APPLICATION NOTE

NEUTRON TRANSMUTATION DOPED (NTD) SILICON FOR HIGH POWER ELECTRONICS

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Abstract.
In this application note the neutron transmutation doping (NTD) technique will be discussed with the focus on the factors limiting the homogeneity of silicon crystals doped by irradiation and properties of the NTD silicon that are important for the end users. In the lower resistivity range irradiation precision is the dominating uncertainty factor, while at higher resistivities the requirements for the starting material become restrictive, if we want to avoid the material properties becoming the dominating uncertainty factor. We will show that the current upper limit for NTD doping of 150 mm FZ crystals is 4000 Ωcm with a record low value of the radial resistivity variation (RRV) ≈ 8%. The upper limit for 200 mm is 800 Ωcm, RRV less than 7%. Also, very good resistivity stability of the NTD silicon under device like process steps will be demonstrated.

Introduction
Use of semiconductors for power electronics requires unique material characteristics because of the high power levels flowing in the devices. Non-perfect semiconductor crystals with non-uniform properties will render the power devices unstable with a potential dangerous impact on many everyday activities. Silicon is by far the most perfect semiconductor and therefore silicon is the preferred material for power semiconductor devices. Since the early 1970’s the use of large monocrystalline silicon material has grown rapidly, and tremendous efforts have been made to develop techniques for growing perfect Si crystals with ever increasing dimensions. These crystals are the basic material for a wide range of electronic and optoelectronic devices.

For the power devices to work at designated power levels and voltage readings it is necessary to dope the silicon with impurities that makes current flow through the bulk of the silicon possible. Doping silicon crystals with phosphorus by neutron irradiation is the
most superior technique in terms of doping control and it effectively removes non-uniformities in the high resistivity silicon crystal.

When neutrons are used for changing the resistivity of silicon, phosphorous dopant atoms are produced by transmutation of $^{30}\text{Si}$ into $^{31}\text{P}$ by thermal neutron irradiation in atomic reactors. Topsil pioneered the production of neutron transmutation doped (NTD) silicon and has been involved in developing this technique since the very beginning in the early 1970’s when the production of crystals sufficiently clean to benefit from the outstanding doping characteristics obtained by NTD became possible.

The NTD technique is used when the application requires doping levels of high precision and uniformity. Typical dopant levels for NTD silicon and the corresponding resistivities are indicated in Table 1. The major market for silicon of this extremely clean and uniform quality is power semiconductor devices. For the performance of thyristors, rectifiers, IGBT’s and Power MOSFET’s uniformity is essential for obtaining high, well-defined breakdown voltages typically above 300 V.

For high resistivity silicon doping (i.e. in the range of Table 1) NTD is by far the most precise and uniform way of introducing phosphorus. For purposes of comparison, the resistivity and the resistivity distribution, which reflect the carrier concentration, are often the key parameters. We will comment on the connection between the dopant concentration and the resistivity later. However, the resistivity is roughly proportional to the reciprocal dopant concentration.

<table>
<thead>
<tr>
<th>Resistivity (Ωcm)</th>
<th>Dopant conc. $(10^{13}$ atoms/cm$^3$)</th>
<th>Ppba Phosphorus</th>
<th>Neutron dose $(10^{16}$ cm$^{-2}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>30</td>
<td>14.5</td>
<td>2.9</td>
<td>86</td>
</tr>
<tr>
<td>100</td>
<td>4.3</td>
<td>0.85</td>
<td>24</td>
</tr>
<tr>
<td>200</td>
<td>2.1</td>
<td>0.42</td>
<td>10</td>
</tr>
<tr>
<td>300</td>
<td>1.4</td>
<td>0.28</td>
<td>7</td>
</tr>
<tr>
<td>500</td>
<td>0.85</td>
<td>0.17</td>
<td>4</td>
</tr>
<tr>
<td>1000</td>
<td>0.45</td>
<td>0.086</td>
<td>2</td>
</tr>
</tbody>
</table>

Table 1: Phosphorus doping concentrations and corresponding neutron doses required to introduce the phosphorous in the typical resistivity range of NTD silicon.

**The neutron transmutation technique**

In conventional chemical doping methods - for example the incorporation of impurities from the melt during crystal growth - thermodynamics plays an important role. Since nearly all impurities of interest have distribution coefficients much less than unity, they tend to remain in the melt. This means that the distribution of impurities in the grown crystals is non-uniform. However, tight control of the melt and the feed rate of the doping impurities are used to decrease this non-uniformity in PFZ products (gas phase doped Float Zone). For NTD the role played by thermodynamics is negligible. Homogeneity is determined by the uniformity of the
neutron flux in the reactor over the volume of the silicon crystal and by the purity of the grown crystals.

The irradiation-induced phosphorus atoms are created by means of neutron capture by a $^{30}\text{Si}$ atom, forming the unstable $^{31}\text{Si}$ isotope. $^{31}\text{Si}$ subsequently transmutes to $^{31}\text{P}$ by $\beta$-emission, with a half-life of 2.6 hours (see Table 2). In addition to this reaction a number of secondary reactions take place, the most important of which are shown in Table 2.

Simple calculations show that these reactions need no concern, as their production of new isotopes is negligible at reasonable doping levels. The crystals are exposed to the required neutron dose by placing them close to the core of a nuclear reactor resulting in very homogenous irradiation fluxes. However, the neutron flux is not perfectly homogeneous, and the background impurity levels in the grown crystals are not negligible. Both give rise to non-uniformities in the final doping profile. The irradiation also partly damages the crystal structure, mainly by introducing point like defects. These defects must be removed before use of the NTD silicon for device making. This is done by annealing at elevated temperatures in clean furnaces. The resistivities of irradiated crystals referred to later in this paper are all measured after an annealing step. The defect annealing aspect of the NTD will be discussed in later parts of this note.

**Irradiation inhomogeneities**

In the present section we disregard the effects of dopant impurities in the as-grown undoped crystals, but for high resistivity NTD crystals (i.e. low number of added dopant impurities) the background doping impurities in the as-grown crystals must be taken into account. The approximation is thus good in the low resistivity crystals. By neglecting the resistivity state of the undoped crystal, the doping profile homogeneity is determined by the irradiation alone. We often distinguish between two types of homogeneity: The doping uniformity along the crystal axis within wafer batch variation, which plays no or little role for the individual wafers cut from the crystal and the radial variation within wafer variation, which is a crucial feature of each wafer cut from crystal.

The radial resistivity variation (RRV), defined as

$$\text{RRV} = \frac{R_{\text{MAX}} - R_{\text{MIN}}}{R_{\text{MIN}}} \times 100\%$$  \hspace{1cm} (1)

<table>
<thead>
<tr>
<th>Reaction</th>
<th>Isotope Change</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^{30}\text{Si}(n,\gamma)\rightarrow^{31}\text{Si}$</td>
<td>$\beta^-$ (2.6 h)</td>
</tr>
<tr>
<td>$^{28}\text{Si}(n,\gamma)\rightarrow^{29}\text{Si}$</td>
<td></td>
</tr>
<tr>
<td>$^{28}\text{Si}(n,\gamma)\rightarrow^{30}\text{Si}$</td>
<td></td>
</tr>
<tr>
<td>$^{31}\text{P}(n,\gamma)\rightarrow^{32}\text{S} + \beta^-$ (14.3 h)</td>
<td></td>
</tr>
</tbody>
</table>

Table 2. Nuclear reactions

In this paper, the radial resistivity variation within wafer batch variation is denoted by the radial variation within wafer variation, which is a crucial feature of each wafer cut from crystal.
where $R_{\text{MAX}}$ and $R_{\text{MIN}}$ are the maximum and minimum resistivities measured on individual wafers, is the most important factor to control in silicon industry, because power semiconductor manufacturers cannot allow for high variations across the wafers cut from the cylindrical crystals.

The undoped silicon crystals are irradiated in vertical or horizontal channels beside the reactor core. The crystals have diameters of up to 200 mm and typical lengths of up to 60 cm during irradiation. In case of an inhomogeneous neutron flux the resistivity profile of the doped silicon crystal will be influenced and it is therefore of importance to control the flux profile during the irradiation cycle. The solution to an inhomogeneous neutron flux problem can be either flattening of the flux at the expense of intensity or moving the crystal during irradiation.

The radial resistivity profile is determined by the attenuation of the neutron flux during penetration of the silicon crystal. The decay length in silicon is about 19 cm, so for stationary crystals the flux inhomogeneity is rather high, as seen in Table 3. This will in turn give rise to high radial resistivity variations.

<table>
<thead>
<tr>
<th>Crystal diameter</th>
<th>Stationary crystal</th>
<th>Rotating crystal</th>
</tr>
</thead>
<tbody>
<tr>
<td>4''</td>
<td>41</td>
<td>1.7</td>
</tr>
<tr>
<td>5''</td>
<td>48</td>
<td>2.5</td>
</tr>
<tr>
<td>150 mm</td>
<td>54</td>
<td>3.9</td>
</tr>
</tbody>
</table>

Table 3: Radial flux variations (%)

To minimise this effect, the crystals must rotate during irradiation. If we consider the cylindrical crystals as surrounded by material with the same decay length as silicon itself we can calculate the non-uniformity of the dose by integrating the time average of the flux at a distance from the crystal axis over a rotation period. To first order the result is:

$$\frac{\Phi(a)}{\Phi(0)} = 1 + \frac{a^2}{4b^2}$$

where $\Phi$ is the average flux at a distance from the crystal axis, $a$ is the radius of the crystal and $b$ the decay length.

Results are shown in Table 3. It can be seen that rotation gives a considerable improvement compared to stationary irradiations. In practice the situation is slightly different, since the material surrounding the crystals normally has decay lengths longer than 19 cm.
Resistivity variations

To convert the inhomogeneities in the flux to resistivity variations the relations between resistivity and neutron dose must be considered. First, however, we must consider the relationship between resistivity and doping levels, taking into account the fact that both acceptor and donor impurities are present in the bulk of the as-grown silicon crystal. The polysilicon is normally phosphorus doped, but due to the significant difference in boron and phosphorous segregation coefficients any boron contamination or content in the polysilicon during growth will impact heavily on the resistivity of the finished monocrystal.

The resistivity of the bulk monocrystal is given by:

$$\rho = \frac{1}{ne\mu_e + p\mu_p}$$

(3)

where e is the charge unit, n and p the electrons and hole concentrations and \(\mu_e\) and \(\mu_p\) the electron and hole mobilities, respectively. In the pure types of silicon crystal we are discussing here, \(\mu_e\) and \(\mu_p\) can be considered as constants, so we only need to estimate n and p. Assuming that the only electrically active impurities are B as the acceptor and P as the donor, a very good estimate is given by

$$n = \frac{1}{2} \frac{\sqrt{(N_p - N_B)^2 + 4n_i^2} + 1/2(N_p - N_B)}$$

(4)

and

$$pn = n_i^2$$

(5)

where \(N_p\) and \(N_B\) are the phosphorus and boron concentrations, and \(n_i\) is the intrinsic carrier concentration, commonly taken to be \(1.0 \times 10^{10} \text{ cm}^{-3}\) at 300K.

We can now make assumptions about the boron impurity level, and calculate the silicon resistivity from equations (3), (4) and (5). Some results are shown in Fig. 1, where the monocrystal resistivity is plotted against phosphorous concentration for boron concentrations in the range 0.2 - 20 ppta. Normal polysilicon for Float Zone growth have a phosphorus concentration of \(\sim 10^{12} \text{ cm}^{-3}\). From Fig. 1 it can be seen that as long as the boron concentration is kept below 10 ppta \((\sim 10^{11} \text{ cm}^{-3})\) there is no practical influence of the boron dopants on the bulk resistivity. This is important because this gives possibilities for stable NTD production up to 500 \(\Omega\text{cm}\) without taking into account the boron doping contribution.
For boron concentrations larger than 10 ppta the bulk resistivity shows distinct peaks at phosphorous concentrations between $10^{11}$ and $10^{12}$ cm$^{-3}$ which is close to the phosphorous content for normal polysilicon. At the peak the silicon type changes from p-type to n-type.

The effect of residual boron impurities is more pronounced for new higher resistivity types of phosphorous doped polysilicon that have phosphorous levels down to $10^{11}$ cm$^{-3}$. In order for these new types of polysilicon to give reliable and predictable outcome in terms of the bulk resistivity of the monocrystal, boron concentrations must be kept below 1 ppta. This is a difficult task for the manufacturers of polysilicon to handle, but they are capable of doing so and ultrapure polysilicon with residual boron impurities in the sub ppta region is on the market.

The almost invariable behaviour of the monocrystal resistivity (given low bulk content of boron) prompts us to characterise the starting material for the NTD in terms of its resistivity alone. If we do so, and assume that the phosphorus donor levels introduced by the irradiation are fully ionised and uniformly distributed, we get a simple indication of the homogeneity of the doped crystals.

![Graph showing resistivity versus phosphorous concentration](image)

Figure 1. Estimated resistivities in near intrinsic silicon at 300 K versus phosphorous concentration for various background levels of boron. $\mu_i$ and $\mu_f$ are arbitrarily fixed at 1500 cm$^2$/V$s$ and 450 cm$^2$/V$s$.

The amount of phosphorous introduced by the transmutation is given by:

$$N_{P,NTD} = k\Phi$$  \hspace{2cm} (6)

where

$$k = d c(30\text{Si})\sigma$$  \hspace{2cm} (7)
Φ is the neutron dose, d is the atomic density of silicon, c (30Si) is the relative presence of the 30Si isotope and s is the cross section for the transmutation reaction. From (3) and (6) the following relation between the neutron dose and target resistivity is obtained:

\[
\frac{1}{\rho_{\text{target}}} = \frac{k\Phi}{\mu_e e} + \frac{1}{\rho_{\text{initial}}}
\]  

(8)

where \(\rho_{\text{target}}\) and \(\rho_{\text{initial}}\) are the resistivity of the doped and as-grown crystal, respectively. \(\mu_e\) is the electron mobility and \(e\) is the electronic charge. Fig. 2 shows this almost universal relation between the neutron dose and the silicon resistivity for various resistivities of the starting material.

Consider an ideal irradiation represented by a horizontal line in Fig. 2; clearly, the resistivity of the starting material, and thus the homogeneity of the as grown crystals, is important for the final result. As in Fig.2, the curves in this figure approach each other at higher doses, i.e. at lower resistivities, illustrating the increasing influence at higher resistivities of the as-grown crystal on the final result. For example, we can see that with undoped crystal resistivities between 3000 and 6000 \(\Omega\text{cm}\), and doping with \(4\times10^{16}\) neutrons/cm\(^2\) we obtain monocystalline silicon resistivities of about 500 \(\Omega\text{cm}\) \(\pm\)4%, whereas doses of \(1\times10^{17}\) neutrons/cm\(^2\) result in 200 \(\Omega\text{cm}\) with a total resistivity variation of only \(\pm\)3%.

![Figure 2. Neutron dose versus the target resistivity obtained for crystals of various starting resistivities.](image-url)
NTD products are all about restricting the bulk resistivity variations beyond what is obtainable with any other means of producing n-type doped silicon. However, the bulk radial resistivity variation (RRV) of the starting undoped silicon material imposes some limitations to the minimum obtainable NTD bulk RRV. In Fig. 3a are shown limitations for the bulk RRV for a virgin undoped monocrystal with an average bulk resistivity of 5000 Ωcm as a function of the target bulk NTD resistivity and for different start bulk radial resistivity variations (in percent). As can be seen from Fig. 5a it is no problem to obtain low RRV NTD products as long as the target resistivity is below 100 Ωcm, but for larger target resistivities it is becoming increasingly more difficult to obtain low resistivity variations if RRV exceeds 20% for the undoped silicon. However, if the average resistivity of the undoped silicon is sufficiently high, RRV can be kept low even for cases where RRV exceeds 50% for the undoped silicon. This is shown in Fig. 3b for the case of an average bulk resistivity of the undoped silicon of 15000 Ωcm.

**Topsil NTD product overview**

Topsil has the broadest range of NTD silicon products among the manufacturers of Float Zone silicon. In Table 4 the full product range for NTD material is shown. Product range covers diameters
from 4” up to 200 mm sizes and bulk resistivities from 5 Ωcm to 4000 Ωcm. In addition, Topsil has different processes of NTD that can support many different applications and requirements. In particular, the NTD silicon from Topsil is stable towards high temperature processing. Topsil is continuously pushing the limits of NTD and has programmes on NTD product improvements and developments. In addition to the bulk properties stated in Table 4 which is numbers for the large cylindrical crystals, Topsil can wafer the crystals into products that can be introduced directly into processing environments. The last column in Table 4 lists the possible surface preparations of the wafers that Topsil can deliver.

<table>
<thead>
<tr>
<th>Tolerance ≤ 4”</th>
<th>5-100 Ωcm</th>
<th>100-200 Ωcm</th>
<th>200-600 Ωcm</th>
<th>600-1000 Ωcm</th>
<th>1000-1999 Ωcm</th>
</tr>
</thead>
<tbody>
<tr>
<td>%</td>
<td>±5</td>
<td>±5</td>
<td>±6</td>
<td>±7</td>
<td>±10</td>
</tr>
<tr>
<td>RRV ≤ 4”</td>
<td>%</td>
<td>&lt;3</td>
<td>&lt;4</td>
<td>&lt;5</td>
<td>&lt;7</td>
</tr>
<tr>
<td>%</td>
<td>&lt;3</td>
<td>&lt;4</td>
<td>&lt;5</td>
<td>&lt;7</td>
<td>&lt;10</td>
</tr>
<tr>
<td>Tolerance 5”/150 mm</td>
<td>%</td>
<td>±6</td>
<td>±6</td>
<td>±7</td>
<td>±8</td>
</tr>
<tr>
<td>RRV 5”/150 mm</td>
<td>%</td>
<td>&lt;4</td>
<td>&lt;5</td>
<td>&lt;6</td>
<td>&lt;8</td>
</tr>
<tr>
<td>%</td>
<td>&lt;4</td>
<td>&lt;5</td>
<td>&lt;6</td>
<td>&lt;8</td>
<td>&lt;12</td>
</tr>
<tr>
<td>Tolerance 200 mm</td>
<td>%</td>
<td>±7</td>
<td>±7</td>
<td>±7</td>
<td>±7</td>
</tr>
<tr>
<td>RRV 200 mm</td>
<td>%</td>
<td>&lt;7</td>
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<td>%</td>
<td>&gt;300</td>
<td>&gt;300</td>
<td>&gt;300</td>
<td>&gt;300</td>
</tr>
<tr>
<td>Resistivity striations</td>
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<td>Not detectable</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Wafering capabilities</td>
<td></td>
<td>As-cut (wire/ID saw), Lapped, Etched, Bright Etched, Grinded, High Reflectivity, IC Grade Polished, MEMS Grade Double Side Polished</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 4: Neutron Transmutation Doped (NTD) products

Please note that 200 mm is shipped unannealed.

**Lifetime**

An important parameter to control for manufacturers of NTD silicon is the minority carrier bulk lifetime (t or bulk lifetime for short). Topsil measures the bulk lifetime after having annealed the irradiated crystals. The annealing effectively removes all of the defects in the silicon generated by the neutrons. Both fast and slow neutrons generate defects. Fast neutrons are responsible for the general disorder in the crystalline structure after irradiation. They have an almost linear transport path in the silicon. Slow neutrons generate large localised disorder and thereby a lot of localised vacancies and interstitials are generated. The disorder from both fast and slow neutrons are readily removed by a simple annealing sequence that restores completely the crystalline structure of the monocrystalline silicon. However, if contaminants like oxygen, carbon and nitrogen are present in ppm or sub-ppm concentrations they can pair with each other and vacancies generated by the slow neutrons to form complexes. The vacancies themselves can form different kind of complexes – i.e.
monovacancies, divacancies and donor-vacancy pairs. Complexes in the silicon monocrystals can act as lifetime killing defects and can donate free carriers to the lattice. Both of these effects can be present after the annealing sequence that restores the monocrystalline silicon structure, but they can also induce instabilities during subsequent device processing.

The annihilation of defects and disorder in the silicon monocrystal can be observed by monitoring the increase in bulk lifetime in the duration of the annealing cycle. At the point where all defects and disorder are removed, the bulk lifetime significantly increases to a stable, predictable value. Topsil measures the bulk lifetime according to the SEMI standard by the photoconductive decay (PCD) method on the irradiated and annealed crystals. Also, a spatially resolved PCD method that allows for local determination of the bulk lifetime is used to control the outcome of the annealing of the irradiated crystals or wafers. Spatial resolution for this method is Ø2 mm with a penetration depth into the bulk of up to 2 mm. By this method it is easy for the operator of the lifetime equipment to determine the right quality of the crystal. In Fig. 4 is shown an example of a measurement with the spatially resolved PCD method.

At high minority carrier densities the surface recombination dominates the measured effective lifetime resulting in a steep decrease for increasing minority carrier densities above \(10^{15}\) cm\(^{-3}\). For low minority carrier densities (< \(10^{14}\) cm\(^{-3}\)) the state of the bulk silicon is the only contributing effect to the bulk lifetime, and there is a straightforward linear dependence (in a log-log plot) of the bulk lifetime on the excess minority carrier density. In cases where the bulk is contaminated by metals or alkali ions or there is a high number of defects/disorder the linear dependence is violated and the silicon crystal is rejected.

Figure 4. Spatially resolved PCD screen dump.
In figure 5 is shown an example of bulk lifetime measurements for silicon crystals along the radial axis on the end faces of the crystals. In this example the distance between individual PCD measurements is set to 10 mm. The radial variation is very small, in the order of 5% only indicating a homogeneous, high quality silicon crystalline material.

**Resistivity Control**

NTD silicon is superior to any other techniques in terms of bulk resistivity control for doping with n-type carriers. Furthermore, NTD silicon is used in device applications where non-equilibrium conditions apply during operation. Therefore, bulk resistivity and bulk lifetime stability towards device processing is mandatory. The bulk resistivity cannot be allowed to shift during device processing and the bulk lifetime should remain stable above a certain limit. The device process that has the biggest potential of changing the properties of the silicon is an oxidation step above 1050°C.

Above this temperature, dislocation loops may form in improperly conditioned silicon (i.e. silicon containing many complexes) and the bulk resistivity may shift as much as 20% if a proper float zone process is not selected from the beginning. Bulk resistivity shifts are associated with impurities such as oxygen and nitrogen that can be activated to form donors in the bulk and defects and complexes that can be electrically activated. For this reason there is a maximum specification of oxygen and carbon on float zone material. If oxygen is present in the float zone growth cycle it is
readily incorporated in the monocrystalline solid state silicon and

this would have negative impact on bulk resistivity stability of NTD material. Likewise, nitrogen can be electrically activated in the silicon bulk, but unlike oxygen in silicon nitrogen is segregated away from the monocrystal solid state silicon. However, if the amount of nitrogen exceeds a certain limit (for instance by a vacuum leak into the growth chamber) it can impact the bulk resistivity upon device processing.

In order to control the NTD products the atmosphere is closely monitored during Float Zone growth and the oxygen, carbon and nitrogen concentrations in the finished product are subjected to concentration analysis. By these extensive control procedures and by monitoring the state of the bulk Topsil is able to guarantee full resistivity control during device processing.

In summary, Topsil is in control of all of the important parameters for high quality NTD products. NTD products are characterised by their low resistivity variations within the individual wafers, from wafer to wafer in the same batch and from crystal to crystal. The control is in effect by accurately controlling the irradiation dose and by control of the undoped silicon growth process. Additional important control parameters include measuring the minority carrier bulk lifetime after recrystallisation to ensure that the state of the bulk is recovered and that the irradiation induced defects are annihilated.

Topsil Semiconductor Materials A/S

Topsil is a world leading supplier of ultrapure silicon to the global semiconductor industry. Engaging in long term relations with customers, Topsil focuses on premium quality, an efficient production process and a safe delivery of products.

Silicon is used in electronic components to aid conversion and control of electrical power. Topsil provides ultrapure silicon mainly for the most demanding purposes, based on extensive knowledge and significant investments in new technology, facilities and equipment.

Headquartered in Copenhagen Cleantech Park, Topsil spans production sites in Denmark and Poland and sales locations in Europe, Asia and the US. Topsil is publicly listed at the Nasdaq OMX Copenhagen stock exchange and was founded in 1959.