Development of Ion Photon Emission Microscopy at JAEA

Shinobu Onoda[*1], Hiroshi. Abe[1], Takashi Yamamoto[1], Takeshi Ohshima[1],
Junichi Isoya[2], Tokuyuki Teraji[3], and Kenji Watanabe[3]
1 Japan Atomic Energy Agency (JAEA), Japan
2 University of Tsukuba, Japan
3 National Institute for Materials Science (NIMS), Japan
*Email: onoda.shinobu@jaea.go.jp
Keywords: Ion Photon Emission Microscopy, Microbeam, Diamond, NV center

Abstract

We are developing the Ion Photon Emission Microscopy (IPEM). Since the spatial resolution is determined by the spot size and the intensity of Ion Beam Induced Luminescence (IBIL), the scintillator is one of the most important parts of IPEM. We propose that the diamond containing Nitrogen Vacancy (NV) centers can be used as a scintillator. For both diamond and YAG:Ce proposed by Branson et al., the minimum spot size is a few micrometers. IBIL intensity from diamond is four times higher than that from YAG:Ce. Therefore, we propose that the diamond containing NV centers is a rival candidate of YAG:Ce for IPEM.

1. Introduction

When a high energy heavy ion passes through microelectronics, an unexpected transient current is generated and leads to microelectronic malfunctions so called Single Event Effects (SEEs). To study the origin of the transient current in specific device regions, a focused microbeam combined with a single ion hitting technique has a key advantage.Therefore, we have developed two systems to acquire two-dimensional maps of transient currents by using focused microbeams connected with the 3 MV tandem and the Azimuthally Varying Field (AVF) cyclotron accelerators at Japan Atomic Energy Agency (JAEA), Takasaki [1-3]. These are called Transient Ion Beam Induced Current (TIBIC) systems. Avariety of heavy ions can be focused such as Carbon (C), Nitrogen (N), Oxygen (O), Nickel (Ni), Gold (Au), etc. with the energies ranging from 3 to 18 MeV. In addition, Neon (Ne) 260 MeV and Argon (Ar)520 MeV can be focused. The similar system called Time Resolved Ion Beam Induced Current (TRIBIC) has been developed at Sandia National Laboratories (SNL) [4]. The powerful advantage of TIBIC mapping is the ability to determine the position dependence of the event induced by an ion. While the microbeam has many advantages for SEE testing, the transport and optimization of the microbeam requires much time and effort, especially for high energy heavy ions. Therefore the mapping system with less effort is required.

A different way to perform mapping has been proposed by SNL [5-7]. It is called the Ion Photon Emission Microscopy (IPEM). To observe map using IPEM, it is not necessary to focusheavy ions at all. Instead of microbeam to control the position of the ion strike, the position where ion strikes the sample is recorded together with the ion induced event at semiconductors. The event can be electrically detected by oscilloscope. To realize the position detection by single ion strike, a scintillator is placed on the semiconductor, and Ion Beam Induced Luminescence (IBIL) on the scintillator is detected by Position Sensitive Detector (PSD).Since the spatial resolution is determined by the spot size and the intensity of IBIL, the scintillator is one of the most important parts of IPEM system. Recently Branson et al. have studied the optical properties of various scintillators [7]. They suggested that $Y_3Al_5O_{12}$:Ce (YAG:Ce) is so far the best option for application in IPEM system, because of its high emission rate, short decay time, and high radiation hardness. Additionally, the emission spectrum is particularly important. In the case of high energy ion irradiation in air, the gas molecules are ionized, the subsequent luminescence of air disturbsefficient IBIL detection because it acts as noise. Therefore, the emission wavelength of the scintillator should be different from that of air (<450 nm) [7].

We have been developing IPEM at JAEA. One major difference between our system and IPEM developed by SNL is scintillator, which is one of the most important parts of IPEM system. At the beginning of IPEM setup, we have used Zinc Sulfide (ZnS) as a scintillator. However, the spot size of ZnS is one order of magnitudebigger than that of YAG:Ce [8]. Recently, we have proposed that the diamond containing a high concentration of Nitrogen Vacancy (NV) centers can be used as a scintillator for IPEM application. In this paper we focus on IBIL of diamonds containing NV centers.

2. Experimental

2.1 Setup

Fig. 1 shows a photograph of the measurement system connected with AVF Cyclotron at JAEA Takasaki. This system contains the following; (1) a beam extraction window (Kapton film, 7 μm in thick) is mounted at the end of beam line. On the film, a mirror with hole is placed. The angle of hole is 45 degrees with a diameter of 1

This document is provided by JAXA

mm, and the mirror is mounted at an angle of 45 degrees to the objective lens.; (2) a scintillator and a semiconductor (diode) mounted on a micro XYZ stage.; (3) electronics for charge measurements including an amplifier, a bias-tee, a bias supply and an oscilloscope. At the moment, fast transient currents cannot be measured.; and (4) a photon detection equipment including the microscope (Olympus, BX51M), the GaAsP Image Intensifier (I.I.) (Hamamatsu, C8600), and the cooled Charge Coupled Device (CCD) camera (Hamamatsu, C4880-50-26A). The objective lens is the most important parts of optics. Working Distance (W.D.) and Numerical Aperture (N.A.) need to be long and large as much as possible. We used two objective lenses. One is the standard plan-achromatic lens for brightfield and darkfield microscopy(Olympus, Model: MPLN5x BD, W.D.: 12mm, N.A.: 0.10) as shown in Fig. 1. Another is the plan-achromatic lens with long W.D. (Olympus, Model: SLMPLN20x, W.D.: 25 mm, N.A.: 0.25).

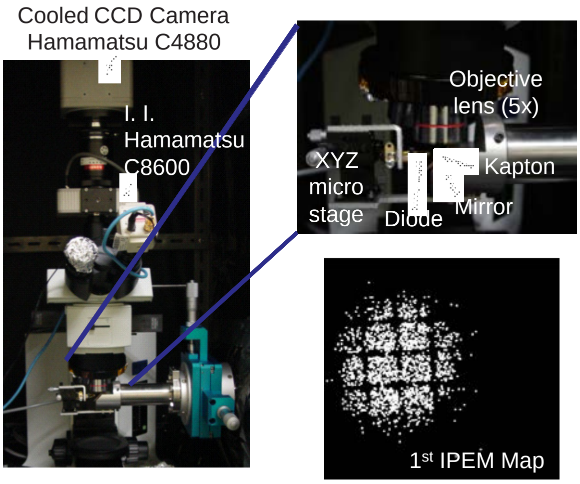

Fig. 1 Photograph of measurement system (left and top right). The first IPEM map of Cupper mesh on diode is represented (bottom right).

The high energy ion beams accelerated by the AVF cyclotron are extracted from vacuum to air via Kapton film. The extracted ion penetrates the scintillator on a semiconductor. The photons from the scintillator are detected by the cooled CCD camera. The position where the ion hit the scintillator can be calculated from the center of mass of ion induced luminescence. At the same time the ion induced transient current pulse generated at a semiconductor is recorded by the oscilloscope. Combining the position where ion hit and the transient current, two dimensional mapcan be observed as shown in Fig. 1.

2.2 Scintillators

Three different varieties of diamonds without NV centers are studied;one is aIb single crystal diamond particles (about 1 mm in diameter) purchased from Element Six (E6). The type Ib diamond contains dense nitrogen as an impurity(labeled as #1); second is an electronic grade IIa single crystal diamond plate (4x4x0.5mm) purchased from E6 (labeled as #2); third is a polycrystalline diamond synthesized at National Institute for Materials Science (NIMS) (labeled as #3). These samples do not contain NV centers.

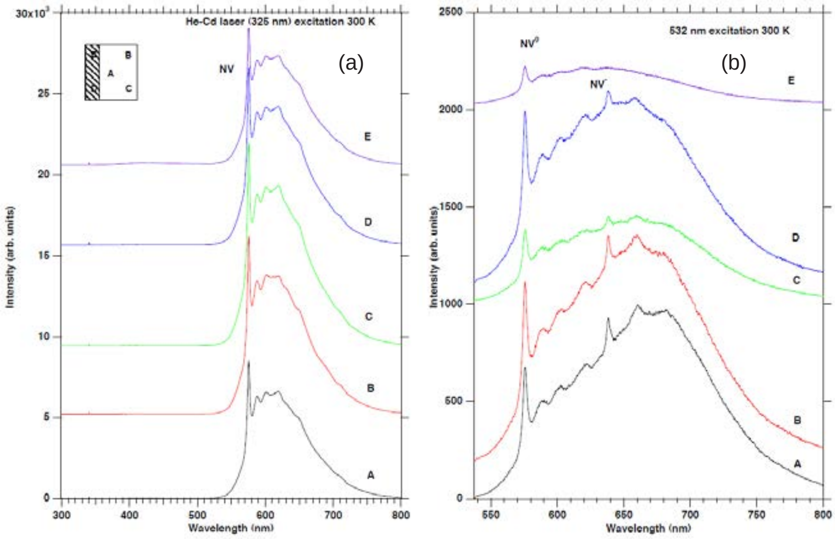

Fig. 2Photo-Luminescence (PL) spectra excited by 325 nm laser (a) and 532 nm laser (b) at room temperature.

On the other hand, a non-commercial High Presser High Temperature (HPHT) IIa single crystal diamond containing NV centers is studied(labeled as #4). NV centers are created by high energy electron irradiation and subsequent annealing. The distribution of NV centers is uniform and theaverage concentration of NV centers is evaluated to be 0.14 ppm ($2.5 x 10^{16}$ cm^{-3}). The distributionuniformity of NV centers is confirmed by Photo Luminescence (PL) spectra at various locations of the diamond. PL spectra excited by 325 nm (see Fig. 2(a)) and 532 nm (see Fig. 2(b)) are measured at NIMS. Inset of Fig. 2(a) shows the schematic top view of sample #4. The capital alphabets in the inset figure show the locations where PL spectra are measured. As shown, NV centers with negatively charged (NV^-) and non-charged (NV^0) are detected from whole area of sample #4. In the case of 532 nm excitation, Zero Phonon Lines (ZPLs) of NV^0 and NV^- are found at the wavelengths of 575 and 637 nm. The broad spectrum (550-800 nm) corresponds to vibronic side bands of each charge state. Although not shown here, Quantum Efficiency (QE) of I.I. decreases at the emission maximum of NV centers in the diamond. However, the portion of the emitted photons from NV centers in the diamond can be detected, since there is partial overlap.

In addition to sample #4, we prepare another diamond containing NV centers. We perform the electron

This document is provided by JAXA

irradiation to type Ib diamond particles (sample #1), and subsequent annealing at 800 degree Celsius for 5 hoursto create NV centers (labeled as #5). Figure 3 (a, b) shows the diamondsbefore (sample #1) and after irradiation (sample #5) under illumination of Ultra Violet (UV) light with the wavelength of 254 nm. The yellow color corresponds to nitrogen impurity (see Fig.3 (a)), and the red color corresponds to NV centers (see Fig.3 (b)). This fact suggests that we successfully convert nitrogen impurity to NV centers by creating vacancies.

Fig. 3.Photograph of sample #1 (a) and #5 (b) under UV light with the wavelength of 254 nm.

3. Results and discussion

Figure 4 (a, b) shows the CCD images when 150 MeV Ar ions penetrate YAG:Ce and diamond containing NV centers (sample #4). The dashed circles highlight the ion induced spots. As shown, IBIL is observed from YAG:Ce and the diamond containing NV centers. However no spot is observed from diamonds without NV centers (samples #1, #2, #3). This fact suggests that NV centersarethe main contributor forIBIL. One possibility of the luminescence mechanism from a diamond containing NV centers is following. According to the ion track distribution theories, the radius of ion track created by the single ion with the several hundreds of MeV exceeds 1 μm. Since a lot of NV centers distribute in the ion track, these are excited. When the excited NV centers change to the ground state,IBIL is emitted. To confirm this, it is necessary to measure IBIL spectrum. Therefore it needs further consideration.

Figure4 (c) shows the typical spot distributions observed from YAG:Ce(closed symbols with red color)and sample #6 (open symbols with black color). The dashed line shows the noise level. The averaged Signal to Noise (S/N) ratios of each scintillator are 3.4 and 4.6, respectively. The Full Width at Half Maximums (FWHMs) of spot are evaluated to be 2.9 and 3.8 μm, respectively. After normalization by the maximum values, the shape of spot distribution of YAG:Ce agrees well with that of the diamond containing NV centers (sample #6). In contrast with our data, it was reported that the spatial resolution of about 5 μm has been achieved by using YAG:Ce [7]. In the case of n-type Gallium Nitride (n-GaN), the spatial resolution of about 2.5 μm has been demonstrated [6]. We suggest that a diamond containing NV centers is a rival candidate of YAG:Ce and n-GaNfrom the point of view of spatial resolution.

We perform the high energy heavy ion irradiation to sample #5. While no spot is observed from sample #1, spots are found in sample #5. This fact supports that NV centeris the main contributor of IBIL. Figure 5 shows typical spotsobserved from sample #5 when 56 MeV-N, 75 MeV-Ne, 150 MeV-Ar, 322 MeV-Kr and 454 MeV-Kr. The dashed circle highlights the ion induced spot. In this study, we successfully demonstrate that the

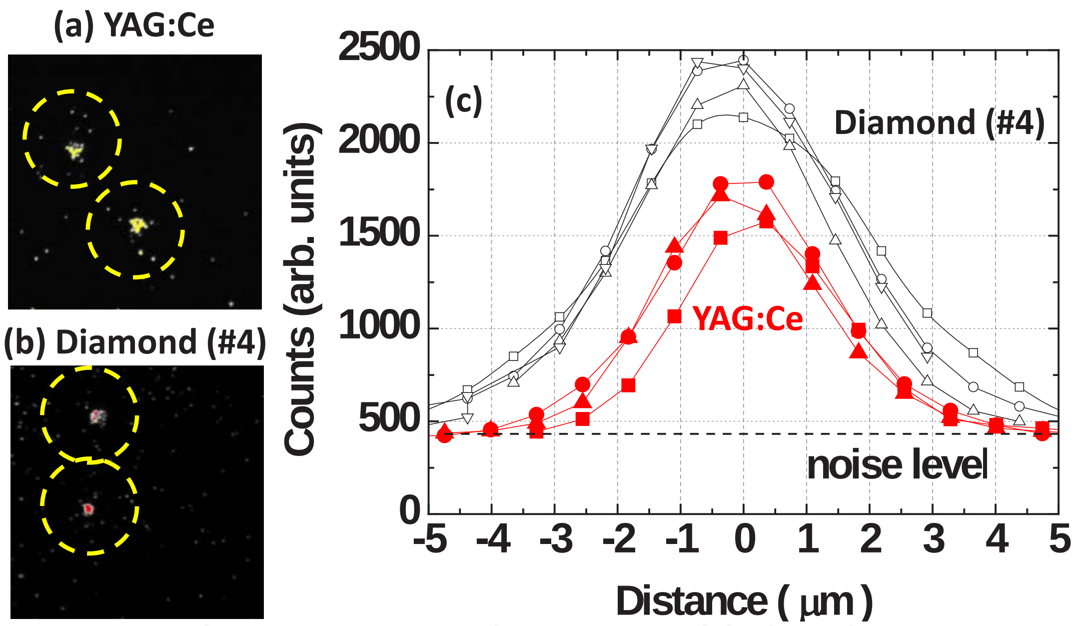

Fig. 4.CCD images when 150 MeV Ar ions strike YAG:Ce (a) and the diamond containing NV centers (sample #4) (b). Two spots are found for both cases. The cross sectional spectrum of luminescence observed fromYAG:Ce and diamond (sample #4) (c).

This document is provided by JAXA

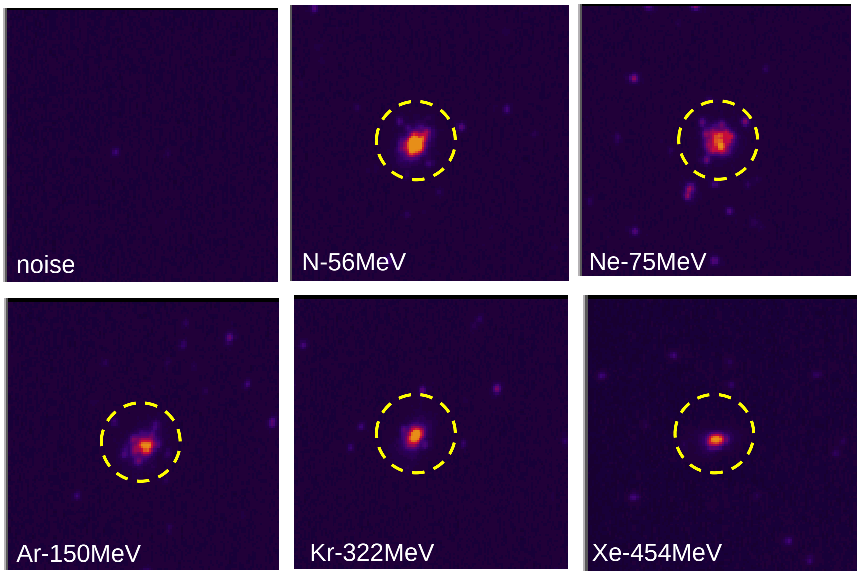

Fig. 5.CCD images when various ions with various energies penetrate the diamond containing NV centers (sample #5).

diamond containing NV centers can be used to detect a wide variety of ion species and energies. It is debatable whether the spot size and intensity of IBIL depends on the ion species and energies.

4. Summary

To achieve high spatial resolution mapping of Single Event Effects (SEEs) by using Ion Photon Emission Microscopy (IPEM) system, the exploration of optimum ion luminescent materials is required. It has been reported that YAG:Ce demonstrates the most promising properties [7]. As a result of ion irradiation, the IBIL is observed from the diamond containing NV centers as well as YAG:Ce. No IBIL is observed from diamonds without NV centers. These facts suggest that NV center mainly contributes to IBIL. We found that the spot size of the diamond containing NV centers is comparable with the YAG:Ce after normalization. S/N ratio of the diamond containing NV centers is better than that of YAG:Ce. To detect a single ion with high sensitivity, it is better to use the diamond containing NV centers instead of YAG:Ce. Finally, we demonstrate that the diamond containing NV centers can be used to detect the single ion with a wide variety of ion species and energies. According to these results, we therefore conclude that a diamond containing NV centers is a rival candidate of a YAG:Ce for IPEM applications.

Acknowledgments

YAG:Ce was provided by Mr. A. Yokoyama of the Department of Advanced Radiation Technology, JAEA. This work was partly supported by the Strategic Japanese-German Joint Research Program in 2009 from the Japan Science and Technology Agency.

References

[1] I. Nashiyama, et al., "Single-event current transients induced by high energy ion microbeams," IEEE Trans. Nucl. Sci.,vol. 40, pp. 1935-1940 (1993).
[2] J. S. Laird, et al., "Development of a new data collection system and chamber for microbeam and laser investigations of single event phenomena,"Nucl.Instr. and Meth.B, 181,pp. 87-94 (2001).
[3] T. Hirao, et al., "Transient current mapping obtained from silicon photodiodes using focused ion microbeams with several hundreds of MeV,"Nucl. Instr. and Meth.B,267, pp. 2216-2218 (2009).
[4] H. Schone, et al., "Time-resolved ion beam induced charge collection (TRIBICC) in micro-electronics,"IEEE Trans. Nucl. Sci., vol. 45, pp. 2544-2549 (1998).
[5] B. L. Doyle, et al., "Nuclear emission microscopies,"Nucl.Instr. and Meth.B, 181, pp. 199-210 (2001).
[6] J. V. Branson, et al., "The ion photon emission microscope on SNL's nuclear microprobe and in LBNL's cyclotron facility,"Nucl.Instr.And Meth.B, 267, pp. 2085-2089, (2009).
[7] J. V. Branson, et al., "Ion beam characterization of advanced luminescent materials for application in radiation effects microscopy,"Nucl. Instr. and Meth.B, 269, pp. 2326-2329(2011).
[8] S. Onoda, et al., "Diamonds Utilized in the Development of Single IonDetector with High Spatial Resolution,"Trans. Mat. Res. Soc. Jap., pp. (2012).

This document is provided by JAXA