

# Novel magnetic-semiconductors in modified iron titanates for radhard electronics

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**Abstract** Some members of the modified iron titanate family show remarkable tolerance to radiation and are well suited for radhard electronics. Of particular interest are solid solutions of ilmenite–hematite (IH) represented by  $(1-x)$   $\text{FeTiO}_3 \cdot x\text{Fe}_2\text{O}_3$  where  $x$  varies from 0 to 1; and pseudo-brookite,  $\text{Fe}_2\text{TiO}_5$  (PsB). These multifunctional oxides can be both ferrimagnetic and wide bandgap semiconductors, and can be exploited in a variety of ways in radhard electronics, microelectronics and spintronics technologies. In this paper we emphasize the potential applications of the modified Fe-titanates with special emphasis on: (a) response of the non-linear current–voltage ( $I$ – $V$ ) characteristics to a magnetic field; (b) how the introduction of a biasing voltage might be used to produce bipolar currents in

circuits and fabrication of voltage tunable varistors; and (c) the response of non-linear current–voltage characteristics when irradiated with neutrons, protons and heavy Fe-ions. Based on these observations, we will identify a few applications for which we can make use of the unique multifunctional nature of modified Fe-titanates.

**Keywords** Tunable varistors · Bipolar currents · Switching voltages · Radhard electronics

## 1 Introduction

Fe-titanates have been known to geologists and geophysicists for a very long time. They exist principally as three minerals, namely, ilmenite ( $\text{FeTiO}_3$ ), pseudo-brookite ( $\text{Fe}_2\text{TiO}_5$ ) and ulvöspinel ( $\text{Fe}_2\text{TiO}_4$ ). Ilmenite is an established raw material as the primary source of  $\text{TiO}_2$ , which is used widely as pigments. Ilmenite is also the most prominent radiation shielding material and is extensively used for shielding buildings and other facilities from dangerous radiation leakage in the vicinity of nuclear reactors. When mixed with hematite it forms wide range of solid solutions. Early on it was realized to possess fascinating magnetic properties in its natural forms. The pioneering work of Ishikawa in 1957 [1] and then of Brown et al. [2] in 1993 deal with the magnetic properties of lab-processed IH ceramic samples with varying concentration of hematite. However, the unique semiconducting properties of synthetically processed ilmenite–hematite solid solution ceramics were systematically studied first by Ishikawa in 1958 [3].

The general formula for ilmenite–hematite solid solutions (IH) can be represented as  $(1-x)$   $\text{FeTiO}_3 \cdot x\text{Fe}_2\text{O}_3$  with  $x$  varying between 0 and 1. Throughout this paper we

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will refer to ilmenite–hematite ceramic with  $x=0.33$  as IHC33 and with  $x=0.45$  as IHC45. Similarly, for ilmenite–hematite films with  $x=0.33$  we will use the abbreviation IHF33 and IHF45 would refer to film of ilmenite–hematite with  $x=0.45$ . Also, pseudobrookite,  $\text{Fe}_2\text{TiO}_5$ , will be abbreviated as PsB.

It was discovered that when the hematite concentration  $x$  exceeds the value of 0.27 per formula unit the semiconducting nature changes from p-type to n-type by doping via defects. This result was modified by Zhou in 2000 [4] when he showed that the p-type nature changes to n-type when  $x>0.20$ . He also reported that pure ilmenite, that is, ilmenite when not doped by defects or impurities, is intrinsic in nature besides identifying IHC 45 to be both strongly magnetic and an n-type semiconductor [4, 6, 7]. Only recently a composition close to IHC 45, namely, ilmenite–hematite with  $x=0.40$ , has been grown as epitaxial film and identified to be an n-type semiconductor as well as ferrimagnetic with the Curie temperature  $>400$  K [8].

IH is perhaps the only natural system that is both n- and p-type semiconductor as well as ferrimagnetic. The combination of these two properties, that are enormously important for the advancement of modern technologies such as integrated circuit, microelectronic, information technology etc., it is no wonder that it has attracted the attention of scientists and engineers. Furthermore, with the recent emergence of spintronics as a viable technology this system has drawn the attention of investigators [8–11].

The system is ideally suited for fundamental studies and exploration of potential applications. It is to be noted that many of its members have magnetic Curie points well above room temperature [1, 2, 5, 8–11] in contrast to actively studied spintronics materials belonging to dilute magnetic systems, for example, Mn-doped GaAs and doped ZnO with the magnetic Curie points below room temperature. Furthermore, the nature of the magnetism in IH system is intrinsic and samples can be processed so that magnetism is uniformly distributed over the bulk of the sample and does not exist simply in clusters [5, 11, 12].

In the last few years we have studied extensively the magnetic, semiconducting and radiation hardness properties of modified Fe-titanates bulk ceramic samples, textured and epitaxial films, and bulk single crystals. The samples were processed after careful optimization of processing conditions. Of special interests are IHF33 and IHC45, and single crystals of pseudobrookite (PsB). Some of these studies have been reported in references [6, 7, 12–19]. In this paper our emphasis will be on materials characterization for radhard electronics, evaluation of magnetically and electrically tuned varistors and effect of a biasing voltage on the non-linear current–voltage ( $I$ – $V$ ) characteristics.

The uniqueness of the IH solid solutions lies in the fact that: (1) they are wide band gap semiconductors ( $E_g >$

2.58 eV) which can be tailor-made as p- or n-type by carefully selecting the level of concentration ( $x$ ) of hematite,  $\text{Fe}_2\text{O}_3$ , in ilmenite,  $\text{FeTiO}_3$ ; (2) their magnetic nature (anti-ferromagnetic or ferrimagnetic) is solely dependent upon the concentration  $x$  and the degree of cation ordering, (3) the bandgap of IH is highly dependent upon the processing conditions. For example, the bandgap of IH film with  $x=0.33$  varies between 2.3 to 2.5 eV for high oxygen pressure, and for low oxygen pressure it varies from 3.0 to 3.4 eV [12]. For ilmenite film grown by laser ablation method (PLD) the bandgap is reported to be 3.55 eV whereas it is 2.58 eV for bulk ilmenite [20]; and (4) their magnetic transitions occurring much above room temperature is of added importance for the development of magneto-electronics and spintronics technology. Also, Mn–PsB ( $\text{Mn–Fe}_2\text{TiO}_5$ ) with Mn concentration of 0.4 atomic percent per formula unit exhibits magnetic and semiconductor properties observed similar to those by ilmenite–hematite solid solutions [17].

As mentioned in the abstract the specific applications that we would propose later in this paper will be based on our observations of the following studies: (a) response of the non-linear current–voltage ( $I$ – $V$ ) characteristics to a magnetic field; (b) how the introduction of a biasing voltage might be used to produce bipolar currents in circuits and fabrication of voltage tunable varistors; and (c) the response of non-linear current–voltage characteristics when irradiated with neutrons, protons and heavy Fe-ions.

## 2 Sample processing

The basis of sample preparations and the methods adopted are the phase diagrams of ilmenite ( $\text{FeTiO}_3$ ), pseudobrookite ( $\text{Fe}_2\text{TiO}_5$ ), ulvöspinel ( $\text{Fe}_2\text{TiO}_4$ ), and ilmenite–hematite solid solutions (IH) [21, 22]. Bulk ceramic samples were prepared using the standard technique of ball milling the raw materials, pressing at high pressure using a uniaxial press followed by sintering and finally annealing the green ceramic at high temperatures. The details are given in many of our papers referenced here but the reference number [5, 13, 15] will be of special interest to those interested in processing dense, homogeneous ceramic samples of IH. We have studied also the textured and epitaxial films of different compositions of IH, primary among them being those with  $x=0.33$  and  $x=0.45$ . These films were grown by using a laser ablation system (PLD) and the experimental details are described in reference [12] for single crystal films of IHF33. The textured films have been dealt with in references [6, 18] for multiple compositions of IH. Bulk single crystals of PsB were grown using the modified technique of high temperature solution growth [17].

### 3 Proposed devices and applications

#### 3.1 Magnetically tunable varistors

Varistors or voltage-dependent-resistors (VDR) are 2-terminal ceramic based devices with highly nonlinear current–voltage ( $I$ – $V$ ) characteristics. They are based on the principles of metal-semiconductor transport and tunneling through the grain boundaries and are analogous to metal-semiconductor Schottky diodes connected back-to-back [23].

The  $I$ – $V$  characteristic of a varistor follows an empirical law given by the relation

$$I = CV^\alpha \quad (1)$$

where  $\alpha$  is called the non-linear coefficient. From Eq. 1 it follows that

$$\alpha = \frac{\log I_2 - \log I_1}{\log V_2 - \log V_1} \quad (2)$$

It is evident from Eq. 2 that the non-linear coefficient  $\alpha$  represents the figure of merit of the device. The larger its value the more efficient is the device [23].

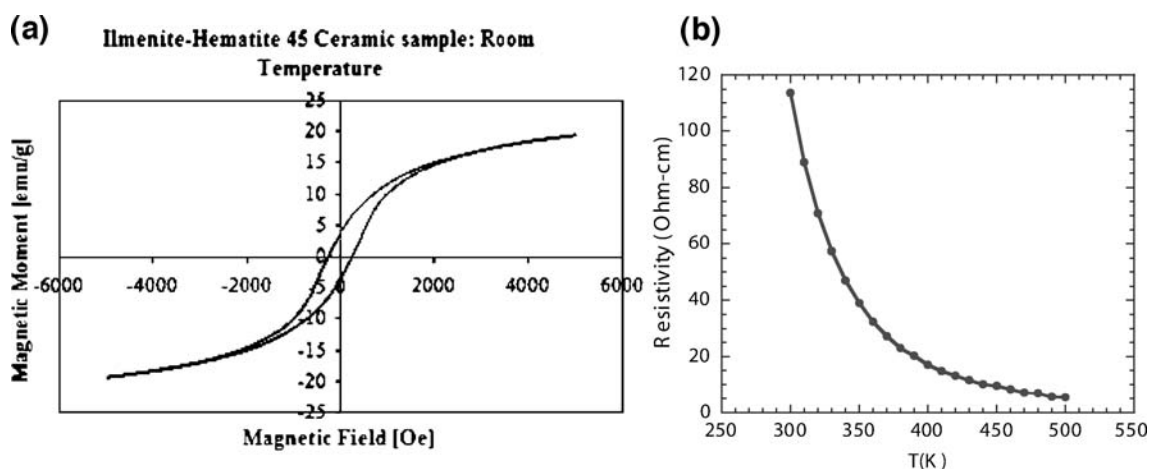
Varistors are normally used in parallel to the load in an electrical circuit for protection from voltage surges. Commercially available varistors are based on ZnO and SiC bulk ceramics and are primarily targeted for high volume applications in power circuits. They switch at high voltage and so cannot be used for protecting electronic circuits and components which are primarily Si-based having the operating voltage limit of approximately 5 V.

Our varistor devices based on IH ceramic switch at low voltages (<5 V) and they have excellent immunity to radiation [13, 15]. Low voltage switching makes them attractive for integration with microelectronics and radiation immunity for radhard electronics.

Recently, the four-point  $I$ – $V$  measurements done on single crystal IHF 33 and reported in reference [19] show the  $I$ – $V$  curve to be linear. However, when two-point measurements were conducted on the same sample highly non-linear  $I$ – $V$  relationship was observed, consistent to our previous observations reported in [4, 7, 13, 15, 17, 18]. A careful analysis suggested the existence of Schottky barriers which is the basis of all metal-semiconductor devices.

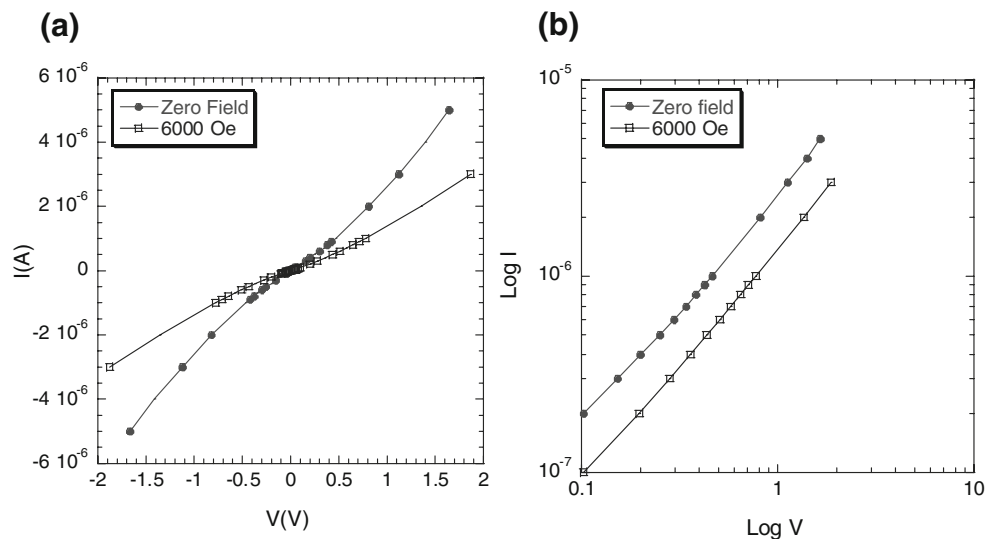
For studying the response of non-linear  $I$ – $V$  characteristics to a magnetic field we chose a highly dense and homogeneous bulk ceramic sample of IHC45. The non-linearity of  $I$ – $V$  for IHC45 was first reported in [13]. It is highly magnetic as well as a wide bandgap semiconductor like its other counter parts in the IH family. The magnetic and semiconductor nature of IHC45 is seen from Fig. 1(a) and (b), respectively. The resistivity vs. temperature curve, shown here, is typical of a semiconductor material as is the well-defined hysteresis loop for a ferrimagnetic material. As mentioned earlier IHC45 is a n-type material with a bandgap,  $E_g \approx 2.3$  eV [17]. Its important magnetic parameter are: Curie point ( $T_c$ )  $\approx 610$  K, coercivity ( $H_c$ )  $\approx 250$  Oe, remnant magnetization ( $M_r$ )  $\approx 3.5$  emu/g and saturation magnetization ( $M_s$ )  $\approx 19.4$  emu/g.

Both Ag and Ni were used as contact materials for the two-point studies that were done at room temperature. To eliminate the possibility of any thermal effect the samples were subjected only up to low voltages so that the current does not exceed more than a few microamperes. From Fig. 2(a) we observe how strongly the non-linear  $I$ – $V$  is influenced by the application of a magnetic field ( $H$ ) of 6 kOe. A similar behavior was observed for Mn-doped PsB film [17]. From Fig. 2(b), we see that the non-linear coefficient ( $\alpha$ ) of this device remains practically unaltered by the application of a magnetic field. The fact that it remains practically constant for our device means that the



**Fig. 1** (a) Magnetic loop and (b) resistivity vs temperature of IHC 45 bulk ceramic at room temperature

**Fig. 2** (a) Non-linear  $I$ - $V$  before and after application of magnetic field  $H=6$  kOe for IHC45 bulk ceramic at room temperature, and (b)  $\log I$  vs  $\log V$  plot of curves in (a)



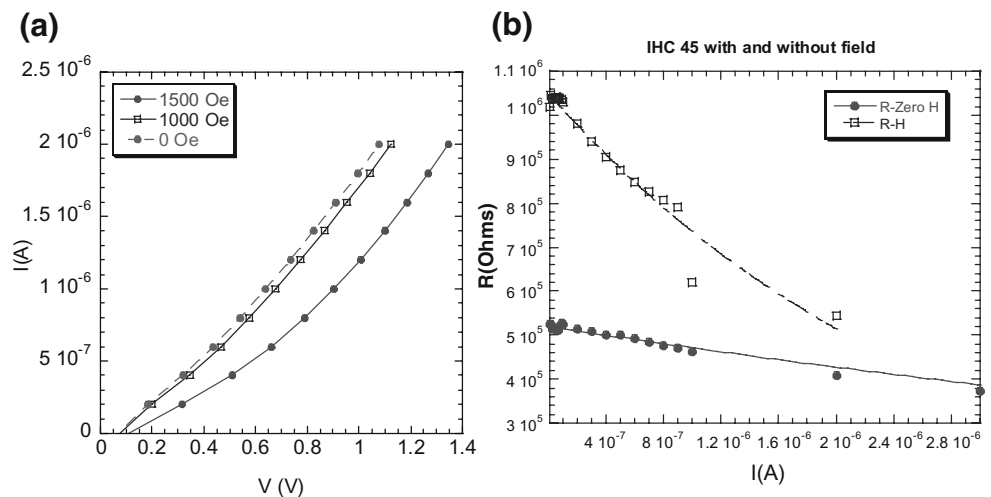
IHC45 based varistor should work efficiently also in the presence of a magnetic field ( $H$ ) as without it.

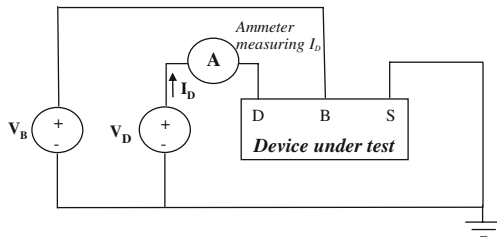
In Fig. 3(a) we show the  $H$ -field dependence of  $I$ - $V$  of IHC45 at 100 K for two different values of the field (1 and 1.5 kOe). As the field increases the effect becomes more pronounced and the magnitude of current for a fixed value of applied voltage ( $V$ ) decreases with the increase in the value of the magnetic field ( $H$ ). The Fig. 3(b) shows resistance as function of current for  $H=0$  and  $H=6$  kOe for this sample. For low values of current the change is large, i.e., we see a very large change in resistance at the application of the  $H$ -field. However, it decreases, as we would expect, as the current is increased. This result is consistent with the decrease of current in the presence of a  $H$ -field such as in Fig. 2(a) and 3(a). Here we can draw two conclusions: (a) magnetic field tunable varistors can be built using the  $I$ - $V$  characteristics of IH materials, and (b) they will operate satisfactorily at room temperature as well as at low temperatures.

### 3.2 Proposed $\mu$ -electronic devices based on three-terminal behavior of $I$ - $V$

Three metallic contacts (either Ni or Ag) were placed, 1–2 mm apart from each other, on the surface of the film sample (IHF33) and bulk ceramic sample (IHC45) to study the effect of a biasing voltage on the non-linear  $I$ - $V$  characteristics. For discussing the results we identify the three contacts as source (S), bias (B) and drain (D). It is to be noted that the three-point  $I$ - $V$  measurements of our devices is not to be confused with the  $I$ - $V$  measurements of FET devices. Bias (B) does not act like a gate in FET circuits. There is no gate oxide in our test devices. Their relative positions and the circuit used for determining the three-point  $I$ - $V$  characteristics are shown in Fig. 4. Figure 5(a) and (b) are the representative  $I$ - $V$  curves of IHF33 and IHC 45, respectively, when the bias voltage ( $V_B$ ) is introduced in the circuit.

**Fig. 3** (a) Non-linear  $I$ - $V$  as a function of  $H$ -field at 100 K, and (b) resistance vs current before and after application of  $H=6$  kOe at room temperature, both for IHC 45 bulk ceramic





**Fig. 4** Circuit diagram for three-terminal  $I$ - $V$  measurement;  $D$  is drain,  $S$  the source and  $B$  the bias contact

An inspection of Fig. 5 reveals the qualitative similarities between the two individual  $I$ - $V$  characteristics in spite of the fact that the magnitudes of the drain current are different. This is only the consequence of device impedance. Impedance of the IHF33 is much greater than that of IHC45. Therefore, the magnitude of the drain current,  $I_D$ , generated in the IHF33 device is in microamperes whereas it is in milliamperes for the IHC45 device.

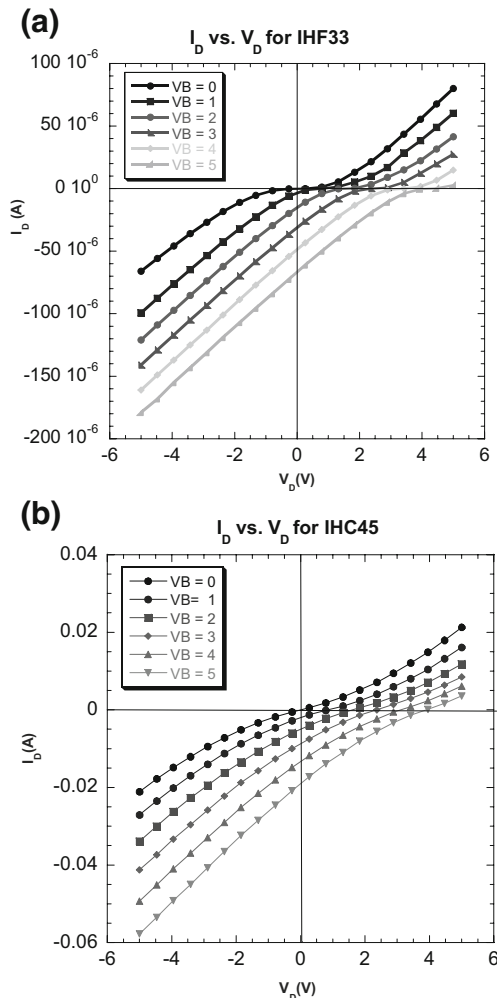
We observe from these two figures that: (1) The asymmetry exists in the current output ( $I_D$ ) between the

positive values of  $V_D$  and the negative values of  $V_D$ . This is the manifestation of the change in the potential barrier heights on application of the bias voltage; (2) The device becomes more resistive (current decreases) as the bias voltage  $V_B$  increases for positive values of  $V_D$ ; whereas exactly the opposite is true when  $V_D$  is negative. That is, the device goes from high resistive states to high conductive states. The reason again being the change in potential barrier height of the device; (3) The switching voltage (it is that voltage,  $V_S$ , at which the current increases rapidly for a small change in the applied voltage  $V_D$ ), is the function of the biasing voltage,  $V_B$ . It increases with the increase in  $V_B$  for all values of  $+V_D$  as seen in Fig. 6. This property allows us to build a biasing voltage tunable varistor; (4) The curvature of the  $I$ - $V$  curve decreases as the  $V_B$  increases. The non-linearity disappears almost around  $V_B \approx 5$  V. The device, though, still retains its diode like property and can be used as a varistor; (5) The currents ( $I_D$ ) are “bipolar” for all values of  $V_B$  and for a wide range of  $+V_D$ . It is most pronounced when  $V_D$  lies between 2.5 to 3.5 V (i.e., close to the bandgap). It is important to note that even negative currents ( $I_D$ ) can be generated while both the  $V_D$  and  $V_B$  are positive.

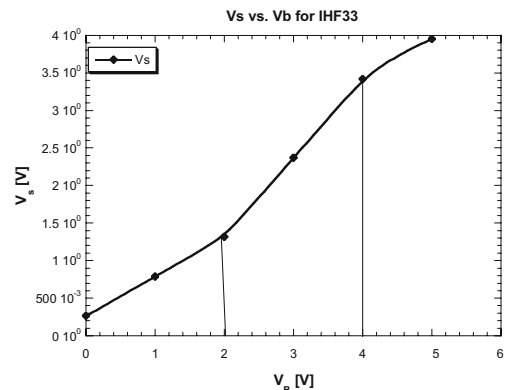
It is interesting to note from Fig. 6 that the slope ( $\Delta V_S / \Delta V_B$ ) between  $V_B = 0$  to  $V_B = 2$  V is 0.5, whereas it is twice this value ( $\approx 1.1$ ) when  $V_B$  lies between 2 and 4 V. It appears that in the vicinity of the bandgap the slope has its maximum value. It is again approximately 0.5 when  $V_B > 4$  V.

Based on these observations, it is possible to build IH based biasing voltage tunable varistors and bipolar current generators. No single crystals or epitaxial films are necessarily required for producing these devices. Ceramic substrates that are less time consuming and cheaper to produce in large volumes are well suited for the fabrication of these devices.

The concept of building a biasing voltage tunable varistor is straight forward. However, building a bipolar current generator warrants some explanation.



**Fig. 5** (a) Three-terminal  $I$ - $V$  measurements of IHF33. (b) 3-terminal  $I$ - $V$  measurements of IHC45



**Fig. 6** Switching voltage  $V_s$  vs biasing voltage  $V_B$  for IHF33



One can observe, for example, that by fixing the value of  $V_D \approx 2.5$  V bipolar current (negative and positive) of different magnitudes can be produced simply by changing the values of the biasing voltage,  $V_B$ . It is to be noted that both  $V_D$  and  $V_B$  remain positive while the current polarity changes. The importance of this feature can be appreciated by considering that this device can “inject” a small amount of controlled positive or negative current into a circuit. The amount of current injected is controlled by  $V_B$ . The device can thus act as a bipolar precision current supply. The bipolar feature is important—even though the values of  $V_D$  and  $V_B$  will always be positive, the device is capable of producing precise positive or negative values of  $I_D$ . In addition to providing current, the device can possibly be used in a balanced bridge-type circuit to provide precise measurement (or precise control) of currents.

### 3.3 Non-linear $I-V$ and irradiation

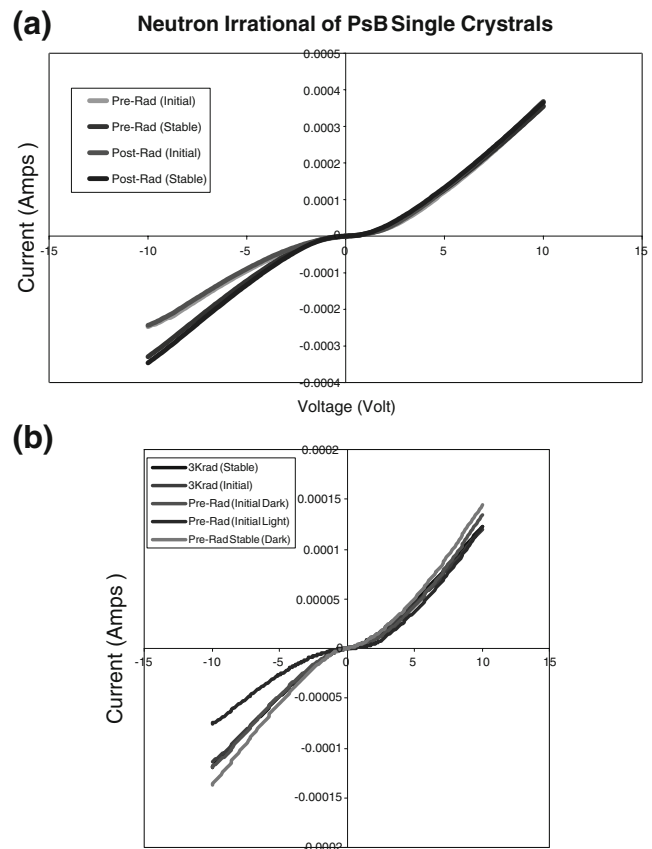
The field of radiation hardened (or, tolerant) materials in  $\mu$ -electronics is becoming increasingly important when it is realized that radiations like neutrons and protons can severely damage the electronic components and ultimately make them useless. Classical wide bandgap semiconductors, in general, have been identified as suitable materials for the advancement of this technology. Semiconductor oxides also appear to be attractive for radhard electronics primarily because they are: wide bandgap materials (usually  $E_g > 2.5$  eV), they can be processed as highly dense and homogeneous bulk ceramics by methods that are relatively less expensive, they can be produced in large volumes and sizes, they are mechanically robust and their particle size can be controlled with precision from sub-microns to almost nanometer scales.

The devices can be exposed to radiation sources present in the upper earth’s atmosphere, low earth orbit (LEO), the trapped radiation belts (van Allen belts), interplanetary space, and planetary surfaces. In the space environment the primary radiation sources of concern to electronics are energetic protons from the sun and galactic cosmic rays [24]. The interactions of the protons and heavier ions with matter can produce high energy secondary neutrons. These secondary neutrons are a major component of the radiation environment in the upper atmosphere and within spacecrafts [25, 26].

Radiation effects on electronics are generally classified into long term total ionizing dose (TID) effects and short term single event effects (SEE). At the single device or component level, TID effects result from radiation induced damage accumulated over a long period of time that produce changes in critical device performance parameters (figures of merit), resulting in system degradation or failure. At the integrated circuit and system level, a single radiation

particle strike can deposit sufficient energy in a device to produce enough excess conduction electrons and holes to flip a logic bit or, in severe cases, cause permanent destruction of the device [27].

We have studied extensively the effect of irradiation by neutrons and protons on IH of different compositions to examine their suitability for radhard electronics. The results have been published in [13–15]. These papers discuss in greater depth the various mechanisms through which radiation can interact with materials and change their basic properties. It is found that the electrical resistivity of IHC with  $x$  varying from 0 to 0.45 decreases rather substantially for compositions with  $x < 0.2$ , and then slightly after being radiated for about 3.5 h at DOE’s Los Alamos National Laboratories (LANL) with neutrons of about 73 MeV, total fluence of  $4.36 \times 10^{10}$ , beam flux of  $3.46 \times 10^6$  neutrons/cm<sup>2</sup>/s [13]. According to this reference, qualitatively the electrical conductivity of the p-type IHC ( $x < 0.2$ ) increases substantially more than of the n-type members of the same family ( $x > 0.2$ ) after irradiation with neutrons. On the other hand, the varistor devices based on IHC retain their non-linearity after irradiation and therefore are expected to perform satisfactorily in neutron environments [13].



**Fig. 7** (a) Non-linear  $I-V$  of PsB single crystal before and after neutron radiation, 73 MeV energy,  $2.1 \times 10^9$  neutrons/cm<sup>2</sup>. (b) Non-linear  $I-V$  of PsB single crystal before and after irradiation by Fe-ions of 307 MeV/nucleon at dose of 3,000 rad

The proton radiation, on the other hand, changes virtually nothing in the shape of the non-linear  $I$ - $V$  of IHC with  $x=0.1$  (p-type IH) and  $x=0.45$  (n-type IH) resulting in no change in the figure of merit and switching voltages of the varistor devices fabricated using these ceramics [15]. This experiment was done at the Cyclotron Institute of Texas A&M University using the proton source of the following parameters: 10 and 40 MeV, total fluence= $5 \times 10^{10}$  p/cm<sup>2</sup>, and exposure time ranging from 10 min to 4 h. From these results we conclude that resistivity remains unchanged from proton irradiation. However, the chemical ordering takes place resulting in increase of magnetic moments for a whole range of IHC [14].

These results confirm the suitability of IH for radhard electronics. However, IH are not the only members of the modified iron-titanate family to have such unique properties. Pseudo-brookite, Fe<sub>2</sub>TiO<sub>5</sub> (PsB), also exhibit excellent radiation resistant properties. We present here also the results of irradiation on the non-linear  $I$ - $V$  characteristics of PsB single crystals. The radiation sources are neutrons and heavy Fe-ions. We see from Fig. 7(a) and (b) that the initial burst of radiation appears to induce a noticeable change in the current output of the device, especially for currents corresponding to negative values of voltage applied, which then rapidly decays back to its original stable value. The performance of the device remains completely unaffected by both the radiation sources. The neutron experiment was done at LANL and the relevant conditions are: average energy  $\approx 73$  MeV and fluence= $2.1 \times 10^9$  neutrons/cm<sup>2</sup>. The heavy Fe-ion experiment was done at the DOE's Brookhaven National Lab and the experimental conditions were: average energy  $\approx 307$  MeV/nucleon and dose  $\approx 3$  krad.

#### 4 Summary

The importance of emerging technologies such as radhard electronics, magnetically and electrically tunable devices, spintronics and oxide based  $\mu$ -electronics has led to the search for new materials. Multifunctional oxides and magnetic-semiconductors are of special interest for the advancement of these technologies. Some members of the family of modified iron-titanates have been identified to be both magnetic-semiconductor and wide bandgap semiconductor. For this research we have concentrated on three such materials, namely, ilmenite-hematite film with 0.33 concentration of hematite per formula unit (IHF33), ilmenite-hematite bulk ceramic with 0.45 concentration per formula unit (IHC45) and pseudobrookite (PsB). We have investigated them for their immunity (or high resistance) to high energy neutrons, protons and heavy Fe-ions and to examine how the device performance might be altered on irradiation. Furthermore, we have investigated

the effect of externally applied magnetic field and biasing voltage on the non-linear device, named varistor, fabricated by using these compositions. We find that these materials are well suited for radhard electronics. Furthermore; we find that they can also form the basis for the development of some novel microelectronic devices. Some examples are: magnetically and electrically tunable varistors; and simple bipolar current generators.

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