Industrial Use of a Sub-Compact Cyclotron

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A Sub-compact cyclotron(k=18) has been used for industrial use in the past decade at neutron radiography, CPAA, TLA. Most of machine time is now occupied with proton and ^{3}He bombardment for switching time modification of power device by introducing localized defect layer. Also, CPAA serves for light element analysis of oxygen and carbon that act for gettering center by forming atom-vacancies complex in silicone LSI. Radio-luminography , a new type of autoradiography, is under investigation for contamination and migration analysis of transition elements by radio-tracer technique on semiconductor wafer.

1. Introduction

A fixed energy sub-compact cyclotron , which was initially developed for PET radio-isotopes as ^{11}C, ^{13}N, ^{15}O and ^{18}F, giving 18 MeV protons, 9 MeV deuterons and 24 MeV ^{3}He with 50 μA, has been used for industrial use(1). This article reviews specially the applications for defects creation and the control on silicone device and the substrate and coloration of Jewry, specially for diamond.

2. Survey of Industrial Applications at Toyo

(1) Thermal Neutron Source and Neutron Radiography[1]

Cyclotron-based neutron can be obtained through ^{9}Be(p, n) reaction with average energy of about 2 MeV.
A properly designed moderator gives thermal neutron fluxes of 1.7 x 10^6 n/cm^2/sec at an L/D = 44 collimator end. The neutron source intensity increases at square of energy and linear to beam current. This neutron beam is periodically used for neutron radiography of both film and real-time imaging for aviation component for defense. Two phase flow visualization in car air-conditioning is under examination for new frons which is chlorine free substitute in order to conserve ozone layer in the stratosphere [2].

(2) Thin Layer Activation(TLA) for Machine Wear/RTM [3]

TLA has been used for wear measurement of mechanical components and was introduced to Toyo from FZK(KfK), Germany in 1993 under technical license agreement . Non-invasive and quantitative wear can be measured by this technique at 1 μg level which saves normal test time and expenditure as 90% and 80%, respectively[4]. The main application field is expected for screening of combustion engine components and lubricants , which strongly requested for the improvement for the ecological problem.

(3) Charged Particle Activation Analysis/CPAA[5]

CPAA is an advantage against NAA in light element analysis. LSI semiconductor chips and opto-devices are continuously pursed for control of light element in ppb level. Table 1 shows light element analysis conducted at Toyo for different kinds of matrix, i.e. most of them are related to semiconductor industry.

Table 1: Some Results of requested analysis

Matrix	Element determined	Analysis process	Content found
Si	O	Wet	2 ppb ~ 12 ppm
	N	Dry	2 ppb ~ 5.9 ppm
	C	Dry	2 ppb ~ 306 ppb
GaAs	O	Wet	2 ppb ~ 50 ppm
	C	Dry	2 ppb ~ 137 ppm
	B	Dry	1.2 ppm ~ 2.4 ppm
GaP	O	Wet	<3 ppm
	C	Dry	<100 ppb
	B	Dry	1~10 ppm
InP	O	Wet	1~10 ppm
	C	Dry	5~25 ppb
Al	O	Wet	0.1~50 ppm
	N	ND*	0.1~3 ppm
	C	ND	<9 ppm

* Nondestructive analysis

In order to avoid matrix activation, chemical separation of volatilization under RF fusion or chemical precipitation of KBF_4, etc. are employed for boron, carbon and oxygen, respectively. Two ppb determination limit for carbon and one ppb for oxygen are obtained. Application of CPAA is now in progress for surface contamination analysis of boron on silicon wafer[6], as traditional high sensitive equipment like SIMS fails the surface analysis due to scattering of surface boron through ion bombardment fusing process and not free from abundant light elements in the surroundings.

(4) Ion Implantation for Power Devices[7][8][9][10]

Such power devices as thyristor, GTO, IGBT (Table 2)are treated by electron radiation to produce lattice

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defects that act as trapping center of deep level of recombination center for the excess carriers created at turn-off with a trade-off relation of forward voltage rise which results power loss.

Table 2 : Kinds of Power Devices

	Thyristor (Light Trigger Thyristor)	GTO Thyristor*	IGBT** & MCT***
Status in market	Customary	Present	Next
Break-down Volt(kV)	12 (8)	8	1.7
Current(kA)	1 (2)	1	0.4
Gate Drive	Current Turn-on	Current Turn-on & off	Voltage Turn-on & off

* Gate Turn-on
** Insulated Gate Bipolar Transistor
*** MOS Controlled Thyristor

High energy ion bombardment produces defect layer near Bragg peak and the trade-off relation can be improved by setting at proper depth looking device

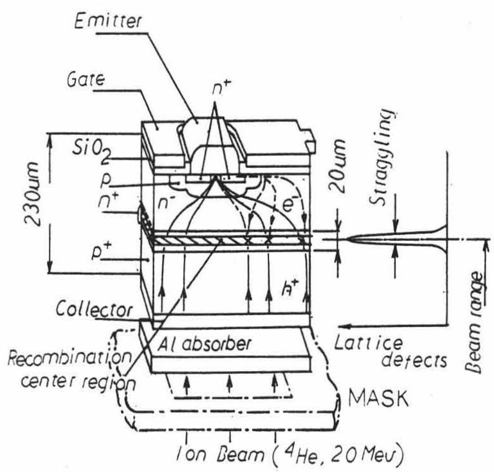

Figure 1: Structure of Insulated Gate Bipolar transistor and typical position of defect layer[11].

Typical results are compared for various particles as shown in Table 3. Masking of beam is reported for further improvement of this structure (Fig. 1) as shown in Table 3.[11].

A set of beam scanning device and wafer handling device ,which can carry one hundred sheets of six inch dia. wafers in the vacuum box, has been installed and daily used for the bombardment service.

Table 3: Comparison of Irradiation Treatment on IGBT of 1 kV reverse voltage

Kind of particle	Forward voltage(V)	Turn-off time(μ sec)
No treatment	2.0	10
Electron	3.5	3
Proton	3.5	1.8
Helium	3.5	1

(5) Positron Source [12][13]

Conventional strong slow positron source is produced from pair creation of γ-ray annihilation, where high energy electron beam has been used for the γ-ray field generation. Recently, the use of positron emitting short-lived radioisotopes, e. g. through $^{27}Al(p, n)^{27}Si$ reaction are proposed by on-line activation of cyclotron beams. Preliminary experiments are now conducted at Toyo and RIKEN(IPCR) cyclotron ,aiming 10^7-10^9 e^+/sec of intensity that is promising and comparable order to electron linac source. The typical results are shown in Table 4. The application fields are expected for ;(1) low energy positron diffraction, (2)positron life time measurement for lattice defect and vacancy analysis, (3)positron remitting microscope, (4)positron annihilation induced ionization of polymer and organic materials for mass spectroscopy , etc.

Analysis of defects through positron beam is now carried out through ^{22}Na source[16][17]. The slow positron beam, accelerated at 50 kV can penetrate the surface of material, i.e. silicone , few μ mm depth. At Toyo, slow positron pulsing beam apparatus is now under construction aiming 200 picosec pulse width to measure decay of positron annihilation through timing technique for the depth profile.

Table 4 : Intensity of slow positron beam obtained with on-line excitation

Target	Aluminum		Carbon	Si_3N_4
Particle	proton		deuteron	
Energy(MeV)	18	14	8	
Arrangement	Transmission	Reflection	Reflection	
Extraction current (e+/μA)	$1x10^5$	$1.8x10^2$	$3.2x10^3$	$2.6x10^3$
Moderation Efficiency	$2x10^{-5}$	$<10^{-7}$	---	---

3. Approach for lattice defects and stabilization

(1)Gettering process

CPAA has been used for carbon and oxygen determination in silicone at ppb to ppm level. These light elements do not affect to electrical conductivity, but act as gettering center for collecting defects and vacancy of lattice by forming atom vacancy clusters. These defects are most related to the leak current of MOS memory of mega-bit or high voltage power device.

The silicone is eventually requested to much lower defects and impurities with reduction rate of 1/5 to 1/10 every three years from the trends of LSI chips development. Typical histogram of carbon and oxygen content in 279 silicone substrates analyzed at Toyo in 1994 shows the low content peak less than few ppb level(Fig. 2). Similar situation is for transition elements of iron, Ni, Cu, Ti, Zn, and Au have been intensely purified less than $10^9/cm^2$ content in both CZ and FZ-Si crystal. However, the contaminants, introduced during such fabrication process like ion implantation and dry etching, etc. are becoming of much more importance. It is now estimated that trace of elements of the light and transition elements even at $10^{10}/cm^2$ acts for stabilization of lattice defects as gettering center.

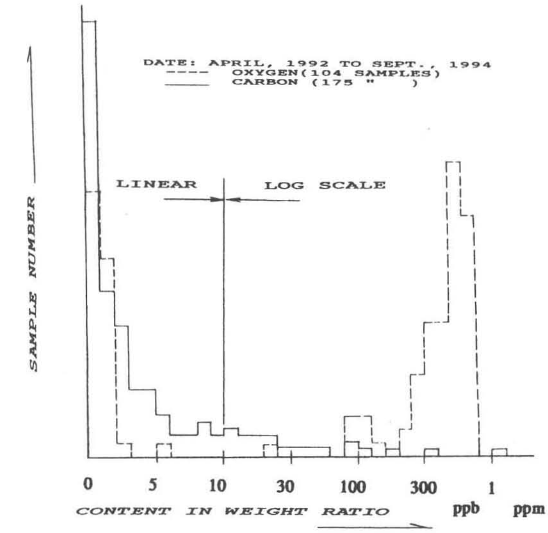

Fig. 2: Histogram of Carbon and Oxygen Analysis in Silicone through CPAA at Toyo

1-1. Gettering process at semiconductor fabrication

For purification , intrinsic gettering(IG) and extrinsic gettering(EG) are presently used. Typical IG process is conducted to insert oxygen segregation layer near active region of device through heat process, plasma etching and ion implantation of carbon or nitrogen. EG is simply creating defects and dislocations by mechanical rough finishing in the back of substrate. The light ion bombardment for power device, above-mentioned, is ,also, a kind of IG in which the defect stability is very important and a series of annealing process is accompanied after irradiation.

1-2. Radio-luminography

This gettering effect has been investigated by radio-tracer method called "Radioluminography[14]; a new version of autoradiography that uses Fuji imaging plate of photo-simulative phosphor of $BaFBr:Eu^{2+}$, which gives five digits of wider dynamic range in stead of hundred and hundred to thousand time more sensitive than conventional silver-halogen film. The radio-isotopes now being used are ^{64}Cu, ^{60}Co, ^{57}Ni, ^{54}Mn and ^{198}Au of reactor-based.
However, the cyclotron-based alternatives of ^{61}Cu, ^{52}Fe, ^{51}Cr, ^{65}Zn, are more desirable in respect of high specific activity. ^{61}Cu production through ^{64}Zn(p, α) is now under investigation at Toyo accompanied with chemical separation process to eliminate by-product elements like ^{67}Ga.

1-3. Experimental procedure and results

Typical experimental procedure[14] is ;(1)adsorption of ^{64}Cu on silicone wafer by immersing into BHF(boron-HF) solution containing 1 ppb doped ^{64}Cu. Then, (2) heat treatment under 700°C for one hour and migration of ^{64}Cu from the mirror face to the EG back side which were observed quantitatively by film density measurement. Calibration curve of the image density versus activity is plotted prior to the experiment. Also, radio-luminography was conducted on a slanted cut sample for depth profile measurement, where the density along the depth was measured for both back and front cutting plane. After correction of the beta-ray attenuation, the depth profile was obtained(Fig. 3). It is concluded that only 20% of ^{64}Cu moves to the back plane and the low temperature annealing process ,normally used, is insufficient.

This method is now being applied for the detergent and air filter test in semiconductor process[15](Fig. 4).

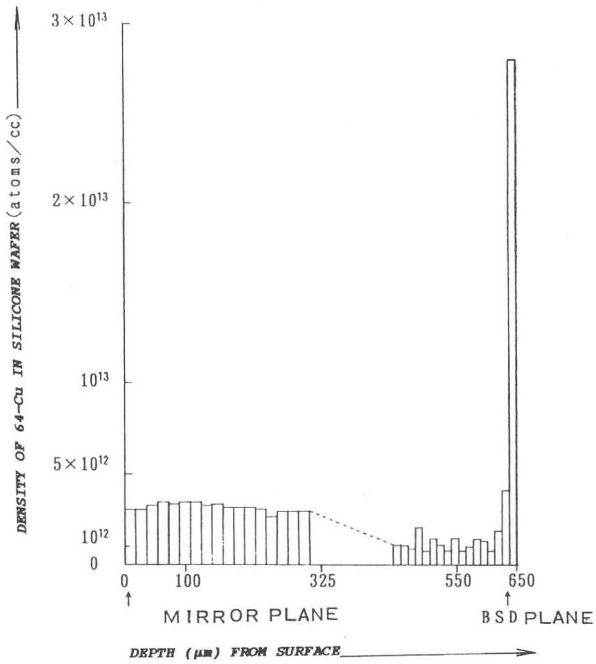

Fig. 3: Depth profile of ^{64}Cu after heat treatment at 700°C, 1 h by Radioluminography method with slanted cut sample of 0.7°, where labeling of 2×10^{11} atoms /cm^2 at mirror plane

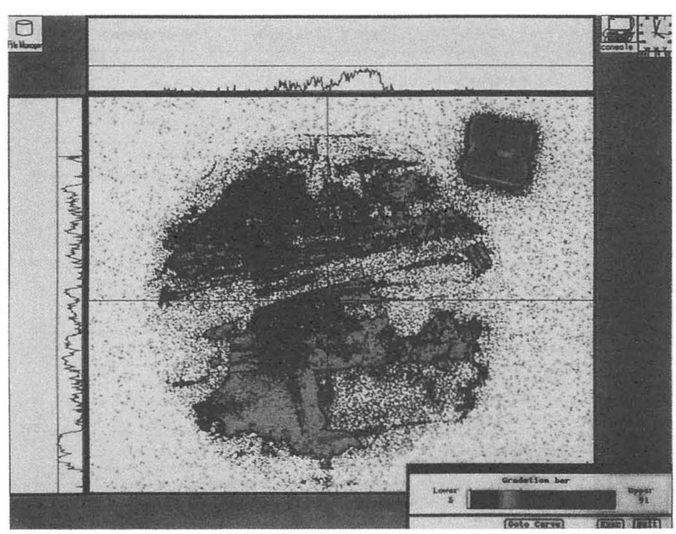

Fig. 4: Contamination of silicone wafer through Radioluminography by immersing in BHF solution containing 1 ppb of ^{64}Cu for the test of the divergent of BHF

(2) Fancy Diamond

The color of diamond is affected largely by the growing process in natural ore, i.e. kind of country rock, e.g. igneous rock or sedimentary rock together with heat hysteresis , stress ,etc. The color of most natural diamond depends on the content of light element of nitrogen (yellow) and boron(pink) which causes absorption band in blue and red region, respectively. The first artificial color was obtained by Sir W. Crookes(1832-1919), immersing in solution containing radium by exposing α particles and blue green colored diamond was obtained.

Since, several attempts ,bombarding electron, neutron and γ-ray, have been carried out to create "Fancy Color" which is not found in natural diamond. A diamond(0.513 carat), treated by proton bombardment to the direction of culet and found an umbrella pattern ,typical mark of ion bombardment and a microscope inspection revealed a thin layer of color zone that causes an umbrella pattern. After bombardment it was treated by heat process in order to stabilize and modify the color[18].

The coloration is based on the similar mechanism to create defects, i.e. impurity level in the band gap, of which study will provide another defect analysis tool.

Finally, typical nuclear reactions ,herein used, are summarized in Table 5.

Table 5: Summary of nuclear reactions used for industrial use

Element	Reaction	Bombarding Energy (MeV)	Half Life (day)	γ-ray Energy (keV)
Nuclear reaction for TLA;				
Carbon	$^{12}C(^{3}He,2\alpha)^{7}Be$	12	53	478
(Alminum	$^{27}Al(\alpha,2\alpha n)^{22}Na$	47	845	511, 1275)
Iron	$^{56}Fe(p, n)^{56}Co$	12	77.3	511, 847,1238
Nickel	$^{58}Ni(p, 2n)^{57}Co$	19	244	1115
Copper	$^{65}Cu(p, n)^{65}Zn$	10	53	478
Lead	$^{206}Pb(p, 2n)^{204}Bi$	21	15.3	1764, 703, 988
			Half Life(min)	Decay Mode
CPAA;				
C	$^{12}C(d ,n)^{13}N$	17.7	9.958	β+100%
B	$^{10}B(d ,n)^{11}C$	9.2	20.385	β+ 99.8%
O	$^{16}O(3He,p)^{18}F$	21.8	109.6	β+ 96.9%
On-line excitation of positron beam;				
	$^{11}B(p ,n)^{11}C$	12 *	20.4 min	
	$^{14}N(p ,\alpha)^{11}C$	12	20.4 "	
	$^{19}F(p, n)^{19}Ne$	9	18 sec	
	$^{23}Na(p, n)^{23}Mg$	14	12 "	
	$^{27}Al(p, n)^{27}Si$	14	4.2 "	
	$^{12}C(d, n)^{13}N$	4	10 "	
	$^{14}N(d, n)^{15}O$	4	2.0 "	
	$^{28}Si(d, n)^{29}P$	5	4.5 "	
Radioluminography;				
Cu	$^{64}Zn(p ,\alpha)^{61}Cu$		3.1 h	β+ & Ec
Neutron production;				
Be	$^{9}Be(p ,n)^{9}B$			evapolation

* Maximum cross section energy

4. Conclusions

Present status of a sub-compact cyclotron for industrial use are summarized, specially related to creation of defects and control in material together with introduction of a new technique of Radio-luminography that is a strong tool for evaluation of gettering effect of lattice vacancy and surface impurity.

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