

# NEUTRON TRANSMUTATION DOPING OF SILICON: HIGHLY HOMOGENEOUS RESISTIVITY SEMICONDUCTOR MATERIAL PRODUCED IN NUCLEAR REACTORS.

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## ABSTRACT

The neutron transmutation doping (NTD) of semiconductor materials is a method in which the quantity of dopant can be precisely controlled and homogeneously distributed throughout the material. The NTD method is based on nuclear reactions where some nuclei of the semiconductor material capture thermal neutrons and form unstable radioactive nuclei which beta decay to nuclei of a different element, i.e. the dopant. The irradiation of silicon produces phosphorous, by means of the neutron capture in  $^{30}\text{Si}$  nuclei producing  $^{31}\text{Si}$  nuclei which decay to the stable isotope  $^{31}\text{P}$ . Since the irradiation of a silicon crystal can be performed in a homogeneous way in nuclear reactors, the resistivity variation is very small throughout the volume of the crystal. The advent of NTD Si made possible the development of high power semiconductor devices as it allows tight resistivity tolerances and excellent homogeneity of the silicon base material. An irradiation rig with simple design has been constructed and installed in the IEA-R1 research reactor for irradiation of silicon crystal ingots. Test irradiations were performed with float zone silicon crystals and the results of radial and axial uniformities in the final resistivity values as well as the doping accuracy, obtained in the test irradiations show an excellent doping quality achieved.

## I INTRODUCTION

The semiconductor industry has change dramatically many aspects of human life in the last decades. Telecommunication, electronics, personal microcomputers, industrial automation, among others, are some examples of the great progress provided by electronic devices.

The material responsible by this revolution is silicon. Pure silicon is the most common material used for the fabrication of electrical and electronic devices in the semiconductor industry. Large volume high voltage thyristors, power diodes and various types of transistors for medium to high power used in power plants, power transmission lines, automotive control and power supplies to integrated circuits for computers and microprocessors are made from silicon wafers. However, in order to be used in these devices, silicon needs to be doped with another element to achieve the desired resistivity value which usually differs for different applications.

The doping procedure is, therefore, a fundamental step in the manufacture of semiconductor materials. Properties like band gap energy, resistivity, etc., critically depends on the nature and concentration of the dopant

element. Doping techniques like diffusion or ion implantation produce highly inhomogeneous dopant concentration and lattice defects, respectively. The quality of a semiconducting material also critically depends on the nature and concentration of the electrically active defects present in the material. The dopant atoms must reside on defect free regular lattice sites to become electrically active. The doping procedure always includes the risk of either creating lattice defects, by ion implantation for example, or the incorporation of unwanted impurity atoms into the material by diffusion.

The elements used to dope Si are phosphorus (for n-type Si) and boron (for p-type Si). The conventional phosphorus doping is carried out by the incorporation of the dopant in the required concentration in the molten stage during crystal growth. The conventional doping however leads to inhomogeneous distribution of the dopant due to segregation caused by low distribution coefficient of phosphorus in silicon and the resultant material presents large resistivity variations. As a consequence, this material is unsuitable for the production of high volume devices such as rectifiers for high-voltage direct current (HVDC) transmission since the microscopic resistivity fluctuations (striations) may cause local break through ("hot spot")

which not only have negative impact on device characteristics but they are usually detrimental to the device itself with eventual possibility of device breakdown.

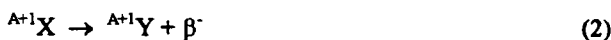
The neutron transmutation doping (NTD) technique is a very convenient and highly reproducible method for introducing a homogeneous distribution of well controlled dopants into certain semiconductors. The main advantage of the NTD method lies on the transformation of the starting semiconductor material itself in the dopant by means of a nuclear reaction. In this way the presence of impurities is avoided, the dopant element is created on the same lattice site of the starting semiconductor material and the generation of defects is smaller than in other methods.

## II. NTD METHOD

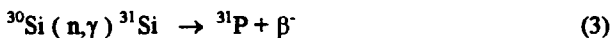
The NTD method is based on the neutron capture reaction where thermal neutrons are captured by nuclei X which increase the mass number A of one unit resulting in new nuclei which are in an excited state. These excited nuclei decay quickly to the ground state by emitting a photon:



These  ${}^{A+1}\text{X}$  nuclei are, however, not stable and decay into new nuclei Y of a different element from the original by, in most cases,  $\beta^-$  particle emission:



In the case of Silicon, neutron transmutation doping (NTD) process is based on the capture of thermal neutrons by the  ${}^{30}\text{Si}$  nuclei (roughly 3.1% of silicon consists of  ${}^{30}\text{Si}$  isotope) producing the radioactive nuclei  ${}^{31}\text{Si}$  which decay with a half-life of 2.62 h to stable isotope of  ${}^{31}\text{P}$ , as is shown below:



The number of dopant atoms  $n_{\text{NTD}}$  produced by neutron transmutation of a certain nuclei present in the semiconductor material is given by:

$$n_{\text{NTD}} = N_0 \sigma_c \phi t \quad (4)$$

where  $N_0$  is the number of atoms per  $\text{cm}^3$  in the starting material,  $\sigma_c$  is the neutron capture cross section for the given isotope and  $\phi t$  is the neutron dose.

For example, in the case of silicon, with specific gravity of  $2.33 \text{ g/cm}^3$ , atomic weight of 28.086 and  ${}^{30}\text{Si}$  isotopic abundance of 3.09%,  $N_0 = 1.544 \times 10^{21} \text{ atoms/cm}^3$ . For  ${}^{30}\text{Si}$ ,  $\sigma_c = 0.11 \text{ b}$ , therefore, the number of P atoms produced by neutron transmutation is given by  $n_{\text{NTD}} = 1.698 \times 10^{-4} \phi t \text{ atoms/cm}^3$ . The P atoms act as donor resulting in the N-type silicon. The neutron flux in

research reactors used for NTD is typically in the range  $0.5$  to  $5 \times 10^{13} \text{ neutrons/cm}^2 \cdot \text{s}$ .

The main advantage of using neutron transmutation doping results from the possibility to produce silicon with extremely homogeneous resistivity distribution since the irradiation of the silicon crystal can be performed in an homogeneous manner in nuclear reactors. The spreading resistance profile measurements have shown[1] that on a microscopic scale the total resistivity fluctuations of a conventionally doped float-zone silicon crystal could be as high as 30%. With neutron transmutation doping the resistivity variation can be reduced to less than 2-3%. Moreover since the neutron dose received by the silicon crystal during the irradiation can be determined with high accuracy, the resistivity targets can be achieved with very tight tolerance.

The starting material is an undoped silicon crystal grown by the floating zone method. The resistivity of the starting material typically ranges from 1,000 to 10,000  $\Omega\text{cm}$ . The main impurities are oxygen and carbon with typical concentrations in the range of  $10^{15} \text{ atoms/cm}^3$ . Metallic concentrations are below  $10^{10} \text{ atoms/cm}^3$ . Electrically active impurities are boron and phosphorous in the range of  $10^{12} \text{ atoms/cm}^3$ . Due to the excess of phosphorous in polycrystalline silicon, the undoped FZ silicon crystals have n-type conductivity. After irradiation, final resistivities as specified by the customer range from 20 to 500  $\Omega\text{cm}$ . As other possible nuclear reactions contribute to a negligible extent, the transmutation nuclear reaction produces n-type semiconductor silicon.

The idea of Phosphorus doping of Silicon by means of thermal neutron transmutation was originally suggested by Lark-Horovitz[2]. Tanenbaum and Mills[3] made the first experiment of producing Silicon with perfectly uniform phosphorus distribution by NTD method which was first applied by Schönller[4] for high-power thyristor manufacturing, followed by extensive research which demonstrated a clear superiority of the resulting device characteristics [5,6].

The impact of this technique was fundamental for the development of high power semiconductor devices because it allows tight resistivity tolerance and excellent homogeneity of the silicon base material. These properties are indispensable for the performance of the power devices and cannot be provided by conventional doping methods.

**NTD Market and Outlook.** The main use of NTD silicon is for electronic devices which need exact resistivity and homogeneity, i.e. thyristors, rectifiers, diodes and transistors for medium and high power. These discrete devices are used in power plants, power transmission lines, automotive control, power supplies, etc. Since the beginning of commercial silicon irradiation, the annual amount has been continuously increasing, and the forecasts of the next years predict growth rates of about 10% per year. Today, NTD silicon is commercially produced in a number of research reactors around the world and the total capacity of production is about 110[7]. The limitation of the future growth of silicon irradiation regards to the

irradiation capacity available in the research reactors throughout the world. The problem is the lifetime of the present reactors which is, in many cases, reaching the limit and there are severe restrictions on the replacement for new ones.

A competing method is the growth of epitaxial thick layers, which can be used for small power discrete devices. The disadvantages of this technology are the high cost, the difficulty to produce layers with high resistivity and the limitation on the thickness of the layer (about 100  $\mu\text{m}$ ). However, despite the improvements, the epitaxial growth is not expected to reach the same performance of the NTD.

In order to achieve the desired resistivity homogeneity, the irradiation of Si crystal ingots is performed in such way that the neutron flux must be as homogeneous as possible throughout the whole crystal volume. Radial flux gradients can be compensated by simply rotating the crystal during the irradiation, resulting in radial resistivity variations less than 1%. The homogenization of the neutron flux in the axial direction is more complicated and depends on the axial neutron flux profile for each reactor. In most cases it is common to use shaped neutron shields to flatten the axial flux profile, or moving periodically the entire crystal in the axial direction.

With the increasing demand for NTD silicon with very tight target resistivity tolerances and considering the fact that only a limited number of research reactors are able to irradiate silicon presently, it is important to improve the silicon irradiation process by introducing new procedures and precise neutron dose control methods.

### III. THE NTD Si EXPERIMENTS AT IEA-R1 RESEARCH REACTOR

This work describes a very efficient and accurate method of irradiating silicon crystals without the use of neutron absorbing shields or moving the crystals in the axial direction. Radial and axial uniformities, doping accuracy and the simplicity of operation are the main objectives which were considered in the design of a test silicon irradiation facility at IEA-R1 reactor.

The IEA-R1 reactor is an open swimming pool-type light water reactor operated at 2 MW on an 8 hours a day, 5 days per week, cycle. The reactor core consists of 31 MTR-type fuel elements including 4 elements with neutron absorbing control plates. About half of the fuel elements are 93%  $^{235}\text{U}$  while others use 20% enriched uranium. The reactor core is surrounded by graphite blocks serving as a reflector. A cross-sectional view of the reactor is shown in Fig. 1. The silicon irradiation position is on the left side top.

**Silicon Irradiation Rig.** The rig essentially consists of two parts, the one fixed on the reactor grid plate is a square cross-sectional aluminum guide tube with dimensions of 12 cm x 12 cm x 150 cm. The other part of the rig consists of a freely rotating cylindrical aluminum tube which is 175

cm long, has 109 mm internal diameter and 2.5 mm thick wall. The cylindrical tube is supported inside the guide tube through an aluminum pin which holds it at the center during rotation. The rotating tube is connected to a long aluminum rod which extends above, beyond the pool water, and is coupled to the shaft of an electric motor through a ball bearing. The rotating cylinder serves as the irradiation tube for silicon crystals. The rotation speed is 2 rpm, and to ensure that the tube does in fact rotate during the irradiation period the rotational motion is constantly monitored through an optical sensor. Figure 2 is the side view of the silicon irradiation rig which is located in the graphite reflector.

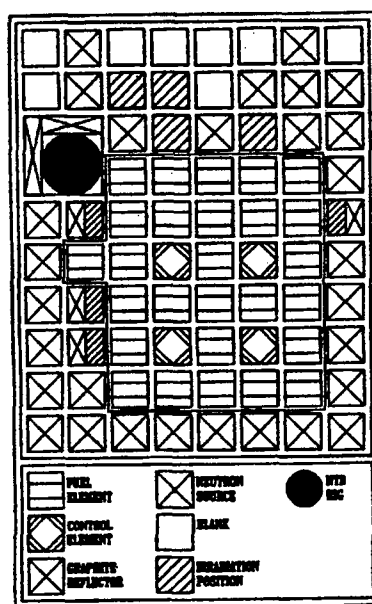


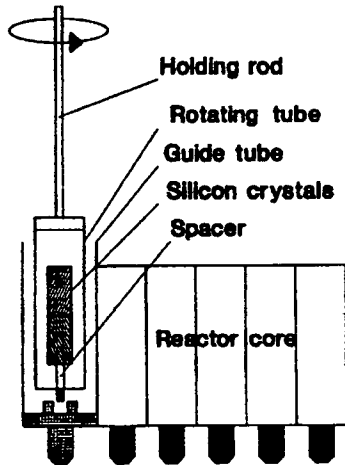
Figure 1. A Cross-sectional View of the IEA-R1 research reactor core.

**Neutron Flux Profile Measurement.** To obtain the desired resistivity uniformity, the neutron flux must be as homogeneous as possible over the entire crystal volume. While the small radial flux gradient of the reactor can be greatly reduced by rotating the crystal around its axis during the irradiation, which usually results in the radial resistivity variation of the order of 1% or less, the detailed knowledge of the neutron flux variation in the axial direction is essential. The axial flux profile depends on the reactor type and dictates the method to be used for optimizing the maximum to minimum resistivity variation over the ingot length and consequently the maximum ingot length which can be irradiated.

The thermal neutron flux profile has been measured by the activation method. The selected reaction was  $^{59}\text{Co}(n,\gamma)^{60}\text{Co}$  due to the fact that it is almost insensitive to epithermal neutrons and provides an adequate activity for

quick measurement by an ionization chamber or HPGe spectrometer.

Cobalt wire monitors wrapped in thin aluminum sheets were positioned at regular distances on the surface of aluminum rods having the same dimensions as the silicon ingots. Aluminum was chosen because of its similarity to silicon with respect to the neutron scattering and absorption cross-sections. Irradiation of the aluminum rods was carried out by following the identical procedure as adopted for the silicon crystals and the data were obtained for cobalt monitors covered with and without cadmium sheets. The neutron dose was calculated using



the Westcott

Figure 2. Side View of the Silicon Irradiation Rig at IEA-R1 Research Reactor.

formalism[8]. Most of the correction factors involved canceled out and the only remaining important factor was the cobalt wire self-shielding to neutrons which was determined in a separate experiment[9] for the used wire. The measured flux profile is shown in figure 3. The cadmium ratio was determined to be  $29.3 \pm 0.6$ .

**Irradiation Method.** The measurement of the axial neutron flux profile at the silicon irradiation position (Fig. 3) shows that the flux peaking occurs at about the geometrical center of the irradiation tube and that the neutron flux drops rather steeply but quite symmetrically above and below this central position reaching about 50% of the peak value at approximately 25 cm on either side.

In order to utilize the maximum possible irradiation capacity of the rig as well as the consideration of the cost for NTD irradiation it was decided to irradiate a maximum of 50 cm long silicon crystals in the installed facility. The approach to axial uniformity involving flux profile modification using neutron absorbers was immediately rejected as this method would reduce about 50% of the available flux capacity.

Excellent characteristics of the flux profile at the irradiation position in our reactor has provided us a better solution to achieve the desired axial uniformity without

sacrificing the irradiation capacity. In this method two silicon crystals 20 cm long each, are irradiated together in an aluminum can, with their interface adjusted at the maximum flux position inside the irradiation tube. The irradiation tube is rotated about its axis at 2 rpm during irradiation. At precisely 50% of the total necessary dose, the crystals are pulled out of the core and their positions are interchanged as shown in figure 3. The flux profile being symmetrical, the crystals now see almost mirror images of the respective profiles at previous positions. The irradiation then continues till the remaining dose is complete. Precise detection of the 50% dose is the key to

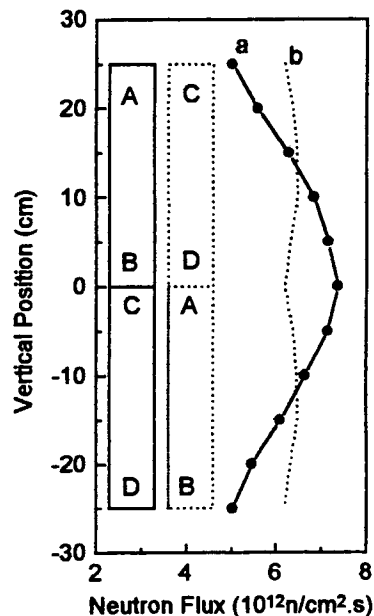


Figure 3. Experimentally Measured Thermal Neutron Flux Profile in the Vertical Direction (a), The Final Flux Profile as Would be Seen by the Crystal Pair When Their Positions are Interchanged at 50% of the Desired Dose (b)

the success of this method. This is accomplished with the help of self powered neutron detectors placed close to the irradiation tube as well as the cobalt wire monitors which are irradiated together with the silicon crystals.

The resistivity,  $\rho$  of n-type silicon containing  $N$  atoms per  $\text{cm}^3$  of dopant concentration of phosphorous can be written as:

$$\rho = (N\mu_e q_e)^{-1} \Omega \cdot \text{cm} \quad (5)$$

where  $\mu_e$  is the drift mobility of electrons in the crystal lattice ( $\text{cm}^2/\text{V}\cdot\text{s}$ ) and  $q_e$  is electronic charge ( $1.602 \times 10^{-19}$  Coulomb). The final concentration of  $^{31}\text{P}$  atoms  $N_f$  in the neutron irradiated silicon is given by the sum of dopant phosphorous concentration  $N_i$  in the starting material and

the number of atoms  $n_{\text{NTD}}$  of  $^{31}\text{Si}$ , produced by neutron capture in  $^{30}\text{Si}$ , which subsequently decay to  $^{31}\text{P}$ , given by:

$$N_f = N_i + n_{\text{NTD}} \quad (6)$$

Combining equations (4), (5) and (6) the relation between the neutron dose  $\phi \cdot t$  ( $\text{n/cm}^2$ ) received by the silicon crystals and the final resistivity  $\rho_f$  ( $\Omega \cdot \text{cm}$ ) attained after irradiation can now be written as [10]:

$$\phi \cdot t = K [1/\rho_f - 1/\rho_i] \quad (7)$$

where  $\rho_i$  is the initial resistivity of the crystal and,  $K = (N\sigma_c \mu_e q_e) \cdot l$ .

The constant  $K$  ( $\Omega/\text{cm}$ ) depends mainly on the neutron capture cross section of  $^{30}\text{Si}$  which in turn is determined by the neutron energy spectrum at the irradiation position and is therefore characteristic of each reactor. Precise experimental determination of the value of  $K$  is essential for the calculation of the required neutron dose to achieve a given target resistivity.

All the silicon crystals are encapsulated in aluminum cans which are 50 cm tall and have 0.5 mm wall thickness. The internal diameter of the can varies according to the diameter of the silicon crystal to be irradiated (3" or 4"). The aluminum cans are provided with holes at the bottom and at the top to permit the flow of water around the crystal during irradiation.

The neutron dose is measured by two silver wire self-powered neutron detectors (SPND) placed very close to the irradiation tube. The neutron detectors utilize the  $\beta^-$  decay of the radioactive silver isotopes  $^{108,110}\text{Ag}$  to produce a current which is proportional to the neutron flux. The current is measured by digital current integrator and counted in a preset scaler. When the integrated counts reach the present value, an alarm sounds prompting the reactor operator to take the necessary action to pull the irradiation tube out of the reactor core. The self-powered neutron detectors are calibrated periodically against cobalt wire monitors irradiated simultaneously. The activity of cobalt monitors is measured by an ionization chamber.

A series of test irradiations were performed with float-zone silicon crystals having diameters of 3 and 4 inches and a length of 20 cm each. The silicon crystals were furnished by a Japanese manufacturer. A total of 125 crystals were irradiated for the test experiments. The initial resistivity of the crystals varied from 1000 to 20000  $\Omega \cdot \text{cm}$  and the target resistivities varied between 33 to 220  $\Omega \cdot \text{cm}$  and specified in each case by the manufacturer. The irradiation of silicon crystals was carried out following the procedure outlined in the preceding section. After irradiation, the crystals were checked for the residual radioactivity and finally shipped back to the manufacturer where they were submitted to an appropriate thermal annealing process before measuring the final resistivity. The data on the final resistivity of each crystal were furnished by the manufacturer. The resistivity

measurements were carried out on both flat faces of each ingot using a four point probe.

#### IV. RESULTS AND DISCUSSION

Results show an excellent doping quality. More than 80% of the crystals showed axial variation of the resistivity, defined as the ratio of average resistivity of the upper and lower crystal face, equal or less than 3%. The maximum observed variation in all cases is 5%. More than 95% of the crystals attained radial doping uniformity, which is the difference between maximum and minimum resistivity relative to the average value in any given face of the crystal, equal or less than 2%. In more than 80% of the cases the doping accuracy, defined as the ratio between the final average resistivity and the target value, was better than 3%. These results can be considered entirely satisfactory when compared with the commercially produced NTD silicon in other reactors.

Experimental data obtained in the test irradiations also served to determine a precise value of the calibration constant  $K$  appearing in the equation (7). A least square fit of the data to equation (7) gave, in the case of 4 inch crystals,

$$\phi \cdot t = (2.751 \pm 0.012) \times 10^{19} [1/\rho_f - 1/\rho_i] \quad (\text{n/cm}^2) \quad (8)$$

A simple design irradiation rig has been constructed and installed in the IEA-R1 research reactor for the neutron transmutation doping of 3 and 4 inches diameter silicon crystals. A reasonably symmetric thermal neutron flux profile at the irradiation position permitted the irradiation of two silicon ingots, each 20 cm long simultaneously. By interchanging the position of ingots at precisely half the required neutron dose, it has been possible to achieve an excellent axial doping uniformity. Test irradiation of 100 silicon crystals demonstrated excellent doping uniformities as well as doping precision.

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# ESTUDO DA ESTABILIZAÇÃO DO TETRAFLUOROETILENO E DA TEMPERATURA DE IRRADIAÇÃO NA SEGURANÇA DO PROCESSO DE POLIMERIZAÇÃO DO MONÔMERO INDUZIDA POR RADIAÇÃO GAMA

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## RESUMO

Neste trabalho, estudou-se a influência da temperatura inicial de irradiação na segurança do processo de polimerização do tetrafluoroetileno (TFE) líquido e a eficiência dos inibidores terpenos: dipenteno,  $\alpha$ -pineno e terpinoleno na estabilização do TFE, usando a radiação gama, para induzir as reações de polimerização e de desproporcionamento do monômero. Estudou-se a influência da temperatura irradiando 40 gramas de TFE líquido, introduzido previamente em um vaso de reação (reator), dotado de sistema de proteção contra explosões, nos intervalos de temperatura entre  $-78^{\circ}\text{C}$  e  $-64^{\circ}\text{C}$  e entre  $20^{\circ}\text{C}$  e  $24^{\circ}\text{C}$ . A eficiência dos terpenos: dipenteno, terpinoleno e  $\alpha$ -pineno na polimerização do TFE, foi testada, irradiando-se o TFE sem e com terpenos, introduzido, previamente, em um reator similar ao utilizado nos experimentos anteriormente descritos. Os resultados mostraram que as reações de polimerização foram facilmente controladas, quando iniciadas no intervalo de temperatura entre  $20^{\circ}\text{C}$  e  $24^{\circ}\text{C}$  que entre  $-78^{\circ}\text{C}$  e  $-64^{\circ}\text{C}$ . Nos ensaios com TFE contendo terpenos a polimerização foi efetivamente inibida pela adição de até 0,1% de dipenteno,  $\alpha$ -pineno e terpinoleno ou da mistura 1:1:1 destes terpenos. Os resultados mostraram que o uso da radiação ionizante é um método seguro, adequado e facilmente controlável para estudar a estabilização do TFE e a sua polimerização no intervalo de temperatura entre  $20^{\circ}\text{C}$  e  $24^{\circ}\text{C}$ .

## I INTRODUÇÃO

O tetrafluoroetileno (TFE), um monômero altamente instável, é a matéria-prima para a produção de polímeros como o politetrafluoroetileno (PTFE) e os perfluoropoliéteres (PFPEs) e copolímeros que constituem a maior parte dos produtos comerciais fluorados da atualidade. A razão para a diversidade e quantidade destes produtos no mercado reside nas suas excepcionais combinações físico-químicas, conferindo-lhes excelentes resistência química e à degradação mecânica, estabilidade térmica alta e propriedades dielétrica, antifricção e antiaderente boas<sup>[1,2]</sup>. Embora estes produtos despertem grande interesse comercial, os seus preços são elevados em decorrência da presença do flúor e da complexidade dos processos de fabricação. Estes processos envolvem altos riscos de explosões e requerem equipamentos projetados adequadamente para suportarem altas pressões, elevando

ainda mais o custo final e, conseqüentemente, limitando-os a utilizações específicas bem como o desenvolvimento de novas aplicações. As explosões são causadas, fundamentalmente, pela polimerização exotérmica e instabilidade do monômero TFE. O monômero TFE polimeriza-se espontaneamente liberando grande quantidade de calor ( $\Delta H = -41\text{kcal/mol}$ )<sup>[2]</sup>, formando blocos poliméricos que obstruem dutos e válvulas. A condutividade térmica baixa do PTFE ( $k = 15,5\text{mW/m.K}$ ,  $30^{\circ}\text{C}$ )<sup>[2]</sup> dificulta a dissipação do calor gerado na polimerização favorecendo a reação de desproporcionamento exotérmica e explosiva do monômero que libera, aproximadamente, a mesma quantidade de energia ( $2567\text{J/g}$ ) liberada na explosão da pólvora negra ( $2842\text{J/g}$ )<sup>[1]</sup>. Muitos compostos químicos podem inibir estas reações, como o ácido metacrílico e seus derivados