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# ***Neutron Transmutation Doping of Silicon at Research Reactors***



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# Neutron Transmutation Doping of Silicon at Research Reactors

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# **NEUTRON TRANSMUTATION DOPING OF SILICON AT RESEARCH REACTORS**

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## FOREWORD

The IAEA has for many years been conducting several missions to its Member States in order to assist them in ensuring and enhancing the performance of their neutron irradiation facilities. One area that can serve as a significant source for commerce and cooperation for these facilities is the irradiation of materials for added value, including the production of semiconductor material and gemstone colouration through neutron bombardment and transmutation, a process in which research reactors (RRs) possess prevalence over chemical techniques with regard to uniformity and enhanced electro-conductivity. In the Asia-Pacific region in particular, its research reactors are uniquely positioned to gain substantially through implementing demanding standards and promoting their burgeoning irradiation and processing capacity to the robust electronics industry and gemstone dealers. In order to gather the region's leading producers and interested research reactor staff, the IAEA sponsored under a Regional Cooperative Agreement (RCA) the project RAS4026, 'Adding Value to Materials through Irradiation with Neutrons,' which concluded in 2010. The participating Member States were Bangladesh, China, India, Indonesia, the Republic of Korea, Pakistan, Thailand and Vietnam, and the project's objectives were:

- To expand utilization of RRs in the region with a specific focus on neutron irradiation of materials to increase their material value and thereby generate additional revenue for the utility;
- To gather valuable information to improve the design and capability of such irradiation activities in new research reactors being planned by some Member States in the region.

Within the eight participating Member States are eight operational research reactors already practicing neutron transmutation techniques on various materials, and 33 research reactors overall of a wide spectrum in terms of design, power rating and age. Also, each is at a different stage in the management of its irradiation facilities, making the exchange of experiences very important to achieving safe and effective practices as well as optimized production in collaboration with end users and industrial partners. Among the several research reactor utilization fields in the region that need information exchange, silicon doping, gem colouration and membrane production activities have not been collaboratively organized so far, but Member States assert high interest because such technologies are growing more important, especially regarding the semiconductors necessary for a rapidly advancing electronics sector.

This report is the culmination of the project, and its purpose is to relate guidance on the preparation, design, execution and quality control of neutron transmutation doping (NTD) for silicon in a single document. The publication also contains a detailed history of techniques used at research reactors over the past 50 years and guidelines for quality design and operation of a Member State's irradiation facilities. In addition, an extensive database containing information on experiences world-wide and existing capabilities in using neutron irradiation for the increase of material value has been included for assessing reactor capacity for providing irradiation services. Ultimately, the report is intended to improve research reactor utilization by promulgating the best practices in use today and the quality control demanded by industrial partnership in the field of neutron irradiation services.

The IAEA officers responsible for this publication were D. Ridikas of the Division of Physical and Chemical Sciences and N.D. Peld of the Division of Nuclear Fuel Cycle and Waste Technology.

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# **1. INTRODUCTION**

## **1.1. Background**

For many years, the IAEA has conducted several missions to promote the research and development activities of research reactors. Among various areas of research reactor utilization, the neutron transmutation doping of silicon is one well established technology desired by industry. Silicon semiconductors doped via the NTD method have been widely used in various industrial fields, especially for high quality semiconductor power devices.

NTD can provide a direct commercial income to research reactors, and many research reactor operators desire to implement NTD at their facilities. However, because of its commercial features, detailed up to date techniques have been shared rarely since the last Consultants' Meeting on the topic organized by the IAEA in 1988. This may have given a negative effect to industry's motive for adopting NTD technology for their products. Industry is basically reluctant to use nuclear reactors as a part of their production process based on worries about the availability of NTD, since many NTD reactors across the world are aged, some of them operate with reduced capacity or may even be closed. Therefore, enhancing NTD availability by involving more research reactor facilities would reduce the burden of potential industrial partners developing another technology or material that is costly and needs further R&D efforts. Benefitting not only industry but also the research reactor community, collaboration in NTD is needed.

The worldwide demand for NTD increased rapidly during the 1980s but decreased in the subsequent decade due to the development of gas doping technology, and then fluctuated between 2000 and 2005. Since then, NTD has faced another new circumstance. The demand for high power semiconductors was increasing constantly given the rapid increase of green energy technologies such as alternative electricity generation by wind mills and solar cells as well as hybrid and electric vehicles. The NTD method has several advantages in the production of high power semiconductors in comparison with other conventional doping technologies such as ion implantation or the diffusion method.

The IAEA conducted a regional TC project on 'Adding Value to Materials through Irradiation with Neutrons' during 2007 and 2008. This project implemented various activities in Asia-Pacific region to increase the Members States' ability in NTD, initiated and contributed significantly to the preparation of this document. Certainly, this publication will be useful for other Member States interested in NTD technology.

## **1.2. Purpose and scope**

The general purpose of this publication is to provide comprehensive guidelines in order to start NTD based Si production or modernize/upgrade an existing NTD facility. This document is composed of three major components: basic knowledge of NTD, technical requirements and quality control. The guidance on basic knowledge constitutes a standardization of the concept and theory. In fact, a large part of the theory in NTD and related neutron irradiation is well known. However, standardization of the entire process still needs to be documented and made available in all interested parties. Technical requirements for NTD are a key part in the design of an irradiation facility. As a matter of fact, these requirements will vary depending on the characteristics of each irradiation site of a research reactor. Therefore, in this document, several typical conditions had to be assumed in order to provide more unified description. Finally, the guidance for quality control is intended to define an efficient and constant control

of material irradiation process through all its stages and to ensure a high quality of the final product.

The present publication is applicable to the following types of work which are necessary to initiate and implement NTD:

- Obtaining detailed theoretical knowledge for NTD;
- Designing a new irradiation facility for NTD;
- Maintaining good quality in irradiation results;
- Improving existing facilities to increase the available quantity and irradiation quality;
- Obtaining information on the present status and future prospects of the NTD market.

### **1.3. Structure**

The present report consists of 11 sections including references. Sections 2, 3 and 4 present the definition, necessity, history and future prospects of NTD. The fifth section presents the basic and theoretical explanation for NTD and neutron irradiation. Sections 6 and 7 present detailed guidelines for aligning a neutron irradiation facility for NTD. Section 8 presents the methodology for examining the performance of the facility and neutron irradiation. In section 9, a detailed actual procedure of neutron irradiation for NTD is described. Section 10 presents the documentation procedure which is essential to ensure the quality of the overall process of NTD technology.

## **2. WHAT IS NTD?**

### **2.1. Definition of NTD**

Nuclear transmutation means the change of a nucleus to another or multiple other nuclides through a nuclear reaction. The transmutation is usually instigated by interaction with neutrons, photons or high energy charged particles. If a nucleus absorbs a neutron, the resulting compound nucleus may be in an unstable state and undergo some processes to become stable. If its atomic number is changed in this process, the original atom is changed into a new element and therefore will have different properties.

Doping is a process of intentionally adding a small amount of an impurity into a material to improve its properties for dedicated purposes. Particularly, doping in semiconductor technology is the insertion or creation of impurities in an intrinsic or extrinsic semiconductor material to improve its electrical properties. An intrinsic semiconductor is a pure semiconductor without any significant intentionally introduced dopant species present. Thus, its electrical properties are determined by the properties of the material itself. An extrinsic semiconductor, on the other hand, is a doped semiconductor.

Therefore, NTD is defined as the process by which neutron irradiation creates the impurity in an intrinsic or extrinsic semiconductor to increase its value for various uses [1, 2, 3, 4].

### **2.2. Targets of NTD**

The most prominent target or candidate materials for NTD are Si, Ge, GaAs, GaN, GaP [5], InP [6], InSe [7] and HgCdTe [8]. Among them, silicon and germanium belong to the carbon group, which is group 14 in IUPAC classification in the periodic table.

Each of the elements in this group has four valence electrons in its outermost orbital. The valence electrons of Si or Ge enter into covalent bonds with the valence electrons of neighbouring atoms resulting in the formation of a diamond lattice, i.e. two penetrating face centred cubic Bravais lattices, displaced along the body diagonal of the cubic unit cell by one quarter the length of the diagonal. The covalent bonds in the diamond lattice make Si and Ge a semiconductor or in other words an insulator at low temperatures.

Natural silicon atoms are composed of three isotopes,  $^{28}\text{Si}$  (abundance: 92.23%),  $^{29}\text{Si}$  (abundance: 4.67%) and  $^{30}\text{Si}$  (abundance: 3.10%). Among them, when  $^{28}\text{Si}$  or  $^{29}\text{Si}$  atoms absorb a thermal neutron, they are changed into other stable silicon atoms. The absorption of a fast neutron leads to the direct or indirect (via decay) production of Al or Mg isotopes. The probability of this type of absorption, however, is lower by about two orders of magnitude. The absorption of fast neutrons disturbs the goal of Si doping. It is therefore desirable to suppress it by the provision of a well thermalized neutron spectrum for doping purposes.

In the case of  $^{30}\text{Si}$ , thermal neutron capture leads to the unstable isotope  $^{31}\text{Si}$ , which undergoes beta decay. The product of this process is a phosphorous atom,  $^{31}\text{P}$ , resulting in the n type impurity doping in silicon material. The process is as follows without accounting for the associated antineutrino emission:



The  $^{31}\text{P}$  atom is a stable pentavalent impurity from group 15 in the periodic table. All of the component elements from this group have five electrons in their outermost shell, i.e. n type impurity doping is achieved through this process. The fact that the irradiation of Si with thermal neutrons results only in a single nuclear reaction and the short half-life of  $^{31}\text{Si}$  of only 2.62 h are parameters of crucial importance with respect to the use of the NTD doping technique on an industrial scale.

When natural germanium is irradiated by thermal neutrons, gallium-31, arsenic-33 and selenium-34 atoms are formed by electron capture and some  $\beta^-$  decay. Among these products, the yield of gallium generated by the following reaction is the largest:



Since  $^{71}\text{Ga}$  is a stable trivalent impurity from group 13 in the periodic table, p type impurity doping is possible by the neutron irradiation of germanium [9]. The NTD method for germanium doping demonstrates its superiority in the microdistribution of resistivity (0.7%) compared to reference resistances prepared by metallurgical techniques (4.3%) [10]. Germanium doped by the NTD method is used for the far-infrared p-Ge laser and several sensors [11], including extremely low temperature measurement devices like germanium cryogenic thermistors [12].

Gallium arsenide (GaAs) is an important semiconductor material in the manufacture of devices such as microwave frequency integrated circuits, infrared light-emitting diodes, laser diodes and solar cells. GaAs has some superior electronic properties in comparison with silicon. When GaAs is irradiated by neutrons, germanium or selenium atoms are generated, and the amount of generated selenium is much larger than that of germanium. Thus, n type impurity doping in GaAs is possible by neutron irradiation [13, 14, 15].

Gallium nitride (GaN) can be used to produce an n type semiconductor through two doping processes:



HgCdTeSe is a very useful sensor material, and it can be doped into a p type semiconductor by neutron irradiation using the process:



While many significant commodities are produced from germanium, the main target of NTD is yet silicon. A large commercial amount of NTD is applicable only for silicon because of the following reasons:

- Silicon is the most widely used semiconductor material;
- The demand for an extremely high quality silicon semiconductor useful for power devices and sensors has been significant and is still increasing;
- Si ingots are available in the form of extremely pure perfect single crystals;
- Unlike a pure silicon base, compound semiconductors in certain cases are more difficult to manufacture, primarily in the introduction of the dopant into the correct sub-lattice;
- NTD technology is far superior for the production of a semiconductor with an extremely uniform dopant concentration.

### 3. WHY IS NTD NEEDED?

#### 3.1. Doping of a semiconductor

##### 3.1.1. Intrinsic semiconductors

The electrical conductivity of a semiconductor can be controlled over a wide range either permanently or dynamically. Semiconductor devices are essential in modern technology. Silicon is instrumental as a matrix material to be doped in a majority of semiconductor devices, while other materials, although at lesser extent, are used as well.

An intrinsic semiconductor [16], also called an undoped semiconductor, is a pure material without any significant dopant species present. Elements from the group 14 in the periodic table such as silicon and germanium have four valence electrons, and they are called tetravalent elements. In their crystalline structure (diamond lattice), they form covalent bonds with four adjacent atoms. The periodic lattice of a semiconductor crystalline structure establishes certain energy bands for electrons. The energy of an electron is confined to one of these energy bands. Figure 1 shows a simplified representation of the two uppermost bands in silicon. The lower lying energy bands are of no importance for the electrical conduction

The lower band is called the valence band, in which outer shell electrons are bound to specific lattice sites by the covalent bonding that constitutes the interatomic forces within the crystal. The next higher band is called the conduction band, in which electrons are free to migrate through the crystal. Only these electrons and the corresponding holes (see below) contribute to the electrical conductivity of the material. The two bands are separated by the band-gap. Electrons in the valence band must cross the band-gap to reach the conduction band. This scenario is similar to insulators, but the band-gap for an insulator is usually 5 eV or more, which is much higher than that of semiconductors.

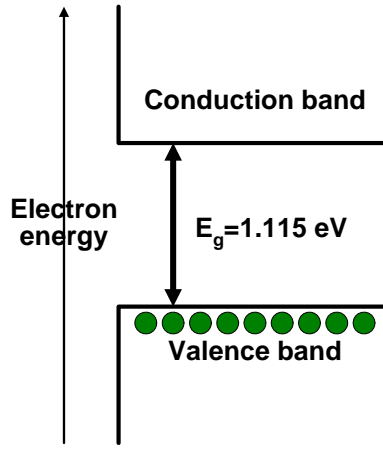


FIG. 1. Simplified representation of the electron energy bands of silicon.

In intrinsic semiconductors at room temperature, just as in insulators, very few electrons gain enough thermal energy to leap over the band-gap. Therefore, intrinsic semiconductors and insulators have roughly the same conductivity at ambient temperature.

If an electron in the valence band leaps over the band-gap, the electron can leave the specific bonding site and drift in the conduction band throughout the crystal. The excitation process not only creates an electron in the conduction band, but also leaves a vacancy called a hole in the valence band, creating what is called an electron–hole pair. The hole can also move under the influence of an applied electric field in the opposite direction of the electron. The mobility of both of these charges contributes to the observed conductivity of the material.

After their formation, both the electron and the hole take part in random thermal motion that results in their diffusion away from their point of origin. The diffusion coefficient  $D$  can be estimated using the following relationship [16]:

$$D = \mu \frac{kT}{e}, \quad (3.1)$$

where  $\mu$  is the mobility of the charge carrier,  $k$  is the Boltzmann constant and  $T$  is the absolute temperature.

If an electric field is applied, the electrons and holes undergo a net migration. In the presence of a low-to-moderate electric field, the drift velocity  $v$  is proportional to the applied field  $E$ . The mobility for both electrons and holes is defined by:

$$v_h = \mu_h E, \quad (3.2)$$

$$v_e = \mu_e E. \quad (3.3)$$

In a completely pure intrinsic semiconductor, the number of electrons in the conduction band must exactly equal the number of holes in the valence band. However, this condition is practically impossible to achieve due to the very small levels of residual impurities. However, neglecting the residual impurities, the concentration of electrons and holes in an intrinsic semiconductor under thermal equilibrium is equal, i.e.

$$n = p = n_i, \quad (3.4)$$

where  $n$  represents the electron concentration in the conduction band,  $p$  represents the concentration of holes in the valence band and  $n_i$  is the material's intrinsic carrier concentration. The intrinsic carrier concentration is dependent on the material and temperature. Table 1 shows several properties of intrinsic silicon and germanium, including their hole and electron densities.

TABLE 1: PROPERTIES OF INTRINSIC SILICON AND GERMANIUM [16]

	Si	Ge
Atomic number	14	32
Atomic weight	28.09	72.60
Stable isotope nucleon numbers	28-29-30	70-72-73-74-76
Density (300 K), g/cm <sup>3</sup>	2.33	5.32
Atomic density, cm <sup>-3</sup>	4.96×10 <sup>22</sup>	4.41×10 <sup>22</sup>
Dielectric constant (relative to vacuum)	12	16
Forbidden energy gap (300 K), eV	1.115	0.665
Forbidden energy gap (0 K), eV	1.165	0.746
Intrinsic carrier density (300 K), cm <sup>-3</sup>	1.5×10 <sup>10</sup>	2.4×10 <sup>13</sup>
Intrinsic resistivity (300 K), Ω·cm	2.3×10 <sup>5</sup>	47
Electron mobility (300 K), cm <sup>2</sup> /V·s	1350	3900
Hole mobility (300 K), cm <sup>2</sup> /V·s	480	1900
Electron mobility (77 K), cm <sup>2</sup> /V·s	2.1×10 <sup>4</sup>	3.6×10 <sup>4</sup>
Hole mobility (77 K), cm <sup>2</sup> /V·s	1.1×10 <sup>4</sup>	4.2×10 <sup>4</sup>
Energy per electron-hole pair (300 K), eV	3.62	
Energy per electron-hole pair (77 K), eV	3.76	2.96

### 3.1.2. Doped semiconductors [16]

A semiconductor's intrinsic electrical properties are often permanently modified by doping. The amount of dopant added to an intrinsic semiconductor determines the level of conductivity. Silicon is tetravalent and its normal crystalline structure forms covalent bonds with four adjacent atoms as shown in Figure 2(a). In this figure, each dot represents a normal valence electron involved in the covalent bond. One of these covalent electrons can be lost by thermal excitation, leaving behind an unsaturated bond or hole. In silicon doped by an element in group 15 of the periodic table, i.e. phosphorus, arsenic or antimony, some silicon atoms in the lattice are substituted by the dopant atoms as shown in Figure 2(b). Because there are five valence electrons surrounding a dopant atom, one electron is left over after all covalent bonds have been formed. This extra electron is only weakly bound to the atom at the original site. From the perspective of the energy bands, it populates a level within the band gap but is separated by only a comparatively small energy (i.e. 44 meV for P in Si [17]) from the conduction band and can be excited at moderate temperature easily into the conduction band to form a conduction electron without a corresponding hole. At normal temperature, virtually all such electrons are excited into the conduction band. Since excitation of these electrons does not result in the formation of holes, the number of electrons far exceeds the number of holes. In this case, the electrons are the majority carriers, and the holes are the

minority carriers. As the dopant atoms have extra electrons to donate, they are called donor atoms. Semiconductor materials doped with donors are called n type semiconductors from the negative charge of the majority charge carrier electrons. Note that each movable electron within the semiconductor has always been donated by an immobile positive dopant ion, and the doped material has a net electric charge of zero.

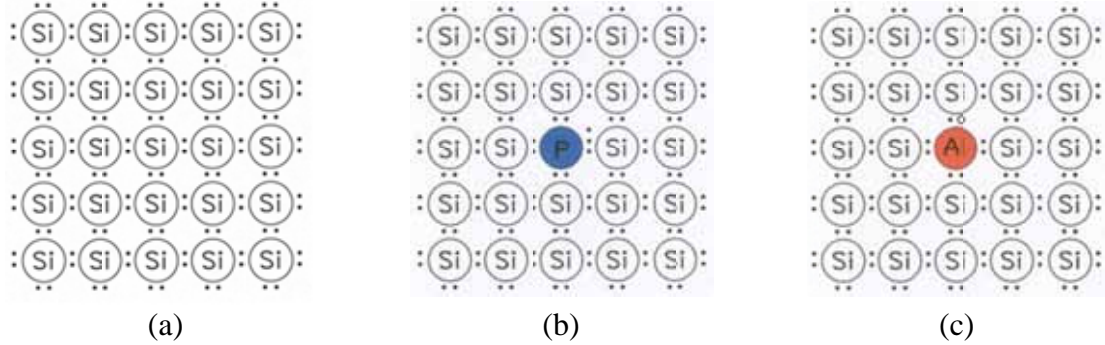


FIG. 2. Schematic representation of the covalent bond of the intrinsic silicon crystal (a), doped silicon crystal with n type impurity (b) and p type impurity (c).

In nearly all cases of n type semiconductors, the donor concentration  $N_D$  is much larger than the electron concentration in the conduction band of the intrinsic material. Therefore, the number of conduction electrons is dominated by the contribution from the donor, and we can write:

$$n = N_D. \quad (3.5)$$

As the number of electrons in the conduction band is much more than the intrinsic value, the number of holes becomes much less due to recombination. At equilibrium, the product of  $n$  and hole concentration  $p$  is the same as for the intrinsic material:

$$np = n_i p_i. \quad (3.6)$$

Intrinsic silicon has  $1.5 \times 10^{10} \text{ cm}^{-3}$  electrons and holes (Table 1). Therefore, as an example, if it is doped by  $10^{17} \text{ cm}^{-3}$  (about 2 parts per million) donor atoms, the density of conduction electrons  $n$  will be  $10^{17} \text{ cm}^{-3}$  and the concentration of holes  $p$  will be in the order of  $10^3 \text{ cm}^{-3}$ . Because the total number of charge carriers is now much greater than in intrinsic silicon, the electrical conductivity of the doped semiconductor is much larger than the one of the corresponding pure (un-doped) material.

The charge neutrality in the doped material is maintained by a balance between the electrons and the immobile ionized donor impurities in the n type semiconductor. The electrical conductivity is determined almost exclusively by the flow of electrons. The resistivity of doped material can be calculated with the dopant concentration and the mobility of the majority carriers.

A p type semiconductor, with positive holes as the dominant charge carrier, is obtained by doping a trivalent element in group 13 of the periodic table, typically boron, aluminium or gallium. This type of doping agent is also called an acceptor. If a silicon atom is replaced by an acceptor atom as shown in Figure 2(c), the missing valence electron leaves one covalent bond among the surrounding silicon atoms unsaturated, i.e. one electron is missing from one



of the four covalent bonds. Analogous to the description given above, for donors the energetic levels of acceptors are located within the band gap but in this case closely above the valence band edge. This vacancy represents a hole similar to that left behind when a normal valence electron is excited to the conduction band. If an electron is captured to fill this vacancy, it participates in a covalent bond, albeit one not identical to the bulk of the crystal because of the trivalent atom. Consequently, the electron is slightly less firmly attached than a typical valence electron. Some electrons are always available to fill the holes created by acceptors by virtue of thermal excitation. These electrons come from other normal covalent bonds throughout the crystal and therefore leave holes behind in the valence band, meaning that an extra hole is created in the valence band for every acceptor atom.

If the concentration  $N_A$  of acceptors is sufficiently large compared to the intrinsic concentration of holes  $p_i$ , then the number of holes is completely dominated by the concentration of acceptors, or:

$$p = N_A. \quad (3.7)$$

By the equilibrium constant discussed above, the equilibrium number of conduction electrons becomes much smaller. In this material, holes are the majority carrier and dominate the electrical conductivity. The filled acceptor sites represent fixed negative charges that balance the positive charges of the majority holes.

Semiconductors doped with donor impurities are called n type, while those doped with acceptor impurities are known as p type. The n- and p type designations indicate which charge carrier acts as majority carrier. The opposite carrier is called the minority carrier, which exists due to thermal excitation at a much lower concentration than the majority carrier.

Degenerately, or very highly, doped semiconductors have conductivity levels comparable to metals. From the point of view of the energy bands, a degeneracy means that the majority carrier levels interact and form a band on their own which merges with the conduction band in case of n type semiconductors or the valence band in case of p type semiconductors. Like in metals, in degenerate semiconductors occupied and un-occupied energy levels are no longer separated by a band-gap. Degenerate semiconductors are often used in modern integrated circuits as a replacement for metal. Superscript plus and minus symbols are often used to denote relative doping concentration in semiconductors. For example,  $n^+$  denotes an n type semiconductor with a high, often degenerate, doping concentration. Similarly,  $p^-$  indicates a very lightly doped p type material.

Even at degenerate levels of doping, the concentration of dopant with respect to the base material is much lower. The doping concentration for silicon semiconductors may be in the range from  $10^{13} \text{ cm}^{-3}$  to  $10^{18} \text{ cm}^{-3}$ . Doping concentrations above  $10^{18} \text{ cm}^{-3}$  is considered degenerate at room temperature. Degenerately doped silicon contains dopant atoms in the order of parts per thousand of silicon.

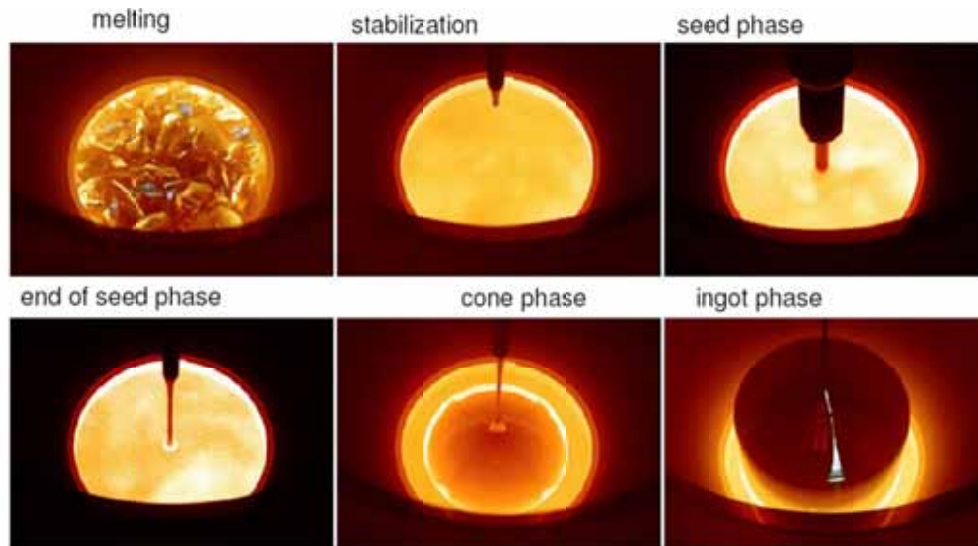
## 3.2. Crystal growth

### 3.2.1. Czochralski process

The Czochralski (CZ) process is a method of crystal growth using a melted material [18]. The most important application of this process may be the growth of large cylindrical ingots of single crystal silicon (CZ-Si). High purity, semiconductor grade silicon, i.e. having only a few

parts per million of impurities is melted in a crucible, which is usually made of quartz. Subsequently, a seed crystal is touched to the molten silicon and a crystal ingot is pulled by slowly withdrawing the seed as shown in Figure 3 and Figure 4.

For homogeneity, the seed crystal and the crucible are rotated. The small single crystal seed is mounted on a rotating axis and put in contact with the surface of the liquid. The rotation speed can vary from 2 to 50 rpm (rotations per minute). The temperature gradients are adjusted in such a way that crystal growth occurs at the surface of the seed. Crystals with diameters up to 50 cm are obtainable by this method [20].



*FIG. 3. The phases of crystal growth during the Czochralski process [19].*



*FIG. 4. A 200 mm Czochralski crystal pulled from a melt. [19].*

As the crystal is grown, the impurity concentration including the dopants will increase in the melt due to segregation, and therefore some portion of the impurities is incorporated into the crystal. The temperature profile of the whole system will also change. All these factors influence the homogeneity and the radial and lateral doping level of the crystal. In this process, an especially important impurity is interstitial oxygen. In general, the concentration of oxygen increases from 'head' to 'tail'.

Semiconductors made through this method are mainly used in low power semiconductor devices such as personal computers. In this method, the crucible is a major source of pollution. At the melting point of silicon, 1418°C, the crucible always chemically attacks and pollutes the melt. Due to impurities from the crucible and dissolved oxygen, the resulting semiconductor has relatively low purity and quality. Also, it is difficult to obtain a semiconductor with a resistivity larger than several tens of  $\Omega\text{cm}$  by this method.

Impurity atoms can be added to the molten intrinsic silicon in precise amounts for the purpose of doping. Since the dopant distribution in the axial direction varies, a proper part of the grown crystal is chosen and cut. In the radial direction of the crystal, the semiconductor grown by this method shows variations in doping concentration in a rather regular pattern known as striation. This is because the temperature of the crystal surface varies by convection of the molten silicon in the crucible. Since the temperature gradients in the crucible cannot be avoided, there will be some convection in the liquid silicon.

In order to suppress the natural convection and pollution within the crucible, the semiconductor crystal can also be grown under a strong magnetic field resulting in the product called Magnetic-Field-Applied Czochralski Silicon (MCZ-Si) [21]. Since the molten silicon has metallic properties, it undergoes convective motion under the magnetic field. The intensity of the applied magnetic field is about 50–4000 G. In this case, it is possible to compensate for the original convection of the CZ method. Therefore, non-uniformity of resistivity in the radial direction can be much smaller than with the CZ-Si, although the dopant distribution in the axial direction is as large as in the CZ type. Since a high purity silicon crystal with about several thousand  $\Omega\cdot\text{cm}$  can be obtained by this method, if NTD is applied to MCZ-Si, a silicon semiconductor with a very uniform dopant distribution is obtainable. Floating zone silicon (FZ-Si) in several power devices could be replaced by MCZ-Si. MCZ-Si is also used for charge coupled devices (CCDs) and highly integrated circuits.

### *3.2.2. Floating zone method*

The floating zone method is a crystal growing method that ensures a very high purity. When the molten liquid silicon is in contact with solid silicon, the impurity atoms will diffuse in a process called segregation to the liquid region since they prefer to sit in the liquid phase. Thus, by passing a boule of silicon through a thin section of a furnace very slowly such that only a small region of the boule is molten at any time, the impurities will collect in the molten region and migrate with it as the boule is moved through the heat zone. This is called the zone refining method developed by William Gardner Pfann, and the concept is shown in Figure 5.

### Float-zone pulling

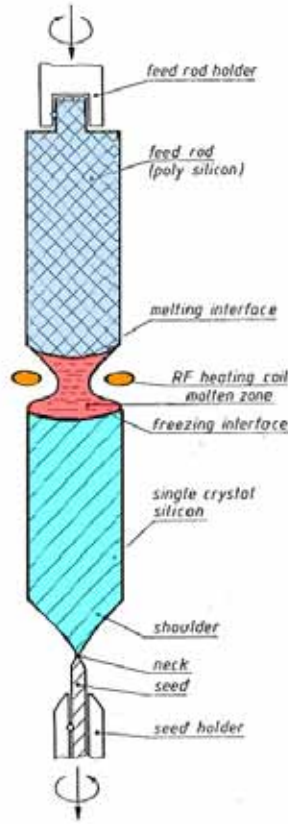


FIG. 5. Concept of the floating zone method [22].

The narrow part of the ingot is heated by a coil attached to a high frequency generator. The local induction heating forms a narrow molten zone. By slowly moving the heating coil upward, a slow displacement of the molten zone takes place followed by solidification. Once the whole polycrystalline ingot has been passed through by the molten zone, a single crystal ingot is obtained. By repeating this process one can obtain an extremely pure crystal.

The ratio of the impurity concentration in the liquid phase  $c_L$  to that in the solid phase  $c_s$  is represented by the segregation coefficient  $K$ , i.e.:

$$K = \frac{c_L}{c_s}. \quad (3.8)$$

In general,  $K$  is much larger than unity because impurities are more soluble in the liquid than in the solid phase. When the impurity concentration in the starting ingot is  $c_0$ , the initial molten zone also has impurity concentration  $c_0$ , but the first solid segment obtained after the passage of the molten zone has the concentration given by:

$$c' = c_0/K. \quad (3.9)$$

Since  $K > 1$ , the solid segment has a lower impurity concentration than at its initial state. As the molten zone progresses, it becomes enriched with impurities until its impurity concentration reaches the product of  $K$  and  $c_0$ . Further motion of the molten zone does not give additional purification.

After the first passage of the molten zone is completed, the ingot is cooled down. A second passage can be started from the bottom of the ingot with the lowered impurity concentration. Many passages can be made, starting always at the bottom of the ingot. Eventually the impurity concentration profile reaches a steady state which cannot be improved upon by further passages [20].

Since the melt never comes into contact with anything but vacuum or inert gases, there is no incorporation of impurities in the melt. This is especially vital considering oxygen impurities, which cannot be avoided with CZ crystals. FZ crystals therefore are always used when a very low oxygen concentration is important, but FZ crystal growth is limited in terms of crystal diameter.

Silicon crystals are basically grown by one of the two methods; CZ-Si is by the Czochralski method and FZ-Si is by the floating zone method. While FZ-Si has much a higher crystal quality than CZ-Si, CZ-Si occupies more than 90% of the silicon market. The diameters of these crystals have become larger as the process has been refined. The largest crystal diameter of the major CZ-Si market recently jumped from 8 to 12 inches. For the case of FZ-Si, the major size had been 5 and 6 inches for several years, but recently 8 inch FZ-Si has been seen.

For the case of CZ-Si, the dopant is mixed with silicon before crystal growing. Then, the dopant distribution in the silicon crystal is less uniform than in FZ-Si due to the aforementioned striation. Also, the longitudinal variation of dopant concentration gradually increases or decreases over the axial length of CZ-Si, which limits the usage of the entire ingot for applications with a tight resistivity tolerance. The incorporation of oxygen in CZ-Si is much higher than in FZ-Si as well.

FZ-Si shows much higher quality than CZ-Si, and gas diffusion doping for FZ-Si (DW-Si) results in much more uniform resistivity than for CZ-Si as well. Currently, NTD is applied mainly to FZ-Si (NTD-Si) in order to attain more uniform resistivity than DW-Si. As the technology developed, resistivity uniformity of DW-Si was enhanced, which was the reason that NTD demand decreased during the 1990s. NTD-Si holds about 20% of the current FZ-Si market.

## **4. HISTORY, PRESENT STATUS AND FUTURE PERSPECTIVES OF NTD-SI**

### **4.1. History of NTD-Si**

#### *4.1.1. The beginning stages of NTD*

The possibility of producing silicon semiconductors with perfectly uniform phosphorus dopant distribution by neutron transmutation was first identified and described by Karl Lark-Horovitz in 1951 [23]. In 1961, Morris Tanenbaum and A.D. Mills at Bell Telephone Laboratories carried out the first NTD experiment [24], by irradiating small pieces of silicon with neutrons while measuring the phosphorus distribution.

While the NTD technique had been known and practiced for a decade in research laboratories, it was not commercially used to any significant extent until the mid-1970s. In 1973, a German high power device manufacturer used NTD-Si for the production of thyristors, which was a practical commercial breakthrough [25]. Due to the superiority of the resulting device

characteristics, between 1974 and 1976, NTD was offered by a number of research reactors in the USA, England and Denmark, and quickly the amount of irradiated silicon reached several tonnes.

At the Danish reactor DR2 of RISØ National Laboratory, the first industrial production of a 2 inch diameter silicon ingot began in 1974 in cooperation with the Danish company Topsil of Frederikssund [26]. They were called T-silicon, which was the registered trademark of commercial transmutation doped silicon. Two inch silicon ingots were irradiated in 53 mm diameter aluminium cans in a horizontal graphite stringer in the reactor's thermal column. Upon the final shutdown of DR2 in November 1975, about 100 kg silicon had been irradiated. Afterwards an irradiation facility for a 3 inch silicon crystal was constructed for the DR3 reactor, and its commercial service started in 1976. Additional irradiation facilities were constructed in 1977 and installed in the graphite reflector surrounding the heavy water tank. In 1997, they installed an additional 5 inch irradiation facility [27]. DR3 continued irradiations of 3, 4 and 5 inch ingots and produced a large amount of the NTD-Si supply until it was permanently shut down in 2000 because of a small leak of heavy water.

In the United Kingdom, NTD was first undertaken by the Materials Test Reactors DIDO and PLUTO at the Harwell site of the United Kingdom Atomic Energy Authority (UKAEA) in 1975 [28]. Following the first successful trial for a silicon irradiation in 1975, the amount of NTD production increased from 2 t during 1976 to a level of around 30 t in 1985. According to the Harwell reactor's report in 1987, the demand for NTD had rapidly risen, and that the application fields of NTD Si had been extended. In order to satisfy an even higher demand in the future, the Harwell reactors had prepared for a capacity of about 35 t per annum [29]. Then in early 1990, the DIDO and PLUTO reactors at the Harwell site were shut down. At that time, about 206 t of the silicon had been doped in these reactors over the course of 15 years [30]. To compensate for the shutdown of the Harwell reactors, a joint venture was set up between AEA Technology (the trading name of the UKAEA) and the Centre for the Study of Nuclear Energy (SCK/CEN) involving silicon irradiation in the Centre's BR2 reactor in Mol, Belgium. A silicon irradiation facility named Silicon Doping by Neutron Irradiation Experiment (SIDONIE) with an annual capacity of 28 t was designed and installed for this reactor in early 1992 [31, 32].

In Switzerland, the first irradiations in the SAPHIR reactor were carried out as early as 1975. The total annual quantities remained modest at less than 200 kg until about 1990, but in 1992, NTD activity was stepped up to about 2 t of silicon [33].

MURR, a 10 MW open pool reactor at the University of Missouri, USA, has performed NTD actively, starting with several test runs in 1975. It was reported that the reactor's capacity in 1978 was on the order of 15 t with 50  $\Omega\cdot\text{cm}$  resistivity, and that it could be enhanced to 60 t with some modification [34].

In Japan, NTD tests began in 1975 in compliance with a local semiconductor company's request, and commercial service began in 1977. Irradiation rigs in the JRR-4 reactor were developed for 2.5, 4 and 5 inch silicon ingots of 45 cm length. JRR-2 had irradiated silicon up to 3 inch ingots from 1983 till its shutdown in 1996.

In the Russian Federation, NTD semiconductor technology was first realized in the early 1970s based at the WWR-C nuclear reactor developed at the Obninsk Branch of the Karpov Scientific Research Institute of Physical Chemistry. In 2003, several tonnes of NTD-Si and other semiconductor materials were processed at this facility along with simultaneous

development and testing of different NTD procedures, while several other institutes were also participating in NTD [35].

HIFAR in Australia had irradiated a relatively large amount of silicon ingots for many years [36] until its final shutdown in 2007.

In Seibersdorf, Austria, the ASTRA, a 10 MW swimming pool type reactor was in parallel to scientific applications intensively used for Si doping until its final shut down in 1999. According to an Austrian press release in 1991 about 5 % of the global demand of NTD Si was produced in Seibersdorf. In the former German Democratic Republic the RFR, a 10 MW WWR-SM reactor of the former USSR design was used for isotope production but also for Si doping. The reactor was finally shut-down in 1991 and has been decommissioned meanwhile.

#### *4.1.2. Major reactors for NTD at present*

BR2 in Belgium has been an active reactor for NTD irradiation. This reactor underwent a refurbishment shutdown for about two years and then resumed operation in April 1997 [37]. The irradiation facility for silicon installed in 1992 was for ingots of 4 and 5 inches in diameter. In anticipation for the shifting of the crystal diameter to larger sizes, they recently realized a larger irradiation facility [32, 38].

JRR-3M in Japan has a 17 cm diameter irradiation hole for NTD. The irradiation of 5 inch ingots began in 1990, and it was reconstructed to irradiate 6 inch ingots throughout 1994 and 1995. It can irradiate ingots up to 60 cm in length. The amount of NTD-Si produced was about 3.7 t at JRR-3 and about 0.7 t at JRR-4 in 2006 [39].

In the Republic of Korea, the HANARO reactor at the Korea Atomic Energy Research Institute (KAERI) has two vertical holes in the heavy water reflector region for NTD. The development of an NTD irradiation facility began in 2001, and commercial service at a smaller hole began at the end of 2002 using 5 inch ingots. In 2005, service for 6 inch ingots also began at the same hole. The irradiation record in 2007 totalled about 15 tonnes in spite of the reduced operation time of the reactor. From 2007, 6 and 8 inch irradiation facilities have been studied at a larger hole [40].

OPAL, a new reactor in Australia has six vertical irradiation holes in the heavy water reflector vessel for 5 inch (one hole), 6 inch (three holes) and 8 inch (two holes) ingot irradiations. The possible ingot diameters are 4-8 inches. It uses a combination of water bearing and drive rotation utilizing reactor pool water. OPAL achieved its full power rating of 20 MW at the end of 2006 [41].

South Africa's SAFARI-1 research reactor has a silicon irradiation facility installed in the pool side region next to the reactor core. The commissioning of the facility took place in 1992, and it has been upgraded and improved to meet the growing international demand for NTD-Si. An initial test irradiation was performed for 4 inch ingots. In 2008, it was redesigned to accommodate a maximum diameter of 6 inches and a maximum length of 600 mm [42, 43].

FRM-II in Germany was successfully commissioned in 2004 and 2005. An automatic doping system in FRM-II was designed to accommodate up to 8 inch silicon ingots exhibiting a total stack length of 500 mm. FRM II offers a single irradiation channel. In order to allow 5 inch and 6 inch ingots to be irradiated suitable Al-spacers are used to minimize the gap within the standard 8 inch irradiation basket. Irradiation service began in 2007 [44], and that year, a total

of 3.8 t was irradiated by the semiautomatic silicon doping facility, which increased its output to 9.8 t in 2008 [45] and about 15 t in 2010.

#### *4.1.3. Other reactors related to NTD*

Other reactors that have some activities related to NTD or plan to carry out similar irradiation services are as follows:

- MITR-II (5 MW): Massachusetts Institute of Technology in US [46]
  - Two horizontal through ports
  - 4TH1-3: 4 inch crystals, 6TH1-2: 4, 5, 6 inch crystals
  - 400 mm long magnesium cans
- MURR (10 MW): Missouri University Research Reactor in US
- IRT-T (6 MW): Tomsk Polytechnic University in the Russian Federation [47]
  - 150 mm in diameter, horizontal GÉK-4 experimental channel
  - Reciprocal motion at a velocity of 270 mm/min passing through Be reflector
  - In 1995, capacity of ingots with 12.7 cm in diameter: 4.5 t/yr
- JEEP II (2 MW) in Norway [48] [Annex]
- HFR (45 MW) in the Netherlands [48] [Annex]
- MARIA (30 MW) in Poland [Annex]
- SPR IAE (3.5 MW), HWRR-II (15 MW) and CARR (60 MW): the Institute of Atomic Energy (CIAE) in China [Annex]
- SPRR-300 (3 MW): Tsinghua University in China
- HFETR (125 MW) [Annex], MJTR (5 MW): the Nuclear Power Institute of China
- CIRUS (NTD test, 40 MW) in India
- ETRR-2 (22 MW) in Egypt [49]
  - Conception for 3 and 5 inch crystals with resistivity lower than 50  $\Omega\text{cm}$ , using two vertical holes in the thermal column
- LVR-15 (10 MW) in the Czech Republic [Annex]
- TRR (5 MW) in the Islamic Republic of Iran
  - Conception for ingots up to 10 cm in diameter [50]
- IEA-R1 (5 MW) in Brazil [Annex]
  - Suitable for ingots of 5 inches up to 500 mm in length, used for low resistivity Si of 30  $\Omega\text{cm}$ , capacity 1.2 t/year [51, 52]
- OSIRIS (70 MW) and ORPHEE (14 MW) in France [Annex]
  - 4 irradiation channels for ingots of 4 and 6 inches [31]

The reader should note that the above list should be considered as not fully complete, i.e. other research reactors involved in NTD of Si technology do exist according to the existing information available through the IAEA RRDB [48], i.e. in Kazakhstan, the Russian Federation, Ukraine, USA, and Uzbekistan.

#### *4.1.4. NTD conferences and meetings*

The first formal conference associated with neutron transmutation technology was organized in 1976 by John Cleland and Richard Wood. It had become clear that the NTD process would be implemented on a commercial scale to prepare uniformly doped starting material for the power device industry. The Second International Conference on Neutron Transmutation Doping in Semiconductors was held on 23–28 April, 1978 at the University of Missouri in Columbia, Missouri. Various aspects of NTD-Si technology were discussed at this conference including irradiation technology, radiation induced defects and the use of NTD-Si for device



applications. The Third International Conference on Neutron Transmutation Doped Silicon was held in Copenhagen, Denmark on 27–29 August, 1980. The Fourth International Neutron Transmutation Doping Conference was held at the National Bureau of Standards in Gaithersburg, Maryland on 1–3 June, 1982. In addition to the conferences, an IAEA Consultants Meeting on Silicon Transmutation Doping Techniques and Practices was held at the Institute of Atomic Energy, Otwock-Świerk, Poland, during 20–22 November 1985, resulting in the publication of IAEA-TECDOC-456 in 1988 [4]. Unfortunately, this publication is neither available electronically nor for additional printing.

From the related literature, we can confirm that in the early stage of NTD technology, neutron irradiations not only for FZ-Si but also Ge, GaAs, etc., had been examined. Also, as a possible way to overcome resistivity variations, mainly axial resistivity variations in Czochralski grown silicon ingots, additionally controlled neutron irradiation for CZ crystals had been proposed.

Most probably due to the growing commercial importance of Si doping to the research reactors and to the related non-disclosure agreements between the reactors and the Si suppliers no more topical meetings dedicated exclusively to NTD of semiconductors were held in more recent years. However, individual contributions dealing with NTD continue to be presented during the following conferences:

- International Conference on Isotopes (ICI);
- Research Reactor Fuel Management (RRFM);
- Meeting of the International Group of Research Reactors (IGORR);
- International. Conference on Research Reactors: Safe Management and Effective Utilization.

#### *4.1.5. A shift of the worldwide NTD capacity and demand*

The estimated worldwide production rate of NTD-Si in 1982 was approximately 50 tonnes per year [53]. According to future prospects at that time [54], with slow economic growth, demand for NTD-Si in the 1990s could be 75–80 tonnes per year, whereas with higher world economic growth and the reasonable probability of innovation in applications, demand could be in the range 100–120 t. Since it was impossible to predict the exact demand, different rates of market growth, 3, 5 and 7% per annum were applied. It was expected that the demand could increase to about 150 t until 2000, at which point the 7% increase rate of the market growth was applied. Considering the irradiation capacity of reactors, while about 50 t was estimated in 1982, a potential capacity up to 110 t was expected. Subsequently, beyond the early 1990s, it was expected to decrease since the primary reactors responsible for production would be approaching an average age of more than 30 years [54].

The actual demand for NTD in 1991 reached about 160 t with a growth rate about 10% per annum, which was higher than the above prediction, yet this increased demand was accommodated by the research reactors. Following 1992, the demand for NTD decreased to 70 t in 1999 because of developments of MCZ- and improved FZ-Si technologies that could substitute for the NTD process. The demand for NTD rebounded after 2000, and currently it is more than 100 tonnes per year with a growing trend that is roughly 20% of FZ-Si production.

In 1982, since a shortage of irradiation capacity was predicted, a reactor dedicated to silicon irradiation was examined [54]. An average annual cost of operating a medium power research reactor, without the capital cost or depreciation of the facility, was estimated to be \$6.5 M,

which consisted of \$5 M for operation and \$1.5 M for fuel. Assuming 6–7 cents/g for the price charged for irradiation, about 100 t/year would have to be irradiated to support the upkeep of the reactor alone. Since a tenth of this amount was the practical quantity at that time, it was concluded that silicon irradiation must be carried out in conjunction with a large contingent of other activities.

The current NTD market is dominated by 5 and meanwhile even more important 6 inch ingots. 2.5 inch ingots and 3 inch ingots are rarely found in the current market. The share of 4 inch ingots has been decreasing drastically. Recently, the demand for irradiation of 8 inch ingots has grown, and its market share could expand in the future.

At present, since the typical diameter of silicon ingots has become much larger, a large amount of silicon can be irradiated at an irradiation site. It may not be easy for many existing reactors to make new larger irradiation holes, but their inclusion in the design of new reactors would be possible and will be necessary to satisfy the demand of the semiconductor industry. As found at, i.e. OPAL, but also at FRM II and BR2 — all of them providing irradiation facilities for ingot diameters up to 8 inch — a large scale NTD facility may be possible while satisfying other needs of a research reactor. After all, the construction of a large irradiation capacity for NTD could be profitable, though it is closely dependent on industrial demand for NTD-Si.

The CZ-Si market has jumped from 8 to 12 inches. Therefore, the next wave of NTD ingots would be 12 inches rather than in between 8 and 12 inches such as a 10 inch ingot unless special processing lines for NTD ingots and wafers are justified. It seems that the industry is now developing technology for 18 inch CZ-Si production but NTD for 18 inch ingots may be difficult to satisfy not only in terms of irradiation uniformity but also accommodation at reactors.

Besides the increasing diameter of Si ingots to be irradiated it has to be noted that an increasing demand for high target resistivity up to  $>1000 \Omega \cdot \text{cm}$  NTD Si arose in the last few years. On the one hand this development is connected with an increasing capacity because high targeted resistivity is achieved by short irradiation times. On the other hand for this type of NTD Si high quality neutron sources providing excellent thermalized neutron spectra are required because the effect of extended irradiation defects by fast neutrons on the electrical properties of a semiconductor is much more important at high resistivity

## **4.2. Parameters for the future of NTD-Si**

### *4.2.1. Future demand and supply*

As mentioned above, the worldwide demand for NTD had increased rapidly up to the early 1990s. During the 1990s, the demand decreased due to the development of other competitive technologies, and remained stagnant until the mid-2000s at about 100 t/year. However, since then NTD has been facing a new circumstance. The demand for high power semiconductors may increase rapidly according to the rapid increase of alternative and much wider use of ‘green energy’ technologies.

One significant example is the quickly expanding production of hybrid electric vehicles. Interest in electric vehicles, including plug-in hybrid cars and fuel cell engines, is also surging. In addition to transportation, electricity generation by wind, solar and fuel cell systems is increasing rapidly. These advances are, in principle, motivated by efforts to decrease the

discharge of air pollution, especially carbon dioxide, which is supposed to be the major cause of global warming. Furthermore, various activities to increase efficiency in electricity utilization like smart grid projects have been proposed. Each of these fields requires high quality power devices, and NTD can supply the best base material to manufacture them.

From the aspect of supply, the ingot diameter has become larger, and recent new reactors are apt to possess a large NTD capacity. Irradiation of large diameter ingots allows for a large NTD capacity from the same number of irradiation holes. The 6 inch ingot has become the major product in the NTD market, and the share of 8 inch ingots will gradually increase. Meanwhile, shutdowns of aged research reactors serving NTD will continue. In 1999 ASTRA in Seibersdorf, Austria, as well as in 2000 DR3 in Denmark were shut down, and then the R2 reactor in Sweden was shut down in 2005. None of them was replaced so far. Although many research reactors currently serving NTD are aged, world-wide NTD capacity will likely increase because replacement reactors, even at smaller number than shutdown facilities, will have a larger and better optimized capabilities than their predecessors.

A very rough but more positive projection of NTD is as follows: in 2005, the worldwide production of new cars was about 60 million, and could be more than 100 million in 2030. If 50% of new cars will contain hybrid, electric or fuel cell engines in 2030, then the number of new cars equipped with electric motors is assumed to be about 50 million. If one 6 inch NTD silicon wafer is used per car, since a silicon wafer requires about 1 mm thickness including the sawing part, NTD-Si of about 2,000 ton per annum would be needed to satisfy this demand. It is not certain whether this future demand will be met by reactors. If the supply of NTD-Si is not sufficient, the industry may use another, likely more expensive, technology.

At any rate, since the pressure on industry from environment groups is becoming stronger, 'green energy' technology could rapidly expand. Therefore, it is likely that the future demand for NTD will increase.

#### *4.2.2. Development of other technologies*

##### *(1) Silicon carbide (SiC)*

Theoretically the most ideal semiconductor material for a power device is a diamond, but it is expensive and would not be practical. At the moment, a wide band gap semiconductor such as SiC is considered to be one of the most promising candidates that can replace the pure silicon semiconductor. SiC has properties in the middle of the range between silicon and diamonds. Power devices made from SiC could have electrical characteristics approaching those of the ideal device. SiC is a ceramic compound of silicon and carbon that is manufactured on a large scale for various applications from semiconductors to even nuclear fuel. In their use as a power semiconductor, SiC Schottky diodes with a breakdown voltage of 1200 V are commercially available, and they can be operated for high speed switching. Bipolar devices made of SiC are being developed for higher voltages, up to 20 kV. Among its advantages, SiC devices can be operated at higher temperatures, up to 400°C, and have a lower thermal resistance than silicon devices, which allows for better cooling.

SiC does not have a liquid phase available for the growth of single crystals by similar silicon crystal methods. To make the SiC crystal, a crystal growing method from gas phase to solid state should be used, although this is much more difficult and expensive than the silicon crystal method. In summary, even if the manufacturing method of SiC makes great strides, and the use of devices made of SiC is extended, SiC devices will hardly encroach on the

territory of the silicon based devices. They will probably be used in the range of a very high frequency or a very high power that silicon semiconductors cannot cover.

## (2) Epitaxial growth

The epitaxy is a kind of interface between a thin film and a substrate, and epitaxial growth describes a process of depositing a thin layer, typically 0.5 to 20 microns, of single crystal material over a single crystal substrate, usually through the chemical vapour deposition method. The epitaxy enables various applications in the semiconductor fabrication, and it can be the source of high crystalline quality growth for use in many semiconductor materials. The epitaxy is also used to grow layers of pre-doped silicon on the polished sides of silicon wafers before they are processed into semiconductor devices. An epitaxial layer can be doped during deposition by adding dopant to a source gas such as arsine, phosphine or diborane. By growing a lightly doped epilayer over a heavily doped silicon substrate, a high breakdown voltage across the collector–substrate junction is achieved while maintaining a low collector resistance. Lower collector resistance allows a higher operating speed at the same current. This epitaxial growing method is utilized for a large portion of the modern power device fabrication process, for example, in the punch through insulated gate bipolar transistor (PT-IGBT) fabrication procedure. However, since the thickness of an epitaxial layer is limited, the areas in which the epitaxy is applied also remains limited.

These two competing technologies have their own weaknesses compared with the NTD method at present. While SiC is an ideal material for power devices, the associated technology has not matured for mass production so far. For epitaxial growth on silicon wafers, growing an epitaxial layer with enough thickness for sufficient endurance against high voltages is very difficult. Consequently, SiC or epitaxial wafers presently are much more expensive than NTD wafers. Their higher cost would not allow competition in the market, in which other lower cost devices are available, but they may establish their own market where other substitutes are not available, similar to the role of NTD in the silicon market. However, since in the future these technologies could have a great influence on the NTD market, their progress or development, including new technologies, should be carefully observed.

## 5. NEUTRON IRRADIATION AND RESISTIVITY

### 5.1. Resistivity in semiconductors

While only the flow of negatively charged electrons can contribute to the electrical conductivity of a metallic conductor, both electrons and positively charged holes contribute to the conductivity of a semiconductor. A measure of a material's ability to conduct an electric current is represented by either conductivity mostly denoted  $\sigma$  or its inverse, resistivity, denoted by  $\rho$ . Resistivity is widely used, and it is determined by the intrinsic carrier density and mobility. If we have a slab of a semiconductor with thickness  $t$  and surface area  $A$ , the current  $I$  that will flow, when a voltage  $V$  is applied across the thickness is [16]:

$$I = \frac{AV}{\rho t} \quad \text{or} \quad \rho = \frac{AV}{It}. \quad (5.1)$$

The current is composed of the flow of holes  $I_h$  and electrons  $I_e$ . Although these two types of charge carriers move in opposite directions, the separate currents are additive because of the

opposite charges of holes and electrons. Thus an external measurement of the current alone cannot distinguish between the flow of holes or electrons. The total observed current in an intrinsic semiconductor will be their sum, with each term given by the product of area  $A$ , intrinsic carrier density  $n_i$ , electronic charge  $\varepsilon$  and drift velocity  $v$  of the charge carrier. Thus,

$$I = I_e + I_h = An_i\varepsilon(v_e + v_h). \quad (5.2)$$

From Eqs. (3.2), (3.3), (5.1) and (5.2),

$$I = An_i\varepsilon E(\mu_e + \mu_h) = An_i\varepsilon \frac{V}{l}(\mu_e + \mu_h). \quad (5.3)$$

Then, the resistivity becomes,

$$\rho = \frac{1}{n_i\varepsilon(\mu_e + \mu_h)}. \quad (5.4)$$

For an intrinsic silicon at room temperature,

$$\rho = \frac{1}{(1.6 \times 10^{-19} \text{ C})(1.5 \times 10^{10} / \text{cm}^3)(1350 + 480 \text{ cm}^2 / \text{V} \cdot \text{s})}, \quad (5.5)$$

$$\rho = 2.3 \times 10^5 \text{ V} \cdot \text{s} \cdot \text{cm} / \text{C} = 230,000 \text{ } \Omega \cdot \text{cm}.$$

This is the maximum resistivity which is theoretically obtainable. In practical silicon material, this resistivity cannot be achieved due to residual impurities. A typical initial resistivity of Si to be irradiated for NTD purposes ranges between 3000 and 20 000  $\Omega\text{cm}$ .

When impurity atoms are introduced into an intrinsic semiconductor material, the densities of the charge carriers and impurities are represented by the charge neutrality [55]. Thus, most donors and acceptors are ionized at room temperature, so the neutrality condition can be approximated by

$$n + N_A = p + N_D, \quad (5.6)$$

where  $n$  and  $p$  are the electron and hole densities, and  $N_A$  and  $N_D$  are the acceptor and donor densities, respectively. Since the following product of electron and hole densities is always independent of the added impurities, thus

$$np = n_i p_i = n_i^2 = p_i^2 \quad (5.7)$$

where  $n_i$  and  $p_i$  are the intrinsic carrier densities. Combining above two equations, in an n type semiconductor where  $N_D > N_A$ ,

$$n = \frac{(N_D - N_A) + \sqrt{(N_D - N_A)^2 + 4n_i^2}}{2}. \quad (5.8)$$

If  $|N_D - N_A| \gg n_i$  or  $N_D \gg N_A$ ,

$$n \approx N_D, \quad p \approx \frac{n_i^2}{N_D}. \quad (5.9)$$

Similarly, carrier concentrations in a p type semiconductor ( $N_A \gg N_D$ ) is given by

$$p \approx N_A, \quad n \approx \frac{n_i^2}{N_A}, \quad (5.10)$$

Therefore, the final resistivity of silicon after n type doping is as follows,

$$\rho = \frac{1}{en\mu_e}. \quad (5.11)$$

As an example, assuming a silicon sample with a donor density of  $10^{13} \text{ cm}^{-3}$ , then the resistivity will be

$$\begin{aligned} \rho &= \frac{1}{eN_D\mu_e}, \\ \rho &= \frac{1}{(1.6 \times 10^{-19} \text{ C})(1.0 \times 10^{13} / \text{cm}^3)(1350 \text{ cm}^2 / \text{V} \cdot \text{s})}, \\ \rho &= 463 \text{ } \Omega \cdot \text{cm}. \end{aligned} \quad (5.12)$$

## 5.2. Doping concentration for NTD-Si

The resistivity of irradiated silicon is inversely proportional to the total concentration of the produced dopants such as  $^{31}\text{P}$  atoms and initially existing impurities. In NTD, the added  $^{31}\text{P}$  concentration is proportional to the irradiated neutron fluence, which is a product of the neutron flux, time of irradiation with a constant neutron flux and the reaction cross-section. As the neutron cross-section varies by neutron energy, it is influenced from the neutron spectrum in the irradiation site. In the semiconductor industry, the resistivity rather than the dopant concentration is usually used. Therefore, the relationship between the resistivity and the dopant concentration should be established first.

For n type silicon doping with phosphorous, the resistivity ( $\Omega\text{cm}$ ) is given by

$$\rho = \frac{1}{[P]\mu_e}, \quad (5.13)$$

where  $[P]$  is the phosphorus atomic concentration in  $\text{cm}^{-3}$ ,  $e$  is the electron charge,  $1.602 \times 10^{-19} \text{ C}$ , and  $\mu$  is the drift mobility of the electrons in the silicon crystal lattice. Electron mobility depends on the temperature, and it is in the range of  $1220 \sim 1500 \text{ cm}^2 / \text{V} \cdot \text{s}$ . In normal conditions at  $300 \text{ K}$ , it is usually  $1350 \text{ cm}^2 / \text{V} \cdot \text{s}$  for silicon as shown in Table 1. Then we can easily obtain the relationship between the dopant concentration and the resistivity:

$$\rho = \frac{K_P}{[P]} \quad \text{for phosphorus doping}, \quad (5.14)$$

$$\rho = \frac{K_B}{[B]} \text{ for boron doping,} \quad (5.15)$$

where  $K_P$ ,  $K_B$  are the proportional constants and  $[B]$  is the boron atomic concentration in  $\text{cm}^{-3}$ .

Figure 6 shows the relationship between dopant concentration and resistivity. The resistivity is basically inversely proportional to the doping concentration, but in the heavily doped range, this relationship is broken as shown in Figure 6. The difference between the resistivity of p type and n type silicon is due to the difference in the mobility of the electrons and holes. Since the minimum resistivity practically obtainable through NTD is regarded as several ohms due to the residual radioactivity of an irradiated ingot, NTD-Si is in the region of proportionality. Thus, Eqs. (5.14), (5.15) can be applied to NTD irradiation.

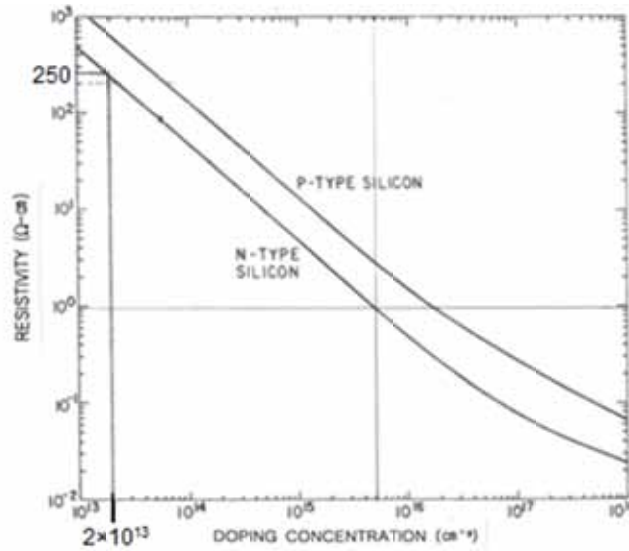


FIG. 6. Relationship between doping concentration and resistivity.

At the Japan Atomic Energy Agency (JAEA), previously Japan Atomic Energy Research Institute (JAERI), the following constants were used [56]:

$$\rho = \frac{5 \times 10^{15}}{[P]} \text{ for n-type, } \rho = \frac{1.3 \times 10^{16}}{[B]} \text{ for p-type,} \quad (5.16)$$

while at the Harwell reactors [28], the following constant was used:

$$\rho = \frac{4.63 \times 10^{15}}{[P]}. \quad (5.17)$$

In order to determine the amount of impurity needed to attain a target resistivity  $\rho_t$ , first of all, the concentration of the impurities already existing in the crystal should be known, which is usually represented by an initial resistivity  $\rho_i$ . Then we can easily deduce the additional dopant concentration needed to achieve the target resistivity:

$$[P]_{\text{doping}} = K_P \left[ \frac{1}{\rho_t} - \frac{1}{\rho_i} \right] \text{ for initially n-type silicon,} \quad (5.18)$$

$$[P]_{\text{doping}} = \frac{K_P}{\rho_t} - \frac{K_B}{\rho_i} \text{ for initially p-type silicon.} \quad (5.19)$$

### 5.3. Neutron flux and cross-section

From the above equations, we can determine the increment of phosphorus atom concentration to be achieved by neutron irradiation. When silicon is irradiated in a neutron field, the neutron capture reaction rate of  $^{30}\text{Si}$  in the silicon is given by [57]

$$R = \int_0^\infty \phi(E) \sigma^{30\text{Si}}(E) N^{30\text{Si}} dE = N^{30\text{Si}} \bar{\sigma}^{30\text{Si}} \phi_T, \quad (5.20)$$

where  $\phi(E)$  and  $\sigma(E)$  are the energy dependent flux and cross-section for the (n, $\gamma$ ) neutron capture reaction, respectively,  $\phi_T$  is the thermal flux intensity,  $\bar{\sigma}$  is the average neutron capture cross-section and  $N$  is the atomic density of target isotope.

The thermal flux is usually defined as an integral of the energy dependent flux over a thermal energy range. For a well thermalized condition as in a reflector region of a research reactor, the energy dependent flux is represented by a Maxwellian spectrum. The Maxwellian spectrum dies off rapidly beyond  $5kT$ , that is,

$$\phi_T = \int_0^{5kT} \phi(E) dE \cong \int_0^\infty \phi(E) dE. \quad (5.21)$$

For a Maxwellian neutron spectrum,

$$\phi_T = \frac{2\pi n}{(\pi k T_n)^{3/2}} \left( \frac{2}{m} \right)^{1/2} \int_0^\infty E e^{-E/kT_n} dE = \frac{2}{\sqrt{\pi}} n \left( \frac{2kT_n}{m} \right)^{1/2} = \frac{2}{\sqrt{\pi}} n v_T = 1.128 n v_T, \quad (5.22)$$

where  $m$ ,  $n$  is the neutron mass and density,  $T_n$  is the neutron temperature in Kelvin,  $k$  is the Boltzmann constant and  $v_T$  is the speed of the neutrons having the most probable energy.

Since the most probable energy of the above distribution is  $kT_n$ ,  $v_T$  is given by

$$\frac{1}{2} m v_T^2 = k T_n. \quad (5.23)$$

From the above equations,  $v_T$  and the neutron flux depend on the neutron temperature even though the neutron density is unchanged.

If the absorption cross-section of  $^{30}\text{Si}$  varies as  $1/v$ , then

$$\sigma^{30\text{Si}}(E) = \sigma_0^{30\text{Si}} \sqrt{\frac{E_0}{E}} = \sigma_0^{30\text{Si}} \frac{v_0}{v}, \quad (5.24)$$



where  $E_0$  is an arbitrary relative energy, but for usual practice is taken to be 0.0253 eV, which is the most probable energy in the Maxwellian spectrum at 20.46 °C and also corresponds to a neutron speed of 2200 m/s.  $\sigma_0$  is the cross-section at  $E_0$ .

Then, the reaction rate is given by

$$R = N^{30Si} \frac{2\pi n}{(\pi k T_n)^{3/2}} \left( \frac{2E_0}{m} \right)^{1/2} \sigma_0^{30Si} \int_0^\infty \sqrt{E} e^{-E/kT_n} dE. \quad (5.25)$$

For integration, we can use the parameter change  $x = E/kT_n$ , then,

$$R = N^{30Si} \frac{2n}{\sqrt{\pi}} v_0 \sigma_0^{30Si} \int_0^\infty \sqrt{x} e^{-x} dx. \quad (5.26)$$

Performing the integration, and if we insert a 2200 m/s flux,

$$\phi_0 = n v_0. \quad (5.27)$$

Then,

$$R = N^{30Si} n v_0 \sigma_0^{30Si} = N^{30Si} \sigma_0^{30Si} \phi_0. \quad (5.28)$$

Thus, the (n, $\gamma$ ) capture reaction rate of the  $^{30}\text{Si}$  inside the neutron field can be simply deduced by the 2200 m/s flux and the cross-section. From Eq. (5.22), we can obtain

$$n = \frac{\sqrt{\pi}}{2} \frac{\phi_T}{v_T}. \quad (5.29)$$

Also, if we introduce an average cross-section at 0.0253 eV,  $\overline{\sigma_0^{30Si}}$ ,

$$R = N^{30Si} \frac{\sqrt{\pi}}{2} \frac{v_0}{v_T} \sigma_0^{30Si} \phi_T = N^{30Si} \frac{v_0}{v_T} \overline{\sigma_0^{30Si}} \phi_T. \quad (5.30)$$

If we define a useful quantity known as the average cross-section at a given neutron temperature,

$$R = N^{30Si} \overline{\sigma^{30Si}} \phi_T, \quad (5.31)$$

where the average cross-section is defined as

$$\overline{\sigma^{30Si}} = \frac{\sqrt{\pi}}{2} \left( \frac{T_0}{T} \right)^{1/2} \sigma_0^{30Si}. \quad (5.32)$$

Therefore, we can obtain that

$$R = N^{30\text{Si}} \sigma_0^{30\text{Si}} \phi_0 = N^{30\text{Si}} \overline{\sigma^{30\text{Si}}} \phi_T. \quad (5.33)$$

Thus, the reaction rate can be deduced by either a 2200 m/s formulation or an integral formulation, with varying flux and cross-section values. The 2200 m/s formulation does not represent an actual situation, but however, it has been often used because the neutron flux can be easily determined with  $1/\nu$  neutron flux monitors and commonly available 2200 m/s cross-section tables regardless of the neutron temperature and spectrum. In contrast, reactor physics calculations usually give the neutron flux based on the integral formulation. When the neutron spectrum is a well thermalized Maxwellian at 20.46°C, the integral thermal flux is 1.128 times that of a 2200 m/s flux as shown in Eq. (5.22). Therefore, the thermal neutron flux should be given with its definition in order to avoid confusion.

For some materials with non- $1/\nu$  cross-sections, we must correct the deviation from  $1/\nu$  cross-sections by introducing a new factor,

$$R = g(T) N^{30\text{Si}} \sigma_0^{30\text{Si}} \phi_0, \quad (5.34)$$

where  $g(T)$  is the non- $1/\nu$  factor, and is also called the  $g$ -factor. It is temperature dependent, and for  $^{30}\text{Si}$  capture at room temperature, the  $g$ -factor is 1.0071. Figure 7 shows the typical capture cross-section of  $^{30}\text{Si}$  [58]. From the figure, it is confirmed that the neutron capture cross-section of  $^{30}\text{Si}$  has very good  $1/\nu$  characteristics up to  $\sim 1$  keV.

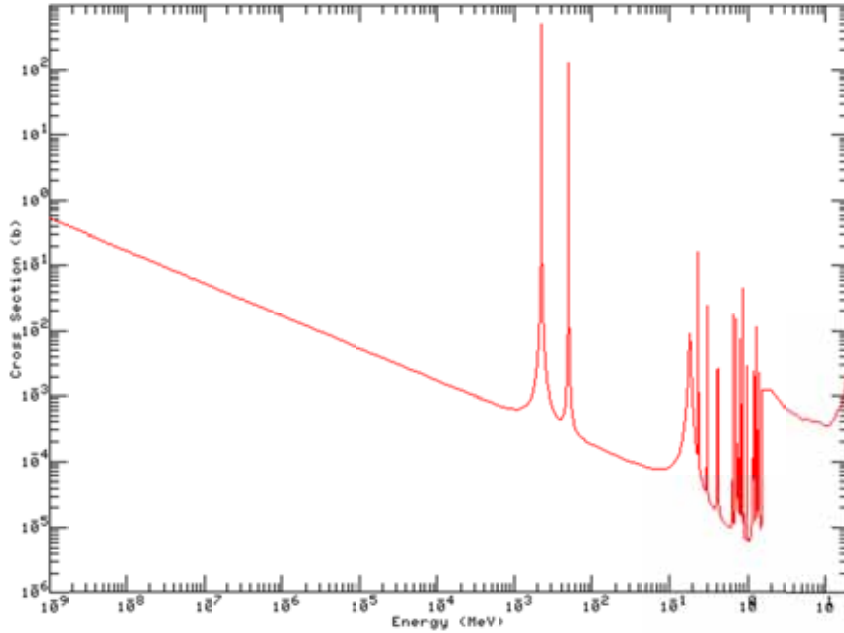


FIG. 7. Neutron capture cross-section of  $^{30}\text{Si}$ .

#### 5.4. Estimation of the $K$ value

From the above equations, we can obtain the production of phosphorus atoms by neutron irradiation of silicon crystal for an irradiation time  $t_i$ , namely

$$[\text{P}]_{\text{doping}} = \int_0^{t_i} \int_0^\infty \phi(E, t) \sigma^{30\text{Si}}(E) N^{30\text{Si}} dE dt = N^{30\text{Si}} \overline{\sigma^{30\text{Si}}} \phi_T t_i. \quad (5.35)$$

Then, the neutron fluence that provides a phosphorous concentration corresponding to the target resistivity can be deduced from Eq. (5.19) as follows:

$$\phi_T t_i = K \left[ \frac{1}{\rho_t} - \frac{A}{\rho_i} \right], \quad (5.36)$$

where:

$$\begin{aligned} A &= 1 \text{ for initially n type,} \\ A &= K_B/K_P \text{ for initially p type.} \end{aligned}$$

A new constant  $K$  is introduced to represent the product of fluence and resistivity for an infinite case of  $\rho_i$ , and it is defined by

$$K = \frac{K_P}{N^{30Si} \bar{\sigma}^{30Si}}. \quad (5.37)$$

Since  $\bar{\sigma}$  is dependent on the neutron spectrum including non-thermal neutrons, the  $K$  value must be determined at each irradiation site. If the neutron spectrum is a well thermalized Maxwellian,  $\bar{\sigma}$  is a Maxwellian-averaged value at a specific neutron temperature. In a real situation, the  $K$  value for an irradiation site should be determined for each customer and each diameter based on the measured resistivity values' feedback. An example of an estimation of  $K$  is shown in Table 2. It is found that the  $K$  values show a rather wide range as listed in Table 3. Since the neutron spectrum does not vary significantly across NTD irradiation sites, it cannot be the major reason for this wide range. The value of resistivity for the silicon wafer is an accepted standard in the industry. It seems that different definitions of the neutron flux and differences in neutron flux measurement would be major causes of these variations.

TABLE 2: AN ESTIMATION OF  $K$  FOR A MAXWELLIAN SPECTRUM AT 20.46°C

Specifications	Typical values
$^{30}\text{Si}$ abundance	0.0310
Silicon nominal density	2.33 g/cm <sup>3</sup>
Silicon atomic weight	28.086
Avogadro number	6.023×10 <sup>23</sup>
$^{30}\text{Si}$ atomic density	1.549×10 <sup>21</sup> cm <sup>-3</sup>
$^{30}\text{Si}$ capture cross-sections at 25.3 meV	0.1075 barns [10 <sup>-24</sup> cm <sup>2</sup> ]
Maxwellian avg. capture cross-section	0.09599 barns [10 <sup>-24</sup> cm <sup>2</sup> ]
Electronic charge	1.609×10 <sup>-19</sup> C
Electron mobility in silicon crystal at 300 K	1350 cm <sup>2</sup> /s
$K_P$	4.630×10 <sup>15</sup>
$K$ for 2200 m/s flux	2.780×10 <sup>19</sup>
$K$ for Maxwellian integral flux	3.114×10 <sup>19</sup>

TABLE 3: *K*-VALUES USED IN SEVERAL SITES

Irradiation site	<i>K</i> value
Topsil	0.8~1.0 times of $2.612 \times 10^{19}$
Harwell	$2.54 \times 10^{19}$
SAPHIR	$2.628 \times 10^{19}$
JAERI	$2.94 \times 10^{19}$ (in 2200 m/s flux)
FRM-II [44]	$4.86 \times 10^{19}$ for 150 mm ingot, $4.26 \times 10^{19}$ for 200 mm ingot (in 2200 m/s flux)
HANARO [59]	$2.40 \times 10^{19}$ (in 2200 m/s flux)

## 6. TECHNICAL REQUIREMENTS FOR NTD

The foremost advantage of NTD compared to other doping methods is the high uniformity of the final resistivity after doping a whole silicon ingot, which entitles NTD-Si to the claim of the best quality silicon for high power applications. Since the neutron flux in a reactor is not uniform, the overall uniformity in an ingot depends on the ingot size and irradiation technology. It is usually possible to control the neutron fluence very accurately, and therefore the resistivity target can be specified and accomplished within a very tight tolerance [60]. The accuracy of the target resistivity is a basic requirement for the doping process. Thus, the accuracy of doping combined with the overall uniformity will govern the quality of the product.

The physical volume of a batch of ingots is rather large, and the flux variation in such a volume is much more vital than the required accuracy in the target resistivity. Irradiation uniformity is expressed by radial and axial, or longitudinal, variations of the final resistivity after irradiation. Thus, radially and axially uniform irradiation is the prime target in the design and operation of an irradiation device.

The initial resistivity variation has a direct impact on the final resistivity variation after irradiation. The initial variation can be only partially offset (smoothed) by irradiation. Therefore large local variation in initial resistivity is not a major cause of final resistivity variation in NTD wafers. In order to minimize the impact of initial resistivity variation, the initial resistivity should be high enough compared to the target resistivity. Also, uncertainty (error) in the resistivity measurement impacts on accuracy of the results received from the customer. In general, uncertainty is larger when measuring high resistivity. Therefore, these considerations need to be acknowledged during irradiation of high resistivity targets.

In practice typically several Si ingots are put together to form a batch of material to be irradiated simultaneously. Obviously it is important to combine only ingots with similar starting value of resistivity to obtain high accuracy with respect to the target resistivity to be met by the irradiation

Neutrons used for NTD are mainly thermal neutrons, and a higher thermal neutron flux results in a shorter irradiation time. If the irradiation time is too short, however, an accurate irradiation would be difficult unless the ingots are inserted to and withdrawn from the neutron

field very rapidly. If the flux is too low, a very long irradiation time is needed, which limits the practical application for NTD. The resistivity of NTD Si ranges from about 10 to about 1000  $\Omega\cdot\text{cm}$ . For a neutron flux of  $10^{13} \text{ cm}^{-2}\text{s}^{-1}$ , about 17 hours are needed to obtain 50  $\Omega\cdot\text{cm}$ . When one hour of irradiation is applied, about 800  $\Omega\cdot\text{cm}$  would be a reasonable upper bound at a flux of  $10^{13} \text{ cm}^{-2}\text{s}^{-1}$ . In the case with a flux five times higher, the upper bound would be about 160  $\Omega\cdot\text{cm}$ . Low flux reactors with a thermal neutron flux approximately  $10^{12} \text{ cm}^{-2}\text{s}^{-1}$  would be proper for the accommodation of high resistivity targets.

Since fast neutrons create extended charged lattice defects in a crystal, the fast neutron flux in the irradiation position must be as low as possible. This requirement is in particular important for high target resistivity NTD Si, because the screening radius of charged defects (Thomas Fermi screening length) is longer in materials exhibiting a low charge carrier density.

Gamma rays are the major source of heat generation in the ingot, so the gamma ray flux should also be as low as possible, and the ingot must be sufficiently cooled during the irradiation. In addition, due to the short half-life of only 2.62 h also the  $\beta$  decay of  $^{31}\text{Si}$  itself contributes considerably to the heat generation within the Si ingot under irradiation.

The diameter of an irradiation hole should be large enough to accommodate an irradiation rig containing the ingots, coolant paths and online flux monitors. Because the NTD service charge is usually based on the number of irradiations (cycles), the irradiation of large volume ingots is profitable. However, loading and unloading of large ingots significantly distort the neutron flux at nearby experimental facilities and neutron detectors for reactor operation. This interference should be carefully examined.

Since a silicon ingot is very brittle, careful design and operation of the ingot handling tool is required so as not to damage the ingot. Particular attention has to be paid to the use of gloves during all handling procedures and to the cleanliness of all storage locations within and outside the reactor pool. Si ingots must only be in contact with Si, Al or plastics. The contact to steel and other metals must be strictly avoided.

Irradiated ingots must be cooled to reach the exemption level of residual activity, so a proper space is needed to cool down the irradiated silicon. In addition, a cleaning facility, e.g. an ultrasonic bath to be connected to the radioactive waste water tanks of the reactor, is needed to remove potential contamination from the contact of the Si ingot with reactor pool water. Equally, a surveying system for the residual radioactivity of the irradiated ingot is part of the necessary equipment. In addition, it is crucial for the reactor operator to have the license to issue an official radioactivity release document. Finally, for commercial irradiation services, systematic quality assurance, traceability of the product and control are necessary. Many customers of NTD service require the certification of the irradiation facility according to the ISO 9001:2008 standard because they are urged by their customers to only cooperate with ISO 9001 certified subcontractors.

The uniformity and accuracy criteria of NTD are determined in an individual contract between an irradiation site and a wafer company. When irradiations do not meet the criteria, the vendor of the spoiled ingots typically will require compensation.

## **6.1. Radial uniformity**

The planar distribution of the resistivity of a NTD-Si wafer is represented by the radial resistivity gradient (RRG) defined by

$$RRG = 100 \times \frac{\rho_{\max} - \rho_{\min}}{\rho_{\min}} \quad (\text{in } \%). \quad (6.1)$$

where  $\rho_{\min}$  and  $\rho_{\max}$  are the minimum and maximum values, respectively, among the measured resistivities at several points on a wafer.

In general, there is a radial flux gradient in the reflector region of any research reactor, i.e. the neutron flux becomes smaller away from the core centre. The effects of the radial non-uniformity of the neutron flux are compensated by rotating the ingot during irradiation. Due to the comparatively long irradiation times a rotational speed of only few rpm is sufficient. Although silicon is rather transparent to neutrons, some attenuation of the neutrons inside the ingots will occur. Therefore, the neutron fluence at the inner part of an ingot becomes lower than the periphery. This difference becomes larger as ingot diameter increases. If the RRG is only due to the neutron attenuation in the ingot, tight specifications from customers can be met even with eight inch ingots. On the other hand, asymmetric irradiation of an ingot can increase the RRG. Also, the initial resistivity and uncertainty in the resistivity measurement can affect the RRG directly. Above effects may be reflected in the specifications. While the specification of the RRG would depend on the customer, it is usually less than or equal to 4–5% nowadays.

## 6.2. Axial irradiation uniformity

The longitudinal variation of the resistivity in a cylindrical silicon ingot is represented by an axial resistivity variation (ARV) given by

$$ARV = 100 \times \frac{\rho_{\max}^{plane} - \rho_{\min}^{plane}}{\rho_{\min}^{plane}} \quad (\text{in } \%). \quad (6.2)$$

where  $\rho_{\min}^{plane}$  and  $\rho_{\max}^{plane}$  are the minimum and maximum values among the plane resistivities in an ingot. The plane resistivity may be represented by an average, a medium or a centre value in the plane, depending on the customers. When the plane resistivity is represented by the value at centre, since the ARV is determined by only one measured value each for the maximum and minimum resistivity planes as similar to RRG, it is more sensitive to the initial resistivity variation and uncertainties in resistivity measurements than other variables.

The requirement for axial resistivity uniformity depends on the customers, but this requirement has become stricter. In the early 1990s, a general variation of 10–20% was allowed, but in the 2000s a variation within 5–8% has been required. If the uncertainty in the resistivity measurement and the deviation from the target resistivity are combined into the axial variation, the requirement is extremely precise. Therefore, it is recommended to design the irradiation facility to minimize the axial variation of the neutron fluence as much as possible.

## 6.3. Irradiation accuracy

The irradiation accuracy (IRRACC) may be represented by the deviation of the average resistivity in an ingot from the target resistivity.

$$IRRACC = 100 \times \frac{\rho_{\text{average}} - \rho_{\text{target}}}{\rho_{\text{target}}} . \quad (6.3)$$

where  $\rho_{\text{target}}$  is the target resistivity, and  $\rho_{\text{average}}$  is the average or medium of all the measured resistivity values for an ingot or plane. When many IRRACC measurements are accumulated, their statistics may be close to a Gaussian distribution. The irradiation site should give an effort to make its average zero and its standard deviation as small as possible. The standard deviation is usually less than 3% at an irradiation site with sufficient experience in NTD. The standard deviation becomes larger in cases of higher target resistivity due to the propagation of the uncertainties.

Wafer companies want every measured resistivity to be within their criteria for maximum yield. The maximum deviation of plane resistivity values from the target may be specified rather than the ARV and the IRRACC, depending on customers.

#### 6.4. Interfering radiation and NTD

Exposure to fast neutrons and gamma rays is not desirable for NTD. Fast neutrons are a major source of permanent lattice defects, and gamma rays are a major source of heat generation in ingots.

Lattice defects are the displacement of silicon atoms from their normal lattice positions. Low energy neutrons create lattice defects in an indirect way due to their absorption by  $^{30}\text{Si}$  and the subsequent  $\beta$  decay of  $^{31}\text{Si}$ , the recoil of which leads to the production of single interstitials and vacancies. These defects, however, are usually recovered by appropriate heat treatment after irradiation. The same applies to defects caused by energetic gamma rays. The most important interference in the NTD process is the fast neutron knock-on displacement leading to defect clusters which cannot be easily recovered. These defects introduce defect levels into the band gap that cause free carrier removal, reducing carrier mobility and minority carrier lifetime. It should be emphasized that an in-core fission neutron spectrum produces about  $10^3$  more displaced silicon atoms than a graphite moderated spectrum. Thus, a low fast neutron flux is needed to avoid the irreversible lattice defects in a silicon crystal caused by fast neutrons [61, 62]. After neutron irradiation, the ingot is annealed by the wafer company in order to recover the crystal defect [63]. Wafer companies may judge the quality of neutron fields by measuring the carrier lifetime.

In the IAEA consultants meeting, it was recommended that the thermal-to-fast flux ratio should be at least of the order 7:1 [64]. Particular high values for the thermal/fast neutron flux can be achieved in heavy water moderated reactors. E.g. in the case of the OPAL reactor, they designed their thermal-to-fast neutron flux to be 900:1; at FRM II a ratio of thermal/fast flux of  $> 1000$  was measured. This ratio for the irradiation facility of SIDONIE in (the light water moderated) BR2 was reported to be about 10:1. SIDONIE also reported a cadmium ratio of approximately 25 [30]. For the new Si doping facility POSEIDON at BR2 a cadmium ratio of 50 is obtained [32]. It is noteworthy that the efficiency of lattice defects as scattering centres for the charge carriers is the more important the lower the charge carrier density is. Consequently high target resistivity ( $>400 \Omega\text{cm}$ ) NTD Si is preferentially to be produced in a well thermalized neutron spectrum (i.e. a heavy water moderated reactor).

The thermal-to-fast flux ratio varies depending on the low energy boundary of the fast neutron flux. The Cd ratio, on the other hand, reveals information on the epithermal neutron flux,

which is not included in the thermal-to-fast flux ratio, but depends on neutron detectors. For example, the classical Cd ratio calculated or measured by the reaction rate of gold is surely different from that by silicon or cobalt. Thus, the thermal-to-fast flux ratio and the Cd ratio should be explicitly specified in order to be meaningful. In brief, it is recommended that a Cd ratio larger than 10 in the case of gold would be appropriate for NTD.

Gamma rays and  $\beta$  decay of  $^{31}\text{Si}$  are the major source of heat generation, which may cause excessive ingot temperature and formation of bubbles at the ingot surface. The IAEA consultants meeting recommended the temperature at the irradiation position be below 180°C [64] to avoid the diffusion of the minority carrier and to minimize lattice defects such as a swirl. In BR2, the gamma ray heating was rather high, 3 W/g, and the target temperature of the NTD facility process was set below than 250°C [30]. Although it is difficult to define a concrete limit on the ingot temperature during the irradiation process, but it is certain that the temperature should be as low as possible. Table 4 lists the neutron flux and gamma ray heating data reported by various reactors.

TABLE 4: THE NEUTRON FLUX AND GAMMA RAY HEATING OF SEVERAL IRRADIATION SITES

Reactor	Thermal power	Irradiation rig	Neutron flux [cm <sup>-2</sup> s <sup>-1</sup> ]	Gamma ray heating or temperature
MARIA in Poland	30 MW		$2.1 \times 10^{13}$ , (thermal), Fast (>1 MeV)/thermal=0.02	0.5 W/g
BR2 in Belgium	56 MW (Nominal: 85 MW)		$1.74 \times 10^{14}$ (thermal), $1.9 \times 10^{13}$ (fast),	< 200°C Si core temp.
SAFARI-1 in South Africa	20 MW	4 inch SILIRAD	$2.5 \times 10^{13}$ - $8 \times 10^{13}$ (thermal)	Measured temperature at Si: ~80°C
FRM II in Germany	20 MW	8 inch	$1.6 \times 10^{13}$ (thermal) Thermal/fast=1700	Max. temp.: 110°C (Si core temp.) [44]
OPAL in Australia	20 MW	5, 6, 8 inch	$2.5 \times 10^{12}$ – $1.5 \times 10^{13}$ (thermal) Thermal/fast=900	
HANARO in the Republic of Korea	30 MW	5,6 inch NTD2	Thermal/fast=400 Cd ratio for gold: 16~22	0.2–0.9 W/cm <sup>3</sup>

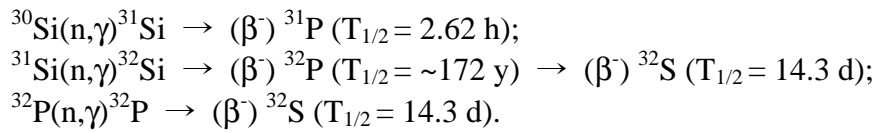
## 6.5. Residual radioactivity

There are three kinds of regulations specified regarding the radiation level of irradiated silicon [28]:

- Transport regulations — the radiation level at any point on the external surface of a package shall not exceed certain criteria;
- Non-fixed contamination;
- The level of radioactivity of the material itself.

To ship the irradiated ingot back to the customer, its residual radioactivity must be lower than the international criteria for exemption from regulations for radioactive material. Reactions of interest in the NTD process according to the aspect of the residual radioactivity are as follows:





In addition to the above reactions, there are several threshold reactions involving fast neutrons such as (n,p), (n,np)Al, (n, $\alpha$ ) and (n,2p)Mg, but their short half-lives together with small cross-sections make their contribution to the residual radioactivity negligible. Also, since the impurity concentration inside the silicon ingot is extremely low, their activation is negligible.

$^{31}\text{Si}$  has a relatively short half-life of 2.62 hours, and it will sufficiently decay away in a proper cooling time of two or three days. The activity of  $^{32}\text{Si}$  is also very low because of its very low production rate despite very long half-life. Therefore, the main source of residual radioactivity is  $^{32}\text{P}$  ( $T_{1/2}=14.3 \text{ d}$ ) produced by the  $^{31}\text{P}(n,\gamma)$  reaction. The activity of  $^{32}\text{P}$  is slightly dependent on the neutron flux level, but it is essentially governed by the neutron fluence, i.e. the inverse of resistivity.

The exemption criterion from the regulation for  $^{32}\text{P}$  may be different in each country. At the Harwell reactors in 1985, the criterion of 7.4 Bq/g was applied [28] according to the IAEA criterion that was published in 1977. They also used a surface contamination limit of 3.7 Bq/cm<sup>2</sup>. 74 Bq/g was used in Japan, which produces the largest amount of NTD wafers. The level of  $^{31}\text{Si}$  or  $^{32}\text{P}$  for exemption from regulation is 1000 Bq/g in the current IAEA Safety Standards.

As the resistivity is inversely proportional to the irradiation time, a lower resistivity causes a higher residual radioactivity. The specific activity of  $^{32}\text{P}$  increases very rapidly as the resistivity decreases below 10  $\Omega\cdot\text{cm}$ . Since the half-life of  $^{32}\text{P}$  is 14.3 days, a very long cooling time is needed when the resistivity is less than a few  $\Omega\text{cm}$ . This is one of the reasons why NTD is not practical for specimens with a low resistivity field. NTD is mainly applied when resistivity surpasses about 20  $\Omega\cdot\text{cm}$ . The actual demand for NTD Si ranges between 30  $\Omega\text{cm}$  and 1100  $\Omega\text{cm}$  with an increasing interest in high target resistivity. The average resistivity of NTD Si was for a long time usually estimated as 40–50  $\Omega\text{cm}$ ; hence an irradiation time of a 5  $\Omega\cdot\text{cm}$  sample is about 10 times of that for the average resistivity. The practical lower limit is about 10–15  $\Omega\cdot\text{cm}$ .

In practice, the activation of impurities and contaminants on the ingot surface sometimes causes a problem in particular since the Si ingot is in most of the irradiation facilities in direct contact with reactor pool water. Careful cleaning of ingots not only after irradiation but also before irradiation is important. At FRM II good experience was made by cleaning Si ingots by isopropanol before the start of the irradiation and cleaning the irradiated Si ingot in an ultrasonic bath at 65°C using a special decontamination non foaming, non-ionic detergence which, is free of halides followed by keeping the ingot in de-ionized water for 24 h.

## 6.6. Interference with other facilities

An irradiation hole for NTD is relatively large considering the size of an average research reactor. If the hole is filled with light water, the neutron flux around the hole changes significantly when ingots are inserted into or withdrawn from the hole. If other experimental facilities or neutron detectors for reactor operation exist near the hole, NTD irradiation could perturb the experiments or reactor operation. Thus, the location of an NTD hole must be carefully placed during the design stage and, if possible, a method minimizing the

perturbation should be considered. For example, a material having similar neutronic characteristics to silicon (typically Al or a Si dummy) may replace the ingot when it is withdrawn from the irradiation hole.

On the other hand also interference of neighbouring installations (like uranium target irradiation positions for  $^{99}\text{Mo}$  production as a very drastic example) has to be taken into account. Today, however, careful mapping of an entire reactor with all its installations is possible by full scale Monte Carlo programs like MCNP(X) [65].

### **6.7. Handling of silicon ingots**

A silicon ingot is a glassy cylindrical single crystal, so its handling tools should be designed and utilized very carefully. Of course, a rig for efficient and safe irradiation should be furnished. A vacuum sucking system is used in Japan and at FRM II to handle silicon ingots, and an automated mechanical transfer system using canisters is used at OPAL. The silicon ingot is also very brittle. The edge of an ingot is easily broken upon impact, especially during insertion to the irradiation rig or to the irradiation canister. Generally, the clearance between the ingot and the inner surface of the rig should be minimal to locate ingots at the centre of irradiation rig. Thus, a specially designed handling tool is required for the easy and safe manipulation of the ingot. As a general practice, if an ingot is broken, the vendor should be reimbursed.

An ingot's diameter has some tolerance from a typical diameter. The tolerance range may depend on customers. The irradiation rig or the irradiation canister should be designed to accommodate all ingots in the tolerance range. Since at FRM II for example only one irradiation hole is available for NTD, the irradiation canisters may be equipped with ring-shaped spacers made like the canister itself from an AlMg<sub>3</sub> alloy to allow the irradiation of ingots having a diameter of 5, 6 and 8 inches.

### **6.8. Neutron fluence monitoring**

Generally, at an NTD irradiation site, online monitoring of the neutron flux is needed to achieve accurate irradiation. It is usually accomplished by the self-powered neutron detector (SPND). When the irradiation time is not long enough to neglect the response time of the SPND, a dynamic compensation of SPND signals may be needed. As the SPND can be located only outside of the ingot, its signal must be calibrated to the neutron flux in the ingot, which can be measured by attaching neutron activation foils or wires in between ingots. But the calibration factor may vary depending on many factors like geometrical core configuration, control rod position, fuel burn up, SPND burn up, etc. Therefore, a careful investigation of the calibration factor over a long term is required.

### **6.9. Washing, decontamination and radioactivity monitoring**

Ingot are usually cleaned by chemicals before shipment to the wafer companies, but additional cleaning before irradiation is recommended. While the liquid used for cleaning before irradiation can be disposed as non-radioactive waste, decontamination after irradiation produces radioactive waste. The pre-cleaning may reduce the burden incurred by decontamination, so a washing procedure using an appropriate cleanser should be determined. Since in most cases the Si ingots are already polished before being irradiated it is sufficient to clean the un-irradiated ingots with a soft tissue and alcohol in order to remove traces from fingerprints, etc.

It is, however, even more important to clearly define the washing procedure for the irradiated Si, including levels of temperature of the cleaning bath, duration of the cleaning procedure, and chemicals to be used. Before carrying the ingot out of the reactor, an inspection against radioactive contamination is necessary. Therefore, all necessary equipment and procedure must also be well established and available.

## **7. METHODS FOR UNIFORM IRRADIATION**

The uniformity of neutron irradiation is usually expressed by radial (planar) and axial (longitudinal) uniformities. The principle to achieve uniform irradiation is rather simple, and the methods can be classified into a few types. However, even when the same method is used, the design and operation of an NTD facility can vary greatly depending on the circumstances of each irradiation site. Therefore, it is recommended that a reactor starting an NTD project should search for the appropriate method by considering the characteristics and conditions of their own reactor, rather than imitating an example.

### **7.1. Radially uniform irradiation**

The radial uniformity of resistivity in a semiconductor ingot can be represented by the RRG (radial resistivity gradient) given by Eq. (6.1). Wafer companies always require that the RRG be as low as possible. Because there is a neutron flux gradient in the radial direction within a reactor, the core side neutron flux in an irradiation hole is higher than that in a position around the vessel. Therefore it is to be recommended to choose the location for a Si doping irradiation hole in a position where the flux gradient is known to be low. In addition, the silicon ingot must be rotated during irradiation in order to guarantee the complete radial uniformity. The rotational speed should be chosen high enough as to guarantee a sufficiently high number of revolutions, typically at least 100 during the irradiation time. The sufficiently high number of revolutions during the irradiation time makes sure that the rotation will not be the reason for a major RRG. It also should be noted that for higher neutron fluxes, with a larger gradient and shorter irradiation time, a faster rotation might be needed.

Nevertheless, the RRG cannot be zero because the neutron flux at the inner part of a silicon ingot is slightly lower than that at the periphery due to neutron self-attenuation by silicon. Also, the non-uniformity of the initial resistivity distribution contributes to the RRG. Figure 8 shows the calculated radial neutron flux distribution inside a silicon ingot [66]. In this case, RRGs by neutron attenuation are expected to be less than 1.5% and 2% for 5 and 6 inch silicon ingots, respectively. If the neutron cross-section of non-crystal silicon is used for the calculation, the RRG becomes larger by a factor between two and three because the scattering cross-section of a single crystal at a thermal neutron energy range is smaller than that of powder. In this calculation, neutron cross-sections at 300 K were used. In the actual irradiation of the ingot, since the ingot temperature is usually higher than 300 K, a slightly higher RRG is expected.

The misalignment and asymmetry in relation to the rotation centre of ingots and irradiation rigs also increase the RRG. Furthermore, it is even more sensitive for an irradiation facility cooled by light water since these neutrons attenuate rapidly in light water.

The rotation concept is simple but its actual realization is not so easy. In a vertical irradiation hole, a metallic chain connected to a stepping motor can be used for rotation. A water bearing concept has been adopted in the OPAL reactor.

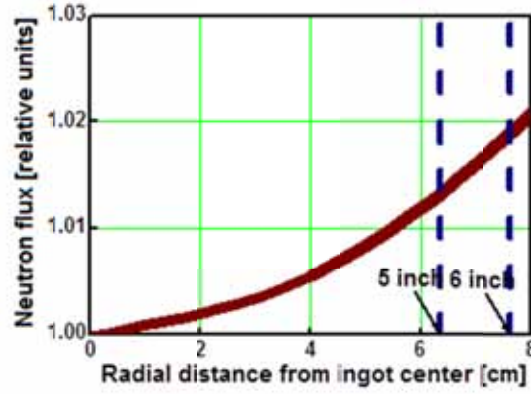


FIG. 8. Example of calculated radial neutron flux distribution inside a silicon ingot.

## 7.2. Axially uniform irradiation

Actually, a uniform axial irradiation is the most challenging in NTD from the viewpoint of irradiation technology, specifically during the design stage of an irradiation device when determining the best irradiation method at each irradiation site. Basically, three irradiation processes have been found to achieve an axial uniformity. The choice of an irradiation process is primarily dependent on the characteristics of each irradiation hole, and the selected process must also be adapted for various equipment and devices and additionally, the preferences of designers.

### 7.2.1. Reciprocating motion

If an ingot moves inside an irradiation hole from one end to the other as shown in Figure 9, the ingot experiences the entire axial variation of the neutron flux. Consequently, when the moving speed is controlled appropriately, a uniform irradiation is possible.

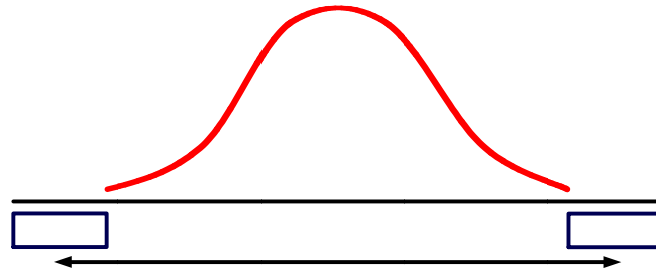


FIG. 9. The uniform irradiation method to use the reciprocation motion of an ingot.

This method is useful for an irradiation hole that has enough space at both ends of the hole such as a penetrating beam tube. The irradiation uniformity in this method is less affected by changes in reactor core conditions such as the control rod position than in other methods since every longitudinal position in an ingot passes through the same neutron flux field. However, the movement of the silicon ingot itself may change the neutron flux distribution. Also, since a silicon ingot undergoes the average neutron flux of the travelling zone, the efficiency in neutron utilization is relatively low. SIDONIE at BR2 [30], GÉK-4 at the Tomsk complex [47] and SILIRAD at SAFARI-1 [42] use this method.

### 7.2.2. Inversion

If the neutron flux distribution in a certain part of the irradiation hole is linear in the longitudinal direction, an ingot can be irradiated through this linear region for half of the total irradiation time, and then the ingot is inverted for the remaining half as shown in Figure 10.

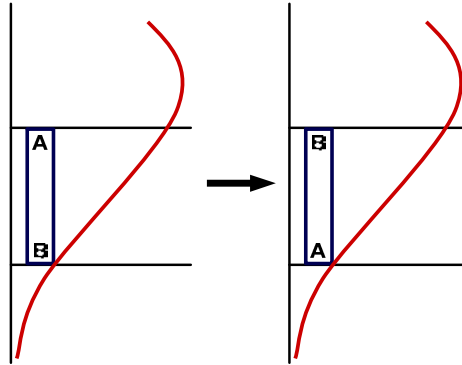


FIG. 10. The uniform irradiation method using the inversion of an ingot.

This method can be applied at an irradiation hole having sufficiently linear neutron flux distribution. Its axial irradiation uniformity is relatively less sensitive to core conditions as compared to the flux screen method. However, the improvement of the uniformity is limited if the linearity of the flux distribution is not sufficient. While it utilizes the neutron flux in the irradiation zone fully, the highest flux region may not be utilized as shown in the figure. Also, this method needs more manpower for the two irradiations of a batch, and the time loss between the two irradiations decreases neutron utilization. JRR-3M and JRR-4 in Japan use this method [56] as well as the POSEIDON facility at BR2 in Belgium [64].

### 7.2.3. Flux screen

If a proper flux screen is installed to flatten a neutron flux as shown in Figure 11, a uniform neutron irradiation can be achieved. The flux screen is composed of relatively strong neutron absorbers in a high neutron flux region and weak absorbers in a low flux region. This may be accomplished by using different materials with different neutron absorption cross-sections or by appropriate thickness variation of the neutron absorbers. The choice of neutron absorbents depends on the available thickness for the neutron screen. If the thickness is a few millimetres, stainless steel or nickel can be used. As the neutron flux in a high flux region decreases, the flux at both ends of the screen decreases as well. In order to increase neutron efficiency, an effort to increase the flux at the both ends of screen should be given.

The irradiation procedure of this method is relatively simple compared to other methods. However, since the axial neutron flux distribution varies especially according to the control rod position, significant planning should be considered before undertaking this method. An appropriate methodology in the development of a suitable flux screen is to irradiate a batch of Si ingots which had been equipped with flux monitors in small holes, drilled into the ingots. The analysis of such experiment would typically follow by complementary modelling by means of MCNP calculations [45]. If the location of the flux screen can be adjusted to compensate for a change of control rod position, rather consistent axial uniformity can be achieved regardless of rod height. This method has already in the 1990s been used at the ASTRA reactor in Austria and has more recently been adopted at OPAL, HANARO [67] and FRM II [44, 45].

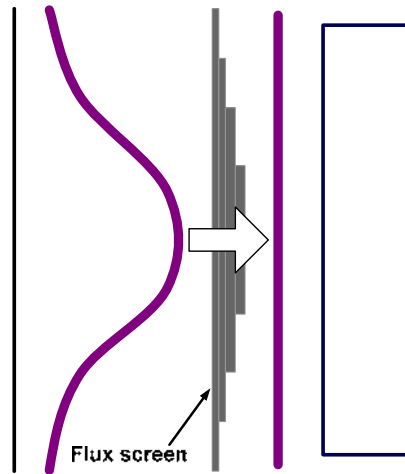


FIG. 11. The uniform irradiation method using a neutron screen.

### 7.3. Effect of initial resistivity

The relationship between the initial and final resistivity is expressed by Eq. (5.36).-There can be an irradiation request for an ingot with a rather low initial resistivity, as in the case of a repeated irradiation or an ingot doped by another method. If there is no local distribution of the initial resistivity value, an irradiation site can easily meet the final resistivity as long as the initial resistivity value is known. In case of a high RRG or ARV the required irradiation time has to be calculated from the average initial resistivity and the target resistivity. In a second step, the effect of this irradiation time on the parts of the ingot exhibiting the minimum and maximum initial resistivity has to be evaluated in order to decide whether or not an acceptable accuracy and homogeneity in the irradiated ingot may be achieved. So, it is important to acquire information on the initial resistivity and its distribution within the ingot from a wafer company, and to convince the company of the necessity to irradiate only ingots showing high homogeneity of the initial resistivity. The same applies of course also when several ingots are put together to form a batch of Si to be irradiated simultaneously.

## 8. NEUTRON FLUENCE MONITORING

Neutron flux measurements are indispensable in the NTD process to ensure the irradiation quality. First, in the commissioning stage of an irradiation facility, the neutron flux distribution over the ingots, Cd ratio, etc., should be measured to confirm the design and performance of the installed NTD equipment. This can be accomplished by the activation method. During routine irradiation of the silicon ingot, the neutron flux or fluence should be monitored in order to guarantee accurate irradiation. The fluence may be monitored by real time flux monitors, neutron activation monitors or simply by tracking the irradiation time. If the neutron flux in the ingot including its trend during a reactor operation cycle is fully understood and reliable, accurately tracking irradiation time alone may be sufficient. Against potential influence on the neutron flux at the NTD hole from changes of reactor conditions, however, real time neutron monitoring calibrated to the neutron flux in the ingot is recommended. Activation monitors calibrated to the resistivity can give the basic data needed for the determination of the irradiation time and for the calibration of online flux monitors. Routine use of activation monitors is reliable until the NTD process is fully stabilized, and afterwards may be occasionally utilized for quality control purposes.

## 8.1. Self-powered neutron detector

In the NTD process, real time neutron flux monitoring is usually accomplished by SPNDs. The SPND operates by directly measuring the beta decay current following the capture of neutrons, so it is always used in the current mode. The SPND is widely used for measurements of the flux variation in nuclear reactors and is available with a small size and diameter. Since it needs no external bias voltage applied to the detector, it requires simple electronics. A good current meter and proper wiring are enough to measure the neutron flux. However, its response is slow, and therefore dynamic compensation may be needed when irradiation time is not sufficiently long as compared to the response time. Since the SPND cannot be installed inside an ingot, the monitored flux is different from the flux in the ingot. This relation between the SPND signal and neutron flux in the ingot should be well understood.

Figure 12 shows a typical structure of a rhodium SPND [16]. The rhodium wire emits beta particles after absorbing neutrons, and these beta particles are transferred to the current meter through a lead wire. MgO or  $\text{Al}_2\text{O}_3$  is used for insulation.

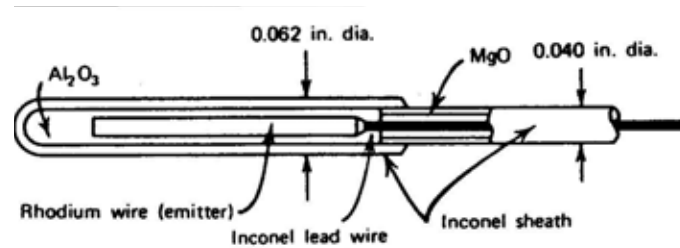


FIG. 12. Typical structure of a rhodium based SPND.

An SPND's emitter should be a conductor so that the electric current may flow through it. Its neutron absorption cross-section should be large enough to produce measurable current, but not be too large for a sufficient life time. The most common emitter materials are rhodium and vanadium, and their basic characteristics are shown in Table 5 [16].

TABLE 5: THE BASIC CHARACTERISTICS OF THE RHODIUM AND VANADIUM

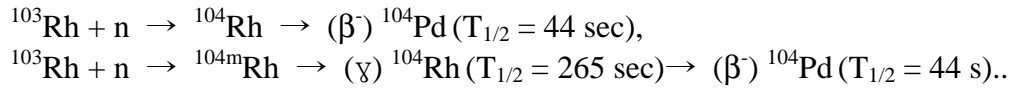
Emitter material	Nuclide of interest	Activation cross-section at thermal energy [barns]	Half-life of induced beta activity [s]	Beta end-point energy [MeV]	Typical neutron sensitivity* [ $\text{A}\cdot\text{cm}^2\text{s}$ ]
Vanadium	$^{51}\text{V}$ (99.750 %)	4.9	225	2.47	$5\times 10^{-23}$
Rhodium	$^{103}\text{Rh}$ (100 %)	139 11	44 265	2.44	$1\times 10^{-21}$

\*Sensitivity quoted for emitter of 1 cm length and typical diameter

The vanadium SPND has a simple neutron absorption reaction scheme producing  $^{52}\text{V}$ , which has a half-life via beta decay of 225 s. Therefore, the current from the SPND after an abrupt reactor shutdown decreases in a simple decay form. Thus the compensation for reactor power variation is simple. Compared to rhodium, vanadium has a smaller absorption cross-section and the half-life of its activation product is longer. Thus the vanadium SPND has a slower

response and a smaller burn-up rate than the rhodium SPND. In a typical reactor flux, the vanadium SPND can be used for several years.

Rhodium has two neutron absorption reactions:



Thus, the decay scheme is somewhat more complex but can be easily evaluated numerically. The high neutron sensitivity of the rhodium SPND provides a great advantage for low neutron flux applications.

## 8.2. Flux monitoring using activation

Neutron activation wires and foils for NTD applications are sufficiently small to attach at the surface of ingots. Thereby, neutron fluxes at the surface, including those between ingots, can be measured. There are several neutron flux monitors that can be utilized, and the radioactivity of the irradiated monitor can be measured by several detectors such as a high purity germanium (HPGe) detector, Geiger-Müller counter, proportional counter and coincidence method, etc. The choice of an activation detector may depend on the purpose of the measurement and reactor power. The fluence measurements would be compared with the measured resistivity values by the customer, and both are used for fitting or calibration of the SPND signals.

### 8.2.1. Activation and radiation counting

Neutron monitoring using activation is an indirect neutron measurement that uses the radioactivity induced in materials by neutron interactions. The activation interaction rate  $R$  of neutrons within a material is given by:

$$R = N_t \sigma \phi, \quad (8.1)$$

where  $N_t$  is the number of target nuclei in the sample,  $\sigma$  is the activation cross-section averaged over the neutron spectrum and  $\phi$  is the neutron flux averaged over the sample. Thus, the rate of activation per unit mass is a direct indicator of the neutron flux. As the sample is irradiated, the radioactive nuclear species that are formed also undergo radioactive decay. Thus, the activity of the sample is given by

$$A(t) = R(1 - e^{-\lambda t}), \quad (8.2)$$

where  $\lambda$  is the decay constant. The induced activity builds up over time and approaches the saturated activity,  $A_\infty$ , after an infinitely long irradiation time, as given by

$$A_\infty = R = N_t \sigma \phi. \quad (8.3)$$

If the irradiation has proceeded for a time  $t_0$ , an activity  $A_0$  at the time  $t_0$  is given by

$$A_0 = A_\infty (1 - e^{-\lambda t_0}). \quad (8.4)$$



If a radiation counting of the material is carried out over an interval between  $t_0+a$  and  $t_0+b$ , the number of counts will be

$$C = \varepsilon \int_a^b A_0 e^{-\lambda t} dt + B, \quad (8.5)$$

where  $\varepsilon$  is the overall counting efficiency, and  $B$  is the number of background counts [16]. Then, the saturated activity is

$$A_\infty = N_t \sigma \phi = \frac{\lambda(C - B)}{\varepsilon(1 - e^{-\lambda t_0})(e^{-\lambda a} - e^{-\lambda b})}. \quad (8.6)$$

Finally, the neutron flux can be obtained by

$$\phi = \frac{A_\infty}{N_t \sigma} = \frac{\lambda(C - B)}{N_t \sigma \varepsilon(1 - e^{-\lambda t_0})(e^{-\lambda a} - e^{-\lambda b})}. \quad (8.7)$$

In addition to the above procedure, the emission probability of radiation from the nuclide of interest and the dead time of the counting system must also be considered and accounted for.

### 8.2.2. Activation detectors

General criteria for choosing an activation detector are as follows [16]:

- Shape of the activation cross-section;
- Magnitude of the cross-section;
- Decay constant of the induced activity;
- Purity and interfering activities;
- Nature of the induced activity;
- Physical properties.

The shape of the activation cross-section describes the variation of the neutron capture cross-section for a given neutron energy. It usually varies inversely with neutron velocity,  $v$ , in the thermal neutron energy range, but above the thermal region, it is more dependent upon the material. Since the reaction of interest in NTD is the  $^{30}\text{Si}(n,\gamma)^{31}\text{Si}$  reaction, an activation detector having a similar activation cross-section shape to  $^{30}\text{Si}$  is preferable. However, the usual  $1/v$  detector can be used as well because a well thermalized neutron spectrum is usually employed in NTD.

If the magnitude of the cross-section is high, the mean free path of neutrons in the detector is small. If the thickness or diameter of the detector is not sufficiently small compared to the mean free path, the neutron flux inside the detector material is significantly lower than on the surface, which is called the neutron self-shielding effect. The insulation of the neutron detector also depresses the neutron flux. This may cause a wrong deduction of the neutron flux. Therefore, the common geometric form of the material is a thin foil or a small diameter wire. Most valuable information can be obtained by the irradiation of flux monitors which are placed within a Si ingot, being equipped with tiny drilled holes for this purpose. Because of their negligible self-shielding suitable flux monitor are e.g. an Al: Au wires containing 0.1% or 0.2% of gold [44]

The decay constant of the induced activity must be small to have a sufficiently long half-life as compared to the irradiation and counting times during the fluence measurement. The purity should be high and interfering activities should be as small as possible. The nature of the induced activity must have characteristics for easy and accurate detection, such as gamma rays with appropriate energy levels and high yield. The physical properties must be suitable for handling and corrosion resistant.

Cobalt and gold are useful in NTD applications as the activation detector for thermal neutron detection [68]. They are stable elements, so there is neither interfering radioactivity nor an uncertainty in the abundance. Gold has a strong resonance capture at 4.9 eV, which must be accounted for through the cadmium difference method in order to measure accurately the thermal neutron flux. As its high capture cross-section may cause flux depression near the monitor, the monitor must be very small or thin. Gold is proper for low power experiments like a performance test of an irradiation facility since it has advantages such as a high cross-section, 100% abundance, high detection efficiency for induced gamma rays and an appropriately long half-life. For routine fluence monitoring, however, the cross-section of gold is too high and the half-life of  $^{197}\text{Au}$  is not long enough. Therefore, cobalt is popularly used for fluence monitoring though its cross-section is also very high. If the detector is pure cobalt, the consequently tiny detector makes handling difficult. A cobalt detector diluted with aluminium may be convenient for fluence monitoring. The cross-section shape of  $^{94}\text{Zr}$  is rather similar to  $^{30}\text{Si}$ , and a zirconium foil with enough size for easily handling can be used for fluence monitoring due to its low cross-section. Figure 13 shows a typical activation cross-section for related materials [58].

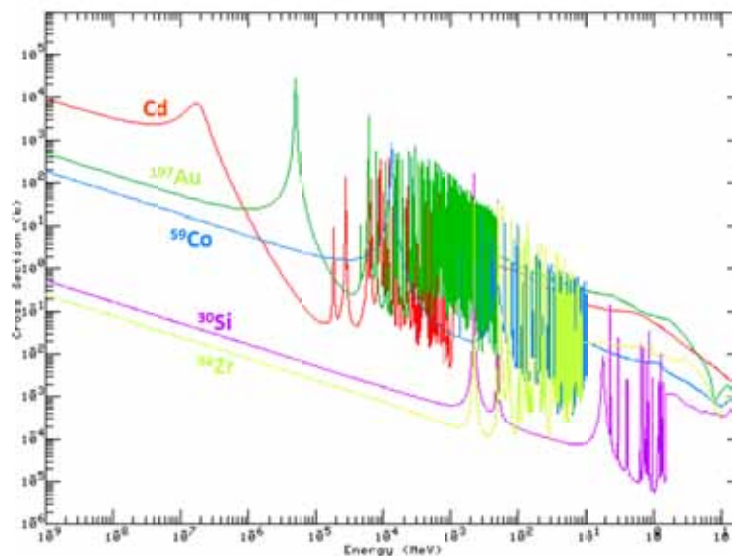


FIG. 13. Neutron capture cross-sections of Cd,  $^{30}\text{Si}$  and typical activation detectors for NTD application (see figure caption).

### 8.2.3. Cadmium difference method

The observed activity of an activation detector corresponds to a mixture of activation caused by thermal, epithermal and fast neutrons. In a well thermalized region, since the contribution of fast neutrons is almost negligible, a majority of the non-thermal neutron reactions are resonance reactions with epithermal neutrons. To confirm the total contribution of non-thermal neutrons to the total reaction rate, the cadmium difference method can be applied.

The neutron absorption cross-section of cadmium is very large for neutron energies up to about 0.4 eV, above which it drops abruptly and remains low for high neutron energies as shown in Figure 13. Cadmium with a thickness of only 0.5 mm acts as a selective neutron filter, blocking low energy neutrons below 0.4 eV but allowing higher energy neutrons to pass through with little attenuation. The cadmium ratio is defined as follows,

$$\begin{aligned} \text{Cd ratio} &= \frac{\text{Activity of the bare detector}}{\text{Activity of the cadmium covered detector}}, \\ &= \frac{\text{Thermal and resonance neutron contribution}}{\text{Only the resonance contribution}}. \end{aligned} \quad (8.8)$$

The Cd ratio can show the degree of thermalization for a given neutron field. However, this value depends on the material used in neutron detection. For example, cobalt and gold have different cross-section shapes as shown in Figure 13, so the Cd ratios for cobalt and gold are different for an identical neutron spectrum.

#### 8.2.4. Procedure of neutron flux determination

A procedure of neutron flux determination using gamma ray detection from an activated monitor is as follows:

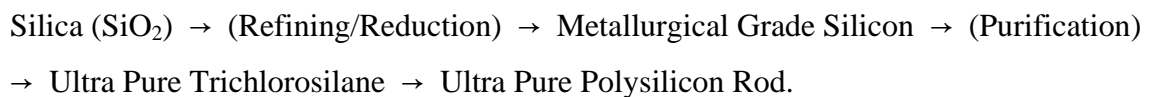
- 1) Set up gamma ray detection electronics;
- 2) Measure the gamma ray spectrum of standard sources for calibration;
- 3) Calculate the absolute photo-peak efficiency;
- 4) Draw the calibration curve using experimental condition;
- 5) Choose the proper activation monitor;
- 6) Place the activation monitor in the irradiation facility and activate it for a certain time;
- 7) After proper cooling time, transfer it to a counting position and count until sufficient statistics are attained; one should note that the geometry of the flux monitor must be as similar as possible to the one of the calibration source.
- 8) Read the data from the multi-channel analyzer and determine the area under the photopeak;
- 9) Remove the sample and measure the background spectrum;
- 10) Substitute the data values into the proper equation;
- 11) Determine the neutron flux of the irradiation facility.

## 9. PROCEDURES OF NTD FOR SILICON

### 9.1. Overall procedure of NTD-Si wafer production

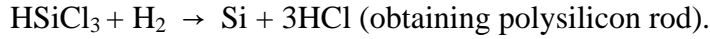
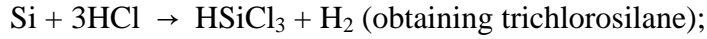
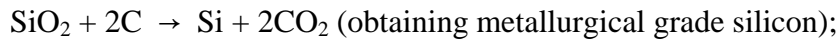
#### 9.1.1. Production of ultra pure polysilicon rods

The production procedure of ultra pure polysilicon is as follows:



This process is common for CZ-Si and FZ-Si, but the polysilicon rod of FZ-Si has a higher price because it is purer than one of CZ-Si. The starting material, silica, is very abundant in earth. After refining, metallurgical grade silicon with about 98% purity is obtained through

the reduction process using graphite in a kiln. Ultra pure trichlorosilane is made from a reaction with hydrochloric acid followed by distillation. Finally, it is reduced by hydrogen to obtain the ultra pure polysilicon rod. Its purity is about the ration of eleven-to-nine. The reactions corresponding to the respective steps are as follows:



### 9.1.2. FZ single crystal growth

This process is common for both gas doped FZ-Si and NTD Si:

(Polycrystal Grinding) → (Polycrystal Etching) → (FZ Single Crystal Growth) →  
(Block Cutting) → (Ingot Inspection) → (Ingot Surface Grinding).

While the CZ-Si crystal is made from broken polysilicon pieces, the FZ-Si crystal is made from a polysilicon rod whose surface is ground and etched. The FZ-Si crystal is then cut into a cylindrical ingot. An orientation of the crystal is measured by X-ray and its surface is polished. For the case of gas-doped FZ-Si, doping is accomplished during crystal growth. MCZ-Si is also sometimes treated with NTD. The NTD process for MCZ-Si after single crystal growth is the same as FZ-Si.

### 9.1.3. NTD

An overall NTD process is as follows:

(Ingot Etching) → (Initial Resistivity Measurement) → (Ship to a Reactor) → (Storage under suitable conditions e.g. no contact with stainless steel from storage racks) →  
(Neutron Irradiation and Decay of Induced Radioactivity) → (Cleaning and Residual Radioactivity Measurements) → (Ship Back) → (Heat Treatment) → (Resistivity Measurement) → (Feedback of Measured Resistivity to the Reactor).

A silicon ingot is etched to remove impurities on the surface and the initial resistivity on the top and bottom are measured before sending the ingot to a reactor site. The measured initial resistivity is forwarded to the irradiation site to determine the irradiation fluence. The procedure of NTD at the reactor site is shown in Figure 14.

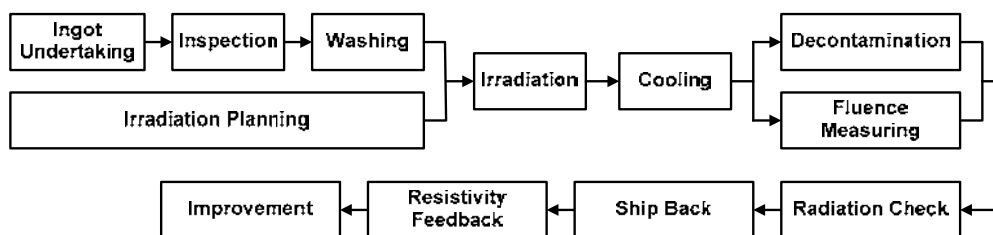


FIG. 14. NTD procedure at the reactor site.

Ingots may arrive at the reactor site earlier or later than their data, including target resistivity. The ingots are inspected and washed before irradiation. An irradiation plan may be set for a reactor operation cycle or for a certain time period. After irradiation and sufficient cooling, irradiated ingots are decontaminated. In parallel, the neutron fluence may be measured by activation detectors irradiated along with the ingots. The resistivity of each irradiated ingot may be predicted based on the fluence determined either by the activation detectors or irradiation records. The residual radioactivity is inspected for each decontaminated ingot. If the radioactivity is low enough, the ingot is shipped back to the customer. Preferably the packaging which had been provided by the supplier for the shipment to the reactor is reused. Final resistivity data returned by the customer are compared with predictions and utilized for the improvement of future neutron irradiations.

#### *9.1.4. Post NTD process*

The post NTD process in the wafer company is as follows:

(Ingot Inspection) → (Annealing) → (Surface Grinding) → (Slicing) → (Lapping) → (Annealing) → (Etching) → (Polishing) → (Resistivity Measurement). After inspection, the ingot is heat treated for annealing. A wafer is produced from each end of the ingot. Then the resistivity of each wafer is measured.

The method for measuring the resistivity is a four probe method. The uncertainty in the measurement is about 2%. The measuring positions are usually seven points — at centre, four points at a half radius away from the centre, and two points at five mm inside the edge. During the test irradiation step that begins the irradiation procedure, several characteristics such as resistivity variations, the annealing temperature, the carrier life time, impurities and the crystal deficiency may be investigated. The main impurities are usually oxygen and carbon.

After their return from the reactor, the ingots go through a special thermal process to anneal irradiation induced lattice damage. Thermal neutrons merely generate interstitial-vacancy pairs, whereas fast neutrons produce a large disordered region around the recoil track. In the case of excessive crystal damage, extensive trapping results in a behaviour similar to an intrinsic semiconductor and the irradiated silicon's resistivity is immeasurably high [53]. At any rate, it has been confirmed that the composition of the fast neutron spectrum affects the performance of NTD-Si. Thus, it seems that NTD customers prefer to irradiate their silicon ingots in heavy water reactors.

## **9.2. NTD procedures at the reactor site**

### *9.2.1. Ingot undertaking*

Some terminologies used by the wafer companies for the NTD irradiation are as follows:

- Batch: A quantity of silicon ingots which will be irradiated at the same time at an irradiation hole.
- Carton: A packing box for holding ingots at an irradiation site. It could contain multiple ingots.

- Packing list: A document containing a list of items related to the packing of the ingots. Generally, it contains the quantity of ingots, identification numbers, dimensions and weights, information on n or p type conduction in the starting material and initial and target resistivities.
- Invoice: A customs invoice. It may be required for the import and export processes of ingots.
- Instruction on neutron irradiation: A document containing the items related to neutron irradiation. It may be enclosed in the packing carton or transferred by fax or e-mail.
- Order: In addition to the instructions for neutron irradiation, a document which requests irradiations for specific ingots.

When a carton containing the ingots arrives at an irradiation site, first of all, a packing list or invoice should be confirmed. Then items inside the carton should be checked with the packing list. If the packaging was broken or raises concerns over ingot damage, it has to be recorded with a photograph and the ingot supplier should be notified immediately. If multiple companies have ordered the ingot irradiation, the documentation has to be classified by each company.

#### *9.2.2. Ingot inspection and washing*

After arrival of an ingot shipment, the ingots should be examined for defects, whether they originated before arriving at the irradiation site or not. The inspection should be done very carefully because a very small crack on the ingot may cause a large break during processing. Thus, if a small crack is found on the ingot, then the ingot supplier should be notified immediately. Ingots should be treated very carefully and only using gloves to avoid any mechanical damage and contamination. In some cases, each silicon ingot is covered by aluminium foil to protect its surface or to protect it against gross contamination [63, 68]. Also, it may be protected by a specially fabricated aluminium container [69].

The dimensions, including diameter, length and any eccentricity in shape, may be measured. During the measurements, the following matters should be confirmed. If the measured values are larger than the criteria below, a normal irradiation is impossible:

- Longitudinal length of the silicon ingot: Lengths at four points 90° apart are usually measured to confirm the eccentricity of the ingot;
- Deviation in the flank: Upper, middle and lower points are measured. Large deviations can cause an abnormal rotation of the irradiation rig or its precession;
- Diameter: The diameter should be within the allowed tolerance.

Careful washing of the ingot before irradiation is recommended to reduce the production of radioactive waste during decontamination. An ingot may be washed by demineralized water in an ultrasonic cleaner, and the moisture on the surface of the ingot is then dried. Further cleaning by wiping it with a soft tissue containing alcohol to remove organic contamination like fingerprints etc. is strongly recommended. Finally, any fine powder remaining on the surface is removed by a dry air gun.

Due to the delicacy of single crystalline Si ingots the efficacy of the cleaning effort is difficult to determine quantitatively. In any case careful optical inspection should ensure that the surface is homogeneously shiny, depending on the finish of its polish which may vary according to the supplier, and without any streaks or reams. In addition, once contamination of an irradiated ingot has been observed, the contaminating isotopes need to be identified, i.e.

by gamma spectrometry analysis of the solvent used, in order to optimize the cleaning procedure before further irradiations.

### *9.2.3. Irradiation planning*

Irradiation planning includes:

- Determination of the neutron fluence for each ingot based on the initial and target resistivity and an estimation of the irradiation time using the neutron flux predicted from previous irradiation results and other available data;
- Preparation of an irradiation schedule;
- Determination of the loading scheme of the ingots, taking into account the initial resistivity and length of each ingot;
- Determination of limiting radiation levels during irradiation and unloading of Si ingots and provision of storage positions sufficiently deep below the surface of the reactor pool to accept ingots with an unacceptably high dose rate due to, i.e. unexpected, highly activated trace elements within the ingot;
- Providing a data sheet for each ingot that contains relevant information on the ingot and will include the irradiation record, feedback resistivity, etc.;
- Working schedule and manpower assignment;
- Information of the wafer company on the irradiation and return schedule.

During irradiation planning, the following matters should be taken into account:

- The constant  $K$  may be slightly different depending on the wafer company if various irradiation channels or ingot canisters are used. A proper  $K$  value should be used for each company;
- The type of silicon ingot (initially n or p type) should be checked. Initially n type ingots are the usual case, but sometimes p type ingots are also found;
- The deviation of ingot diameter from the nominal diameter should be checked. This may influence the neutron flux at the ingot;
- The number of ingots per batch and their total length should be checked. When the total length of an irradiation batch is significantly short for a uniform irradiation, an addition of dummy silicon ingots with an appropriate length is recommended;
- The utilization plan for nearby irradiation facilities around the NTD hole should be checked. The neutron flux at the NTD hole can be affected by changes at nearby experimental facilities, especially at irradiation holes between the NTD hole and the core centre;
- If the irradiation time is not long enough compared to the time for ingot loading and unloading as well as to the ingot rotation speed, its impact upon a uniform irradiation has to be checked;
- Initial resistivity values for both ends of the ingot are usually provided from the wafer company. They are usually different, and proper compensation may be possible by careful arrangement of ingots in the irradiation rig.

### *9.2.4. Ingot irradiation*

After finishing the preparations for an irradiation, ingots are moved into the reactor site. Subsequently, neutron activation monitors are attached if necessary, and then ingots are

loaded into the irradiation rig. The rig is inserted into the irradiation hole for irradiation, as shown in Figure 15. The irradiation time and measurements of the neutron fluence by on line neutron flux monitor are marked. When the irradiation time or the neutron fluence reaches pre-set targets, the rig is unloaded. The process may be computerized for an automatic unloading of the rig. When the flux screen method is applied, the rig may function as a neutron screen as well, and its axial position may be adjusted for an optimum location. Rotation of the rig reduces the radial nonuniformity of the irradiation.

In the case that the relation between the signal of the on line flux monitor and the neutron flux in the ingot is not well known, a partitioned irradiation would be preferable. An accurate flux in the ingot and the axial non-uniformity of the irradiation can be obtained from the activation fluence monitors at the first irradiation. The first irradiation should be appropriately short to achieve an accurate irradiation during the second irradiation.

In automatic or semiautomatic irradiation facilities an automatic stop of the irradiation and unloading of the irradiation canisters can be foreseen in case of unstable rotation of the ingot or unexpected changes in reactor power.



*FIG. 15. Start of Si doping using the semiautomatic facility at FRM II. The irradiation channel is marked by a yellow circle.*



### *9.2.5. Ingot cooling*

After irradiation, the ingots are unloaded from the rig, and they are moved to a proper place for cooling. When a water pool is used for cooling, baskets may be utilized to store the ingots in the pool. If the baskets are not irradiated, they can be used for the withdrawal of the ingots from the pool as well. It is important not to use steel baskets but only baskets made from Al or organic materials. The cooling time is dependent on the fluence, but generally, two days of cooling is enough for the handling of ingots without any shielding. If the number of silicon ingots to be handled is large, sufficient space for cooling should be available.

### *9.2.6. Decontamination and radiation inspection*

After enough cooling to enable simple handling of the ingot, it should be decontaminated. Various cleansers such as pure water, soapy water, alcohol or acetone may be used. The highly contaminated areas are washed with an acid solution [69]. It is highly recommended to agree upon the chemicals to be used for cleaning the ingots with the Si supplier and to provide strict written rules for the cleaning procedures to the staff in charge. Finally, the ingot is sufficiently rinsed with pure water and dried. The use of a chemical for decontamination complicates waste management. The careful washing of the ingot before irradiation reduces the burden for decontamination and waste management.

A primary contamination inspection is done by a survey for surface contamination. The smearing test is a typical way to check the surface contamination. A scanning device using a Geiger-Müller detector for measuring  $\beta$  rays coming from the ingot is also used [70]. If the residual radiation level is higher than the criterion for carrying an ingot out of the irradiation site, it should be decontaminated again. It is desirable that this inspection should be done by members of a health physics team who are independently responsible for radiological safety at the irradiation site.

For the final radiation inspection an automatic device for release measurements, commercially available from various suppliers, is recommended. The advantage of such a device is the fixed geometry and calibration, and the issue of a reliable standard release document which will be accepted not only by reactor operators and customers but also by supervision authorities. Of course these devices need to be recalibrated periodically, and the calibration records need to be kept available for future control purposes.

Finally the acceptable radiation level for release needs to be determined. Typically this value is defined in national legislation for the producing reactor's country, but also additional or more stringent limits may be in place in the customer's home country. At FRM II in Germany, for example, the surface contamination must be  $<0.5$  Bq/cm<sup>2</sup> and the specific activity  $<0.09$  Bq/g in order to allow the unrestricted release of the ingot.

### *9.2.7. Neutron fluence measurement and quality control*

The neutron fluence measurement after irradiation can be done by using neutron activation monitors irradiated with the ingot simultaneously. In most cases the readings of the SPND monitors and/or irradiation time are sufficient. When the ingot comes out of cooling, the fluence monitors are detached from the ingot. If the monitors were attached on the ingot surface by adhesive tapes, these tapes and residual bonding agent should be completely removed. These materials can be extra sources of residual radioactivity.

Various neutron activation monitors can be utilized, and the radioactivity of the irradiated monitor can also be measured by various methods. The measured neutron fluence is compared with the signals of real time neutron monitors during the irradiation so that the signals can be calibrated. If the neutron fluence is within the irradiation criterion, it can be concluded that the irradiation was successfully completed.

#### *9.2.8. Ingot return shipment*

If the radiation level is low enough, then the ingot can be taken out of the irradiation site and shipped back. Documents required for shipping are usually an invoice, a traveller's sheet, a certification sheet from the radiation inspection and if applicable a customs clearance. On the traveller's sheet, the number of packages, ingot identification number, weight, target resistivity and the surface radioactivity contamination level may be included. In the return shipment, ingots are usually packed in the same cartons as in the initial undertaking.

#### *9.2.9. Irradiation improvement*

Achieving an accurate neutron fluence corresponding to the target resistivity as well as a uniform irradiation are the prime targets of neutron irradiation with NTD in order to maintain a high irradiation quality. Though the proportional constant between the resistivity and the neutron fluence can be determined theoretically, in practice this constant is determined experimentally for each company. Neutron fluence data measured at the irradiation site are compared with the measured resistivity data from NTD customers so the proportional constant can be precisely determined. As the amount of data increases, a more reliable proportional constant can be generated, and the quality of NTD can be continuously improved.

### **9.3. Warnings and irradiation control**

#### *9.3.1. Warnings during irradiation and treatment*

Warnings for abnormal situations during the irradiation and ingot treatments should be conveyed by sound or lamps on a display [71]. A failure in ingot rotation during irradiation is one of the most frequent cases. If the time without rotation is not short in comparison with the total irradiation time, it could severely affect the final radial resistivity distribution and result in resistivity gradient. The irradiation system may be designed to unload the irradiation rig automatically when rotation is stopped. These warnings can be connected to the instrumentation panel, the reactor control room and the NTD workers. Warnings may be needed for the following cases:

- In the unlikely event of a failure in ingot rotation;
- In the unlikely event of a non uniform reactor power;
- Abnormal signal from the SPND;
- Abnormal temperature rise due to a failure in ingot cooling;
- Abnormal high or low level of an ingot radioactivity in the storage rack or during the transportation of an ingot;
- When an ingot is strongly shocked;
- Occurrence of a transport error;
- End of an irradiation.

### 9.3.2. Abnormal results

The abnormal results that can occur after an irradiation process of silicon ingots are as follows:

- Damage to an ingot during the irradiation process;
- Preposterous over- or under-irradiation;
- Deviation from the irradiation criteria for accuracy and uniformity;
- Significantly different data between the final resistivity and the value received from the ingot supplier;
- Unexpected high residual radioactivity or hardly removable contamination;
- Abnormally high RRG.

For the case of considerable under-irradiation, it can be corrected by additional irradiation if the reactor operation time is available. For other abnormal cases, they must be reported to the ingot supplier as soon as possible. Close communication with the ingot suppliers is the best way to gain trust and resolve such abnormal events on mutual and transparent basis.

### 9.3.3. Computer based irradiation control

Computer based control of irradiation, such as typified by Figure 16, is not only convenient but also can guarantee more accurate irradiation. It may include automatic driving of the irradiation rig based on the pre-set irradiation time or fluence, adjustment of rig position depending on the control rod positions, monitoring of the rotation status, automatic withdrawal of the rig in case of no rotation, monitoring of real time signals, generation of warning signals, transmission of data to remote places, etc.

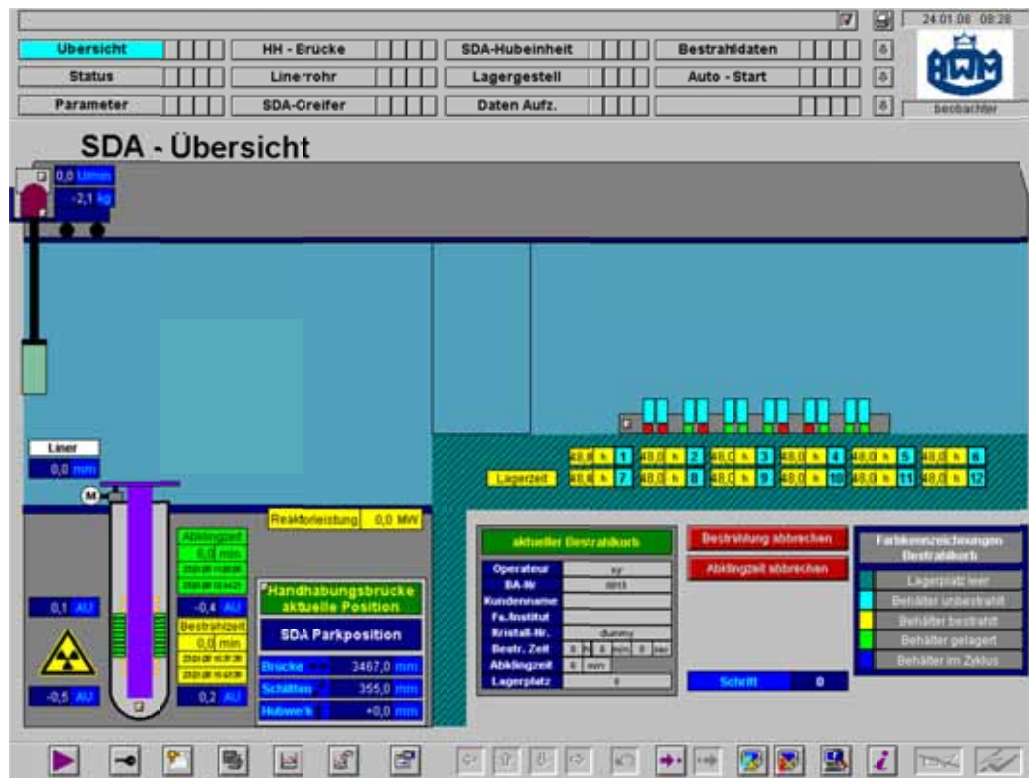


FIG.16. Computed assisted process control of Si doping at FRM II.

## 10. DOCUMENTATION

To ensure the efficient control of silicon throughout all the stages of the irradiation process, a good documentation system should be maintained. Generally, documentation for the NTD process can be divided into two groups, such as documentation for business purposes and for quality assurance. At the Harwell reactors, each piece of silicon was documented by the customer's name, consignment number (i.e. a collection of crystals delivered at the same time), arrival date, irradiation number, crystal identifications, diameter, length, neutron dose required, irradiation facility used, date and times of irradiation, details of rotation, date of health physics check, financial details, invoice numbers and date of dispatch [28]. Also, at the MURR reactor, the database was divided into three groups, such as the lot file, the schedule file and the position table [72].

The following items may be included in the documentation for NTD quality:

- Customer name;
- Ingot identification number;
- Rod number;
- Weight;
- Length;
- Diameter ;
- Initial resistivity of upper side;
- Initial resistivity of lower side;
- Average initial resistivity;
- Target resistivity;
- Requested thermal neutron;
- Delivery date;
- Name of irradiation facility;
- Target resistivity;
- $K$  value for each company or each period;
- Control rod position;
- Irradiation position and direction in the rig;
- Expected thermal neutron flux;
- Expected irradiation time;
- Number of irradiations for each ingot;
- Signals of SPND before, during, after irradiation;
- Started timing of irradiation;
- Ended timing of irradiation;
- Measured irradiation fluence;
- Ratio of irradiated fluence to targeted fluence;
- Estimated resistivity after irradiation;
- Result of the residual radioactivity check;
- Abnormal events;
- Feedback resistivity from ingot supplier.

Since more and more wafer companies are obliged by their customers to cooperate only with sub-contractors being certified according to the regulations of the EN ISO 9001 quality control system, it is recommended to research reactors working in the field of NTD to get licensed according to this system as it has been done long in the past by DR3 in Risø (Denmark) and more recently by BR2, FRM II and other major NTD players.

## 11. POSSIBLE EMERGENCIES

In general the physical properties of Si make it a favourable and easy going target for being irradiated in a nuclear reactor. In this respect the most important parameters are:

- Its moderate neutron capture cross section;
- Only one natural isotope of silicon is radioactive;
- The short half-life of Si-31 (2.62 h);
- The absence of activation induced gamma radiation.

Nonetheless there are few concerns which must be kept in mind during the design of a Si doping facility. First, single crystalline Si ingots are extremely brittle. Consequently the reactor facility should provide a loading/unloading area guaranteeing that Si flakes, which will unavoidably occur during handling of large amounts of Si, are collected and not conveyed into more sensitive regions of the reactor pool. In addition the loading/unloading area has to be easily accessible for cleaning, for example, by means of a vacuum assisted system.

Also, Si ingots are considerably heavy. At FRM II, for example, Si batches up to 35 kg are irradiated simultaneously using a single irradiation canister. Consequently devices for lifting and transporting Si batches within the reactor pool must be laid out with a sufficient safety margin against undesired drops that might cause serious damage to important reactor installations. The same applies to the mechanical design of the interlocking device between the lifter and the canister containing the Si ingots.

Finally, Si is usually provided by suppliers in batches possessing excellent purity. Nonetheless a strategy dealing with irradiated Si ingots containing an unacceptable amount of activated impurities should be available before the start of the irradiation service. If an unexpected high level of radioactivity has been detected upon measurement of an irradiated Si ingot, it must be verified at once whether the unexpected radioactivity is due to contamination or activation. In case of contamination the entire process of Si handling at the reactor must be checked in order to determine at which stage the ingot was polluted. In case of activation, on the other hand, the supplier's fabrication process most likely introduced the foreign atoms, and the supplier must be informed immediately to allow him to inspect his manufacturing process. In that case the Si ingot will most probably need to be disposed by the reactor according to national regulations regarding radioactive waste classification and treatment, since Si suppliers usually do not have a handling license for radioactive materials. Developing a protocol in response to such scenarios is a recommended course of action during negotiations between the reactor facility and Si supplier and should be included in the subsequent contract.

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## ANNEX I.

### Database of Research Reactors Involved in NTD

This document has been developed as a supporting database of research reactors to describe their functional characteristics and NTD capabilities. The intention is to promote, share and provide an overall picture of the usefulness and effectiveness of irradiation facilities available among the Member States as well as to increase cooperation between different research reactor centres.

The information was collected through questionnaires sent to research reactor owners. All data pertaining to the general description of reactor parameters; irradiation holes or facilities; available expertise; and experience and availability for NTD of Si are stored in the database. It is expected that this database will be useful to nuclear facility operators, nuclear system designers, professional engineers and scientists among the Member States. This database should be in particular useful for potential customers of NTD of Si.

It has to be recognized that in addition to 18 research reactors included in this database, several other research reactors offer the service of Si doping on commercial basis and contribute considerably to the worldwide production of NTD-Si, i.e. in the Russian Federation and the US. Unfortunately more detailed information on these facilities was not made available due to confidentiality restrictions.

The IAEA is grateful to all research reactor managers who have responded to the questionnaires and made this information available for public use for other Member States.

#### I-1. Australia: OPAL (20 MW)

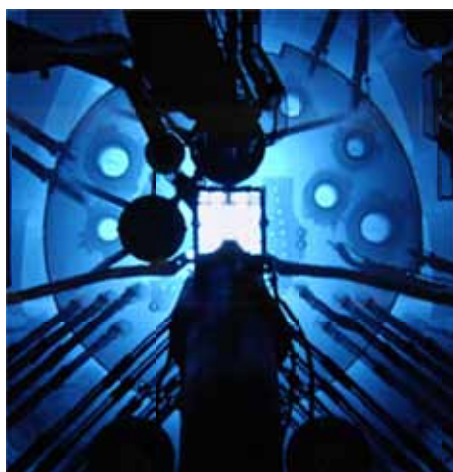
##### I-1.1. General description of the reactor parameters

Reactor name	OPAL	
Reactor type	Pool	
First criticality	August 2006	
Power level	20 MW	
Maximum flux [ $\text{cm}^{-2}\text{s}^{-1}$ ]	(thermal) $2 \times 10^{14}$ (fast) $2 \times 10^{14}$	
Fuel description	Material/Density	$\text{U}_3\text{Si}_2\text{-Al}$ 4.8 g/cm <sup>3</sup>
	Enrichment	19.7% wt.
	Type (element/bundle)	MTR, flat plate
	Clad	Al
Absorber	Hf	
Coolant	Light water	
Moderator	D <sub>2</sub> O	
Reflector	D <sub>2</sub> O	
Core cooling	Forced light water at 2000 m <sup>3</sup> /h	

##### I-1.2. Irradiation hole or facility

Name	Position	Utilization field	Flux [ $\text{cm}^{-2}\text{s}^{-1}$ ] (thermal/fast)	Status
HF (2)	Reflector	Isotope	$2 \times 10^{14}$	In operation

		production		
MF (3)	Reflector	Isotope production	$1.3 \times 10^{14}$	In operation
LF (12)	Reflector	Isotope production	$8 \times 10^{13}$	In operation
LRT (55)	Reflector	Isotope production	$2 \times 10^{12} - 1 \times 10^{14}$	In operation
SRT (2)	Reflector	Isotope production	$6 \times 10^{12} - 2 \times 10^{13}$	In operation
NTD (6)	Reflector	Isotope production	$1.7 \times 10^{12} - 1.6 \times 10^{13}$	In operation



*FIG. I-1.1. Core view of OPAL reactor.*

### **I-1.3. Available expertise**

Field	Computer code	Equipment or facility	Capability including man power	Application or experience
Neutronic design	MCNP			
Thermohydraulic design	CFX			
Mechanical design	CAD			
Safety analysis	PARET/RELAP			
Instrumentation and control				
Radiation dosimetry				
Radiation protection or residual radioactivity monitoring	MCNP/ Microshield			
Decontamination				
Theoretical background for material irradiation				

### I-1.4. Experience and available facility for silicon doping

Objective	Si doping
Irradiation material	Silicon
Position	Channel in Reflector
Name of hole or facility	NTD1 - NTD6 (6 facilities in total)
Thermal neutron flux (with or without target)	$1.7 \times 10^{12} - 1.6 \times 10^{13}$
Fast neutron flux	$1 \times 10^8 - 2 \times 10^{10}$
Gamma ray flux	
Longitudinal length of hole or facility	600 mm (usable)
Diameter or size	237 mm
Diameter of the accessible irradiated material	4", 5", 6" and 8"
Way of uniform irradiation	SS and Al flux screen, Rotation at $\approx 15 \text{ min}^{-1}$
Way of reduction for background radiation	Light water
Irradiation time (max., min. and average)	3 h – 2 d; average 15 h
Way of rotation	Hydraulic turbine system
Way of cooling for irradiation object	Forced light water
Way of monitoring the irradiation fluence	SPND + irradiation time
Way of decontamination	Wash in demin water and cloth dry
Way of residual radioactivity survey	Gamma ray counter
Exemption criteria for residual radioactivity	0.09 Bq/g, 0.5 Bq/cm <sup>2</sup>
Cadmium ratio for gold or other material	> 30
Capacity per year	$\approx 25 \text{ t}$ (current)
Production during last 5 years	$\approx 43 \text{ t}$ (last 2 years)
Present status	In operation

## I-2. Belgium: BR2 (100 MW)

### I-2.1. General description of the reactor parameters

Reactor name	BR2	
Reactor type	Tank in Pool MTR	
First criticality	June 1961, major refurbishment in 1995 - 1997	
Power level	up to 100 MW	
Maximum flux [cm <sup>-2</sup> s <sup>-1</sup> ]	(thermal) $1 \times 10^{15}$ (fast) $6 \times 10^{14}$	
Fuel description	Material/Density	Cermet UAl <sub>x</sub> ; 1.3 g U <sub>tot</sub> /cm <sup>3</sup>
	Enrichment	92,5 % wt
	Type (element/bundle)	Typically 32 assemblies
	Clad	Ag <sub>3</sub>
Absorber	Hf	
Coolant	Light water	
Moderator	H <sub>2</sub> O + Be	
Reflector	Be	
Core cooling	forced light water stream at 1.8 m <sup>3</sup> /s	

### I-2.2. Irradiation hole or facility

Name	Position	Utilization field	Flux [ $\text{cm}^{-2}\text{s}^{-1}$ ] (thermal/fast)	Status
6 PRF	Reflector	Mo-99 production.	$2.5 \times 10^{14}$ /	In operation
7 H1	Central Be plug	Isotope production	Up to $1 \times 10^{15}$ /	In operation
In core	Within fuel assembly	Isotope production by fast neutrons	Up to $6 \times 10^{14}$ (fast neutrons)	In operation
BRIGITTE	pool	Gamma irradiation	--/	commission

### I-2.3. Available expertise

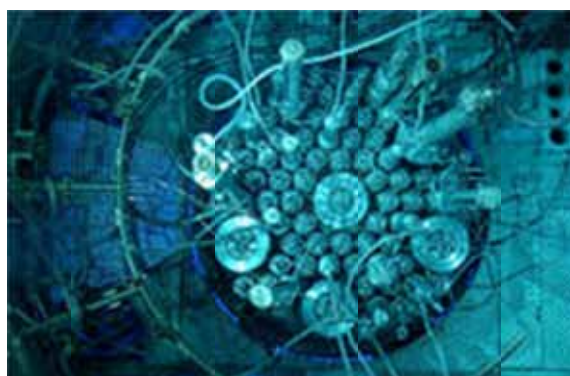
Information not available.

### I-2.4. Experience and available facility for silicon doping, gemstone colouration and membrane fabrication (Separately described for each facility)

ITEM-A	DESCRIPTION
Objective	NTD-Silicon
Irradiation material	Silicon
Name of hole or facility	SIDONIE
Position	Within Be channel
Thermal neutron flux (with or without target)	$5.5 \times 10^{13} \text{ cm}^{-2}\text{s}^{-1}$
Fast neutron flux	$0.2 \times 10^{13} \text{ cm}^{-2}\text{s}^{-1}$
Gamma ray flux	2 W/g Al
Longitudinal length of hole or facility	Batch length up to 800 mm
Diameter or size	200 mm
Diameter of the accessible irradiated material	125 mm
Way of uniform irradiation	Translation and rotation
Way of reduction for background radiation	ALARA principle
Irradiation time (max., min. and average)	15 min – 25 h; average 3.5 h/35 $\Omega\text{cm}$
Way of rotation	Electrical drive
Way of cooling for irradiation object	Forced convection by $\text{H}_2\text{O}$
Way of monitoring the irradiation fluence	SPND + irradiation time
Capacity per year	15 t
Cd ratio	25

ITEM-B	DESCRIPTION
Objective	NTD-Silicon
Irradiation material	Silicon
Position	6 channels in pool outside reactor vessel
Name of hole or facility	POSEIDON
Thermal neutron flux (with or without target)	$5.3 \times 10^{12} \text{ cm}^{-2}\text{s}^{-1}$
Fast neutron flux	$0.1 \times 10^{13} \text{ cm}^{-2}\text{s}^{-1}$
Gamma-ray flux	0.8 W/ $\text{cm}^3$

Longitudinal length of hole or facility	Hole length 1275 mm, batch length: 500 mm
Diameter or size	200 mm
Diameter of the accessible irradiated material	150 mm, 200 mm
Way of uniform irradiation	Inversion and rotation
Way of reduction for background radiation	Irradiation in graphite moderator
Irradiation time (max., min. and average)	average 27 h/48 Ωcm
Way of rotation	Electrical drive
Way of cooling for irradiation object	Natural convection by H <sub>2</sub> O
Way of monitoring the irradiation fluence	SPND + irradiation time
Way of decontamination	Ultrasound in a demineralized water medium
Way of residual radioactivity survey	By independent authority
Exemption criteria for residual radioactivity	IAEA regulations for 'exempt' radioactive material
Capacity per year	60 t (SIDONIE + POSEIDON)
Production during last 5 years	10 t (In core) and ~2 t (Out core)
Present status	In operation



*FIG. I-2.1 Core view of BR2 reactor.*

### **I-3. Brazil: IEA-R1 (5 MW)**

#### **I-3.1. General description of the reactor parameters**

Reactor name	IEA-R1	
Reactor type	MTR Open swimming pool reactor	
First criticality	September 1957	
Power level	5 MW	
Maximum flux [cm <sup>-2</sup> s <sup>-1</sup> ]	(thermal) 2.7x10 <sup>14</sup> (fast) 1.1x10 <sup>14</sup>	
Fuel description	Material/Density	U <sub>3</sub> Si <sub>2</sub> -Al , 3.0 U <sub>t</sub> /cm <sup>3</sup>
	Enrichment	19.75%
	Type (element/bundle)	31 MTR assemblies/ core
	Clad	Aluminium
Absorber	Cd+In+Ag	
Coolant	Light water	
Moderator	Light water	
Reflector	Graphite, Be	
Core cooling	Forced light water	

### I-3.2. Irradiation hole or facility

Name	Position	Utilization field	Flux [ $\text{cm}^{-2}\text{s}^{-1}$ ] (thermal/fast)	Status
Beam tubes (9)		diffraction radiography BNCT Photonuclear reactions		Operational
In core positions		Isotope production Material testing	Thermal: $4.6 \times 10^{13}$ Fast: $1.3 \times 10^{14}$	Operational
Pneumatic	Reflector border	NAA		Operational

### I-3.3. Available expertise

Information not available.

### I-3.4. Experience and available facility for silicon doping

ITEM	DESCRIPTION
Objective	Si doping
Irradiation material	Silicon ingots
Position	within graphite reflector
Name of hole or facility	Si irradiation rig
Thermal neutron flux (with or without target)	Without up to $7 \times 10^{12} \text{ cm}^{-2}\text{s}^{-1}$
Fast neutron flux	
Gamma ray flux	
Longitudinal length of hole or facility	Ingot length: 500 mm
Diameter or size	175 cm
Diameter of the accessible irradiated material	125 mm
Way of uniform irradiation	Inversion of 2 ingots and rotation
Way of reduction for background radiation	Position in graphite reflector
Irradiation time (max., min. and average)	
Way of rotation	Electrical drive
Way of cooling for irradiation object	Natural convection
Way of monitoring the irradiation fluence	SPND, time
Way of decontamination	Distilled water
Way of residual radioactivity survey	Beta counter
Exemption criteria for residual radioactivity	7.4 Bq/g,
Cadmium ratio for gold or other material	$\approx 29$ for Cobalt
Capacity per year	$\approx 1.2 \text{ t} - 30 \text{ } \Omega\text{cm}$
Production during last 5 years	
Present status	Operational

## I-4. China: CARR (60MW)

### I-4.1. General Description of the Reactor Parameters

Reactor name	CARR	
Reactor type	Pool	
First criticality	2010	
Power level	60 MW	
Maximum flux [ $\text{cm}^{-2}\text{s}^{-1}$ ]	(thermal) $8 \times 10^{14}$ (fast) $1 \times 10^{15}$	
Fuel description	Material/Density	$\text{U}_3\text{Si}_2\text{-Al}/4.3 \text{ g/cm}^3$
	Enrichment	19.75 % wt.
	Type (element/bundle)	MTR
	Clad	Al
Absorber	Hf	
Coolant	Light water	
Moderator	$\text{D}_2\text{O}$	
Reflector	$\text{D}_2\text{O}$	
Core cooling	Force	

### I-4.2. Irradiation hole or facility

Name	Position	Utilization field	Flux [ $\text{cm}^{-2}\text{s}^{-1}$ ] (thermal/fast)	Status
CI	Reflector	Cooling needed IRS	$8 \times 10^{14} /$	prepared
I-125	Reflector	I-125	$4 \times 10^{14} /$	prepared
MT	Reflector	Material irradiation monitoring	$/1 \times 10^{14}$	prepared
AT	Reflector	NAA	$6 \times 10^{14} /$	prepared
NI	Reflector	RIs production	$6 \times 10^{14} /$	prepared

### I-4.3. Available expertise

Field	Computer code	Equipment or facility	Capability including man power	Application or experience
Neutronic design	WIMS/CITAT ION/MCNP			
Thermohydraulic design	CFX/CFD			
Mechanical design	CAD/TDM			
Safety analysis	Relap			
Instrumentation and control				
Radiation dosimetry				
Radiation protection or residual radioactivity monitoring				
Decontamination				
Theoretical background for material irradiation				



#### I-4.4. Experience and available facility for silicon doping, gemstone colouration and membrane fabrication

ITEM	DESCRIPTION
Objective	Silicon doping
Irradiation material	Silicon
Position	D <sub>2</sub> O tank
Name of hole or facility	NTD Channel
Thermal neutron flux (with or without target)	$1 \times 10^{14} \text{ cm}^{-2} \text{ s}^{-1}$ (without target)
Fast neutron flux	$< 1 \times 10^{12} \text{ cm}^{-2} \text{ s}^{-1}$
Gamma ray flux	
Longitudinal length of hole or facility	600 mm for NTD
Diameter or size	
Diameter of the accessible irradiated material	3 in (1), 4 in (1), 5 in (5)
Way of uniform irradiation	upside down and rotate
Way of reduction for background radiation	
Irradiation time (max., min. and average)	
Way of rotation	Hydraulic
Way of cooling for irradiation object	nature
Way of monitoring the irradiation fluence	SPND
Way of decontamination	rinsed by hydrochloric or hydrofluoric acid
Way of residual radioactivity survey	
Exemption criteria for residual radioactivity	surface $< 10^{-4} \mu\text{Ci}/\text{cm}^2$
Cadmium ratio for gold or other material	
Capacity per year	50 t/yr (estimate)
Production during last 5 years	
Present status	Under construction and installation

#### I-5. China: HFETR (125 MW)

##### I-5.1. General description of the reactor parameters

Reactor name	High Flux Engineering Test Reactor (HFETR)	
Reactor type	Pressure vessel	
First criticality	Dec. 27, 1979	
Power level	125 MW	
Maximum flux [ $\text{cm}^{-2} \text{ s}^{-1}$ ]	(thermal) $6.2 \times 10^{14}$ (fast) $1.7 \times 10^{15}$	
Fuel description	Material/Density	<sup>235</sup> U-Aluminium
	Enrichment	High: 90%, LFU: 20%
	Type (element/bundle)	Multi-tubular fuel element
	Clad	305-Aluminium
Absorber	Cd-In-Ag	
Coolant	Water	
Moderator	Water	
Reflector	Beryllium	
Core cooling	Force water cooling	

### I-5.2. Irradiation hole or facility

Name	Position	Utilization field	Flux [ $\text{cm}^{-2}\text{s}^{-1}$ ] (thermal/fast)	Status
$\Phi$ 230 mm	Reflector	Fuel, NTD	$1.2 \times 10^{14} / 3.4 \times 10^{14}$	In use
$\Phi$ 150 mm	Core	Fuel, material	$4.2 \times 10^{14} / 1.6 \times 10^{15}$	Core center
$\Phi$ 150 mm	Core	NTD	$2.9 \times 10^{14} / 1.0 \times 10^{15}$	In use
$\Phi$ 120 mm	Reflector	Material, NTD	$1.2 \times 10^{14} / 2.5 \times 10^{14}$	In use
$\Phi$ 63 mm	Core	Fuel	$3.1 \times 10^{14} / 1.2 \times 10^{15}$	Other

### I-5.3. Available expertise: Division and dedicated persons for each field

Information not available.

### I-5.4. Experience and available facilities for silicon doping, gemstone colouration and membrane fabrication

ITEM	DESCRIPTION
Objective	Silicon doping
Irradiation material	Silicon
Position	HFETR's hole
Name of hole or facility	$\Phi$ 230, $\Phi$ 150, $\Phi$ 120 mm
Thermal neutron flux (with or without target)	$1.2 \times 10^{14} \text{ cm}^{-2}\text{s}^{-1} \sim 4.2 \times 10^{14} \text{ cm}^{-2}\text{s}^{-1}$
Fast neutron flux	$0.25 \times 10^{15} \sim 1.6 \times 10^{15} \text{ cm}^{-2}\text{s}^{-1}$
Gamma ray heating	0.9 ~ 11.0 W/g
Longitudinal length of hole or facility	1000 mm (reactivity), 500 mm for NTD
Diameter or size	$\Phi$ 120 ~ $\Phi$ 230 mm
Diameter of the accessible irradiated material	six inches
Way of uniform irradiation	upside down and rotate
Way of reduction for background radiation	
Irradiation time (max., min. and average)	
Way of rotation	automatic rotation (hydraulic)
Way of cooling for irradiation object	water-naturally cooling
Way of monitoring the irradiation fluence	
Way of decontamination	supersonic wave + hydrochloric
Way of residual radioactivity survey	clean, cleanse
Exemption criteria for residual radioactivity	survey $\beta < \text{criteria(GB)} = 10^{-4} \mu\text{Ci}/\text{cm}^2$
Cadmium ratio for gold or other material	
Capacity per year	10 t
Production during last 5 years	
Present status	in use

## I-6. China: MJTR (5 MW)

### I-6.1. General description of the reactor parameters

Reactor name	Ming Jiang Test Reactor(MJTR)	
Reactor type	Pool	
First criticality	1992	
Power level	5 MW	
Maximum flux [ $\text{cm}^{-2}\text{s}^{-1}$ ]	(thermal) $8.03 \times 10^{13}$ (fast) $1.43 \times 10^{14}$	
Fuel description	Material/Density	$^{235}\text{U}$ -Aluminium
	Enrichment	HFU: 90% , LFU: 20%
	Type (element/bundle)	Multi-tubular fuel element
	Clad	305-Aluminium
Absorber	Cd-In-Ag	
Coolant	Water	
Moderator	Water	
Reflector	Beryllium	
Core cooling	Fore water cooling	

### I-6.2. Irradiation hole or facility

Name	Position	Utilization field	Flux [ $\text{cm}^{-2}\text{s}^{-1}$ ] (thermal/fast)	Status
$\Phi 220$ mm	Reflector	NTD	$4.23 \times 10^{13}/0.96 \times 10^{14}$	In use
$\Phi 180$ mm	Reflector	NTD	$4.52 \times 10^{13}/0.98 \times 10^{14}$	In use
$\Phi 120$ mm	Reflector	NTD	$5.82 \times 10^{13}/1.09 \times 10^{14}$	In use
$\Phi 63$ mm	Core	Material, NTD	$6.03 \times 10^{13}/1.21 \times 10^{14}$	Other

### I-6.3. Available expertise

Information not available.

#### I-6.4. Experience and available facility for silicon doping, gemstone colouration and membrane fabrication

ITEM	DESCRIPTION
Objective	Silicon doping
Irradiation material	Silicon
Position	reflector, core
Name of hole or facility	Φ 220 ~ Φ63 mm
Thermal neutron flux (with or without target)	
Fast neutron flux	
Gamma ray flux	0.4 W/g
Longitudinal length of hole or facility	1000 mm (reactivity), 500 mm for NTD
Diameter or size	Φ220 ~ Φ63 mm
Diameter of the accessible irradiated material	2 ~ 6 in
Way of uniform irradiation	upside down and rotate
Way of reduction for background radiation	
Irradiation time (max., min. and average)	
Way of rotation	automatic rotation (hydraulic)
Way of cooling for irradiation object	water-naturally cooling, nature
Way of monitoring the irradiation fluence	
Way of decontamination	supersonic wave + Hydrochloric
Way of residual radioactivity survey	cleanse
Exemption criteria for residual radioactivity	survey $\beta < \text{criteria (China GB)} = 10^{-4} \mu\text{Ci/cm}^2$
Cadmium ratio for gold or other material	
Capacity per year	20 t
Production during last 5 years	
Present status	in use

### I-7. Czech Republic: LVR-15 (10 MW)

#### I-7.1. General description of the reactor parameters

Reactor name	LVR-15	
Reactor type	Tank reactor	
First criticality	1989	
Power level	10 MW	
Maximum flux [ $\text{cm}^{-2}\text{s}^{-1}$ ]	(thermal) $1.8 \times 10^{14}$ (fast) $2 \times 10^{14}$	
Fuel description	Material/Density	UO <sub>2</sub> +Al
	Enrichment	36%/19,7%
	Type (element/bundle)	IRT-2M/IRT-4M, 4/8 quarter tube in one fuel assembly
	Clad	Al, SAV 1
Absorber	Boron carbide	
Coolant	Light water	
Moderator	Light water	
Reflector	Be blocks/light water	
Core cooling	Forced light water flow 1750 m <sup>3</sup> /h	

### I-7.2. Irradiation hole or facility

Name	Position	Utilization field	Flux [ $\text{cm}^{-2}\text{s}^{-1}$ ] (thermal/fast)	Status
DONA	C, D 9, 10 Behind reflector	Si	$2.7 \times 10^{13} / 2.2 \times 10^{12}$	In operation
Vertical channel	H5, H6, H8 Reflector	Radioisotope Production	H5, H8 $1.3 \times 10^{14}$ H8 $8 \times 10^{13}$	In operation
Central channel	D.E,4,5	Ir or U for $\text{Mo}^{99}$ Irradiation	$1.7 \times 10^{14}$	In operation
Rabbit system	H1 reflector	NAA	$5 \times 10^{13}$	In operation

### I-7.3. Available expertise

Field	Computer code	Equipment or facility	Capability including man power	Application or experience
Neutronic design	MCPN, NODER			
Thermohydraulic design	RELAP 5, LVR-15 STA			
Mechanical design	CAD			
Safety analysis	2010			

### I-7.4. Experience and available facility for silicon doping

ITEM	DESCRIPTION
Objective	Si doping
Irradiation material	Silicon
Position	C, D, 9, 10 channel behind reflector
Name of hole or facility	DONA 5
Thermal neutron flux (with or without target)	$2.7 \times 10^{13} \text{ cm}^{-2}\text{s}^{-1}$
Fast neutron flux	$2.2 \times 10^{12} \text{ cm}^{-2}\text{s}^{-1}$
Gamma ray flux	
Longitudinal length of hole or facility	300 mm
Diameter or size	4"
Diameter of the accessible irradiated material	2 – 4"
Way of uniform irradiation	Stainless steel shielding, rotation of ingot 1.3 times per min
Way of reduction for background radiation	-
Irradiation time (max., min. and average)	20 min – 20 h; average 2 h
Way of rotation	Electrical driver
Way of cooling for irradiation object	Forced reactor cooling water
Way of monitoring the irradiation fluence	Neutron coax and irradiation time
Way of decontamination	Bath and decontamination
Way of residual radioactivity survey	Gamma ray counter
Exemption criteria for residual radioactivity	$4 \text{ kBq/m}^2$ for $\text{P}^{32}$
Cadmium ratio for gold or other material	5.3

Capacity per year	500 kg/y depending on orders
Production during last 5 years	1500 kg
Present status	In operation



FIG. I-7.1. Core view of LVR-15 reactor.

## I-8. France: ORPHÉE (14 MW)

### I-8.1. General description of the reactor parameters

Reactor name	ORPHEE	
Reactor type	pool	
First criticality	1980	
Power level	14 MW	
Maximum flux [ $\text{cm}^{-2}\text{s}^{-1}$ ]	(thermal) $3 \times 10^{14}$ (fast) $3 \times 10^{12}$	
Fuel description	Material/Density	UAl
	Enrichment	93%
	Type (element/bundle)	plates
	Clad	Al
Absorber	Hf	
Coolant	$\text{H}_2\text{O}$	
Moderator	$\text{D}_2\text{O}$	
Reflector	$\text{H}_2\text{O}$ + Beryllium	
Core cooling	forced light water stream	

### I-8.2. Irradiation hole or facility

Name	Position	Utilization field	Flux [ $\text{cm}^{-2}\text{s}^{-1}$ ] (thermal/fast)	Status
I1	$\text{D}_2\text{O}$ tank	Isotope production	(thermal) $6 \times 10^{13}$	In operation
R1, R2 for Ø 125 mm	$\text{D}_2\text{O}$ tank	Silicon NTD	(thermal) $1 \times 10^{13}$	In operation
E3, E4 for Ø 151 mm	$\text{D}_2\text{O}$ tank	Silicon NTD	(thermal) $5 \times 10^{11}$	In operation

### I-8.3. Available expertise

Information not available.

### I-8.4. Experience and available facility for silicon doping

ITEM	DESCRIPTION
Objective	Si doping
Irradiation material	Silicon
Position	4 channels in reflector (D <sub>2</sub> O tank)
Name of hole or facility	-
Thermal neutron flux (with or without target)	$1.0 \times 10^{13} \text{ cm}^{-2} \text{ s}^{-1}$
Fast neutron flux	$1.0 \times 10^{10} \text{ cm}^{-2} \text{ s}^{-1}$
Gamma ray flux	
Longitudinal length of hole or facility	600 mm
Diameter or size	160 mm
Diameter of the accessible irradiated material	152 mm
Way of uniform irradiation	flux screen, Rotation of ingot at 2 rpm
Way of reduction for background radiation	-
Irradiation time (max., min. and average)	2 h – 20 h; average 8 h
Way of rotation	Electrical drive
Way of cooling for irradiation object	Natural convection by H <sub>2</sub> O
Way of monitoring the irradiation fluence	SPND + irradiation time
Way of decontamination	Ultrasonic bath +
Way of residual radioactivity survey	Gamma ray counter
Exemption criteria for residual radioactivity	IAEA
Cadmium ratio for gold or other material	500
Capacity per year	≈ 10 t (depending on Si diameter)
Production during last 5 years	≈ 30 t
Present status	In operation

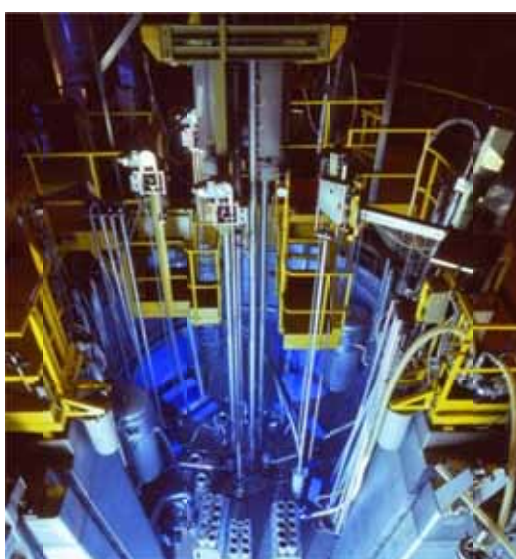


FIG. I-8.1. Core view of Orphée reactor.

## I-9. France: OSIRIS (70 MW)

### I-9.1. General description of the reactor parameters

Reactor name	OSIRIS	
Reactor type	pool	
First criticality	1966	
Power level	70 MW	
Maximum flux [ $\text{cm}^{-2}\text{s}^{-1}$ ]	(thermal) $3 \times 10^{14}$ (fast) $2.3 \times 10^{14}$	
Fuel description	Material/Density	$\text{U}_3\text{Si}_2\text{Al}$
	Enrichment	19.75%
	Type (element/bundle)	plates
	Clad	Al
Absorber	Hf	
Coolant	$\text{H}_2\text{O}$	
Moderator	$\text{H}_2\text{O}$	
Reflector	$\text{H}_2\text{O}$ + Beryllium	
Core cooling	forced light water stream at $5300 \text{ m}^3/\text{h}$	

### I-9.2. Irradiation hole or facility

Name	Position	Utilization field	Flux [ $\text{cm}^{-2}\text{s}^{-1}$ ] (thermal/fast)	Status
12 channels	Core	Material testing	Fast $1\text{--}2.3 \times 10^{14}$	In operation
15 channels	reflector	Material or fuel testing	Thermal $1\text{--}3 \times 10^{14}$	In operation
		Mo-99 & isotope production		In operation

### I-9.3. Available expertise

Information not available.



#### I-9.4. Experience and available facility for silicon doping

ITEM	DESCRIPTION
Objective	Si doping
Irradiation material	Silicon
Position	2 channels in reflector (pool)
Name of hole or facility	DIODON P1 and P2
Thermal neutron flux (with or without target)	$4.0 \times 10^{13} \text{ cm}^{-2} \text{ s}^{-1}$
Fast neutron flux	$5.0 \times 10^{12} \text{ cm}^{-2} \text{ s}^{-1}$
Gamma ray flux	
Longitudinal length of hole or facility	500 mm
Diameter or size	130 mm
Diameter of the accessible irradiated material	125 mm
Way of uniform irradiation	Ni flux screen, Rotation of ingot at $2 \text{ min}^{-1}$
Way of reduction for background radiation	-
Irradiation time (max., min. and average)	1 h – 10 h; average 5 h
Way of rotation	Electrical drive
Way of cooling for irradiation object	Natural convection by $\text{H}_2\text{O}$
Way of monitoring the irradiation fluence	SPND + irradiation time
Way of decontamination	Ultrasonic bath +
Way of residual radioactivity survey	Gamma ray counter
Exemption criteria for residual radioactivity	IAEA
Cadmium ratio for gold or other material	10
Capacity per year	$\approx 12 \text{ t}$ (depending on Si diameter)
Production during last 5 years	$\approx 20 \text{ t}$
Present status	In operation



*FIG. I-9.1. Core view of Osiris reactor.*

## I-10. Germany: FRM II (20 MW)

### I-10.1. General description of the reactor parameters

Reactor name	FRM II	
Reactor type	Pool	
First criticality	2004	
Power level	20 MW	
Maximum flux [ $\text{cm}^{-2}\text{s}^{-1}$ ]	(thermal) $8 \times 10^{14}$ (fast) $2 \times 10^{14}$	
Fuel description	Material/Density	$\text{U}_3\text{Si}_2\text{-Al}$ 3 g/cm <sup>3</sup> , 1.5 g/cm <sup>3</sup>
	Enrichment	92.5% wt.
	Type (element/bundle)	Compact Core
	Clad	AlFeNi
Absorber	Hf	
Coolant	Light water	
Moderator	D <sub>2</sub> O	
Reflector	D <sub>2</sub> O	
Core cooling	forced light water stream at 300 kg/s	

### I-10.2. Irradiation hole or facility

Name	Position	Utilization field	Flux [ $\text{cm}^{-2}\text{s}^{-1}$ ] (thermal/fast)	Status
KBA	Reflector	Cooling needed Isotope prod.	$1.3 \times 10^{14}$ /	In operation
RPA	Reflector	NAA	Up to $7 \times 10^{13}$ /	In operation
JBE70	Reflector	NAA, Fission track	$2 \times 10^{14}$	In operation
GAB	pont	Gamma irradiation	--/	commission

### I-10.3. Available expertise

Field	Computer code	Equipment or facility	Capability including man power	Application or experience
Neutronic design	MCNP			
Thermohydraulic design	CFX/CFD			
Mechanical design	CAD			
Safety analysis				
Instrumentation and control				
Radiation dosimetry				
Radiation protection or residual radioactivity monitoring				
Decontamination				
Theoretical background for material irradiation				

#### I-10.4. Experience and available facility for silicon doping, gemstone colouration and membrane fabrication

ITEM	DESCRIPTION
Objective	Si doping
Irradiation material	Silicon
Position	Channel in moderator tank
Name of hole or facility	SDA
Thermal neutron flux (with or without target)	$1.7 \times 10^{13} \text{ cm}^{-2} \text{ s}^{-1}$
Fast neutron flux	$1.0 \times 10^{10} \text{ cm}^{-2} \text{ s}^{-1}$
Gamma ray flux	
Longitudinal length of hole or facility	Batch length 500 mm
Diameter or size	237 mm
Diameter of the accessible irradiated material	125, 150, 200 mm
Way of uniform irradiation	Ni flux screen, Rotation of ingot at $5 \text{ min}^{-1}$
Way of reduction for background radiation	
Irradiation time (max., min. and average)	20 min – 2 d; average 1-2 h
Way of rotation	Electrical drive
Way of cooling for irradiation object	Natural convection by $\text{H}_2\text{O}$
Way of monitoring the irradiation fluence	SPND + irradiation time
Way of decontamination	Ultrasonic bath + Decopur FS500
Way of residual radioactivity survey	Gamma ray counter
Exemption criteria for residual radioactivity	0.09 Bq/g, 0.5 Bq/cm <sup>2</sup>
Cadmium ratio for gold or other material	> 50
Capacity per year	≈ 15 t (depending on Si diameter)
Production during last 5 years	≈ 40 t
Present status	In operation

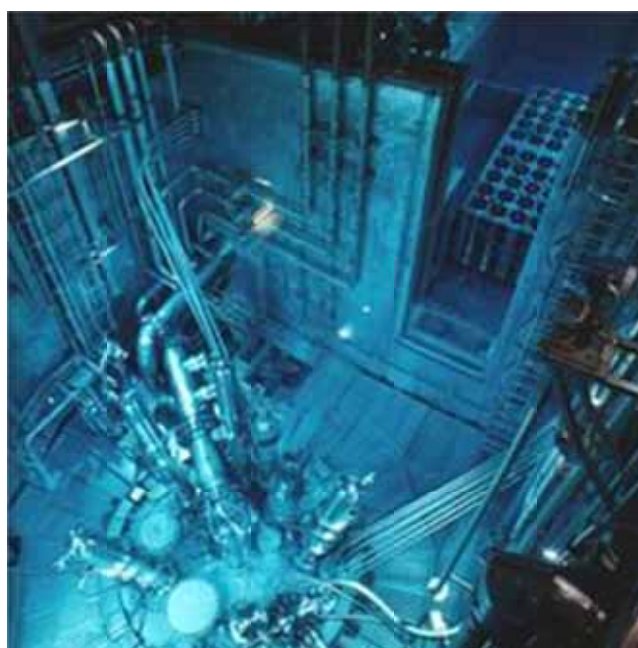


FIG. I-10.1. Core view of FRM II reactor.

## I-11. Indonesia: RSG-GAS (30 MW)

### I-11.1. General description of the reactor parameters

Reactor name	RSG-GAS (Multipurpose Reactor)	
Reactor type	Open pool	
First criticality	Jul. 29, 1987	
Power level	30 MW	
Maximum flux [ $\text{cm}^{-2}\text{s}^{-1}$ ]	(thermal) $2.43 \times 10^{14}$ (fast) $2.8 \times 10^{14}$	
Fuel description	Material/Density	$2.96 \text{ g/cm}^3$
	Enrichment	19.75%
	Type (element/bundle)	MTR
	Clad	AlMg
Absorber	Ag-In-Cd	
Coolant	$\text{H}_2\text{O}$	
Moderator	$\text{H}_2\text{O}$	
Reflector	Be	
Core cooling	$\text{H}_2\text{O}$	

### I-11.2. Irradiation hole or facility

Name	Position	Utilization field	Flux [ $\text{cm}^{-2}\text{s}^{-1}$ ] (thermal/fast)	Status
IP	B6	Topaz	$1.67 \times 10^{14}$	Operation
IP	D9	Isotope	$2.14 \times 10^{14}$	Operation
IP	G7	Isotope	$1.50 \times 10^{14}$	Operation
IP	E4	Topaz	$2.04 \times 10^{14}$	Operation
CIP	D6	Isotope	$2.23 \times 10^{14}$	Operation
CIP	D7	Isotope	$2.43 \times 10^{14}$	Operation
CIP	E6	Isotope	$2.39 \times 10^{14}$	Operation
CIP	E7	Isotope	$2.36 \times 10^{14}$	Operation

### I-11.3. Available expertise

Field	Computer code	Equipment or facility	Capability including man power	Application or experience
Neutronic design	WIMSD, SRAC 2006, Batan-3DIFF, Batan-EQUIL-2D, MCNP, MCNPX	PC	Excellent, 4	Core design, In-core fuel management, Irradiation service
Thermohydraulic design	COOLOD-N, PARET, EUREKA	PC	Excellent, 3	T/H core design
Mechanical design				

Safety analysis	RELAP	PC	Excellent, 3	T/H accident analysis
Instrumentation and control				
Radiation dosimetry	GammaVision-32	HPGe system	Excellent, 2	NTD, NAA, Radionuclide analysis
Radiation protection or residual radioactivity monitoring				
Decontamination				
Theoretical background for material Irradiation				

#### I-11.4 Experience and available facilities for silicon doping, gemstone colouration and membrane fabrication

ITEM	DESCRIPTION
Objective	Gemstone colouration
Irradiation material	Topaz
Position	B6, E4, D9 (In core) and TIF (Out core)
Name of hole or facility	IP (In core) and TIF (Out core)
Thermal neutron flux (with or without target)	$1.50 \times 10^{14}$ up to $2.43 \times 10^{14} \text{ cm}^{-2} \text{ s}^{-1}$
Fast neutron flux	
Gamma-ray flux	
Longitudinal length of hole or facility	60 cm
Diameter or size	7.3 cm
Diameter of the accessible irradiated material	7.0 cm
Way of uniform irradiation	rotation at TIF
Way of reduction for background radiation	N/A
Irradiation time (max., min. and average)	2 up to 10 hours (In core) and 8 up to 12 days (Out core)
Way of rotation	manually (by hand)
Way of cooling for irradiation object	Primary Water (In core) and Air cooling (Out core)
Way of monitoring the irradiation fluence	Survey meter
Way of decontamination	washing by ultrasonic
Way of residual radioactivity survey	Survey meter
Exemption criteria for residual radioactivity	N/A
Cadmium ratio for gold or other material	N/A
Capacity per year	1.8 t (In core) and ~400 kg (Out core)
Production during last 5 years	10 t (In core) and ~2 t (Out core)
Present status	Operation



FIG. I-11.1. Core view of RSG-GAS reactor.

## I-12. Republic of Korea: HANARO (30 MW)

### I-12.1. General description of the reactor parameters

Reactor name	HANARO	
Reactor type	Open tank in pool	
First criticality	Feb. 1995	
Power level	30 MW <sub>th</sub>	
Maximum flux [cm <sup>-2</sup> s <sup>-1</sup> ]	(thermal) 4.0×10 <sup>14</sup> (fast) 2.1×10 <sup>14</sup>	
Fuel description	Material/Density	U <sub>3</sub> Si/
	Enrichment	19.75 w/o
	Type (element/bundle)	element : rod bundle: hexagonal with 36 rods circular with 18 rods
	Clad	Aluminium
Absorber	Hafnium	
Coolant	H <sub>2</sub> O	
Moderator	D <sub>2</sub> O	
Reflector	D <sub>2</sub> O	
Core cooling	Forced up flow	

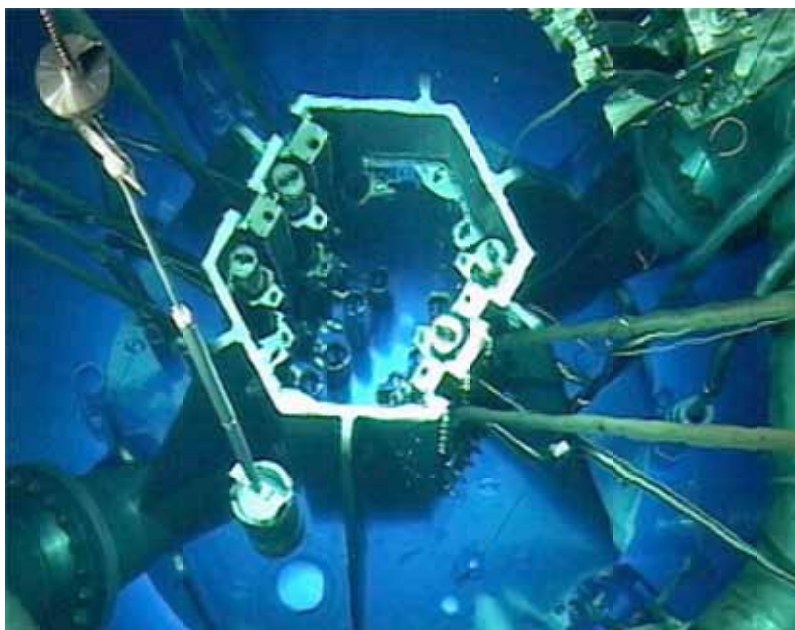


FIG. I-12.1. Core view of HANARO reactor.

### I-12.2. Irradiation hole or facility

Name	Position	Utilization field	Flux [ $\text{cm}^{-2}\text{s}^{-1}$ ] (thermal/fast)	Status
CT	Inner core	Material test, RI production	$4.4 \times 10^{14}$	In service
IR1	Inner core	FTL	$3.9 \times 10^{14}$	Commissioning
IR2	Inner core	Material test, RI production	$3.9 \times 10^{14}$	In service
OR	Outer core	Material test, RI production	$3.4 \times 10^{14}$	In service
CN	Reflector	Cold neutron source	$9 \times 10^{13}$	Developing
NTD-2	Reflector	Silicon doping	$5 \times 10^{13}$	In service
NTD-1	Reflector	Silicon doping	$5 \times 10^{14}$	Testing
HTS	Reflector	RI production	$1 \times 10^{11}$	In service
IP-4, 5, 15	Reflector	RI	$\sim \times 10^{13}$	In service
NAA-1, 2, 3	Reflector	NAA	$6 \times 10^9 \sim 1.5 \times 10^{14}$	In service

### I-12.3. Available expertise

Field	Computer code	Equipment or facility	Capability including man power	Application or experience
Neutronic design	HANAFMS, HELIOS, MCNP, etc.	Workstation, PC Cluster, PC.	Excellent, 4	Core design, In-core fuel management, Irradiation service

Thermohydraulic design	MATRA-h, CFX, etc.	Workstation, PC Cluster, PC.	Excellent, 4	T/H core design, CFD analysis
Mechanical design	ANSYS	PC	Excellent, 4	HANARO construction, New facility installation
Safety analysis	MARS(RELAP5)	PC	Excellent, 2	T/H accident analysis
Instrumentation and control	-	DCS, PLC	Excellent, 4	Reactor power automatic control
Radiation dosimetry	GammaVision-32	HPGe System	Excellent, 2	NTD, NAA, Radionuclide analysis
Radiation protection or residual radioactivity monitoring	-	RMS	Excellent, 5	Radiation monitoring

#### I-12.4. Experience and available facility for silicon doping, gemstone colouration and membrane fabrication

ITEM	DESCRIPTION
Objective	Neutron Transmutation Doping for Si ingots
Irradiation material	Single crystal ingot
Position	Vertical irradiation hole at D2O reflector
Name of hole or facility	NTD1 & NTD2
Thermal neutron flux (with or without target)	$\sim 5.2 \times 10^{13} \text{ cm}^{-2} \text{ s}^{-1}$
Fast neutron flux	
Gamma ray flux	
Longitudinal length of hole or facility	120 cm
Diameter or size	220 cm (NTD1), 180 cm (NTD1)
Diameter of the accessible irradiated material	6" & 8" (NTD1), 5 & 6" (NTD2)
Way of uniform irradiation	Rotating and Neutron screen
Way of reduction for background radiation	N/A
Irradiation time (max., min. and average)	0.2 ~ 9.5 hours
Way of rotation	Stepping motor
Way of cooling for irradiation object	Natural convection and semi-forced pumping by lower floater
Way of monitoring the irradiation fluence	SPND (for real time monitoring) Zr foils (for actual fluence)
Way of decontamination	Washing with water
Way of residual radioactivity survey	Smearing test
Exemption criteria for residual radioactivity	B/G/ level, Beta < 4 kBq/m <sup>2</sup>
Cadmium ratio for gold or other material	~ 20
Capacity per year	20 t using NTD2 only
Production during last 5 years	45 t (2003~2007)
Present status	In service at NTD2 Test irradiation at NTD1



## I-13. The Netherlands: HFR (45 MW)

### I-13.1. General description of the reactor parameters

Reactor name	High Flux Reactor (HFR)	
Reactor type	Tank in pool	
First criticality	1961	
Power level	45 MW	
Maximum flux [ $\text{cm}^{-2}\text{s}^{-1}$ ]	Depending on experiment details	
Fuel description	Material/Density	$\text{U}_3\text{Si}_2$
	Enrichment	About 19.75%
	Type (element/bundle)	Plate
	Clad	Aluminium-alloy
Absorber	Cd	
Coolant	Water	
Moderator	Water	
Reflector	Beryllium	
Core cooling	Force water cooling	

### I-13.2 Irradiation hole or facility

Information not available.

### I-13.3. Available expertise

Information not available.

### I-13.4. Experience and available facilities for silicon doping

ITEM	DESCRIPTION
Objective	Silicon doping
Irradiation material	Silicon
Position	PSF1-4
Name of hole or facility	SIFA
Thermal neutron flux (with or without target)	$2.5 \times 10^{13} \text{ cm}^{-2} \text{ s}^{-1}$
Fast neutron flux	
Gamma ray heating	Approximately 0.5 W/g
Longitudinal length of hole or facility	500 mm for NTD
Diameter or size	~ $\Phi 160$ mm
Diameter of the accessible irradiated material	Six inches
Way of uniform irradiation	Neutron absorption shield
Way of reduction for background radiation	
Irradiation time (max., min. and average)	A few hours
Way of rotation	Automatic rotation (electric)
Way of cooling for irradiation object	Water cooling
Way of monitoring the irradiation fluence	SPND
Way of decontamination	Rinse
Way of residual radioactivity survey	Radiation monitoring

Exemption criteria for residual radioactivity Cadmium ratio for gold or other material Capacity per year Production during last 5 years Present status	IAEA regulations    In use
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## I-14. Norway: JEEP II (2 MW)

### I-14.1. General description of the reactor parameters

Reactor name	JEEP II	
Reactor type	Tank	
First criticality	1966	
Power level	2 MW	
Maximum flux [ $\text{cm}^{-2}\text{s}^{-1}$ ]	(thermal) $3 \times 10^{13}$ (fast) $3.5 \times 10^{13}$	
Fuel description	Material/Density	$\text{UO}_2/10.3 \text{ g/cm}^3$
	Enrichment	3.50%
	Type (element/bundle)	Bundle
	Clad	Aluminium
Absorber	Cadmium	
Coolant	Heavy water	
Moderator	Heavy water	
Reflector	Heavy water	
Core cooling	Forced	

### I-14.2. Irradiation hole or facility

Name	Position	Utilization Field	Flux [ $\text{cm}^{-2}\text{s}^{-1}$ ] (thermal/fast)	Status
I1 - I5	Reflector	NTD	$9 \times 10^{12}/7 \times 10^{10}$	Operating
I6	Reflector	Isotope prod.	$1.3 \times 10^{13}/\text{NA}$	Operating
E3,E4	Reflector	NTD	$4 \times 10^{12}/10^{10}$	Operating
E2	Reflector	Isotope prod.	$8 \times 10^{12}/\text{NA}$	Operating

### I-14.3. Available expertise

Information not available.

#### I-14.4. Experience and available facility for silicon doping

ITEM	DESCRIPTION
Objective	Si-doping
Irradiation material	Silicon
Position	Reflector
Name of hole or facility	I1 – I5, E3, E4
Thermal neutron flux (with or without target)	$4 \times 10^{12} - 9 \times 10^{12} \text{ cm}^{-2}\text{s}^{-1}$ (Meas. Co-activat.)
Fast neutron flux	
Gamma ray flux	$10^{10} - 7 \times 10^{10} \text{ cm}^{-2}\text{s}^{-1}$ (Calc. $E > 0.82 \text{ MeV}$ )
Longitudinal length of hole or facility	90 cm
Diameter or size	12.7 cm
Diameter of the accessible irradiated material	12.7 cm
Way of uniform irradiation	Rotation
Way of reduction for background radiation	
Irradiation time (max., min. and average)	Max 80 h, Min 2 h, Avg. 14 h
Way of rotation	Electrical drive
Way of cooling for irradiation object	Heavy water, forced
Way of monitoring the irradiation fluence	Irradiation time
Way of decontamination	
Way of residual radioactivity survey	
Exemption criteria for residual radioactivity	
Cadmium ratio for gold or other material	
Capacity per year	
Production during last 5 years	20 t
Present status	Operating at full capacity

#### I-15. Poland: MARIA (30 MW)

##### I-15.1. General description of the reactor parameters

Reactor name	MARIA	
Reactor type	Pool	
First criticality	December 17, 1974	
Power level	30 MW	
Maximum flux [ $\text{cm}^{-2}\text{s}^{-1}$ ]	(thermal) $2.5 \times 10^{14}$ (fast) $5.0 \times 10^{13}$	
Fuel description	Material/Density	$\text{UO}_2 - \text{Al}/2.8 \text{ g/cm}^3$
	Enrichment	36%
	Type (element/bundle)	
	Clad	Al Alloy
Absorber	$\text{B}_4\text{C}$	
Coolant	Light water	
Moderator	$\text{Be}/\text{H}_2\text{O}$	
Reflector	Graphite, $\text{H}_2\text{O}$	
Core cooling	Forced light water flow (6-9 m/s)	

### I-15.2. Irradiation hole or facility

Name	Position	Utilization field	Flux [ $\text{cm}^{-2}\text{s}^{-1}$ ] (thermal/fast)	Status
Irradiation channels	graphite reflector	Radioisotopes production	$\Phi_{\text{th}} = 2.5 \times 10^{14}$	in operation
Irradiation channels with rabbit system	beryllium reflector	Radioisotopes production	$\Phi_{\text{th}} = 1.3 \times 10^{14}$	in operation
Facility for minerals irradiation	graphite reflector/special boron shielding	Topaz colouring	$\Phi_{\text{f}} = 2.2 \times 10^{12}$ ( $E > 0.5 \text{ MeV}$ )	in operation

### I-15.3. Experience and available facility for silicon doping

ITEM	DESCRIPTION
Objective	NTD of Silicon ( <i>Neutron Transmutation Doping of Single Crystals</i> )
Irradiation material	Single silicon crystals
Position	Channel located in graphite reflector zone
Name of hole or facility	NTD-1
Thermal neutron flux (with or without target)	$6.0 \times 10^{12} \text{ n/cm}^2 \text{ s}$
Fast neutron flux	$4.5 \times 10^{10} \text{ n/cm}^2 \text{ s}$
Gamma heat generation	$< 0.1 \text{ W/g}$
Longitudinal length of hole or facility	500 mm
Diameter or size	157 mm
Diameter of the accessible irradiated material	5" or 6"
Way of uniform irradiation	Neutron flux flattening: rotation plus flipping with flux profile linearization
Way of reduction for background radiation	Well moderated neutron spectrum in graphite reflector
Irradiation time (max., min. and average)	Dependent on target resistivity ( $7.5 \text{ } \Omega \cdot \text{cm}$ – $100 \text{ } \Omega \cdot \text{cm}$ )
Way of rotation	Electric drive
Way of cooling for irradiation object	Forced convection by pool cooling flow
Way of monitoring the irradiation fluence	SPND – detector system
Way of decontamination	Ultrasonic bath
Way of residual radioactivity survey	Gamma & beta ray counter
Exemption criteria for residual radioactivity	$^{32}\text{P}$ : $< 2.5 \text{ Bq/g}$
Cadmium ratio for gold or other material	Epithermal index $r = 0.02$
Capacity per year	6200 kg/y (for ingots diameter 5 in) 9500 kg/y (for ingots diameter 6 in)
Production during last 5 years	ca. 300 kg (tests)
Present status	Ready for operation; calibration tests for new customer

## I-16. South Africa: SAFARI-1 (20 MW)

### I-16.1. General description of the reactor parameters

Reactor name	SAFARI-1	
Reactor type	Tank in pool, Oak Ridge design type	
First criticality	1965	
Power level	20 MW	
Maximum flux [ $\text{cm}^{-2}\text{s}^{-1}$ ]	(thermal) $2.7 \times 10^{14}$ (fast) $1.1 \times 10^{14}$	
Fuel description	Material/Density	$\text{U}_3\text{Si}_2\text{-Al}$ , $4.88\text{U}_t/\text{cm}^3$
	Enrichment	19.75%
	Type (element/bundle)	19 fuel plates/element
	Clad	Aluminium
Absorber	Cd control rod section	
Coolant	Light water	
Moderator	Light water	
Reflector	Be	
Core cooling	Forced light water	

### I-16.2. Irradiation hole or facility

Name	Position	Utilization field	Flux [ $\text{cm}^{-2}\text{s}^{-1}$ ] (thermal/fast)	Status
Beam tubes (6)		diffraction radiography SANS	$\approx 2 \times 10^7$ thermal	Note 1 Operational
In core positions		Isotope production	Thermal: $2.7 \times 10^{14}$ Fast: $1.1 \times 10^{14}$	Operational
Hydraulic	Reflector border	Activation Isotope production	Thermal: $2 \times 10^{14}$ Fast: $1.1 \times 10^{14}$	Operational
Ringas	Reflector border	Activation analysis	Thermal: $1 \times 10^{14}$ Fast: $1.1 \times 10^{14}$	Operational
Note 1: Under reconstruction				

### I-16.3. Available expertise

Field	Computer code	Equipment or facility	Capability including man power	Application or experience
Neutronic design	MCNP			
Thermohydraulic design	RELAP, ANSYS			
Mechanical design	Solid Edge			
Safety analysis				
Instrumentation and control				
Radiation dosimetry				

Radiation protection or residual radioactivity monitoring				
Decontamination				
Theoretical background for material irradiation				

#### I-16.4. Experience and available facility for silicon doping

ITEM	DESCRIPTION
Objective	Si doping
Irradiation material	Silicon ingots
Position	Pool side
Name of hole or facility	SILIRAD
Thermal neutron flux (with or without target)	Without $1.5 \times 10^{14} \text{ cm}^{-2}\text{s}^{-1}$
Fast neutron flux	Without $2.6 \times 10^{13} \text{ cm}^{-2}\text{s}^{-1}$
Gamma ray flux	
Longitudinal length of hole or facility	Ingot length $\leq 600 \text{ mm}$
Diameter or size	
Diameter of the accessible irradiated material	Diameter $\leq 152 \text{ mm}$
Way of uniform irradiation	Translation and rotation
Way of reduction for background radiation	
Irradiation time (max., min. and average)	$\geq 1 \text{ hour}$
Way of rotation	Electrical drive
Way of cooling for irradiation object	Natural convection
Way of monitoring the irradiation fluence	SPND, time
Way of decontamination	Ultrasonic wash
Way of residual radioactivity survey	Beta counter
Exemption criteria for residual radioactivity	1000 Bq/g, 1MBq total activity
Cadmium ratio for gold or other material	$\approx 13$ for cobalt
Capacity per year	$\approx 20 \text{ t}$ depending on resistivity and diameter
Production during last 5 years	$\approx 90\text{t}$
Present status	Operational



FIG. I-16.1. Core view of SAFARI-1 reactor.

## I-17 Thailand: TRR (2MW)

### I-17.1. General description of the reactor parameters

Reactor name	Thai Research Reactor	
Reactor type	Swimming pool type TRIGA Mark III Reactor	
First criticality	Nov. 7, 1977	
Power level	2 MW (maximum)	
Maximum flux [ $\text{cm}^{-2}\text{s}^{-1}$ ]	(thermal) $3 \times 10^{13}$ (fast) $1 \times 10^{13}$	
Fuel description	Material/Density	UZrH
	Enrichment	20%
	Type (element/bundle)	element
	Clad	SS-304
Absorber	$\text{B}_4\text{C}$	
Coolant	$\text{H}_2\text{O}$	
Moderator	$\text{H}_2\text{O}$	
Reflector	Graphite	
Core cooling	Natural convection	



FIG. I-17.1. Core view of TRR reactor.

### I-17.2. Irradiation hole or facility

Name	Position	Utilization field	Flux [ $\text{cm}^{-2}\text{s}^{-1}$ ] (thermal/fast)	Status
In-core facility	CT, C8, C12, F3, F29, G5, G33, G22	Isotope production, NAA	$1-3 \times 10^{13}$ /	In use
Out-core facility	Rotary specimen rack (lazy susan), A1, A2, A4, CA2, CA3, TC Wet tube and void tank	NAA NAA Gemstone colouration	$4 \times 10^{11} / 1.2 \times 10^{11}$	In use
Neutron beam port	8" south 8" North 6" North	Neutron radiography Neutron diffractometry PGNAA	$7 \times 10^5$ /	In use

### I-17.3. Available expertise

Field	Computer code	Equipment or facility	Capability inclu. man power	Application or experience
Neutronic design	SRAC MVP	Personal computers, Computer cluster	Design and Calculation (4 persons)	Core design and analysis, Core management
Thermohydraulic design	COOLODN	Personal computers	Design and Calculation (2 persons)	Fuel Temperature distribution analysis
Mechanical design	AUTOCAD	Personal computers, Fabrication facility	Design and fabrication (4 persons)	A number of irradiation facility designs and fabrications
Safety analysis	EUREKA2/RR	Personal computers	Analysis and Calculation (2 persons)	Transient analysis of TRR-1/M1
Instrumentation and control	-	Electronics centre	Design and production (3 persons)	Design and production of survey meter, Design and production of MCA
Radiation dosimetry	-	TLD program for neutron and gamma dosimetry	Calibration and measurement 2 personnel	Providing TLD service within the country
Radiation protection or residual radioactivity monitoring	-	A wide range of radiation protection equipment available from Radiation Safety Section	Radiation Safety program (5 Radiation Safety officers)	Health physics operation for TRR-1/M1, Practice of Radiation Safety program
Decontamination	-	Waste management centre	Decontamination (5 persons)	Waste management and decontamination in support of TRR-1/M1
Theoretical background for material irradiation	-	XRF XRD NAA UV spectrophotometer	Analysis, R&D (6 persons)	R&D in gemstone irradiation, R&D in membrane fabrication, R&D in polymerization by radiation processing



#### I-17.4. Experience and available facility for silicon doping, gemstone colouration and membrane fabrication

ITEM	DESCRIPTION
Objective	Gemstone Colouration
Irradiation material	Topaz
Position	Wet tube, Void tank
Name of hole or facility	W1 - 5
Thermal neutron flux (with or without target)	$4 \times 10^{11} \text{ cm}^{-2} \text{ s}^{-1}$
Fast neutron flux	$1.2 \times 10^{11} \text{ cm}^{-2} \text{ s}^{-1}$
Gamma ray flux	
Longitudinal length of hole or facility	60 cm
Diameter or size	12.5 cm
Diameter of the accessible irradiated material	-
Way of uniform irradiation	rotation
Way of reduction for background radiation	Cd Filter
Irradiation time (max., min. and average)	400 h
Way of rotation	Manual
Way of cooling for irradiation object	-
Way of monitoring the irradiation fluence	flux and time control
Way of decontamination	ultrasonic cleaner
Way of residual radioactivity survey	NaI(Tl) system, HPGe system
Exemption criteria for residual radioactivity	2 nCi/g
Cadmium ratio for gold or other material	3.85
Capacity per year	100 kg
Production during last 5 years	~ 0.5 t
Present status	Fully utilized

#### I-18. Vietnam: DNRR (500 kW)

##### I-18.1. General description of the reactor parameters

Reactor name	Dalat Nuclear Research Reactor (DNRR)	
Reactor type	Pool type	
First criticality	Nov. 01, 1983	
Power level	500 kW	
Maximum flux [ $\text{cm}^{-2} \text{ s}^{-1}$ ]	(thermal) $2.19 \times 10^{13}$ (fast) $9.66 \times 10^{12}$	
Fuel description	Material/Density	U-235 /1.4 g/cm <sup>3</sup> (36.0% enrichment) U-235 /2.5 g/cm <sup>3</sup> (19.7% enrichment)
	Enrichment	36% and 19.7%
	Type (element/bundle)	Fuel element of tube type
	Clad	Al alloy

Absorber	B <sub>4</sub> C
Coolant	H <sub>2</sub> O
Moderator	H <sub>2</sub> O
Reflector	Be, graphite
Core cooling	Natural convection



*FIG. I-18.1. Core view of Dalat Research Reactor.*

#### **I-18.2. Irradiation hole or facility**

Name	Position	Utilization field	Flux [cm <sup>-2</sup> s <sup>-1</sup> ] (thermal/fast)	Status
Neutron trap	Centre	RI	$2.1 \times 10^{13} / 2.7 \times 10^{12}$	In use
Rotary specimen rack	Graphite reflector	RI, Gem	$3.5 \times 10^{12} / 1.0 \times 10^{11}$	In use
Channel 1.4	Cell 1.4	RI, Gem	$9.6 \times 10^{12} / 7.1 \times 10^{12}$	In use
Channel 7.1	Cell 7.1	NAA	$6.2 \times 10^{12} / 8.6 \times 10^{12}$	In use
Channel 13.2	Cell 13.2	NAA	$6.6 \times 10^{12} / 7.9 \times 10^{12}$	Upgrading
Thermal column	Next to reflector	NAA	$9.2 \times 10^{10} / 5.3 \times 10^7$	In use
Beam port No.1	Radially oriented tube		$1.30 \times 10^{12} / 1.35 \times 10^{10}$	Not available
Beam port No.2	Radially oriented tube		$5.85 \times 10^{12} / 2.44 \times 10^9$	Not available
Beam port No.3	Tangential tube	(n,2g) reaction study	$9.60 \times 10^{11} / 9.35 \times 10^9$	In use
Beam port No.4	Radially oriented tube	PGNAA, neutron filtered beams	$3.32 \times 10^{12} / 8.75 \times 10^{10}$	In use

**I-18.3. Available expertise**

Field	Computer code	Equipment or facility	Capability including man power	Application or experience
Neutronic design	WIMSD, MCNP4C2, SRAC2006	Workstation	5	For Dalat research reactor calculation
Thermohydraulic design	COOLODN, RELAP5, EURECKA	Workstation	4	
Mechanical design				
Safety analysis	RELAP5, SRAC2006	Workstation	5	
Instrumentation and control		ASUR-14R	1	
Radiation dosimetry		Gamma, neutron dosimeters	5	Dose control
Radiation protection or residual radioactivity monitoring		Radiation monitoring system	2	Radiation monitoring around reactor building
Decontamination				
Theoretical background for material irradiation				

#### I-18.4. Experience and available facility for silicon doping, gemstone colouration and membrane fabrication

ITEM	DESCRIPTION
Objective	Gem irradiation
Irradiation material	Topaz
Position	Cell 1-4, graphite reflector
Name of hole or facility	Channel 1-4, Rotary specimen rack
Thermal neutron flux (with or without target)	Without target: $9.6 \times 10^{12} \text{ cm}^{-2} \text{ s}^{-1}$ (Channel 1-4), $3.5 \times 10^{12} \text{ cm}^{-2} \text{ s}^{-1}$ (Rotary specimen rack)
Fast neutron flux	$7.1 \times 10^{12} \text{ cm}^{-2} \text{ s}^{-1}$ (Channel 1-4), $1.0 \times 10^{11} \text{ cm}^{-2} \text{ s}^{-1}$ (Rotary specimen rack)
Gamma ray flux	
Longitudinal length of hole or facility	200 mm
Diameter or size	28 mm
Diameter of the accessible irradiated material	
Way of uniform irradiation	
Way of reduction for background radiation	
Irradiation time (max., min. and average)	100 h, 30 h, 60 h
Way of rotation	
Way of cooling for irradiation object	
Way of monitoring the irradiation fluence	
Way of decontamination	H <sub>2</sub> O
Way of residual radioactivity survey	
Exemption criteria for residual radioactivity	
Cadmium ratio for gold or other material	4.53 (Au) for Rotary specimen rack
Capacity per year	30 – 50 kg expecting
Production during last 5 years	
Present status	Studying



## **GLOSSARY AND ABBREVIATIONS**

ARV	Axial Resistivity Variation
CCD	Charged Coupled Device
CZ	Czochralski Process
FZ	Floating Zone Method
HPGe	High Powered Germanium Detector
ICI	International Conference on Isotopes
IGORR	International Group on Research Reactors
IRRACC	Irradiation Accuracy
JAEA	Japan Atomic Energy Agency
JAERI	Japan Atomic Energy Research Institute
KAERI	Korea Atomic Energy Research Institute
NTD	Neutron Transmutation Doping
RCA	Regional Cooperative Agreement
RR	Research Reactor
RRFM	Research Reactor Fuel Management Conference
RRG	Radial Resistivity Gradient
SCK/CEN	Center for Nuclear Energy Research
SIDONIE	Silicon Doping by Neutron Irradiation Experiment
SPND	Self-Powered Neutron Detector
UKAEA	United Kingdom Atomic Energy Authority



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