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Cover picture: Peter Siegler loading a U single X-stal into the cryostat sample holder at IRMM for neutron transmission measurement for Doppler broading experiments.
Editor’s note

It is with great sadness that we inform you of the death of Jean Van Audenhove after a long and debilitating illness. Jean was a major figure in the INTDS and founder and leader of the successful target preparation group in Geel. Those INTDS members who knew him will remember a great man and those who did not will certainly have benefited from his contribution.

On a happier note, I would like to express my thanks to those who contributed to the 20th INTDS Conference hosted by IRMM in Antwerp last October. Times have changed and the world of nuclear physics has moved on since the earlier days of INTDS, but our society remains as relevant and important as ever for interaction between target makers faced with new challenges. The 2002 conference will, of course, be hosted by Argonne National Laboratory, and I hope that members will support this meeting with the same degree of enthusiasm and innovation shown in Antwerp.
Farewell Jean...

On December 5th, 2000, Jean Van Audenhove died a couple of months before his 70th birthday at his home in Mol. The last two years of his life had been a period of extreme trial and suffering, which he and his wife Cécile accepted with great courage and dignity. Even though he and his family, and his closest friends knew that his departure would arrive probably sooner than later, the confrontation with reality at the end was hard.

Besides having been a real friend for many of us, Jean has been one of the most important members of both INTDS and IRMM/CBNM. As an exceptional personality of the pioneering generation of nuclear target producers, he was an extraordinary driving force for our Society, for which he became not only a highly appreciated President but also one of the first Award Winners. And who does not remember the exceptional INTDS Conference he organised in 1984 in Antwerp?

He was also one of the most prominent members of the early Central Bureau for Nuclear Measurements and his outstanding accomplishments contributed enormously to the world-wide reputation that the institute built up in the sixties and seventies. He was a pioneer in electron bombardment vacuum deposition, developed high frequency levitation melting as a practical method for quantitative alloying (a technique that was proudly demonstrated to generations of visitors of the institute, and which lead to the development of CBNM’s U-Pu metal spikes). He was also the father of IRMM’s unique collection of reactor neutron dosimetry certified reference materials, and he “re-invented” the dry hydrofluorination of actinides, which allowed CBNM to produce uranium, plutonium, neptunium and thorium deposits of the most exceptionally high quality. But he was also the man with the long-term vision who started the CBNM activity in non-nuclear reference materials as early as 1969! It was his foresight and persistence in cultivating the collaboration with the Bureau Communautaire de Référence (BCR) that led to the inception of the reference materials activity at Geel and he oversaw the first phases of the installation that has become the most advanced CRM production facility in the world.

Thanks for all that Jean: you have always been an example for many of us and we will miss you very much. In the name of his many INTDS friends, I express our most sincere condolences to Cécile, to his children and to his family.

Jean PAUWELS
12th January 2001

Jean Van Audenhove
1931-2000
Meeting Announcement

The Fall 2001 Meeting: "The 7th China Nuclear Target Preparation Technology Meeting" will take place in Mianyang, Sichuan Province, China (date to be decided).

Recent Progress

- 100000~110000 nm thickness CH layers on micro-sphere surfaces have been prepared using low-pressure plasma CVD. The gases sources are H₂ and styrene. RF power is 15 watt and the pressure is 60 mTorr. These targets will be used in ICF experiments.

- Alloy target films of Mg/Si alloy have been prepared. The atom ratio of Si and Mg is 5 - 40%, and thicknesses are 300nm-500nm. The XRD and TEM studies showed that Mg/Si is not in solid solution. Si is present as amorphous nano-particles and these particles are embedded among the Mg crystalline particles. The Mg crystalline particles are of a single orientation ((200) direction).

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Principal directions of research activity at the laboratory of neutron research of the Institute for Nuclear Research of the Russian Academy of Sciences

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The Laboratory of Neutron Research contributes to the design of the INR RAS Neutron Source, with computational support and engineering work on solid state targets (natural W, depleted U) with water and liquid metal cooling. We have developed the calculations of heat transfer, heat deposition, neutron yield, nuclei product distributions, and hydrodynamic and thermo-mechanical behaviour of the target. The experimental effort can also be developed to study the properties of candidate structural materials under the combined influence of mechanical loading, proton irradiation and chemical effect of coolant.

Activity Calculation
The calculation of neutron yield, heat and nuclei product distributions in targets of arbitrary geometries irradiated with proton beams of energy up to 100 GeV can be done by Monte Carlo methods based on the high-energy hadron transport code SHIELD. The SHIELD code is dedicated to simulation of the interaction of high-energy particles with complex macroscopic targets of arbitrary geometry, chemical and isotopic composition. It has been designed as an universal tool for a wide field of studies, such as:

- Study of the spallation-process in heavy targets under proton beam irradiation including generation of neutrons, energy deposition in the target, and radionuclide production in intense neutron generators, accelerator-driven transmutation, and electro-nuclear breeding.
- Radiation shielding and of accelerators and in space.
- Radiation damage of target materials under primary and secondary radiation fields.
- Simulation of background events in experimental accelerator installations and for underground physics.

The main features of the modern version of the SHIELD code are:

- Transfer of nucleons (including low energy neutrons), pions, kaons, antinucleons, and muons in the energy range up to 1000 GeV. Recently, transport of arbitrary (A, Z)-nuclei and ionisation loss and straggling have been taken into account.
- The main modes of the mesons decay are modelled. Hadron-nucleus interactions inside the target are simulated in a unique approach on the basis of known Russian models of nuclear reactions describing the cascade and precompound stages of the reaction as well as evaporation/fission, multi-fragmentation, and Fermi break up of residual nuclei.
- The transfer of neutrons (E<14.5 MeV) is simulated with an original neutron transport code LOENT based on the 28-group neutron data system BNAB. A special interface allows us to connect the known EGS4 code for simulation of eγ-showers initiated by products of meson decay. Each hadron cascade tree is stored completely during its simulation. This permits the full division of the modelling and registration parts of the code and facilitates output. Both direct (analogous) and weighted simulation are provided. The open code architecture facilitates its modification and development.
Calculation of Radiation Damage
The radiation effects in solid under intermediate energy (600-800 MeV) irradiation are defined by elastic (electromagnetic and nuclear) as well as inelastic interaction contributions of primary protons with the target atoms. The inelastic contribution tends to be dominant at the proton energy $E > 100$ MeV. As a result of cascade processes, the nucleons, $2\text{H}$, $3\text{H}$, and $\alpha$-particles escape the rest nucleus, which acquires the kinetic energy of a few MeV. Due to moderation in the target material this rest nuclei produces the primary knocked-on atoms (PKA), which can transfer the energy to other atoms by means of cascades of nucleus-nucleus interactions. The RADDAM code was built up to carry out a computational study of radiation damage induced by middle and high energy nucleon beams in "thin" and macroscopic targets. During the simulations of hadron cascade in the target the SHIELD code gives the individual characteristics of all nuclear reactions occurring including A, Z, and kinetic energy of recoil nuclei, i.e. the primary knocked-on atoms (PKA) sources, which are then used for radiation damage calculation. Compared with the well-known NMTC version, the RADDAM code includes the radiation damage effects of spallation nucleons and geometry-dependent damage energy contribution for moderated neutrons. The calculation results of the RADDAM code have been found to be in reasonable agreement with the data obtained by means of the well-known HETC code. Considering that the DPA number is not, in itself, a fully reliable guide to the changes in properties produced upon irradiation, some further theoretical research will be needed. Therefore, the directions of theoretical radiation research in LNR will be the following:
- Theoretical investigation of stochastic mechanisms for defect clustering kinetics in metals and alloys under irradiation.
- Analysis of atomic displacement cascades and their role in the long-term accumulation of radiation damage.
- The search for possible correlations in structural changes with the spectral effects of primary knocked-on atoms.

Calculations of Thermomechanic and Hydrodynamic Behaviour
The calculations are carried out for structural elements of neutron targets (thermal fields and stresses in three dimensions, thermo-shocks, erosion of target materials by sputtering) under irradiation of proton and ion beams of arbitrary profile by means of analytical and numerical methods. Calculations of lifetimes of carbon stripper foils under ion bombardment are also carried out (see our paper presented at the 18th INTDS Conference).

Accelerator-Driven Experimental Facilities at the Moscow Meson Factory
The facility includes a linear proton accelerator with an energy of up to 600 MeV at an average current up to 1 mA. The temporal structure of the primary beam is characterised by a repetition rate in the range 1-100 Hz. Inside the Experimental Hall of MMF there are three main accelerator-driven installations: the Pulsed Neutron Source, the RADEX facility, and the Lead Slowing-down Neutron Spectrometer.

The Pulsed Neutron Source (PNS) was put into operation in 1998. Its active zone made of tungsten with light water cooling with an average power of 200 kW. The average thermal neutron flux density is $3 \times 10^{12}$/s and the peak thermal neutron flux density is $10^{15}$ n/cm$^2$/s. The
PNS is mainly intended for investigations in the field of condensed matter in the range of neutron energies from thermal (cold) up to several MeV using the time of flight technique. The RADEX facility is the irradiation channel located inside the proton target of the beam stop in the line of the accelerator (see our paper presented at the 20th INTDS conference). RADEX is intended for the study of radiation damage of prospective structural materials for the needs of fusion, fission and radiation waste installation. Two experiments are planned on the RADEX in the near future. The first is the monitoring of neutron flux inside the irradiation channel with the help of a new, compact radiochemical detector which has been used previously as a counter of solar neutrinos. The second is a measurement of the generation rate of point radiation defects at liquid helium temperatures.

The slowing-down neutron spectrometry is based on the fact that, under moderation processes, fast neutrons from the pulsed neutron source break down into many groups around the middle energy E, depending on the moderation time. Therefore the measurement of the time dependence of nuclei production rate allows the determination of the energy dependence of nuclear reaction cross-sections. The Lead Slowing-down Neutron Spectrometer (LNS) has luminosity 103-104 times higher than the time-of-flight spectrometers with the same neutron source intensity and the same energy resolution (~30-45%). The new generation of accelerator-driven LNS, which is now placed inside the experimental hall of MMF contains about 100 tons of lead. This installation will be put into operation this December.

Innovative Research in the Condensed Matter Field

There has been a great deal of interest in recent years in the subject of long range phase coherence in inhomogeneous superconductors. Some systems, such as granular composites, are clearly inhomogeneous at all temperatures. For example, the key feature of carbon arc foil deposition on room-temperature substrates is the low mobility of carbon atoms due to the overcooling state of the carbon condensate. The granular structure is therefore formed. Note that the new high-temperature superconducting oxides are essentially inhomogeneous and granular. Consequently, a different technique is required to verify the superconducting transition when it does not result in a zero-resistance state. Possibly, this technique is the reverse AC Josephson effect which has been shown I have the ability to verify the occurrence of superconductivity in the YBaCuO system, not only at a lower transition temperature (90K), but also at the higher temperature of 240K (J.T.Chen et al., Phys. Rev. Lett., 1987, v.58, p.1972). These observations give some idea that new high-temperature superconductors can be identified initially as granular weak superconducting systems. One carbon-arc compound, that we are studying, is possibly a such system. We are studying the conductive properties of two-dimensional carbon arc condensates in the temperature range of 4-350K. Unusual electrical properties of samples have been observed. The electrical current growth initiates a temperature dependent electrical resistance jump of up to 5 orders of magnitude. This behaviour is likely to be close to the switching phenomena in glassy carbon which has been verified by K. Antonowicz et al (Carbon, 10(1972)81). The switching current value decreases with increasing temperature. The second interesting property which may be useful for electronic devices is the conversion of microwave radiation to direct current, which has been verified by means of the inverse Josephson effect (S.G. Lebedev and S.V. Topalov, Bulletin of Lebedev's Physics Institute, 12(1994)14).
Thin Silicon Foils and Silicon Grating Foils used to Research Spatial Nonuniformities of Driven Lasers in Inertial Confinement Fusion Experiments

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Abstract

The research of spatial nonuniformities of driven lasers is an important experiment for inertial confinement fusion (ICF). Thin silicon foils and silicon grating foils are needed in this experiment. Thin silicon foils with a thickness about 3 to 4 micrometers and with surface roughness about several tens of nanometers were prepared by a semiconductor process together with self-stop etching. The parameters of semiconductor processing and self-stop etching were studied to control the surface roughness of the foils. Combined with ion beam etching, check or stripe patterns were transferred to the surface of thin silicon foils, and the silicon grating foils were obtained. The parameters of the ion beam etching process were studied to control the pattern transfer precision.

Key Words inertial confinement fusion, spatial nonuniformities of driven laser, self-stop etching process, ion beam etching process

1. Introduction

Rayleigh-Taylor instability (RTI) which caused by spatial nonuniformities of driven laser, was studied in Inertial Confinement Fusion (ICF) experiments [1,2]. In China, some researchers put forward a plan to study spatial nonuniformities of laser driven ICF [3] using thin silicon foils and silicon grating foils. Silicon is brittle. It is difficult to obtain self-supporting silicon foils by conventional processes like evaporation or sputtering. Silicon is, however, a material often used in semiconductor technology, where micro-patterns are prepared on silicon wafer by self-stop etching [4]. In this paper, we introduce a process to prepare thin silicon foils by this semiconductor process, and, combined with ion beam etching, to transfer check or stripe patterns to the surface of thin silicon foils and obtain silicon gratings. Surface roughness, microstructure of thin silicon foils and pattern transfer precision of silicon grating foils were measured. The parameters of semiconductor processing, self-stop etching and ion beam etching were studied in order to control the structure of thin silicon foils and silicon grating foils.

2. Experimental details

To research spatial nonuniformities of driven lasers in ICF experiments, thin silicon foils with a thickness about 3 to 4 μm were needed. It was hoped to control the surface roughness to 10nm over a diameter of 600 μm. Silicon grating foils with stripe patterns of about 5 μm width or check patterns with size about 25 x 25 μm, and pattern depth of 1.0 to 1.5 μm were made.
2.1 Preparation processes

An n-type, (100)-oriented silicon wafer with thickness about 260 ± 10 μm, resistivity about 3 - 6 Ω.cm was used. Boron was diffused into one side of the silicon wafer, with B2O3 as dopant. The diffusion depth of boron was about 3 to 4 μm. After diffusion, SiO2 and Si3N4 layers were prepared on the other side of the silicon wafer and used as passivation layers in the self-stop etching process. The SiO2 layer (about 400 nm) was prepared by oxidation and the Si3N4 layer (about 150 nm) was prepared by LPCVD. By photoetching, a pattern about 5mm x 5mm was prepared on the self-stop etching side. Positive-type (A J818) UV photoresist was used in this process. Then self-stop etching was carried out using KOH etching solution. The etching velocity was controlled by the concentration and temperature of the etching liquid. When the silicon wafer was etched to the edge of the boron diffusion side (diffusion depth was 3 to 4μm), etching velocity reduced rapidly to zero. After self-stop process, the passivation layers were removed and the silicon wafer was cut into slices. Thin silicon foils with sizes of 5mm x 5mm were obtained.

Silicon grating foils had the same preparation process as thin silicon foils except that ion beam etching was used after boron diffusion process in the preparation of thin silicon foils. Oxidation was carried out after boron diffusion process, and the SiO2 layer was used as mask layer. On the boron diffusion side, a check mask pattern of size 25μm x 25μm or stripe pattern with width of 5μm were prepared by photoetching. Then ion beam etching was used to transfer the pattern to surface of silicon foils. The parameters of ion beam etching process and etching velocity were controlled to achieve precise pattern transfer. Check pattern or stripe patterns were transferred to the surface of thin silicon foils in this way. Other preparation procedures for silicon grating foils were same as the thin silicon foils.

![Fig. 1 Preparation process of thin silicon foils and silicon grating foils.](image_url)
2.2 Measurement

Type 500 (α-step thickness) apparatus made by the Tencor Company was used to measure the thickness of thin silicon foils and surface roughness on the boron diffusion side and self-stop etching side. A Cambridge S360 scan electron microscope (SEM) was used to study the microstructure and pattern transfer precision of silicon grating foils.

3. Results

By using processes above, thin silicon foils with a thickness at 4.037 μm were obtained measured by α-step thickness apparatus; Silicon grating foils with check or stripe patterns were prepared on thin silicon foils with the same thickness. Further measurements were made on these foils.

3.1 Surface roughness of thin silicon foils

Surface roughness was an important parameter for thin silicon foils. The surface measured by α-step thickness apparatus is shown as Table 1. The measurements showed that the surface roughness was several tens to 100 nanometers for different distances. On the boron diffusion side, the surface roughness was greater for longer measurement distances. This result was caused by defects in the silicon substrate generated during the diffusion process. On the self-stop etching side, the surface roughness was about 25 nm for all measurement distance, and this meant that the self-stop etching process was uniform.

<table>
<thead>
<tr>
<th>Measurement distance / μm</th>
<th>Boron diffusion side</th>
<th>self-stop etching side</th>
</tr>
</thead>
<tbody>
<tr>
<td>3000μm</td>
<td>130.0</td>
<td>25.1</td>
</tr>
<tr>
<td>1500μm</td>
<td>101.1</td>
<td>22.7</td>
</tr>
<tr>
<td>1000μm</td>
<td>48.3</td>
<td>29.4</td>
</tr>
<tr>
<td>600μm</td>
<td>31.4</td>
<td>20.2</td>
</tr>
</tbody>
</table>

3.2 Appearance of silicon grating foils

The appearance and pattern depth of silicon grating foil with check patterns measured by α-step thickness apparatus are shown in fig.2. It can be seen that the check pattern was fairly precisely transferred. The pattern's depth made by ion beam etching process was 962.2 nm. At the same time, the measurement showed the check pattern was changed to a ladder-shape pattern during the ion beam etching process. The width of stripe of the silicon grating foils was too small to measure by α-step thickness apparatus, but the parameters of the ion beam etching process to prepare check or stripe were same, so we assume that the stripe pattern's depth is same as the check pattern depth.
Fig. 2 Silicon grating foils with check pattern measured by $\alpha$-step thickness apparatus

The appearances of silicon grating foils with check or stripe pattern studies by SEM are shown in fig.3. Check or stripe pattern was transferred to the silicon grating foils, but the size of the patterns were smaller than the mask pattern.

Fig. 3(a)      Fig. 3(b)

Fig. 3 Silicon grating foils measured by SEM (x500)

(a) check pattern (b) stripe pattern
4. Discussions

In our experiments, parameters of self-stop etching process and ion beam etching processes were studied to control the surface roughness of the thin silicon foils and the pattern transfer precision of the silicon grating foils.

4.1 Parameters of self-stop etching process

A self-stop etching process was used to prepare the thin silicon foils. In this process KOH etching solution was used. The concentration and temperature of KOH etching liquid were important to control the self-stop etching velocity [5]. In this work, 50 %KOH etching solution was used at a temperature of 50°C. Under these conditions, the etching velocity of silicon wafer was 7 μm/hour. And 30 to 40 hours were needed to etch a silicon wafer with thickness of 260 ± 10 gm to a thickness about 3-4μm.

4.2 Control of the surface roughness of thin silicon foils

In table 1, the surface roughness on boron diffusion side was about several tens of nanometers, and the surface roughness on self-stop etching side was about 30 nanometers. This observation may be explained as follows: on the boron diffusion side, defects were introduced into the silicon wafer by the high temperature in the boron diffusion. Hence the surface roughness on this side was larger than that on the self-stop etching side. The surface roughness was small on the self-stop etching side because the self-stop etching was slow and proceeded uniformly.

Surface roughness was an important parameter for thin silicon foils. There were several ways to control and reduce the surface roughness. For example, (1) using silicon substrates with smaller surface roughness; (2) selecting better experimental parameters, such as diffusion conditions and self-stop etching liquid; (3) using surface modification process after self-stop etching process (sputtering was a good way to deposit a silicon layer about 100 nm, to reduce the surface roughness). All of these methods were investigated.

4.3 Parameters control of ion beam etching

Ion beam etching [6] was an effective process to transfer a pattern to different substrates. In this process, ion beam etching velocity was an important parameter and was influenced by ion power and ion beam density.

In the preparation of silicon grating foils, the parameters of ion beam etching were: vacuum about 7 x 10⁻⁶τ, ion power was 500 eV, density of ion beam 0.35 mA/cm², and the silicon wafer was cooled by a semiconductor cooler. The ion beam etching velocity of boron doped silicon was smaller than that of pure silicon. It needed 100 minutes to get a pattern with a depth about 1 micrometer, and the etching velocity was about 10 nm/minute.

4.4 Appearances of patterns on silicon grating foils

As shown in fig.3, check or the stripe patterns could be transferred to the surface of thin silicon foils, but with some distortion of the patterns. In the patterns, the ion beam etching area was bigger than the non-etching area. Fig.4 gives the appearance of the check pattern enlarged 2500 times by SEM. It showed a ladder-shape pattern. This effect may be caused by a crosswise etching effect. This phenomenon could be avoided by increasing the size of the mask area or by changing the etching parameters.
5. **Conclusions**

Thin silicon foils with thickness about 3 to 4 micrometers were prepared by a semiconductor and self-stop etching processes. The measurements showed that the surface roughness on the both sides of thin silicon foils were several tens nanometers over a distance about 1,000 micrometers. Using ion beam etching, check patterns about 25 μm x 25 μm or stripe patterns about 5 μm wide were transferred to the surface of thin silicon foils to obtain the silicon grating foils. The parameters of ion beam etching were controlled to ensure the precise pattern transfer. The depth of pattern was about 1 μm.

6. **Acknowledgement**

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**References**


Situation for Betaine monohydrate cleared

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In the Newsletter Vol. 27, No. 1 (2000)11 I have asked for help in the delivery problem for betaine monohydrate. Three INTDS members offered help.
Chris Marcus suggested VWR Scientific Products. Their catalogue number is JTC230-5 and their price for 100 g is US$ 35.
John Stoner found out that Fisher Scientific Co. sells it at their stock number AC-204291000. Their price for 100 g is US$ 14.
Since both addresses are in the USA I did not try them.
Adri Michielsen suggested the representative of Aldrich in the Netherlands. That is why we contacted again the Aldrich representative in Germany, where we found out that they could sell again betaine monohydrate which they get from a 85 kg big stock from Fluka in Switzerland. Their new stock number is 14300 and their price for 500 g is 90.80 DM.
Also Lancaster fulfilled their delivery to Hans J. Maier.
I hope I had not alarmed too much all of you. In the moment their seems to be no shortage of the betaine monohydrate.
I thank everyone who looked into this problem.
WHAT’S NEW AT SODERN?
P. Tulle, Sodern, Limeil Brevannes

SODERN is now a subsidiary of the European company EADS. Its historical business, i.e. sealed-tube-neutron-generators studying and manufacturing, remains active.

Neutron Targets
The realization of deuterated and tritiated targets was taken over from CEA/Valduc in the middle of 2000. The small targets, i.e. with backing diameter less than 70mm and activity less than 20 Ci, are prepared entirely at SODERN’s facilities. Shapes for the backing (disc, cup) differ and experience has been gained with different materials (Cu, Al, Mo). The targets, once deposited with titanium, are impregnated individually within metallic containers which are sealed after for a long storage. The T/Ti atomic ratios obtained range between 1.5 and 1.8. (Fig. 1)[photograph N00102]. Larger targets, up to 310mm diameter and less than 1000Ci will be impregnated in a new facility to be operational at CEA/Valduc at the beginning of 2001. The aim of SODERN is to take advantage of their 38 year-expertise to deliver all sorts of tritiated targets according to the specific requirements of the user (Fig. 2). [photograph N00101]

Other Activities
The main activity in neutron instrumentation is still a wide range of neutron generators:
- the transportable GENIE 16, already used in bulk analysers,
- a new compact version for in-field analysis,
- the middle range GENIE 36 with currently being deliveries to nuclear reprocessing plants,
- the high output GENIE 46 (up to 4x10^11 n/s) for imaging or Neutron Activation Analysis.