values for thicker targets (Ag and Au targets). The collection efficiency is also higher when the metal has been deposited on a backing with an earlier deposited layer of one or more metals, i.e. targetnr. 10. A probable explanation, given by Petersen on page 8-15 of [9], is that the sticking coefficient is found to be strongly dependant on the total time, during which the substrate was subject to impingement. Chopra [13] elucidates this by mentioning that the value of the critical beam density for nucleation drops markedly, immediately after the initial nucleation has occurred. This explains that the

eposition rate increases when the sputtering procedure goes on. The collection efficiencies for Al and Fe are significant lower than those for Ag en Au (comparison for the same fluences). However, the Al and Fe beam stops are covered by oxide layers of 15 Å or more. According to Sigmund [6] and Ishitani and Shimizu [14] the majority (95%) of the sputtered atoms originate from a mean depth of $\approx$ 5 Å respectively $\approx$ 7 Å. One can show by calculation and extrapolation of sputter ratios of Al_2O_3 at 10 keV Kr^+ [15] that the fresh sputtered Al layers originate of sputtered Al_2O_3 (most) and Al (less). We could not find sputter ratios for Fe-oxides, but it seems reasonable that for Fe the same is valid as for Al. As sputtering of oxides leads to a surface deficit of oxygen [p. 3-30 of 9] a reliable calculation of the amount of sputtered Al oxide and Al is impossible. The fact, that the comparable collection efficiencies for Ag and Au are 30-100% higher than for Al and Fe, can give some insight into the relative contribution of oxide sputtering.

Table 5 compiles the ratios of the number of oxygen atoms and the number of oxidisable metal atoms in the targets which contain Al, Fe or both. The expected stoichiometric oxygen metal ratio is 1.5 (Al_2O_3 and Fe_2O_3). The ratios in the tables show rather large deviations from the stoichiometric values. The deviations in targets 1 and 10 cannot be ascribed entirely to the error in the RBS analysis. A possible explanation in target 1 is that the layer consists partly or totally of thin Al_2O_3 .H_2O (Aluminiumoxide monohydrate). The deviation of target 2 is within the error, but the value of 1.32 is exactly in accordance with the stoichiometry of Fe_3O_4.

The ratio in target 10 can be ascribed to possible formation of Aluminiumferrate $Al_2Fe_3O_{12}$ [16]. in combination with Al_2O_3.

This RBS analysis showed that the metal layers prepared by Ne^+ sputtering were too thick for obtaining a precise calibration. Especially the peaks for Al and O in target 10 had been shifted clearly (see fig. 1.a). Therefore a new series of targets was prepared, this time by sputtering with 9 kV He^+ ions at a distance of 25 mm between beam stop and backing.

4.   Analysis of He^+ sputtered targets

The targets were produced with 9 keV He^+ ions. The fluences are given in table 6, together with the thicknesses as measured by RBS of 2.0 MeV He^+ ions. The collection efficiencies of atoms of the four utilized elements, deduced from the He^+ fluences in table 6, can be found in table 7. The collection efficiencies of Fe, Ag and Au are also in this case systematically higher if they were sputtered onto a graphite backing, which had been covered already with a thin layer.

Table 8 gives in the second column the sputtering ratio (Y_0) for the four utilized elements with 9 keV He^+ ions at normal incidence derived from the graphs of Matsunami et.al. [11]. These graphs are calculated from their empirical equation. The third column gives the sputtering ratio at 45° incidence to normal incidence (Y_{45}/Y_0), calculated according the calculation example at page 21 of ref. [12]. A Sigmund calculation for light ions doesn't exist. The fourth column gives the quantity of sputtered atoms per ion at 45° incidence (Y_{45}), calculated from the precedent columns. The curves are in good agreement with the experiments as shown in the forementioned graphs, for Al, Fe and Au (second gold curve on p. 254 of ref. [11]). The Ag curve shows only partially agreement with experiments for energies of incident He^+ ions < 1000 eV. The value between brackets for silver has been extrapolated from values, obtained by Keywell [18] and given in the same graph for He^+ ion energies ≤ 6000 eV. Also a value, given by Benninghoven [19] at an ion energy of 1000 eV was more according to these of Keywell than to the curve in table 8. Some of the above mentioned curves of ref. [11] are also reproduced in a reduced size in Nuclear Data [17].

Table 9 gives the calculated collection efficiencies. These are all much higher than the values for the Ne^+ sputtered targets (see table 4), especially for the heavier metals, in spite of the larger distance between beam stop and backing for the He^+ sputtered metals.

A plausible explanation for the discrepancies follows from the conditions for reproducable sputter experiments as given by Andersen and Bay [20]:
- a well defined ion beam
- a high enough beam current

- a uniform current density
- a high enough vacuum and
- a well characterized target.

The situation in our sputtering unit meets tolerable to good to the last four requirements, but because our ion beam has not been mass-separated, one can be sure that an explanation for the discrepancies between the values calculated from litterature and our results can be found in the unselected ion beam. Small impurities of the heavier ions or neutrals of nitrogen and oxygen will be present in the beam. To give information about the influence of impurity, ions sputtering ratios for 9 keV He^+, Ne^+ and N^+ ions are given in table 10 for Al, Fe, Ag and Au at normal incidence. The values were deduced from the relevant curves in ref. [11]. (A N^+ on Al curve was not available)

Table 10 shows that the sputtering ratios of N^+ ions at 9 keV are about one order of magnitude larger than the sputtering ratios of He^+ ions. This means that a small amount of N^+ and eventually O^+ ions in the He^+ beam can influence our sputtering ratios considerably.
The values obtained with Ne^+-ions have been converted to a beam stop to backing distance of 2,5 cm with the square of the distance rule to obtain comparable values for the collection ratios of the metals sputtered by Ne^+ and He^+ ions. The values are presented in table 11.

The collection rates for the samples prepared with an unanalyzed He^+ beam are for Al and Fe about a factor of 3 lower than the corresponding Ne^+ values, which has to be compared to the factor of 10 for the litterature values. These discrepancies are understood in the just described influence of impurity ions. However, for Ag the difference is small, and remarkably, for Au one obtains a higher collection ratio for the He^+ beam than for the Ne^+ beam.

The observed lower collection ratios for Al and Fe can be explained by the lower sputter ratios of the oxides compared with the elements [15] and the in chapter 3 described sputtering of oxide layers [6] and [14]. We have no explanation for the gold targets having a higher collection ratio for He^+ than for Ne^+. Possibly the angle dependence of the sputtered metal atoms is different for Ne^+ and He^+ beams.

Table 12 compiles the atom rates of oxygen atoms to oxidisable metal atoms in He^+ sputtered targets. These rates are for the separate targets about 50% higher than stoichiometric and for the combined target more than two times the stoichiometry for the trioxides. An explanation is given by Vink et al [8] who found that on the top of an iron surface a monolayer of oxygen is present. As the number of atom layers for Al and Fe < 2, the higher stoichiometry can be explained by the existence of this monolayer of oxygen, also in the case of Al.

The O-metal rate in the combined target cannot be explained very well by the existence of $Al_2Fe_3O_{12}$ [16] in combination with absorbed oxygen.

However, the relative error in such thin layers can play a role in the RBS analysis for low Z elements.

Conclusions

Targets of different metals, with a thickness of one or a few monolayers, can be prepared on a graphite backing rather repoducible by sputtering techniques with a not analyzed He^+ beam after some experience. Only a crude prediction can be made for the required ion fluence with the help of the data of Matsunami et al [11] and Yamamura et al [12].

We have no explanation for the higher collection efficiencies of He^+ sputtered gold. Recent applications of thin layers on very smooth backings (silicon and glass) compared to the rather rough graphite show that a notable higher sputtering fluence is necessary in this cases because of the lower sticking coefficient on smooth backings. As we have only a small experience with the smooth backings, we have no knowledge concerning the reproducability of the sputtering deposition of metal layers by noble gas ion sputtering on smooth surfaces.

Table 1
Thicknesses of the targets obtained by sputtering with 9 kV Ne$^+$ ions.

Target number	Element	Fluence: 9 keV Ne$^+$ ions	Number of atoms (x 10^{15})/ cm^2	Average number of atom layers
1	Al	19mC	25.0	16.2
	O		49.6	
10*	Al	19mC	24.0	15.6
2	Fe	16mC	24.4	12.6
	O		32.3	
10*	Fe	17mC	28.2	14.6
	O		109.8	
3	Ag	21mC	107.2	71.2
4	Ag	9mC	34.9	23.2
5	Ag	5.0mC	22.1	14.7
6	Ag	4.4mC	17.3	11.5
7	Ag	3.0mC	11.1	7.4
10*	Ag	3.4mC	13.7	9.1
8	Au	18mC	14.1	26.6
9	Au	7mC	10.9	7.2
10*	Au	7mC	17.1	11.3

(Distance between beam stop and backing 20 mm.)
Used conversion values: amount of atoms/atom layer calculated from data from ref. [2] and ref. [5].
1.536 x 10^{15} for Al
1.933 x 10^{15} for Fe
1.505 x 10^{15} for Ag
1.513 x 10^{15} for Au

* Multi layer target

Table 2
Collection efficiency for 9 keV Ne^+ions.

target number	element	collection efficiency atoms/ion	average
1	Al	0.210	0.21
10	Al	0.202	
2	Fe	0.244	0.26
10	Fe	0.266	
3	Ag	0.818	
4	Ag	0.621	
5	Ag	0.708	
6	Ag	0.630	0.67
7	Ag	0.593	
10	Ag	0.646	
8	Au	0.357	
9	Au	0.250	0.33
10	Au	0.391	

Table 3
Sputtering rates Y_0 at normal and Y_{45} at 45° incidence according to Matsunami et al [11] and Yamamura et al [12]

Element	sputtering rate Y_0 (normal ion incidence) [atoms/ion] [11]	Y_{45} S/ Y_0 according to the Sigmund formula	Y_{45} S	Y_{45} N/ Y_0 according the Nagoya parameters	Y_{45} N
Al	1.7	1.87	3.18	1.67	2.84
Fe	1.8	1.71	3.08	1.53	2.76
Ag	3.9	1.53	5.97	1.40	5.46
Au	2.5	1.36	3.40	1.28	3.20

Table 4
Collection efficiency on the carbon backings for Ne$^+$ sputtered targets.

Targetnr.	Element	Coll. efficiency atoms/cm^2 per sputtered atom Following Sigmund	Coll. efficiency atoms/cm^2 per sputtered atom Following Nagoya
1	Al	0.066	0.074
10	Al	0.064	0.071
2	Fe	0.079	0.089
10	Fe	0.086	0.097
3	Ag	0.137	0.149
4	Ag	0.104	0.114
5	Ag	0.119	0.129
6	Ag	0.105	0.116
7	Ag	0.100	0.108
10	Ag	0..108	0.119
8	Au	0.105	0.111
9	Au	0.073	0.078
10	Au	0.115	0.122

Table 5

Atom ratios of numbers of Oxygen atoms to oxidisable numbers of metal atoms in Ne$^+$ sputtered targets.

Target number	Element	Ratio O to metal
1	Al	1.98
2	Fe	1.32
10	Al + Fe	2.10

Table 6

Thicknesses of the targets, obtained by sputtering with 9 kV He$^+$ ions.

Target number	Element	Fluence 9kV He$^+$ ions	Number atoms/cm^2 (x10^{15})	Number of atoms layers
11	Al	9 mC	2.2	1.4
11	O	9 mC	5.0	
15˙	Al	10 mC	2.6	1.7
12	Fe	8 mC	3.0	1.6
12	O	8 mC	7.4	
15˙	Fe	8 mC	5.0	2.6
15˙	O	8 mC	29.2	
13	Ag	3.7 mC	6.6	4.4
15˙	Ag	4.1 mC	8.3	5.5
14	Au	6.2 mC	11.3	7.5
15˙	Au	6.1 mC	13.6	9.0

Distance beam stop until backing: 25 mm.
˙ Multi layer target.

Table 7

Collection efficiencies for the 9 keV He$^+$ sputtered targets.

Target number	Element	Collection efficiency atoms/cm^2 per ion
11	Al	0.04
15	Al	0.04
12	Fe	0.06
15	Fe	0.10
13	Ag	0.29
15	Ag	0.32
14	Au	0.29
15	Au	0.36

Table 8

Calculated sputtering ratios with 9 keV He$^+$ ions according ref. [11] and [12].

Element	Atoms/ions at normal incidence Y_0	Y_{45}/Y_0	Atoms/ions at 45° incidence Y_{45}
Al	0.081	1.63	0.132
Fe	0.095	1.70	0.162
Ag	0.26 (4)	1.53	0.398 (6.12)
Au	0.17	1.61	0.274

Some experimental values are given between brackets.

Table 9

Collection efficiencies on the carbon backings for He$^+$ sputtered targets.

Targetnr.	Element	Coll. efficiency atoms/cm^2 per sputtered atom
11	Al	0.30
15	Al	0.32
12	Fe	0.37
15	Fe	0.62
13	Ag	0.72 (0.05)
15	Ag	0.81 (0.05)
14	Au	1.06
15	Au	1.30

Table 10

Comparison of the sputtering ratios of Fe, Ag and Au for 9 keV He^+, Ne^+ and N^+ ions at normal incidence, deduced from the curves in ref. [11].

Ion	Element	Sputtering rate Y_0 (atom/ion)	Y_{45}
He^+	Al	0.08	0.13
Ne^+	Al	1.7	2.84
He^+	Fe	0.095	0.16
Ne^+	Fe	1.8	2.76
N^+	Fe	1.2	
He^+	Ag	0.26	0.40
Ne^+	Ag	3.9	5.46
N^+	Ag	2.4	
He^+	Au	0.17	0.27
Ne^+	Au	2.5	3.20
N^+	Au	1.6	

Table 11

Collection ratios of 9 keV Ne$^+$ and 9 keV He$^+$ sputtered targets for a beamstop to backing distance of 25 mm.

Target number	Ion	Element	atoms/cm^2 per ion
1	Ne$^+$	Al	0.13
10	Ne$^+$	Al	0.13
11	He$^+$	Al	0.04
15	He$^+$	Al	0.04
2	Ne$^+$	Fe	0.16
10	Ne$^+$	Fe	0.17
12	He$^+$	Fe	0.06
15	He$^+$	Fe	0.10
3	Ne$^+$	Ag	0.52
4	Ne$^+$	Ag	0.40
5	Ne$^+$	Ag	0.45
6	Ne$^+$	Ag	0.40
7	Ne$^+$	Ag	0.38
10	Ne$^+$	Ag	0.41
13	He$^+$	Ag	0.29
15	He$^+$	Ag	0.32
8	Ne$^+$	Au	0.23
9	Ne$^+$	Au	0.16
10	Ne$^+$	Au	0.25
14	He$^+$	Au	0.29
15	He$^+$	Au	0.36

Table 12

Atom rates of oxygen to oxidisable metals in He$^+$ sputtered targets.

Target number	Element	Rate O metal
11	Al	2.27
12	Fe	2.47
15	Al + Fe	3.84

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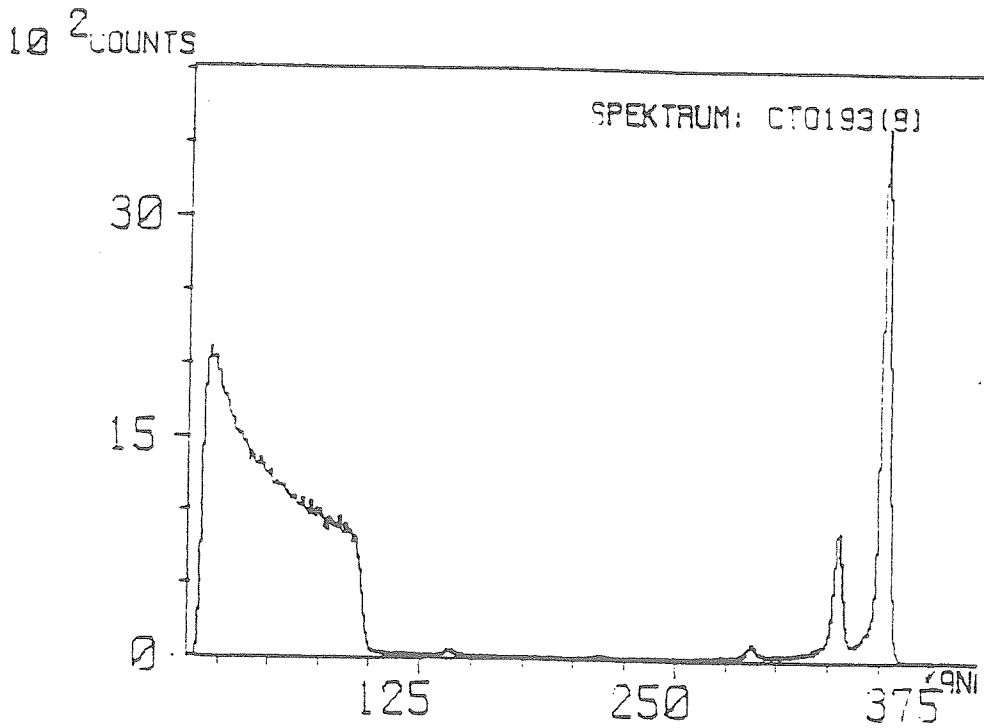
Figure 1.

A.
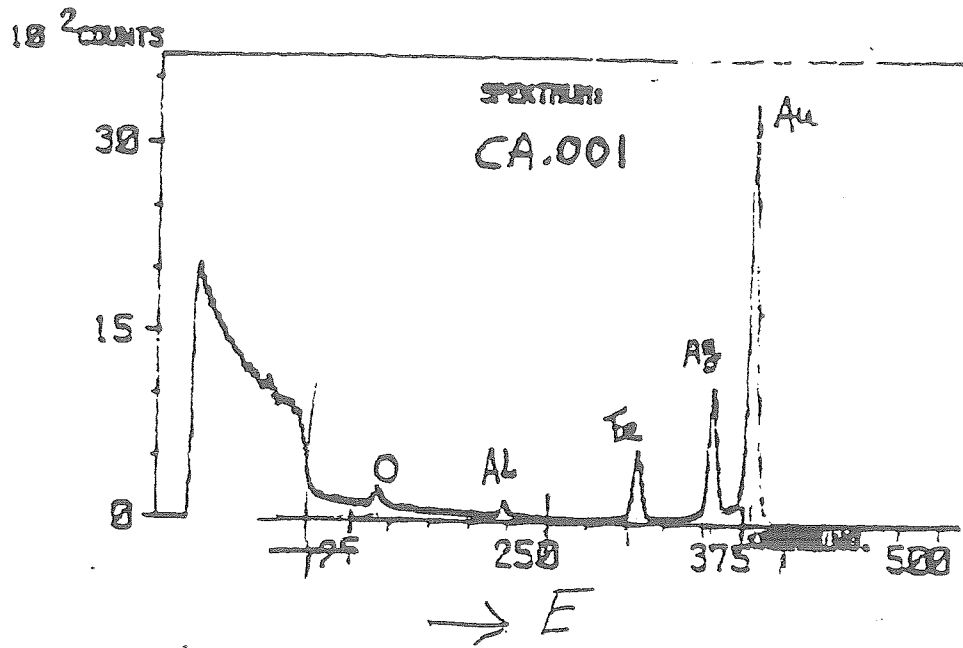
B.

Fig. 1. Demonstration of the shift, due to the thickness of the layers:
A.   RBS-spectrum of the Ne[+]-sputtered multi layer target (no. 10)
B.   RBS-spectrum of the He[+]-sputtered multi layer target (no. 15)

High Efficiency Reduction/Distillation of Yb and Sm Oxides

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The preparation of metals by reduction of oxide powders is routinely carried out in target laboratories for the production of metal stable isotopes (1,2). The scarcity and high value of some isotopes make it important to maximize the collection efficiency of this process.

Apparatus was constructed (Fig. 1) for high yield reduction/distillation of up to 2 g quantities of oxide. The 14 mm diameter tantalum reaction vessel consisted of several tight fitting segments for easy filling and cleaning, and to allow the length of the vessel to be changed to achieve different temperature gradients. Heating was by induction with the coil wound to give intense heating of the crucible base and of the nozzle to reduce distillation of the product at the top of the reaction vessel. The water-cooled collector passes through a sliding vacuum seal and is lowered into position after degassing of the reaction mixture.

Temperatures were monitored by thermocouple and by pyrometer. The key to achieving high yield collections was to minimize vapour leaks from the reaction vessel using tight-fitting joints, and by ensuring close proximity of the·cooled collector to the nozzle.

The equipment was used for the reduction of Yb and Sm oxides using La reductant. The oxide powder was ground together with 50% stoichiometric excess of fresh La filings and was pressed into the Ta crucible at a pressure of 1.5 Mpa using a stainless steel sleeve to prevent distortion of the crucible. The still was then assembled, evacuated to 2×10^{-6} mbar and outgassed at 350°C. The reduction was then carried out at a temperature (950°C for Yb_2O_3 and 1250°C for Sm_2O_3) giving a vapour pressure of the product metal vapour of ≈ 1 torr and above the melting point of La. Yields of about 95% were achieved with both metals (Fig. 2).

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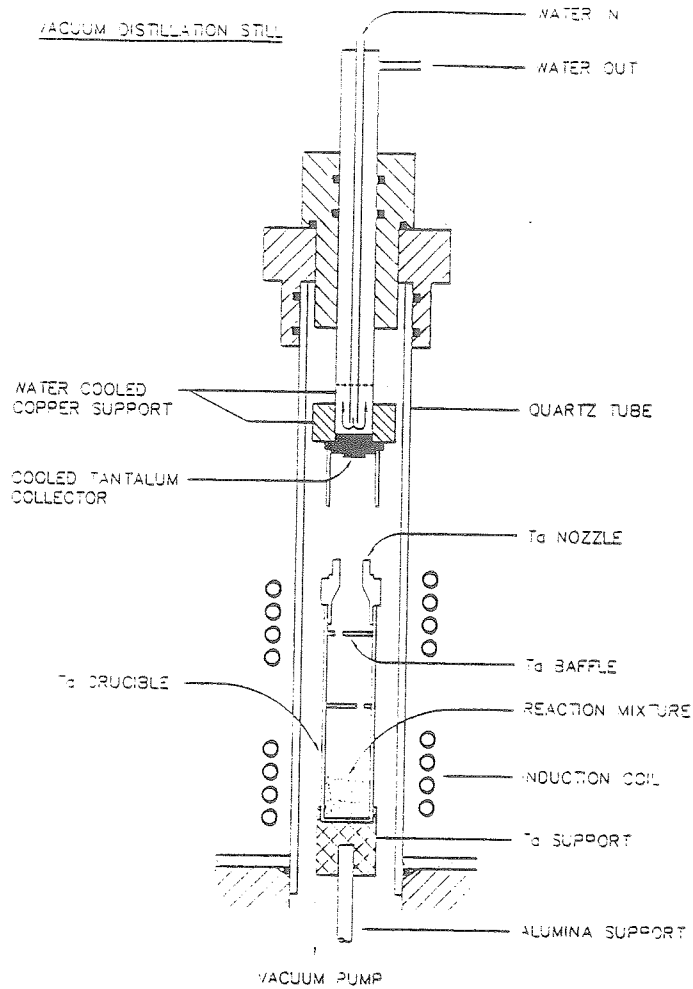
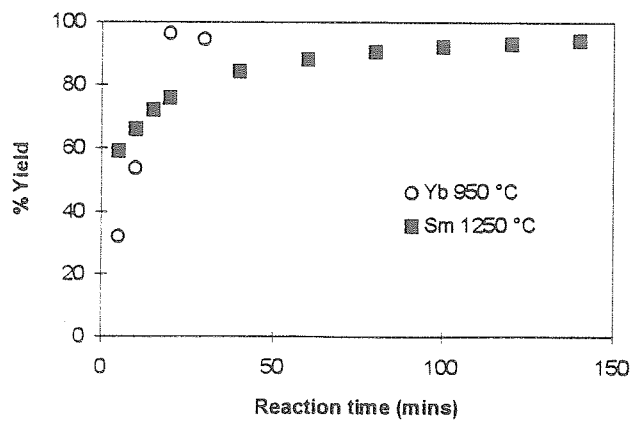Fig. 1   Reduction/distillation apparatus

Fig. 2   Yields of Yb and Sm metal reduced using La.

THE IUCF TARGET LAB TECHNICAL STATUS
(Amended from the IUCF Scientific and Technical Report, May 1995 - April 1996)

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o   A variety of targets for cyclotron experiments were produced, as were skimmer targets and stripper foils for Cooler experiments, and a carbon-fiber support mounting for the Indiana Silicon Sphere (ISIS) targets. The mounting technique consists of two 7-μm diameter C fibers (Union Carbide, Thornel 300 type) which tightly stretch Au foils, typically 2 mg/cm^2 by 20 mm by 20 mm, and centers them over a frame span of 41 mm. To do this, two weight-stretched fibers were glued to opposite sides of a frame, spacing the fibers 21-mm apart. Next, the fibers were temporarily slackened by squeezing two opposite sides of the frame, allowing two 50-μm diameter tungsten wires with hooked ends to be positioned on the fibers. The length of the straight sections of the wires was 19.5 mm. So, when the sides of the frame were released, the distance between the fibers was 19.5 mm. After the fibers were partially coated with glued, they were brought into contact with a dry target foil laid flat on a surface. After the glue cured, the W wires were cut and the target foil was stretched by the tension in the C fibers.

o   Three large-area (3.81 cm × 1.27 cm) targets were produced of both $^{10\,\&\,11}$B in thicknesses of 13.5-15.5 mg/cm^2. Development was directed to minimize the amount of binder necessary for adequate strength during preparation and handling before vacuum baking the pressed powder pellets to achieve adequate strength for use. A total of 300μg/cm^2 of CH binder (1.8 weight %) was sufficient by grinding and sifting to achieve a particle size of ≤ 31 μm, pre coating the die pistons with 5-μg/cm^2 C foils, modifying the technique of distributing the powder into the pressing die (better results were obtained when distribution was conducted along the diagonals of the rectangular die cavity), and baking in a high vacuum at 1600 K. A delicate mounting supported these targets at their edges. The mountings consisted of 190-μg/cm^2 Mylar foils tightly stretched on frame openings of 6 cm × 9 cm, each with a centered cutout for a target pellet. Droplets of five-minute epoxy (Devcon, USA), each <1 mm in diameter and spaced ≈ 5-mm apart, held the target pellets in place.

o   A new mechanical setup was made for thickness determinations with an alpha source inside the lab vacuum evaporator. The setup allowed full x-y direction scans of two thin Si targets and a no-target region, without interrupting the system vacuum.

o   Tests and trials were conducted to make new targets of ^{40}Ca^{127}I$_2$. Although thin-walled Ta tube having flattened-then-welded ends was used successfully in 1989 for direct synthesis from the elements, trouble was experienced with producing leak-free reaction bombs. Several failed efforts generated attempts to make the compound in a quartz tube sealed by heat fusing an open end while the tube was evacuated to high vacuum. A procedure found in the literature described using such a bomb for this reaction. For it, calcium powder is distributed in the middle of the tube and heated to 900 K while the iodine is chased from end-to-end with a torch. Conducted for 1 h, this gave disappointing efficiency. During this attempt, a method allowing a large tube oven in the lab to be used to heat accurately only the middle of a short tube was acquired, as was useful glass-working experience with quartz, and further development of the lab induction heating equipment occurred.

Also tried were other reactions found in the literature: Ca with hydriodic acid (HI) and, separately, Ca with carbon tetra iodide (CI_4) + methanol. In the time available, these efforts had poor conversion efficiencies.

Additional attempts to seal flattened ends of Ta tube produced good reliable results when Ti filler rod was used during welding. Although a literature search had not revealed mention of this, it allowed the reaction to be done. A disadvantage, however, is that Ti forms iodides. Thus, Ti is thought to be the likely source of small but unfortunately placed peaks in data taken with a 43 mg/cm^2 target made from the most recent batch of $^{40}CaI_2$. The probable reason the interfering peaks were seen in this target, and not in a target made earlier, is because the reaction temperature was allowed to be 50-K higher for the contaminated material. If another Ti-free target is required, in a reasonable amount of time it should be possible to refine an acceptable method of synthesis.

During the work to weld-seal the Ta tube, a heat-sinking and clamping device was constructed to minimize oxidation of the tube and to avoid vaporizing the iodine within it during welding. It features massive heat sinking and a recessed area with four jets of deoxidized Ar directed at the exposed end of the Ta.

o   Trials were conducted to chemically polish aluminum pieces of an electrostatic lens for the Cooler Injector Synchrotron. Some of the pieces were too large to chemi-polish with a volume of solution thought practical, so electro-brush polishing was tested. The exercise demonstrated the chief limitation of several brush polishing formulations of perchloric acid + nitric or sulfuric acid when used for 6061 Al. Indeed, under low magnification, surfaces polished by electro-brush and ones chemi-polished by immersion (in ortho-phosphoric + acetic + nitric acid solutions) were pitted. The explanation found widely in the literature is that the 0.4-0.8% Si present in type 6061 Al had been preferentially etched out, although the surfaces appeared smooth and bright to normal vision. Thus, the recommendation made was for mechanical polishing followed with thorough cleaning of the Al pieces. For yet-to-be-made aluminum pieces requiring a highly polished finish, there are aluminum alloys formulated for electopolishing and chemipolishing.

o   For a neutron flux measurement experiment at NIST, the $^6Li_{0.74}Mg_{0.26}$ alloy disk made last reporting period was scraped clean and trimmed to fit tightly in a sealable aluminum box. Until early in 1996, it was stored with continuous vacuum pumping in the glove box of the target lab.

o   Several batches of 450-$\mu g/cm^2$ Teflon films were produced for the original polarized-gas target cell of CE-35, and a new cell which requires somewhat larger films.

o   A brief study was done to estimate the level of O_2 likely to cause accelerated corrosion rates of the materials exposed to the de-ionized water systems. This was prompted because the level of oxygen in the magnet and beam line systems has varied through time from 0.1 to 4 ppm, and the level in the Cooler system had been 4 ppm consistently. Drawing from the corrosion data found in US government literature of problem multi-component systems, it seems that for long-term use of copper and brass components with a minimum of failures, O_2 levels greater than 2 ppm for extended periods are best avoided. A definitive answer to this question should be gained by exposing weighed coupons of copper and brass for perhaps six months.